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PREPARED FOR:

PPG PULVERIZING SERVICES FACILITY
MOORESTOWN, NEW JERSEY

PHASE II SITE INVESTIGATION

- WORK PLAN
- QUALITY ASSURANCE/ QUALITY CONTROL PLAN
- HEALTH AND SAFETY PLAN

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AUGUST, 1994

McLaren Hart
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Prepared For:

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1.0 INTRODUCTION

This document has been prepared by McLaren/Hart Environmental Engineering Corporation (McLaren/Hart) under contract to PPG Industries, Inc. (PPG), to satisfy U.S. Environmental Protection (U.S. EPA) Region II requirements for a Phase II Site Investigation. The Phase II Investigation will be performed at the former PPG Pulverizing Services Site located in Moorestown, New Jersey in accordance with the Administrative Order on Consent (AOC) dated March 31, 1989, issued to PPG by U.S. EPA (II-CERCLA-80109).

The contents of this document are consistent with the Phase I Site Investigation Report (SIR), previously submitted to U.S. EPA on August 12, 1993. Specifically, Section 6.0 of the Phase I SIR has been reformatted (pursuant to U.S. EPA request) and incorporated into a "stand alone" Phase II Investigation Work Plan. The following sections present the background of the site, the current understanding of site conditions (geology and hydrogeology), and the Phase II Investigation objectives.

1.1 SITE DESCRIPTION AND HISTORY

The Pulverizing Services Site is located in an Industrial Park at 332 New Albany Road in Moorestown, Burlington County, New Jersey (Figure 1). Based upon former tax maps, three parcels comprise the site. Each parcel has an area of approximately eight acres. Two of the parcels (Areas A and C) are located on the northwest side of New Albany Road, while the third (Area B) lies almost directly across from areas A and C on the other side of New Albany Road. A plan of the site, including known utility information, is shown on Figure 2.

The Pulverizing Services Site is an inactive facility which formerly formulated pesticides. The plant commenced operations circa 1935. The plant was originally operated by International Pulverizing Company. In 1946, this firm was sold to the Micronizer Company, a subsidiary of Freeport Sulfur Company now Freeport McMoran. PPG purchased this firm in 1948. PPG operated the plant until 1963 when it was sold to Pulverizing Services, Inc.

This firm, which was headed by the former PPG plant manager, operated on the site until January 1979 when it ceased operations because of labor problems.

Industrial activities involving pesticide grinding, micronizing, and blending first occurred at the site in 1935. Initially, inorganic pesticides such as lead arsenate, calcium arsenate, sulfur, and tetrasodiumpyrophosphate predominated. In later years, organic pesticides such as dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichlorethane (DDD), dichlorodiphenyldichloroethylene (DDE), aldrin, malathion, dieldrin, lindane, rotenone, and n-methyl carbamates (sevin, also referred to as Carbaryl), were formulated. The active pesticide ingredients were not actually manufactured at the site. Rather, the active ingredients were brought to the site, then ground, blended, and packaged for distribution under the labels of various companies.

Based upon several interviews of past Pulverizing Services employees (Skacel, 1986), the activities which occurred in on-site buildings are listed below:

Building No.	Building Description
3	Manufacturing
4	Sales, Maintenance Shop, Process Area, and Garage
5	Main Process Area
6	Warehouse
20	Compressor Building
29	Warehouse
30	Employee Services
Green Bldg.	Pharmaceutical Processing

Sulfur piles were previously located where paved areas are currently located on the southeast side of Buildings 5 and 6. This area and the parking lot on the southwest side of Building 30 were paved between 1963 and 1970 (based upon aerial photography).

PPG owned and operated the site from December 30, 1948 until November 29, 1963. During the 1950s and early 1960s, waste was reported to have been disposed behind the main production buildings (landfill area) in several trenches (U.S. EPA, 1988).

In the spring of 1993, Area B was fenced and the office building was boarded up to prevent trespass.

1.2 PREVIOUS INVESTIGATIVE ACTIVITIES

The Pulverizing Services Site is a former pesticide formulating facility that operated from circa 1935 to 1979. The New Jersey Department of Environmental Protection and Energy (NJDEPE) began Investigating the facility in 1985. U.S. EPA began investigating in 1987. In August of 1993, a Phase I Site Investigation Report (SIR) was submitted to U.S. EPA. The following points summarize results of these previous investigative activities:

- In April 1986, the site was sampled by a team from NJDEPE. Samples were obtained of surface soils, stream sediment, floor sweepings from a building, and a building floor drain. Various pesticides and organic compounds were detected. NJDEPE documented the presence of pesticides in the soil from the landfill area. Compounds identified by the NJDEPE analysis were DDT, DDD, and alpha BHC.

- In October 1987, the U.S. EPA Technical Assistance Team (TAT) conducted sampling at the site. According to a sampling report, various pesticides and organics were detected including DDD, DDE, and DDT (U.S. EPA, 1987).

- Soil sampling and analysis performed by U.S. EPA TAT verified the NJDEPE results and revealed widespread occurrences of pesticides in Area A at the site. Areas which, based upon historical photographs and records, were believed not to have been used by the facility (Areas B and C) had pesticide concentrations

in soil up to 110 milligrams per kilogram (mg/kg). Pesticide concentrations of up to 2,400 mg/kg were identified in the disposal area of Area A. Analysis revealed the presence of various pesticides, including DDT, DDD, DDE, alpha BHC, lindane, methoxychlor, heptachlor, and others. The most common and abundant pesticide identified was DDT. DDD and DDE, slightly less common, were often found with DDT (U.S. EPA, 1987).

- Site transformers were sampled and found to have polychlorinated biphenyls (PCB) concentrations less than 50 parts per million (ppm). Site underground storage tanks were also sampled and found to contain no PCBs.

- The U.S. EPA TAT investigated possible off-site migration. A small drainage ditch, which originates in Area A and flows northwest along Area C into a storm sewer that eventually discharges to the north branch of Pennsauken Creek, was identified as a potential pathway for off-site migration of pesticides. It should be noted that this stream is really a constructed drainage ditch and was mislabeled as a stream during initial sampling events. A water sample taken from the ditch along the west boundary of Area C revealed low levels of alpha BHC (20 micrograms per liter (ug/l)) and lindane (21 ug/l). A sediment sample from the same location showed DDT (10 mg/kg) and DDD (27 ug/Kg). A sample of runoff from Area B indicated the presence of DDT (3 ug/l). A soil sample taken in front of a berm near the property line northeast of Building 29 had concentrations of DDT (2,300 mg/kg) and DDD (53 mg/kg) (U.S. EPA).

- The U.S. EPA Environmental Response Team (ERT) performed a ground penetrating radar (GPR) study and obtained soil samples for analysis in December 1987. Soil samples were collected at 14 locations. These locations were determined by stressed vegetation, GPR anomalies, and visible wastes.

Samples were obtained at the surface and at depths of one, three, and five feet for some of the samples (U.S. EPA, 1988).

- The soil samples taken by the ERT contained DDT and its breakdown products, as well as arsenic and sulfur. The sulfur was found to be non-reactive and not a hazard. These other compounds were found at high concentrations in the surface and one-foot deep samples. Significant concentrations of the contaminants were found in deeper samples at two locations, indicating the mixing that may have occurred during trench disposal of these wastes (U.S. EPA, 1988).

- The GPR study indicated a number of areas of disturbance north and northwest of Building 29 which apparently were trenches used for disposal of pesticide wastes and other material associated with site operations (Figure 2). The distribution of waste has also been evaluated on the basis of historical aerial photographs under a U.S. EPA contract (U.S. EPA, 1988). Areas where the ground has been disturbed and where waste may have been buried are shown on Figure 2.

- In July 1991, the New Jersey Department of Transportation (NJDOT) sampled soil along New Albany Road as part of a stormwater construction project. The draft data form this sampling event indicate total pesticide concentrations from 2.0 to 92.7 ppm. The sampling also indicated petroleum odors in the area of the culvert discharge point to the ditch along the railroad tracks in Area B.

The U.S. EPA issued an Administrative Order on Consent to PPG on March 31, 1989 (II-CERCLA-80109). PPG engaged Paul C. Rizzo Associates, Inc. to conduct a Phase I Site Investigation in accordance with a U.S. EPA approved Phase I Site Operation Plan. The intent of the Phase I investigation was to collect soil and groundwater information for the site and determine the constituents of concern.

Preliminary to the investigation, Rizzo conducted both magnetometer and electrical conductivity surveys during early June, 1989. The results of the geophysical surveys were compared to the earlier GPR surveys conducted by the U.S. EPA and were used to help plan the Phase I investigative activities. The field investigative portions of the investigation were conducted between mid-December, 1989 and mid-January, 1990. The Phase I report was submitted to the U.S. EPA on May 25, 1990. Investigative activities for the Phase I included the installation of 20 soil borings and six monitoring wells. Up to three soil samples [shallow (0-2 feet), intermediate (5-7 feet), and deep (10-12 feet)] were collected from each of the soil borings. In addition to these samples, four surface soil samples were collected from the vicinity of the garage in Area B and one sediment sample was collected from the ditch which flows northwest from the area of investigation. Groundwater samples were collected from each of the six monitoring wells. Significant results of the Phase I investigation are summarized below:

- Results of the geophysical surveys indicate that there are two or three areas of buried metal (possibly drums), the most significant of which is the area near the perimeter fence west of Building 29. These areas are indicted on Figure 2.

- Results of the soil sampling activities indicate several constituents of concern in both the surface and subsurface soils of various areas of the site. These constituents include: DDD, DDE, DDT, aldrin, dieldrin, sevin, and alpha, beta, and gamma BHC.

- Analytical results of the sediment sample from the ditch indicate the presence of DDD and malathion. In additional to these pesticides, phenol, benzene, chlorobenzene, ethylbenzene, xylenes, and tetrachloroethane were detected at low levels.

- Results of the groundwater sampling indicate that several constituents of concern are present in the groundwater beneath the site. The following

pesticides were detected in their respective wells: DDT, sevin, malathion, and alpha and gamma BHC in well MW-2; DDT, dieldrin, BHC compounds, and endrin ketone in MW-3; dieldrin and gamma BHC in MW-4; sevin, and BHC compounds in MW-5; and sevin and BHC compounds in MW-6. In addition to these pesticides, organic compounds including xylenes, naphthalene, and 2-methyl-naphthalene were detected in MW-2. 4-methyl-2-pentanone was detected in MW-4. Xylenes, nitrobenzene, 1-methyl-ethyl benzene, carbon disulfide, and diethyl phthalate were detected in MW-5. Tetrachloroethane, 4-methyl-2-pentanone, and endrin ketone were detected in MW-6.

Based on these former investigations, the following groups of analytes were identified at the site:

- VOCs;
- SVOCs including pentachloronitrobenzene (PCNB);
- Pesticides, Malathion, sevin, and Rotenone;
- Dioxins - namely TCDD; and,
- Metals - Arsenic, Lead, Cadmium, and Chromium.

1.3 PROJECT OBJECTIVES

The objectives of the Phase II Investigation Work Plan are to specify investigative procedures that, upon implementation, will provide:

- Completion of the environmental setting characterization of the facility;
- Verification and characterization of the nature and extent of potential constituents of concern;
- Delineation of the nature and extent of the potential constituents of concern source areas;
- Identification of potential environmental receptors, not previously identified; and,

- Assessment of the potential risk, from site conditions, to human health, ecology, and the environment.

The attainment of these objectives and the results of the Phase II Investigation will be adequate to support the evaluation, selection, and development of the appropriate corrective actions during the subsequent corrective measures evaluation.

The Phase II Investigation Work Plan is focused to satisfy specified U.S. EPA requirements for site investigative areas of concern (specified in the AOC). The focus of this investigation is for soils evaluation in the vicinity of specific potential source areas, and groundwater evaluation on a site-wide basis.

1.4 WORK PLAN ORGANIZATION

The main body of this Work Plan is divided into the following 5 Chapters:

- Section 1.0 - Presents introductory comments, background information, and Phase II Investigation Objectives.

- Section 2.0 - Provides the scope of work for field and laboratory activities designed to achieve the Phase II Investigation objectives and fulfill data requirements for corrective action selection. This chapter also describes the methodologies for data analysis and determination of media protection standards.

- Section 3.0 - Presents a discussion on project management and reporting, including monthly reports and agency submittals.

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- Section 4.0 - Summarizes project schedule including the field activities, data analysis, and reporting.

These above chapters are supported by two tables and five figures enclosed herein, as well as the following three appendices:

- Appendix A - Quality Assurance/Quality Control Plan (QA/QC)
- Appendix B - Health and Safety Plan (HASP)
- Appendix C - Field Screening Test Information
- Appendix D - Supporting Information

2.0 PHASE II INVESTIGATION WORK SCOPE

The scope of work for the Phase II Investigation is technically focused and interactive. The interactive approach provides flexibility between successive soil sampling locations to allow for modifications to the site conceptual model as new information is generated. The work plan specifically defines test locations, media to be sampled and quantities, laboratory analyses, and evaluation and assessment procedures required to fulfill Phase II objectives. Specific Project tasks include the following:

TASK 1 - Literature Search
TASK 2 - Soil Sampling and Test Pits
TASK 3 - Groundwater Monitoring
TASK 4 - Surface Water and Sediment Sampling
TASK 5 - Storm Water Sampling
TASK 6 - Building 5 Trench Sampling
TASK 7 - UST Sampling
TASK 8 - Data Analysis
TASK 9 - Potential Migration
TASK 10 - Reporting

The following sections of this chapter present a discussion on the overall technical approach used to develop the above tasks that comprise the project scope of work, followed by a detailed description of the individual tests within the scope of work. This scope of work is supported by the QA/QC plan (Appendix A) and the HASP (Appendix B).

2.1 TECHNICAL APPROACH

The environmental setting of the site and the identification of the respective potential receptors will be addressed on a site-wide basis using existing data and information generated

during the Phase II Investigation. The identification of potential constituents of concern and source characterization will be focused and addressed on an area-specific basis. Table 1 lists the respective areas to be addressed followed by the proposed investigative activities necessary to delineate the area.

2.1.1 Environmental Setting Characterization

Elements of the proposed field program will generate supplementary environmental setting data to be incorporated into the characterization of site conditions. Key elements are as follows:

- Subsurface soil information (geotechnical properties and soil classification data) will be used to characterize and define geologic materials encountered;
- Aquifer characterization data will be used to further understand hydrogeologic conditions; and,
- Groundwater elevation data, for new and existing wells, will be recorded to evaluate the magnitude and direction of hydraulic gradients.

A 1-inch equals 100-ft scale topographic site survey map (similar to Figure 2) will be prepared to illustrate pertinent site conditions. Locations of sample collection completed during the Phase II Investigation will be indicated on this map via additional site survey for graphical presentation.

2.1.2 Source Characterization

To fulfill the requirements of the Phase II Investigation, and to provide sufficient information for potential characterization, source areas must be adequately characterized. A significant portion of this characterization has been addressed in the Phase I SIR, however, additional Phase II activities are necessary to fulfill remaining data requirements of the source characterization. To accomplish source characterization, soils samples will be collected from

areas A, B, and C. In addition, test pits and laboratory analysis will be used to characterize respective trenches.

2.1.3 Potential Constituents of Concern Characterization

The characterization of constituents of concern will be performed through analysis of groundwater and soil samples. The characterization will encompass pertinent existing data (compiled in the Phase I SIR), other historical information, and data generated during implementation of Phase II activities. Implementation of the Phase II activities will provide data to address the following issues:

- Confirm the physical and chemical properties of constituents of concern;
- Delineate the lateral and vertical extent of constituents of concern in the soil;
- Identify the lateral and vertical direction of migration and velocity of constituents of concern;
- Identify factors influencing potential constituent of concern migration; and,
- Determine the potential future movement of identified constituents of concern.

For example, the real-time soil data and the chemical laboratory analytical results of the unsaturated soils will be used to delineate the extent of potential constituents of concern impact in the vadose zone. Soil and groundwater analytical results and aquifer characterization test data will be used to delineate potential constituent of concern migration. Other field/laboratory Phase II activities for different investigative areas are listed in Table 1.

2.1.4 Potential Receptors Identification

The objective of this aspect of the Phase II Investigation is to identify and assess the likelihood of human or environmental receptors being exposed to the potential constituents of concern on site or from off-site migration. The objective of characterizing potential receptors will be made through the completion of a data-search review and compilation (Task

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1) pertaining to both on- and off-site receptors. The potential points of contact with groundwater will be evaluated via information on zoning, determining nearby facility usage, and reviewing existing information. Potential contact with surface water will be evaluated in part by determining potential groundwater contribution. In addition to the evaluation of potential exposure through current uses, exposure through possible future land and groundwater uses will be evaluated.

This process can be used to develop constituent of concern action levels, which can then be compared to existing constituent of concern concentrations to determine if further action at the site is necessary.

2.2 TASK - 1: LITERATURE SEARCH

Preceding or concurrent with the field activities, a literature search will be conducted into the local and regional geology and hydrogeology, local public and private wells, and the potential for migration of contaminants from the site via the groundwater pathway. The literature search will also target information on the following:

- Public wells within one mile of the site;
- Private wells within one mile of the site;
- Groundwater data for the above wells from the last 4 years;
- Floodplain determination; and,
- Wetlands mapping.

This literature search will include, but not be limited to, United States Geological Survey (USGS) reports, NJ Division of Water Policy and Supply reports, and the State of New Jersey well inventory. Some of this information has already been obtained and is included in the previously submitted SIR. Any wells identified will be indicated on a site map.

PPG will obtain a National Wetland Inventory (NWI) map and a USDA Soil Survey for the site from the U.S. Fish and Wildlife Service and the Soil Conservation Service, respectively.

Using these maps, wetlands and/or hydric soils on the property will be identified. Additionally, wetlands will be mapped using the 1987 Corps of Engineers Wetland Delineation Manual.

Flood Plain information will be obtained from FEMA maps maintained at the County Planning Department.

To satisfy the requirements of the National Historic Preservation Act of 1966, Executive Order 11593, and the Archaeological and Historical Preservation Act of 1974, a letter will be submitted to the State Historic Preservation Officer (SHPO) requesting that he identify any potential historic resources in the vicinity of the site. A coy of the request and SHPO response will be submitted to the EPA prior to initiating field activities at the site.

If off-site wells are identified that could be affected by site chemical constituents, these wells will be sampled as described in Section 2.4.2.

2.3 TASK - 2: SOIL SAMPLING AND TEST PITS

General

Additional soil sampling will be performed as part of the Phase II Investigation to define more accurately the extent of contamination found during Phase I and prior investigations. The Phase II soil sampling locations were chosen upon review of existing data and areal coverage. Proposed soil sample locations are depicted on Figure 3. Table 2 summarizes the number of soil samples to be collected (including field screening). The total number of soil samples to be field screened is 199, of which 40 samples are to be sent to a fixed base laboratory (15 from field screening tests and 25 from the remaining soil sampling) for confirmation of the field test methods.

Sample locations include biased sampling of stressed vegetation areas (bare areas), areas which may be potentially impacted by chemicals based on historical aerial photography, drainage areas, or disturbed ground. It is important to note that the soil sampling in Phase II

will be an iterative process guided by field screening results with additional sampling to be conducted if needed after concurrence with the on-site U.S. EPA representative.

The surface soil samples will be obtained from an approximate depth range of zero to six inches. A shovel will be used to remove the surface debris and/or grass. The sample will then be obtained with a stainless-steel trowel. Deeper samples will be obtained using a Geoprobe hydraulically powered sampler and/or drill rig split-spoon sampler, if the location can be accessed with the equipment. The geoprobe sampler consist of a 24 inch long by 3/4 inch internal diameter (ID) stainless steel sample tip with a disposable acetate liner. The split spoon sample is 24 inches long with a two inch I.D. After the sample is collected, it will be removed from the acetate liner, placed in a stainless-steel pan, quartered, and each quarter thoroughly mixed. The quarter portions will then be thoroughly blended and the resultant sample will be placed in the appropriate sample containers. Soil sampling procedures will follow those used in Phase I, and those stipulated in Section 5.2 of the QA/QC plan (Appendix A).

Any material excavated from the sampling point will be replaced in the hole and firmly tamped. The sampling equipment and mixing pan will be decontaminated as described in Section 5.4.2 of the QA/QC Plan.

Field Screening

Field screening for soil contamination will be used to rapidly assess the concentration of pesticides in site soils and to direct additional sampling efforts, if required. Prior to completing Task 2 activities, an initial evaluation of field screening test methods will be conducted. At this time, the two selected methods include a immunoassay method (DDT test kits manufactured by Millipore) and the Dexsil method as described below. Manufacturers' information for these two test kits are included as Appendix C. The scope of the field screening test include the collection of approximately 20 surface soil samples to be analyzed by the two screening methods. The collected samples will represent DDT concentration ranges from low ppm (0 to 5 ppm) to high ppm (> 100 ppm). Sample locations will be

selected based on the results of the Phase I SIR. In addition to field screening, approximately 15 samples will be sent to a fixed based laboratory to be analyzed for TCL pesticides. Based on the results of the screening test, one (or both) test kit(s) will be used for subsequent soil analysis during Task 1. A letter report summarizing the results of field screening (with recommendations) will be submitted to U.S. EPA prior to the initiation of Task 1.

Screening may be conducted using a field chemical extraction method with final chloride measurement (Dexsil). This procedure utilizes sodium metal to remove chloride from a soil sample. The concentration of chloride in the final extract is then measured using a chloride-specific electrode (Finch, *et. al.*, 1990). By measuring the chloride concentration the pesticide concentration can be estimated. The correlation between field screening and actual pesticide concentration will be evaluated initially by analyzing 15 samples, both in the laboratory following Contract Laboratory Procedure (CLP) protocols, and with the field screening protocols. These results will be used to correlate the field results to laboratory values. Data from another U.S. EPA Region II site (Walton Farm) will also be used to assess the correlation. Walton Farm wastes are very similar to the Pulverizing Services waste. The field method assumes all chloride detected is from a pesticide compound.

This screening method is expected to perform well at this site. The screening method was originally developed for PCB screening by Dexsil Corporation of Hamden Connecticut. Our discussions with Dexsil and others that have used the test kits indicate that the same theory applies to pesticides containing chloride such as DDT, DDE, DDD which are the main contaminants of interest at the Pulverizing Services Site. If the field screening results do not correlate well with the laboratory analytical results, total organic halogen (TOX) analysis in the laboratory will be performed. Other screening methods may be considered following discussion with the U.S. EPA prior to field activities.

Soil samples will be collected as described above and taken to the field trailer for preparation. Samples will then be prepped/extracted according to Dexsil's procedures and

analyzed for chloride using the L2000 analyzer. The L2000 has a testing range of 5 ppm to 2000 ppm. The chloride concentration of the sample will be compared to the calibration curves to estimate the pesticide concentration. Testing time is reported to be about 15 minutes per sample. Since the sample analysis time is very rapid, many samples can be analyzed in a short period of time. This will allow a much greater number of samples to be collected to define site contamination rather than waiting several weeks for sample results.

For verification of the screening test sample results, a minimum of 20 percent of the field screened soil samples obtained from Areas A, B and C will also be analyzed for TCL volatiles, TCL semi-volatiles, TCL dioxins/furans, TCL pesticides, TOX, sevin, PCNB, malathion, and cadmium (Table 2). These verification samples will be collected in an attempt to provide a range of sample concentrations within range of the L2000 analyzer. Due to possible sulfur interference, the laboratory will be required to perform a "sulfur cleanup" prior to analysis of the sample.

Proposed Phase II sample analytical parameters are summarized on Table 2. Soil analysis, with the exception of the field test screening, will be conducted in accordance with Contract Lab Program (CLP) standards. Laboratory analysis will be conducted by Enseco-Wadsworth/Alert Laboratories (EWAL) in accordance with Appendix C (laboratory QA/QC Plan) of the Quality Assurance/Quality Control Plan. Soil test kit screening will be conducted in accordance with the manufacturers' specifications included in Appendix C of the Work Plan.

The criteria for selecting the Phase II soil sample locations were guided in part by the following factors:

- The most recent proposed cleanup standards for New Jersey had two cleanup standards; one for zero to two feet and another for two feet deep or greater,
- The site has a relatively high groundwater table beneath it,
- DDT, DDD and DDE were found in greatest concentrations and,

- Except in the disposal areas themselves, significant contamination was limited to the upper few feet of soil.

Appendix D presents a calculation brief supporting the selection of Soil Screening Levels (SSL's) of chlorinated pesticides as a criteria for the collection and analysis of subsurface soil samples.

All soil samples collected will be field screened for arsenic and lead using X-ray fluorescence (XRF) methods. Based on the results of the XRF analysis, 20 percent of the identical samples will then be sent to EWAL for CLP laboratory analysis. Appendix C presents detailed information on XRF field screening methods.

Based on these factors, soil sampling outside of disposal areas will generally be conducted in the first four feet of soil and above the site water table. The field team leader may adjust sample locations with EPA approval. If analytical results indicate significant contamination at these depths, the flexibility of field analysis allows for additional samples to be collected for analysis, if needed, based upon real-time data generated by the field screening approach. The decision for additional samples will be made in the field after discussion with the U.S. EPA on site representative.

In order to evaluate potential soil treatment technologies additional information is required regarding the soil physical characteristics. A total of six soil samples will be selected from the current sample locations in Area A and B for analysis. Laboratory analysis of these samples will be performed for the following parameters:

- Grain size distribution
- Atterberg limits
- Total Organic Carbon (TOC)
- Moisture Content
- pH

In-situ soil moisture content and density testing will also be performed at several locations across the site. This testing will be performed using nuclear measurements per ASTM 2922 and 3017.

2.3.1 Subtask 2.1: Area A Soil Sampling

Surface soil samples will be obtained off-site along of Area A outside the fence along the northeast side (four samples) and southwest side (three samples) of the property (Figure 3) to assess contaminant migration. Two subsurface samples will be obtained along the southeast side of the property along New Albany Road (Figure 3) in the former sulfur storage area. Additional samples will be obtained throughout Area A at various depths. Surface samples collected in paved areas will consist of soil just below the pavement. These sample locations are also shown in plan on Figure 3. A total of 28 soil sample locations are proposed for Area A and the off-site property. Initial Area A soil samples will be collected from 0-0.5 feet, 1-2 feet and 3-4 feet below ground surface. If samples at the 3-4 foot level indicate significant concentrations of concern, then a deeper sample(s) will be collected for field screening. This decision will be made with the concurrence of the U.S. EPA's on-site representative.

In addition to the analysis listed in Section 2.3, soil samples from Area A will be analyzed in the field laboratory for arsenic and lead; based on the results of which, twenty percent of the samples will also be analyzed for cadmium and chromium. In addition 20% of the field-analyzed samples will be sent for confirmatory laboratory analysis.

A minimum of 84 samples will be field screened using immunoassay and XRF techniques. Based on the results, 17 samples will be sent to EWAL for analysis of Table 2 parameters.

2.3.2 Subtask 2.2: Area B Soil Sampling

Twenty soil sample locations are proposed in the area to the southeast of the buildings in Area B. The approximate location of these samples are shown on Figure 3. The results from analysis of these samples should allow evaluation of the extent of surface and subsurface contamination in this area. The same sample depths, analytical parameters, protocols, and screening procedures described above for the Area A samples will be used for Area B soil samples.

These sample locations have been selected to target known and potential areas of contamination and to provide a broad coverage of the area. Soil sample locations are in potential drainage wells and potentially impacted areas as indicated by aerial photographs. There are a total of 21 sample locations for Area B, including one associated with the septic tank adjacent to the garage. In order the address the potential presence of chemicals associated with the septic tank, one sample will be collected at a location adjacent to the observed septic tank disturbance. Based on these results, additional samples may be collected. This decision will be made with the concurrence of the U.S. EPa's on-site representative.

A minimum of 63 samples will be collected and field screened using immunoassay and XRF methods. Thereafter, 13 samples will be sent for analysis of parameters listed in Table 2.

2.3.3 Subtask 2.3: Area C Soil Sampling

Fourteen soil sample locations are proposed in Area C, northwest of Area A. The approximate locations of these samples are shown on Figure 3. The results from analysis of these samples should allow evaluation of the extent of surface and subsurface contamination in this area. The same sample depths, protocols, and screening procedure used in Areas A and B will be used for Area C samples. There is a total of 14 soil sample locations for Area

C which comprise 52 soil samples for immunoassay and XRF field screening. Ten soil samples will be sent to EWAL for analysis of Table 2 parameters.

2.3.4 Subtask 2.4: Area A Test Pits

To better define the extent of subsurface contamination behind Building 29, four to five test pits are proposed for Area A. The locations of the proposed test pits are depicted on Figure 3. Test pits are more cost effective than exploratory borings because the sampling points can be selected on the basis of visual observation across a large area. A substantial number of borings would be required to provide the same amount of information that can be obtained using test pits.

Two to three exploratory test pits will be excavated perpendicular to the assumed direction of the disposal trenches. The exploratory test pits will be excavated to an approximate depth of eight to ten feet. The depth to groundwater in this area is estimated to be between two and six feet, it is unlikely that wastes were disposed below the ground water table. One trench will start near Boring B-6 and progress toward Building 29. When 10 to 20 feet of exploratory trench no longer intersects a disposal trench, the trenching equipment will return to the starting point and work in the opposite direction in a similar manner, until disposal trenches are no longer found. This would constitute one exploratory trench. Another trench would be excavated in an identical manner near Boring B-8. If waste material is visually identified at the limit of the backhoe's reach, alternate equipment such as a larger backhoe with greater reach will be mobilized to assess the vertical limits of waste disposal. An additional test pit may be excavated in this area depending on the results of the previous test pits. The decision to complete an additional test pit in the vicinity of the disposal trenches will be based on apparent lateral extent of the trenches and field screening results of the soul samples from the trenches.

The trenches will be excavated with a backhoe and will be approximately two feet wide. Any material removed from the trench will be placed on polyethylene sheeting or into a

dump truck, and the material will be returned to the trench when sampling is complete. No work will be performed during heavy rainfall when surface transport of any excavated contaminants could occur or when the wind exceeds 20 miles per hour. Any drums encountered during trenching will be left in place. If any drums are encountered, soil sample will be collected from adjacent to the drums.

Soil samples will be taken below the limits of waste disposal and at about five feet below the surface between disposal trenches and near the outside ends of the exploratory trenches. A total of 10 to 25 soil samples will be taken depending on the number of disposal trenches encountered. The soil samples will be analyzed in the field for pesticides using methods described in Section 2.3.

Verification of the screening test sample results will be conducted for a minimum of five samples or 20% of the soil samples collected from the test pits (including those collected during subtask 2.5). These samples will be submitted for analysis of TCL volatiles, TCL semi-volatiles, TCL dioxins/furans, TCL pesticides, TOX, sevin, PCNB, malathion, and cadmium. In addition, at least 20 percent of the soil samples collected from within the disposal trenches (a minimum of five samples) will also be tested for rotenone. The results of this testing will be evaluated to determine whether additional testing of the trenches and/or testing outside of the trenches will be necessary.

In addition to the visual inspection, the trenches will also be photographically documented. Once the documentation is complete and the samples have been obtained, the trench will be lined with plastic sheeting and the excavated material will be returned to the trench. The trench location will then be covered with plastic.

One test pit will be excavated at the edge of the buried drum location to assess the integrity of buried drums (Figure 3, Area A). The test pit will be carefully excavated working towards the drum location. Material around the first drum encountered will be temporarily removed to allow visual assessment of the drum. If the drum is open or rusted through, a

sample of the drum contents will be collected. A second sample will be collected from below the drum location. If the drum appears to be intact, only a sample from below the drum will be collected. A second test pit will be excavated between B-11 and B-12 to assess the EM conductivity anomaly (Figure 3, Area A).

During the trenching and sampling in Area A, air monitoring will be performed at the site perimeter in a downwind direction. Sample collection and preparation will follow U.S. EPA Method T04. Method T04 uses a high-volume sampler consisting of a glass fiber filter with a polyurethane foam (PUF) backup absorbent cartridge. After preparation, analysis will be for TCL pesticides following the methodology identified in the QA/QC Plan. The detection limit using the above procedures is approximately 1.0 nanogram per cubic meter (ng/M^3).

2.3.5 Subtask 2.5: Areas B and C Test Pits

Waste disposal at depth in Areas B and C is not suspected, although the property owner has stated that debris from a plant fire in 1964 may have been buried behind the office in Area B. In order to assess this possibility, additional test pits will be excavated in Areas B and C, three in Area B and four in Area C (Figure 3). If no waste is encountered, excavation will stop at five to six feet below the ground surface. If waste is encountered, excavations will proceed to the bottom of waste or the limit of the backhoe whichever is encountered first.

If waste is encountered, two samples will be collected; one from the waste material and one from below the limits of waste disposal. These samples will be analyzed for Table 8-1 parameters. Additional test pits may be excavated to define the disposal limits.

2.4 TASK - 3: GROUNDWATER MONITORING

In accordance with the AOC, groundwater monitoring will take place to assess the nature and extent of groundwater contamination.

Based on the results of analysis of groundwater samples from the six monitoring wells installed as part of the Phase I Investigation and piezometer water level observations (Subtask 3.3), a minimum of 4 additional wells in the shallow aquifer are proposed. Also, the site's deep production well will be resampled. For all monitoring wells, a geologic boring log of samples obtained will be prepared as well as a schematic drawing showing the details of monitoring well installation. Drilling, sampling, and monitoring well installation procedures will be as indicated in the QA/QC Plan.

2.4.1 Subtask 3.1: Piezometer Installation

Piezometers will be used to refine the groundwater flow direction. Based on this information, monitoring wells will be installed.

Small diameter piezometers will be installed in numerous areas of the site. Since the water level data from Phase I indicates a very flat water table over much of Area A these piezometers will help define the shallow groundwater flow direction. Based on the water levels defined by the existing monitoring wells and the new piezometers and the groundwater analytical data obtained in Phase I, it is currently anticipated that new monitoring wells will be installed during Phase II. This approach will optimize the placement of monitoring wells. Piezometers will be numbered in the sequence of installation. Piezometer depths will be dependent upon the depth at which saturated conditions (i.e. the water table) is encountered. Following installation, piezometer locations will be surveyed to establish horizontal coordinates and vertical elevations. All pertinent installation information (total depth, screened internal, etc.) will be included on a table and locations of numbered piezometers will be depicted on a site map in the final reports.

The piezometers will be installed to below the water table using the Geoprobe system. This system uses a hydraulically powered one-inch diameter probe to make a pilot hole to the required depth. Upon reaching this depth, the probe is extracted and a polyvinyl chloride (PVC) or stainless steel piezometer of the same or larger diameter is inserted in the hole.

Since the probe and the piezometer are the same diameter, there is no annulus to backfill. Given the shallow water table in the site area, several of these piezometers can be installed in a work day.

A total of 21 piezometers will be installed to supplement existing water level data (Figure 4). They will be installed in the following areas:

- Area A - nine piezometers will be installed to further define the shallow groundwater table and to define the groundwater flow direction.

- Area B - six piezometers will be installed to define the groundwater flow direction in this portion of the site. No piezometers are necessary at the swampy southern end of Area B since a surface water staff gage is already installed in this area. The data from these piezometers will be combined with Area A data to assess the potential for contaminant migration.

- Area C - six piezometers will be installed to define the groundwater flow direction in this portion of the site. The data from these piezometers will be combined with Area A data to assess the potential for contaminant migration.

2.4.2 Subtask 3.2: Groundwater Sampling

After the four new monitoring wells are installed on-site, they will be purged and sampled in accordance with the Phase I QA/QC Plan. The Plant Production Well was sampled by the U.S. EPA during preliminary site evaluation and was determined not to be contaminated. If the existing Plant Production well is accessible, it to will be sampled again. Based on the Phase I data, these wells will be analyzed for the following parameters:

- TCL volatiles, semivolatiles, and pesticides;

- arsenic, cadmium, chromium (including hexavalent chromium), and lead (total and dissolved);
- sevin rotenone, and malathion; and
- Dioxins (these will be analyzed for following review of the soil dioxin data).

Off-site wells (public or private) will be considered for sampling if the site production well is contaminated with site contaminants at a level of concern. A letter report will be prepared and submitted to the U.S. EPA which discusses results of the production well sample analysis and assesses the need for off-site well sampling. If the site production well is contaminated, the decision as to which off-site well(s) are to be sampled will be made considering proximity to the site and groundwater pump rates in the site vicinity.

2.4.3 Subtask 3.3: Water Level Elevation Measurements

Water surface elevations in piezometers and new and existing monitoring wells will be periodically measured (a minimum of once a month for three months) to confirm the information gathered from the first round of water elevations collected after installation of the Phase II monitoring wells. Groundwater measurements will be collected with an electronic measuring tape (M-scope or equivalent) and will be recorded to the nearest hundredth of a foot. A groundwater elevation contour map will be prepared based on the results of water levels recorded during Phase II. The map will include the date and time of water level measurements.

A rain gauge will be installed to monitor on-site precipitation. Precipitation will be monitored for at least one week prior to obtaining groundwater elevation measurements. Local airport meteorological data will also be obtained.

2.4.4 Subtask 3.4: Additional Monitoring Well Sampling

If off-site wells are identified (Section 2.2) which could potentially be affected by the site, a program will be implemented to sample these wells. Sampling procedures will be in accordance with the Phase I QA/QC Plan. Analysis parameters will be determined based on the location of the well and the migration potential of site contaminants.

2.5 TASK - 4: SURFACE WATER AND SEDIMENT SAMPLING

Three surface water and sediment samples will be obtained from the current drainage ditch which flows northwest from Area A to Area C (Figure 3). These samples will be collected near the point at which the sediment sample was taken during the Phase I Investigation as well as upstream near the start of the ditch and downstream where the ditch enters the stormwater system. In addition, two surface water and sediment samples will be obtained from the drainage ditch along the railroad track in Area B and two surface water samples and sediment samples will be collected from the swampy area along the southern boundary of Area B. The approximate locations of these samples are shown on Figure 3. Sediment samples will first be screened using the methods described for pesticides in Section 2.3. Surface water sediment samples will be analyzed by CLP methods for TCL volatiles, semivolatiles, pesticides, arsenic, cadmium, chromium (including hexavalent chromium), lead, malathion, rotenone, and sevin. Based on the evaluation of results of surface water and sediment sampling, additional sampling locations may be proposed.

In addition, general water chemistry parameters will include hardness, dissolved organic carbon, pH, temperature and specific conductance for surface water and TOC and pH for sediment. Hydrologic characteristics will also be noted or estimated. These include flow rate, velocity, depth, width of channel, seasonal or perennial nature, and plant and animal life as appropriate.

Data collected during sampling of the Area B drainage ditch in (May 1993) as included in the SIR will be considered during the evaluation of the Phase II data.

2.6 TASK - 5: STORM WATER SAMPLING

The site storm sewer system will be assessed for contamination by sampling the sediment of two catch basins located along the piping system prior to their outlet to surface ditches. The locations of these catch basins are shown on Figure 4. If sediments is present in the catch basin it will be sampled and analyzed for TCL pesticides, arsenic, cadmium, chromium, lead, sevin, and malathion.

2.7 TASK - 6: BUILDING 5 TRENCH SAMPLING

The location of the reported trench behind former Building 5 will be assessed by visual inspection of the area for cleanouts, manholes, etc. If material is found in the trench, it will be first screened for pesticides using methods described in Section 2.3 and then sampled and analyzed for TCL volatiles, semivolatiles (including PCNB), pesticides, dioxins, arsenic, cadmium, chromium, lead, sevin rotenone and malathion.

2.8 TASK - 7: UST SAMPLING

The location of the three underground storage tanks (USTs) will be assessed visually (approximate locations shown on Figure 4). Once located, these USTs will be accessed by opening the fill port or manway. These tanks are reported to have contained fuel oil for use at the site. Tank contents will be sampled and analyzed for RPH using gas chromatography to identify the product. If visible soil contamination is present soil samples may be field screened for TRPH. If product is identified in the tank, it will be removed from the tank and the tank cleaned. Tank cleaning procedures will be submitted to the U.S. EPA, if needed, prior to cleaning.

2.9 TASK - 8: DATA ANALYSIS

Data quality and quantity will be adequate to define the extent of constituents in the soil and groundwater, characterize the constituents, identify potential threats to human health and the environment, and support the requirements of potential corrective measures. The Phase II Investigation report will, at a minimum, identify the following:

- The extent of the source areas;
- The extent and magnitude of constituents present in the soil and groundwater;
- The characteristics (physical and chemical) of the constituents;
- The characteristics of the impacted media units (vadose zone and the saturated fill); and,
- The potential migration pathways.

2.10 TASK - 9: POTENTIAL MIGRATION

Contaminant sources and concentrations in each medium will be analyzed to characterize contaminant migration pathways associated with the Site. Potential exposures to surrounding ecological and human receptors will be identified.

As part of the investigation, data on potential receptors will be collected. Data collected will describe the human populations that are potentially susceptible to releases from the site. The following will be identified:

- Local uses and possible future uses of groundwater, including type of use;
- Local uses and possible future uses of facility surface waters drainage;
- Human use or access to the facility and adjacent lands;
- A demographic profile of the people who use or have access the facility and adjacent land;

- A description of plant and animal species observed near the site.
- Exposure pathways and potential human receptors; and
- Exposure pathways and potential environmental receptors.

Exposure via an airborne pathway will be evaluated using the observations made during the air sampling for TCL Pesticides (During test pit completion). Air sampling activities are as described in Section 2.3.4 of the Work Plan. Local meteorological data will be modeled to determine whether the air pathway poses a significant exposure route.

The intent of the above evaluation will be to obtain sufficient data to develop site specific healthbased cleanup levels.

3.0 PROJECT MANAGEMENT AND REPORTING

To fulfill the requirements of the Order, progress reports will be provided to the U.S. EPA throughout the duration of the Phase II Investigation process; at the conclusion of the Phase II Investigation findings will also be prepared and submitted to the Agency. Both reporting requirements are detailed in the following two sections.

3.1 PROGRESS REPORTS

Progress reports will be submitted to the U.S. EPA on or before the tenth day of every month (from February 1994), until such time as all the requirements of the Order are fulfilled. Four copies of the progress reports will be submitted to the U. S. EPA Project Coordinator, via certified mail, return receipt requested, or hand-delivered.

The progress reports will address, at a minimum, the following items:

- Description of work-in-progress and an estimate of the percentage of the Phase II Investigation completed;
- Summation of new findings during the reporting period;
- Summation of changes or modifications to the approved Phase II Investigation Work Plan during the reporting period;
- Summation of problems of technical or scheduling nature for the reporting period and associated rectifying actions taken;
- Changes in project personnel for reporting period;
- Summation of contacts with representatives of the community, public interest groups, or state government for the reporting period regarding RFI activities; and,
- Projected work for the subsequent reporting period.

3.2 DRAFT PHASE II INVESTIGATION REPORT (Task 10)

All data and information generated throughout the duration of the Phase II Investigation will be compiled, validated (if necessary), and evaluated in the Phase II Investigation Report. A final Phase II Investigation Report, incorporating the Agency's comments, will be submitted to the EPA Project Coordinator in accordance with the AOC.

The findings and conclusions of the Phase II Investigation will be presented in the form of text, tables, and figures. These presentations will include, but not be limited to, the following information:

Tabular Information:

- Summary of sample media, samples collected and analytical program;
- Summaries of ground water and soil chemical laboratory analyses generated during implementation of the field program with select comparison to historical data;
- Summaries of field testing data for ground water, soils, and bedrock;
- Summary of geotechnical data for soils;
- Summary of in-situ aquifer characterization tests;
- Summary of ground water elevation data;
- Summary of monitoring well construction data for new and existing wells.

Graphical Presentation:

- Site location map which depicts residences, schools, and public buildings in the vicinity of the site;
- Site topographic map;
- Plan maps indicating sample locations for all matrices;
- Ground water elevation contour maps;

- Isoconcentration maps for select ground water data;
- Plan view of geophysical survey results;
- Isoconcentration maps for select soil gas survey data;
- Geologic cross sections based on existing and newly generated subsurface data, including lithologies and water elevations, and if appropriate distribution of Constituents of Concern in the subsurface media at various depths;
- Soil boring and test pits lithologies, sample collection intervals and screening data; and,
- Monitoring well construction diagrams for new wells.

These methods of data presentation will be used to support the conclusions of the report with regard to site environmental conditions, source characterization, constituent of concern characterization, identification of potential receptor, and support of the CMS.

Additional information that will be presented as appendices to the Phase II Investigation Report will, at a minimum, include:

- Test pit logs;
- Boring and well logs;
- Analytical summary sheets from the chemical and geotechnical laboratories;
- Field data for the in-situ aquifer characterization tests and subsequent calculations;
- Summary report of electromagnetic survey;
- Summary report of downhole geophysical tests;
- Equipment calibration logs;
- Ground water development sheets;
- Ground water purge and sampling sheets; and,
- Summary of data validation reports.

A discussion of the field tasks, including modifications of additions to the work scope presented herein, will be presented. The conclusions of the report will address the site environmental setting, the source characterization, and characterize the potential receptors of constituents of concern.

4.0 PROJECT SCHEDULE

The anticipated Phase II Site Investigation schedule for Task-1 through Task-10 is presented in Figure 6. The project schedule will be tracked and updated on a monthly basis in conjunction with the monthly report to U.S. EPA. A copy of the schedule will then be included in the monthly report (if any changes in the schedule are made).

REFERENCES

Finch, S. R., Lavingue, D.A., and Scott, R.P.W, 1990, "One Example where Chromatography may not necessarily be the best analytical method", Journal of Chromatographic Science, Vol. 28, July 1990, P 351-356.

Paul C. Rizzo Associates, May 1990, Phase I Site Investigation, Pulverizing Services Site, Moorestown, New Jersey.

Skacel, Wolf, November 1986, Memorandum to Vince Krisak from Wolf Skacel through Mark S. Gruzlovic.

U.S. EPA, October 1987, Pulverizing Services Sampling Report, by U.S. EPA TAT, TAT-02-F-04345.

U.S. EPA, February 1988, Site Analysis, Pulverizing Services Site, Morrestown, New Jersey, for the Environmental Monitoring Systems Laboratory, Las Vegas, Nevada (TS-PIC-88058).

TABLE 1
AREA OF INTREST AND PHASE II INVESTIGATIVE ACTIVITIES

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

INVESTIGATIVE AREA	PHASE II INVESTIGATIVE ACTIVITY
AREA A	Surface/Subsurface Soil Sampling Test Pit Sampling Piezometers Installation Monitoring Well Installation Monitoring Well Sampling
AREA B	Surface/Subsurface Soil Sampling Test Pit Sampling Piezometers Installation Monitoring Well Installation Monitoring Well Sampling
AREA C	Surface/Subsurface Soil Sampling Test Pit Sampling Piezometers Installation Monitoring Well Installation Monitoring Well Sampling
FORMER DISPOSAL TRENCH	Soil Sampling Test Pit Sampling
GEOPHYSICAL ANOMALY	Soil Sampling Test Pit Sampling
BURIED DRUM	Soil Sampling Test Pit Sampling
SURFACE WATER AND SEDIMENT	Soil/Water Sampling
STORM WATER AND SEDIMENT	Soil/Water Sampling
BUILDING 5 TRENCH	Soil Sampling
UST SAMPLING	Soil Sampling Contents Sampling
SEEPAGE PIT	Surface/Subsurface Soil Sampling

TABLE 2
PHASE II SAMPLE SUMMARY

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

SAMPLE MEDIA	NO. OF SAMPLES	DUPLICATES	REPLICATES	TRIP BLANKS	FIELD BLANKS	ANALYTICAL PARAMETERS
SOIL	15(1)	2	2	1	1	Field Test Kit Screening - Total Chlorinated Pesticides Test Kit Analysis - Arsenic, Lead
	199	10	10	4	4	Field Test Kit Analysis - Total Chlorinated Pesticides - Arsenic, Lead
	40(2)	5	5	1	1	TCL, VOCs, SVOCs, Pesticides, TOX(3), Sevin, PCNB Malathion, Arsenic, Lead, Chromium, Cadmium
	10(4)					Dioxins (+5 QA/QC Per Region II SOPs)
	6					Geotechnical Analysis
SURFACE WATER	7	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion, Rotenone Metals: Arsenic, Lead, Chromium, Cadmium General Chemistry: Field pH, temp, and spec. cond.
SEDIMENT	7	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion Metals: Arsenic, Lead, Chromium, Cadmium General Chemistry:pH, TOC
GROUNDWATER	Approx 10	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion Total and Dissolved Metals: Arsenic, Lead - Chromium, Cadmium General Chemistry: Field pH, temp, and spec. cond.
	10					Dioxins (Based on Soil Sampling Results)
TEST PITS	10 to 25	2	2	1	1	Test Kit Field Survey Analysis
	5	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion, Herbicides, PCNB Rotenone, Dioxins, TOX Metals: Arsenic, Lead, Chromium, Cadmium
AIR SAMPLING	3					TCL Pesticides
UST	3	1	1	1	1	Total Petroleum Hydrocarbons

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TABLE 2 (continued)

NOTES:

(1) Indicates that these samples will be collected to select the appropriate field screening test kit (*i.e.* Dexsil or Millipore)

(2) These samples are intended for fixed base laboratory confirmatory analysis, and comprise 20 % of the total number of samples collected for field screening..

(3) Chromium analysis indicates both total chromium and hexavalent chromium will be analyzed for.

(4) Dioxin analysis will be of composite samples from the 40 samples to be sent for confirmatory analysis. QA/QC for these samples will be as per Region II Guidance.

(5) Two additional sediment samples may be obtained from the storm sewer catchment basins (if sediment is present within the basins).

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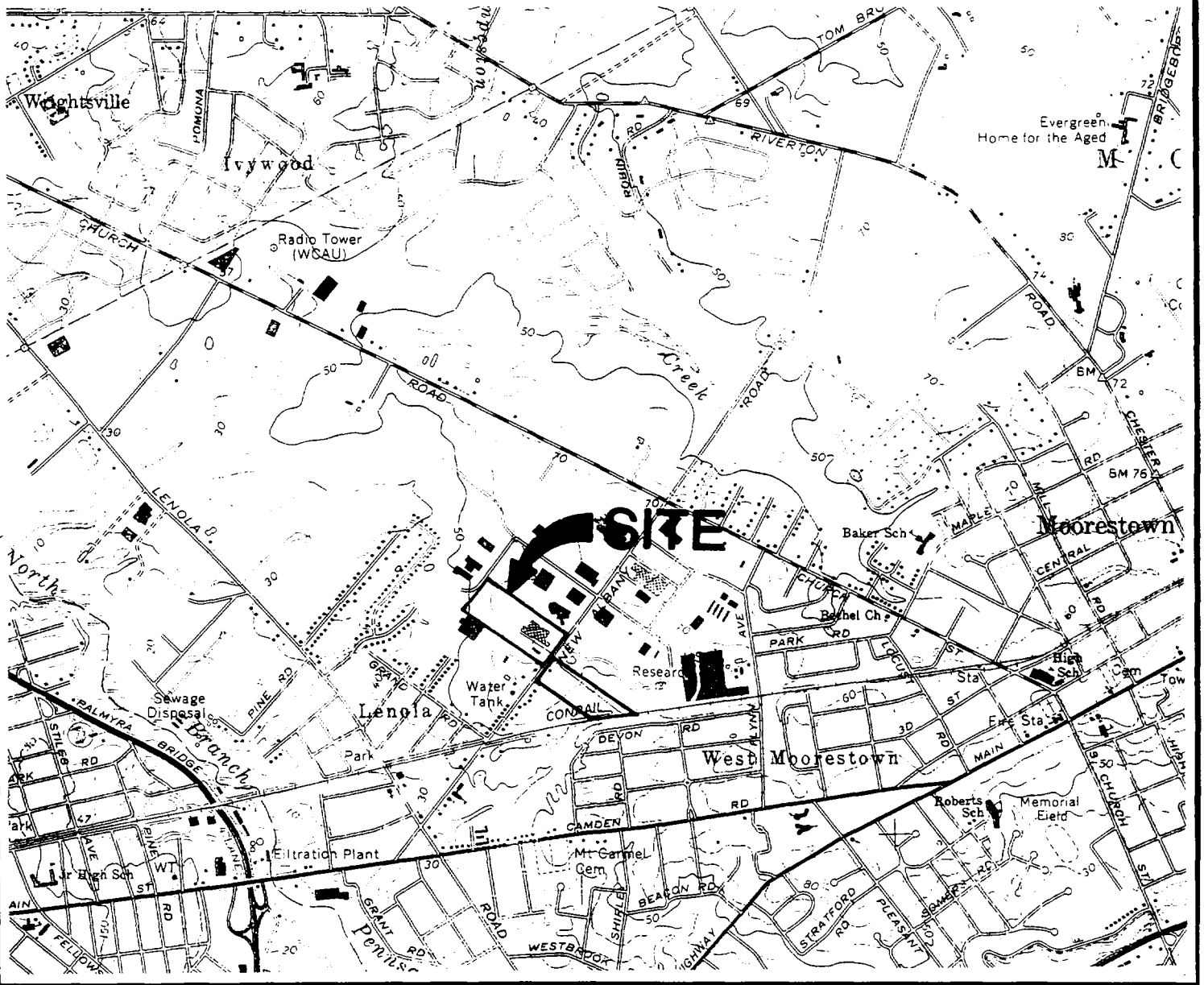
REFERENCED U.S.G.S. MAP

MOORESTOWN, NJ
USGS 7.5 MIN.

1965
PHOTOREVISED 1986

QUADRANGLE LOCATION



SCALE 1:24000

1 1/2 0 1 MILE

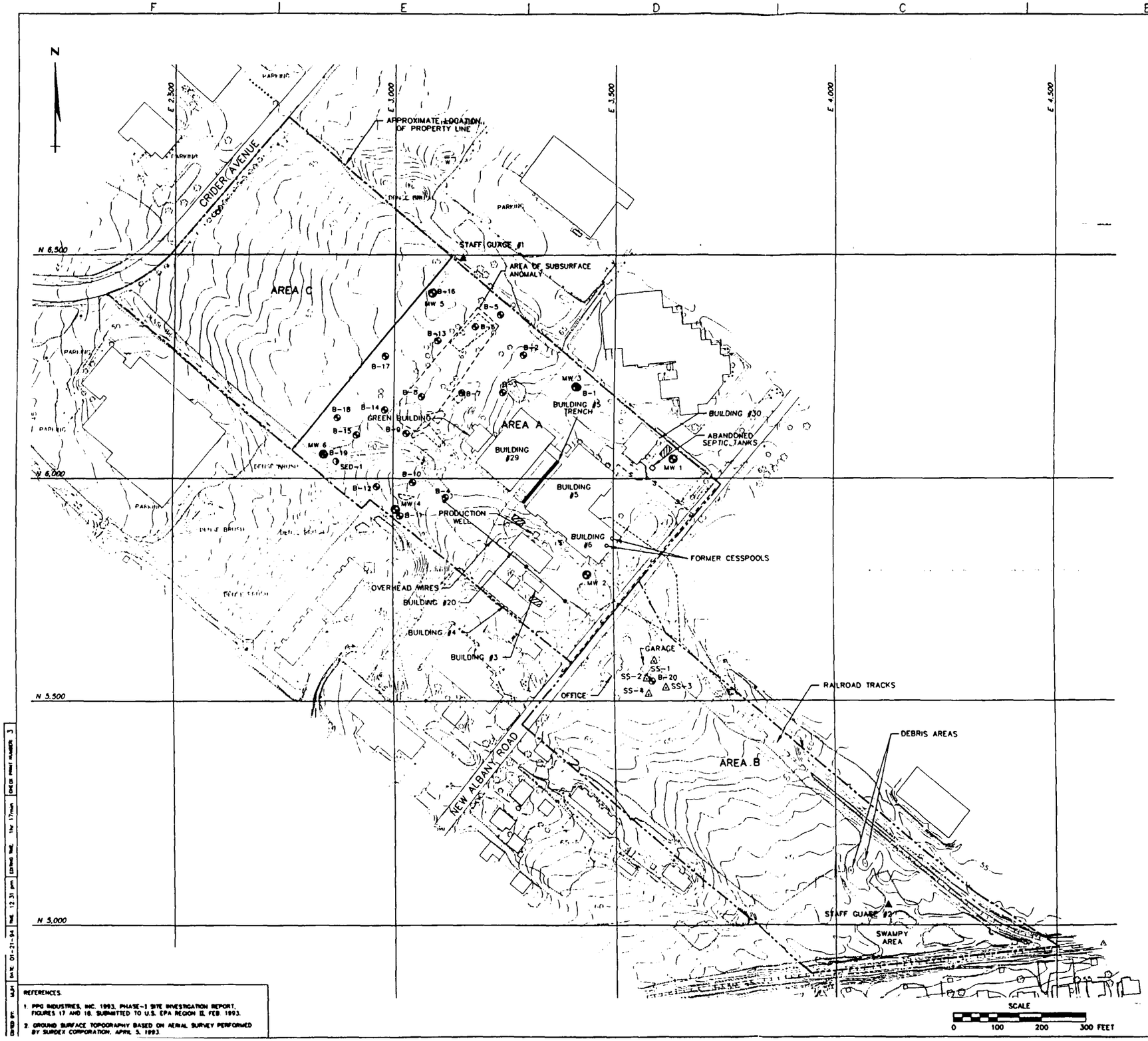
1000 0 1000 2000 3000 4000 5000 6000 7000 FEET

1 .5 0 1 KILOMETER

301634

PHASE-I INVESTIGATION REPORT	DRWN: MJH	CHKD:	APPD:
	SCALE: AS SHOWN		DATE: 1-21-94
PPG INDUSTRIES, INC. PITTSBURGH, PENNSYLVANIA	SITE LOCATION MAP		DRAWING NUMBER 09-0801461 FIGURE 1

(1461A001.DS)



LEGEND:

- EXISTING STAFF GAGE
- MW 1 MONITORING WELL LOCATION
- B-1 PHASE I SOIL BORING LOCATION
- SS-3 PHASE I SOIL SAMPLE LOCATION
- FENCE
- TREELINE
- SANITARY SEWER DRAIN LINE
- STORM SEWER DRAIN LINE
- UNDERGROUND STORAGE TANK
- BARE AREA
- GROUND SURFACE ELEVATION CONTOUR
- PROPERTY LINE (APPROXIMATE LOCATION)
- AREA DIVIDE LINE

NOTES:

1. AREAS A, B, AND C AS DESCRIBED IN SECTION 1.0 OF WORK PLAN.
2. ALL ELEVATIONS IN FEET ABOVE MEAN SEA LEVEL (FT-MSL).

301635

REFERENCES:

1. PPG INDUSTRIES, INC. 1993, PHASE-I SITE INVESTIGATION REPORT, FIGURES 17 AND 18. SUBMITTED TO U.S. EPA REGION II, FEB. 1993.
2. GROUND SURFACE TOPOGRAPHY BASED ON AERIAL SURVEY PERFORMED BY SURDEX CORPORATION, APRIL 5, 1993.

REV #	DATE	DESCRIPTION	APPD

PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

McLaren Hart
ENVIRONMENTAL ENGINEERING CORPORATION

DRWN	MJH	DATE	1-20-94
CHKD		DATE	
APPD		DATE	
SCALE	AS SHOWN		

PHASE II SITE INVESTIGATION
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

SITE PLAN

DRAWING NUMBER 09-0801461

FIGURE 2

DATE

PENN CENTER WEST III
SUITE 106
PITTSBURGH, PA. 15276

FILE 14610001.DWG

CHECK PRINT NUMBER 3

REVISED

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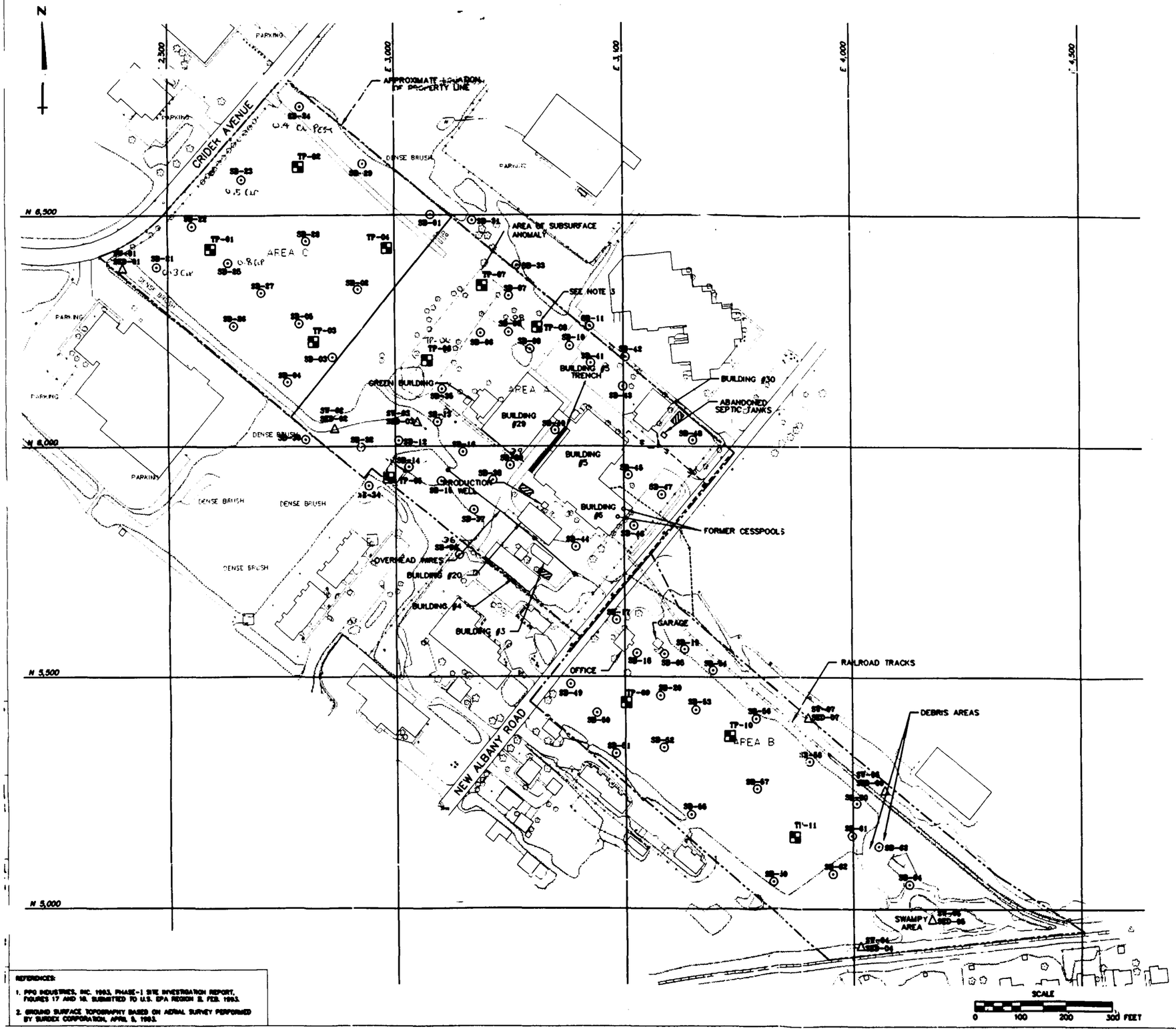


REFERENCES:

1. PPG INDUSTRIES, INC. 1993, PHASE-I SITE INVESTIGATION REPORT, FIGURES 17 AND 18. SUBMITTED TO U.S. EPA REGION III, FEB. 1993.
2. GROUND SURFACE TOPOGRAPHY BASED ON AERIAL SURVEY PERFORMED BY BURDEX CORPORATION, APRIL 8, 1993.

LEGEND:

- PROPOSED TEST PIT LOCATION
- PROPOSED SOIL SAMPLE LOCATION
- PROPOSED SURFACE WATER AND SEDIMENT SAMPLE LOCATION
- FENCE
- TREELINE
- SANITARY SEWER DRAIN LINE
- STORM SEWER DRAIN LINE
- UNDERGROUND STORAGE TANK
- BARE AREA
- GROUND SURFACE ELEVATION CONTOUR
- PROPERTY LINE (APPROXIMATE LOCATION)
- AREA DIVIDE LINE

NOTES:

1. AREAS A, B, AND C AS DESCRIBED IN SECTION 1.0 OF WORK PLAN.
2. ALL ELEVATIONS IN FEET ABOVE MEAN SEA LEVEL (FT-MSL).
3. TEST PIT TO ASSESS BURIED DRUM.

REV #	DATE	DESCRIPTION	APPD

PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

McLaren Hart
ENVIRONMENTAL ENGINEERING CORPORATION

DRWN	W.J.H.	DATE	1-20-94
CHKD		DATE	
APPD		DATE	
SCALE	AS SHOWN		

PHASE II SITE INVESTIGATION
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

PROPOSED SOIL SAMPLING, SEDIMENT SAMPLING AND TEST PIT LOCATIONS

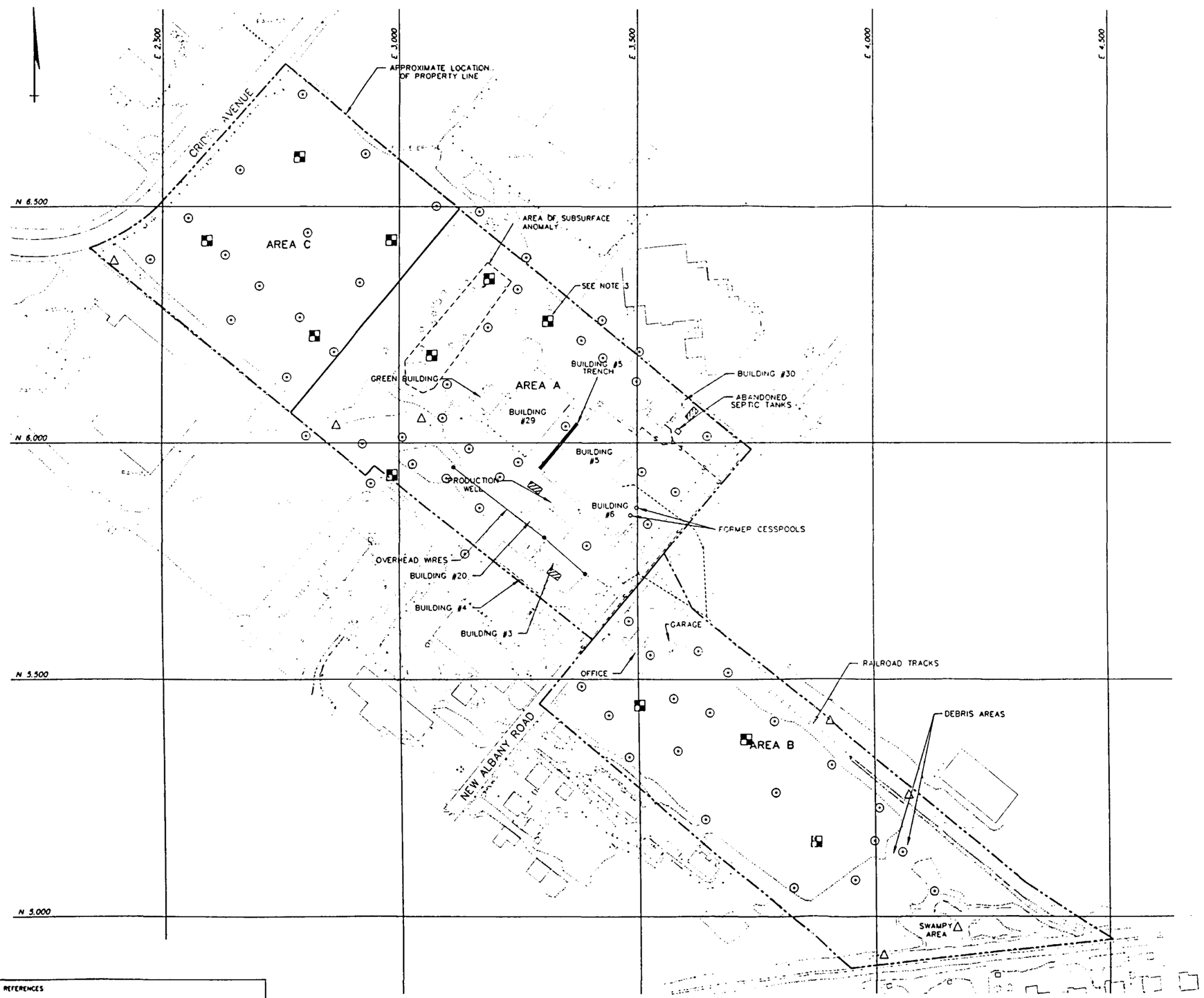
DRAWING NUMBER 00-0001401

FIGURE 3

DATE

8.10C BROOKTREE ROAD
STE. 300
WOXFORD, PA. 15090

FILE: 14010002.DWG



LEGEND

- PROPOSED TEST PIT LOCATION
- PROPOSED SOIL SAMPLE LOCATION
- PROPOSED SURFACE WATER AND SEDIMENT SAMPLE LOCATION
- FENCE
- TREELINE
- SANITARY SEWER DRAIN LINE
- STORM SEWER DRAIN LINE
- UNDERGROUND STORAGE TANK
- BARE AREA
- GROUND SURFACE ELEVATION CONTOUR
- PROPERTY LINE (APPROXIMATE LOCATION)
- AREA DIVIDE LINE

NOTES:

1. AREAS A, B, AND C AS DESCRIBED IN SECTION 1.0 OF WORK PLAN
2. ALL ELEVATIONS IN FEET ABOVE MEAN SEA LEVEL (FT-MSL)
3. TEST PIT TO ASSESS BURIED DRUM

301637

REFERENCES

1. PPG INDUSTRIES, INC. 1993. PHASE-I SITE INVESTIGATION REPORT. FIGURES 17 AND 18 SUBMITTED TO U.S. EPA REGION II. FEB. 1993
2. GROUND SURFACE TOPOGRAPHY BASED ON AERIAL SURVEY PERFORMED BY SURDEX CORPORATION, APRIL 3, 1993

REV #	DATE	DESCRIPTION	APPD

PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

McLaren Hart®
ENVIRONMENTAL ENGINEERING CORPORATION

DRWN	MJH	DATE	1-20-94
CHKD		DATE	
APPD		DATE	
SCALE	AS SHOWN		

PHASE II SITE INVESTIGATION
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

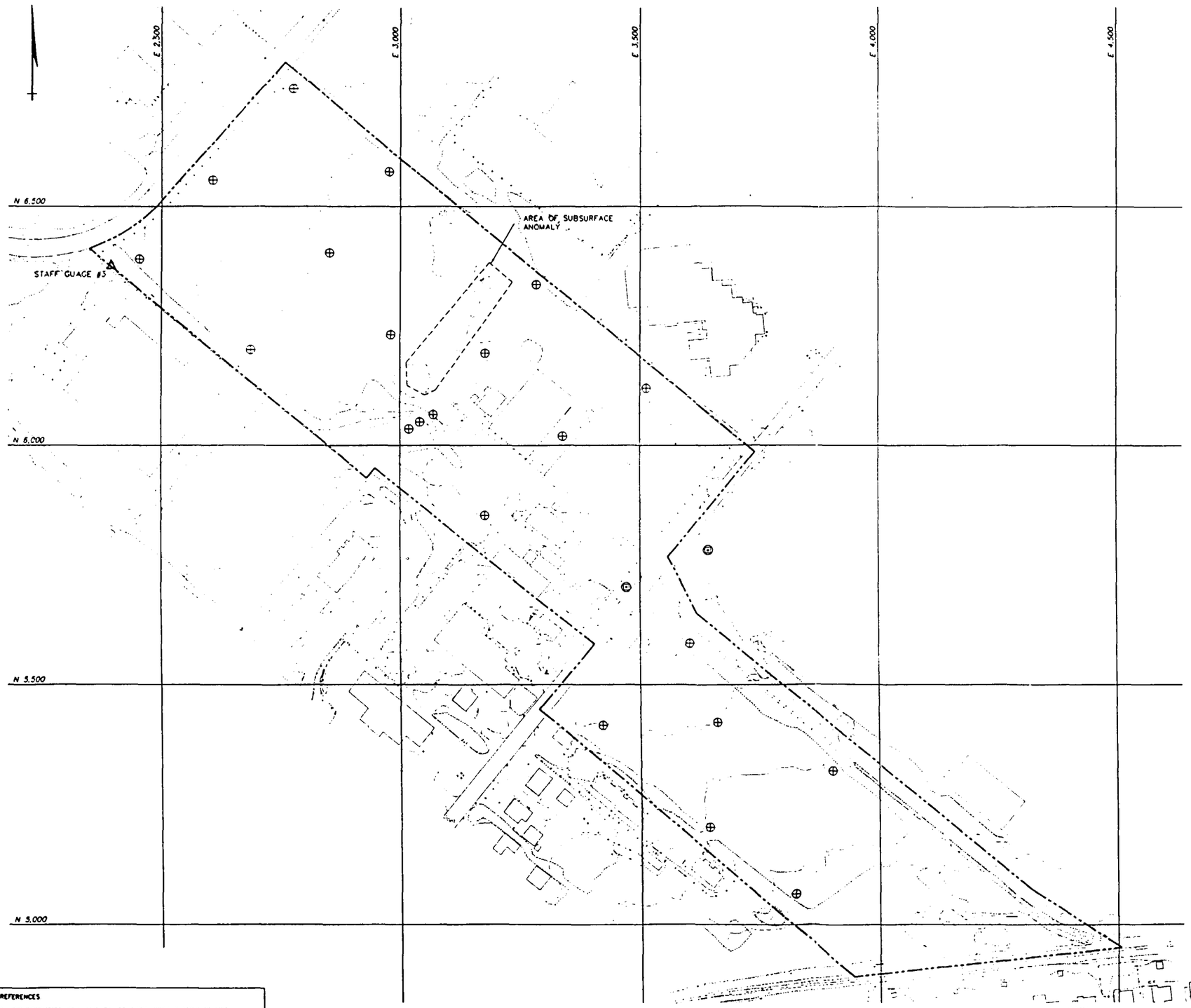
PROPOSED SOIL SAMPLING, SEDIMENT SAMPLING AND TEST PIT LOCATIONS

DRAWING NUMBER 09-0801461
FIGURE 3

DATE

8500 BROOKTREE ROAD
SUITE 300
WEXFORD, PA 15090

FILE 1461D002.DWG



LEGEND

- PROPOSED STAFF GAGE
- PROPOSED PIEZOMETER
- PROPOSED STORM SEWER SAMPLE
- FENCE
- TREELINE
- SANITARY SEWER DRAIN LINE
- STORM SEWER DRAIN LINE
- UNDERGROUND STORAGE TANK
- BARE AREA
- GROUND SURFACE ELEVATION CONTOUR
- PROPERTY LINE (APPROXIMATE LOCATION)
- AREA DIVIDE LINE

NOTES:

1. AREAS A, B, AND C AS DESCRIBED IN SECTION 1.0 OF WORK PLAN
2. ALL ELEVATIONS IN FEET ABOVE MEAN SEA LEVEL (FT-MSL).

REFERENCES

1. PPG INDUSTRIES, INC 1993. PHASE-I SITE INVESTIGATION REPORT, FIGURES 17 AND 18 SUBMITTED TO U.S. EPA REGION II, FEB. 1993
2. GROUND SURFACE TOPOGRAPHY BASED ON AERIAL SURVEY PERFORMED

SCALE

0 100 200 300 FEET

301638

REV #	DATE	DESCRIPTION	APPD

PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

McLaren Hart® ENVIRONMENTAL ENGINEERING CORPORATION

DRWN	MJH	DATE	1-20-94
CHKD		DATE	
APPD		DATE	
SCALE	AS SHOWN		

PHASE II SITE INVESTIGATION
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

DATE

8500 BROOKTREE ROAD
SUITE 300
WEXFORD, PA. 15090

FILE 14610003.DWG

PROPOSED PIEZOMETER LOCATIONS

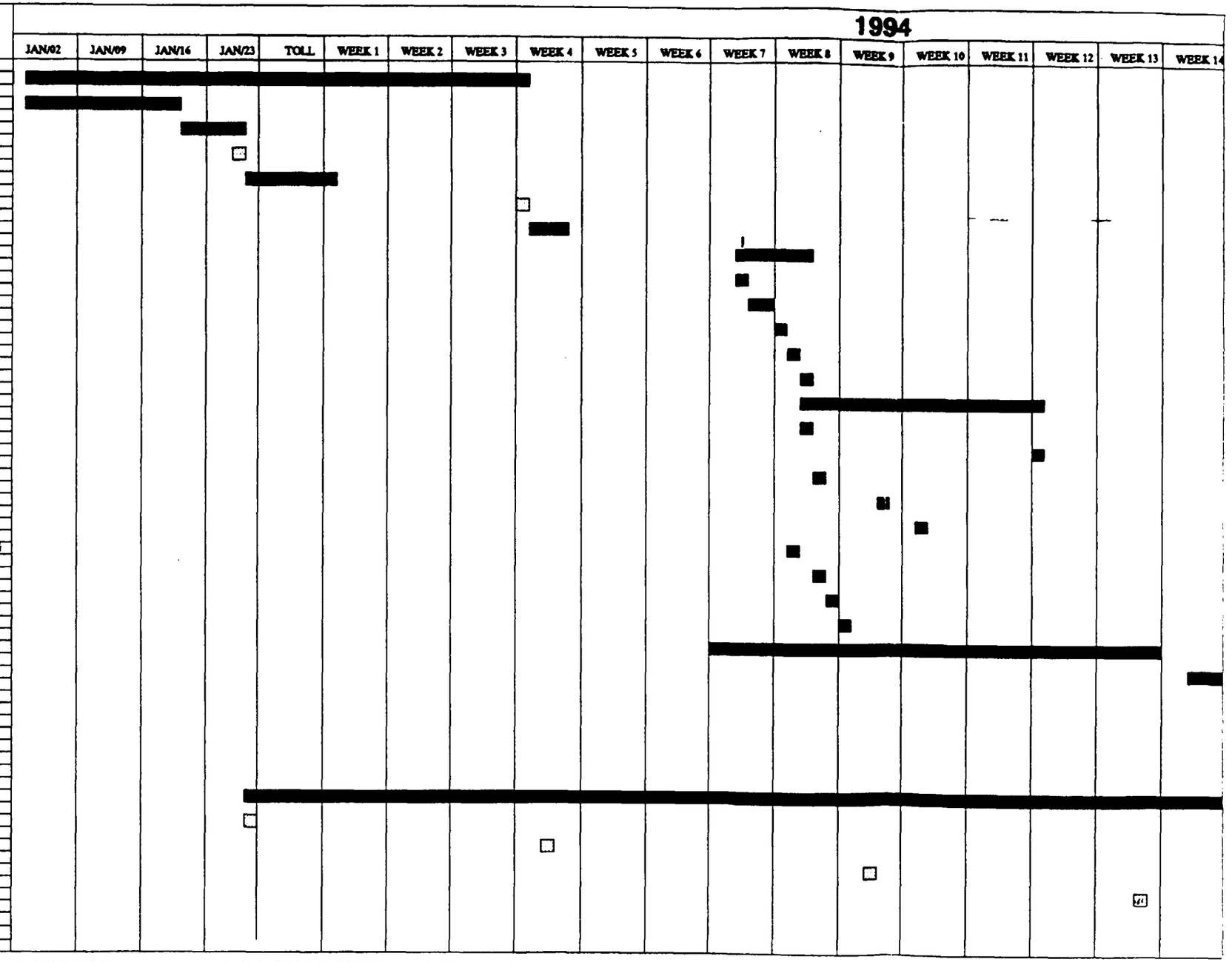
DRAWING NUMBER 09-0801461

FIGURE 4

PHA[illegible] INVESTIGATION SCHEDULE
PULVER[illegible]NG SERVICES FACILITY
MORRISTOWN, NEW JERSEY

FIGURE 5

TASK NUMBER	TASK NAME
	PHASE II WORK PLAN DEVELOPMENT
	Phase II Work Plan Writing
	Phase II Work Plan Internal Review
	Phase II Work Plan Submittal (U.S. EPA)
	Phase II Work Plan Reveiw (U.S. EPA)
	Phase II Work Plan Approval
1	LITERATURE SEARCH
2	SOIL SAMPLING AND TEST PITS
2.1	Area A
2.2	Area B
2.3	Area C
2.4	Test Pits Area A
2.5	Test Pits Area B & C
3	GROUNDWATER MONITORING
3.1	Piezometer Installation
3.2	Groundwater Sampling
3.3	Water Level Elevation Measurements – Round 1
3.4	Water Level Elevation Measurements – Round 2
3.5	Additional Monitoring Well Installation
4	SURFACE WATER AND SEDIMENT SAMPLING
5	STORM WATER SAMPLING
6	BUILDING 5 TRENCH
7	UST SAMPLING
8	DATA ANALYSIS
9	POTENTIAL MIGRATION
10	DRAFT REPORT
	Draft Report Writing
	Draft Report Review
	Draft Report Submittal
	MONTHLY REPORTS
	January Report
	February Report
	March Report
	April Report
	May Report
	June Report



MILESTONE ■ SUMMARY 1 INDICATES SCREENING TECHNOLOGY SELECTION WILL BE PERFORMED FIRST

301639

PHASE II INVESTIGATION SCHEDULE
PULVERIZING SERVICES FACILITY
MORRISTOWN, NEW JERSEY

FIGURE 5 (continued)

TASK NUMBER	TASK NAME	WEEK 16	WEEK 17	WEEK 18	WEEK 19	WEEK 20	WEEK 21	WEEK 22
	PHASE II WORK PLAN DEVELOPMENT							
	Phase II Work Plan Writing							
	Phase II Work Plan Internal Review							
	Phase II Work Plan Submittal (U.S. EPA)							
	Phase II Work Plan Reveiw (U.S. EPA)							
	Phase II Work Plan Approval							
1	LITERATURE SEARCH							
2	SOIL SAMPLING AND TEST PITS							
2.1	Area A							
2.2	Area B							
2.3	Area C							
2.4	Test Pits Area A							
2.5	Test Pits Area B & C							
3	GROUNDWATER MONITORING							
3.1	Piezometer Installation							
3.2	Groundwater Sampling							
3.3	Water Level Elevation Measurements – Round 1							
3.4	Water Level Elevation Measurements – Round 2							
3.5	Additional Monitoring Well Installation							
4	SURFACE WATER AND SEDIMENT SAMPLING							
5	STORM WATER SAMPLING							
6	BUILDING 5 TRENCH							
7	UST SAMPLING							
8	DATA ANALYSIS							
9	POTENTIAL MIGRATION							
10	DRAFT REPORT							
	Draft Report Writing							
	Draft Report Review							
	Draft Report Submittal							
	MONTHLY REPORTS							
	January Report							
	February Report							
	March Report							
	April Report							
	May Report							
	June Report							

301640

FINAL QUALITY ASSURANCE/ QUALITY CONTROL PLAN PHASE II SITE INVESTIGATION PULVERIZING SERVICES SITE MOORESTOWN, NEW JERSEY

Prepared For:

PPG INDUSTRIES, INC.

Prepared By:

McLaren/Hart Environmental Engineering Corporation
8500 Brooktree Road
Suite 300
Wexford, Pennsylvania 15090

August 01, 1994

301641

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1.0 INTRODUCTION

1.1 BACKGROUND

This Quality Assurance/Quality Control Plan (QA/QC Plan) for the Pulverizing Services Site has been prepared in response to the Administrative Order on Consent (Order) of March 31, 1989 executed between the United States Environmental Protection Agency (USEPA) and PPG Industries, Inc. (PPG). In accordance with the Order, this QA/QC Plan and the Phase I Site Operation Plan have been prepared. The purpose of this QA/QC Plan is to set forth in detail data quality objectives, sample collection procedures, data analysis processes, and procedures to ensure that he objectives of the Order are met. In accordance with the Order, this plan is designed to comply with the following:

- Section 10 of the USEPA publication, "Test Methods for Evaluating Solid Waste," second edition (SW-846).
- USEPA Region II QA Guidance Manual
- U.S. EPA RCRA Facility Investigation Guidance

1.2 FORMAT

The contents of this QA/QC Plan are based on the requirements of Section 10 of SW-846 and the format is consistent with USEPA publication QAMS-005/80. Tables and Figures are provided following the text. Additional data or procedures which supplement the text are provided in Appendices A through D.

Each page of this QAPP has document control information provided in the upper right-hand corner. This information includes:

- Section Number
- Revision Number
- Date of Revision
- Page Number in the Section

1.3 ABBREVIATIONS AND CONVENTIONS

Abbreviations for some chemical names and other terms which are frequently used in the text are as follows:

United States Environmental Protection Agency	USEPA
Polychlorinated Biphenyl	PCB
Milliliters	ml
Microliters	ul
Liters	l
Milligrams	mg
Nanograms	ng
Micrograms	ug
Grams	g
Pentachloronitrobenzene	PCNB
Target Compound List	TCL
Contract Laboratory Program	CLP
Administrative Order On Consent	AOC
Quality Assurance	QA
Quality Control	QC
Photoionization Detector	PID
Standard Penetration Test	SPT

301648

• Volatile Organic Compounds	VOCs
• Semi-Volatile Organic Compounds	SVOCs
• Department of Transportation	DOT
• Parts per Billion	ppb

2.0 PROJECT DESCRIPTION

The Phase II Site Investigation Work Plan will be implemented to supplement information collected during the Phase I Site Investigation, and to be consistent with the requirements presented in the AOC. Compilation of the Phase II data will adequately characterize site conditions and support the selection and design of the appropriate corrective action.

The technical objectives of the Phase II Site Investigation are as follows:

- Further define the extents of potential chemical constituents within Area A.
- Delineate the nature and extent of potential chemical constituents within Areas B and C.
- Identification of potential environmental receptors; and,
- Assessment of potential risk, from site conditions, to human health and the environment.

These objectives will be met through the implementation of the proposed work scope comprising the following key tasks:

TASK 1: **Literature Search** - A literature search will be preformed to supplement the current domestic and public well inventory and to gather existing information regarding the geology, hydrogeology, and physical/environmental setting of the site.

TASK 2: **Soil Sampling and Test Pits** - Soil sampling will be performed (in Areas A, B and C) to define the extent of constituents found during Phase I and prior investigations. The soil sampling methodology and locations is described in detail in the Phase II Investigation Work Plan. Initial field screening technology evaluation will be conducted to select an appropriate screening technology which will then be used for the remaining soil sampling activities. Approximately 20%

of field screened samples will be submitted for laboratory confirmatory analysis.

Test Pit excavations will be completed to better define the extent of subsurface contamination at Areas A, B and C, and to evaluate the possibility of waste materials buried at former reported disposal areas. In addition, a septic tank adjacent to the garage in Area A will be investigated.

TASK 3: **Groundwater Monitoring** - Approximately 20 shallow piezometers will be installed to determine shallow groundwater conditions (*ie.* flow direction). Two rounds of water level elevation measurements will be completed. Based on these results, additional monitoring wells will be installed. These additional monitoring wells and the existing site monitoring wells, including plant supply well, will be sampled to determine the groundwater quality. Based on groundwater elevations, the potential for off site migration will be evaluated, and select off-site wells which are potentially affected by site activities may be identified and sampled.

TASK 4: **Surface Water and Sediment Sampling** - Surface water and sediment sampling will be conducted to evaluate the quality of the drainage ditch which flows from Area A to Area C.

TASK 5: **Storm Sewer Sediment Sampling** - The site storm sewer system will be assessed for chemical quality by sampling the sediment of the two catch basins located along the piping system prior to their outlet to surface ditches.

TASK 6: **Building 5 Trench** - The physical and chemical quality of the reported trench behind former Building 5 will be assessed.

TASK 7: **UST Sampling** - The three former site UST's will be located and evaluated.

TASK 8: **Data Analysis** - Qualitative and quantitative analysis of data generated during the Phase II investigation will be performed. Such data analysis must represent site conditions to ensure reasonable selection of corrective actions potentially proposed for the site.

TASK 9: **Potential Migration** - Chemical sources and concentrations in respective media will be analyzed to characterize migration pathways potentially associated with the site. The potential exposure to surrounding ecological and human receptors will be identified, These results will be used to develop preliminary response action objectives and goals.

TASK 10: **Reporting** - This task includes submittal of monthly reports and the Phase II Site Investigation draft and final reports to the USEPA.

A detailed task description is presented in the Phase II Site Investigation Work Plan. The Work Plan specifically defines the test locations, media to be sampled and quantities, laboratory analyses, and the evaluation and assessment procedures required to fulfill Phase II Site Investigation objectives.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

McLaren/Hart Environmental Engineering corporation (McLaren/Hart) has been retained by PPG to perform the Phase II site investigation activities.

A description of the project organization is provided in this section. Table 3-1 lists the addresses and telephone numbers of the individuals identified herein and Figure 3-1 presents a flow chart of the project organization.

3.1 FACILITY COORDINATOR

Mr. Mark Terril is Facility Coordinator for PPG Industries, Inc. Mr. Terril will be the point of contact in the PPG organization for McLaren/Hart. He will monitor performance and schedules.

3.2 PROJECT DIRECTOR

Mr. Greg Peterson is the McLaren/Hart Project Director for the site. His responsibilities include:

- Ensuring that sufficient resources are available to the project team so that it can respond fully to the requirements of the site investigation.
- Providing direction and guidance to the Project Manager as needed.
- Providing senior level review of technical activities.

3.3 PROJECT MANAGER

Mr. David R. Kerschner is the McLaren/Hart Project Manager for the site. He will be responsible for all technical and scheduling matters. Other responsibilities will include:

- Interaction with Quality Assurance Officer and Health and Safety Officer to

assure that these programs are functioning effectively.

- Approval of project-specific procedures and internally prepared plans, drawings, and reports.
- Serving as the "collection point" for project staff reporting of nonconformances and changes in project documents and activities.

3.4 SITE MANAGER

Mr. Neale Misquitta is the McLaren/Hart Site and Task Manager with the following responsibilities:

- Organize and schedule field and lab subcontractor activities
- Establish project records
- Supervise subcontractor activities
- Provide Health and Safety support
- Participate in project meetings with EPA
- Review all project reports

3.5 QUALITY ASSURANCE OFFICER

Mr. Robert Anderson will be the McLaren/Hart Quality Assurance Officer for the project. His responsibilities will include:

- Development, review, and approval of the QA/QC Plan.
- Administration of the project Quality Assurance Program.
- Day-to-day supervision of quality assurance
- Notification of personnel of nonconformances and changes in quality assurance procedures.
- Determination of the audit schedule.

The Quality Assurance Director reports to the Project Director, and may act independently from the Project Manager, if required, to effect compliance with the QA/QC Plan. He will provide the necessary guidance to the project and laboratory staffs on quality-related matters and will be responsible for project audits. He has the authority and freedom to identify quality problems; to initiate, recommend, or provide corrective actions; and to verify the implementation of the corrective actions. He will initiate project audits.

3.6 HEALTH AND SAFETY COORDINATOR

Ms. Julie Panko is Health and Safety Coordinator for the project. Ms. Panko is responsible for the development and implementation of the Health and Safety Plan.

3.7 LABORATORY PERSONNEL

The key laboratory personnel for this project will be the Laboratory Project Manager and the Laboratory Quality Assurance Officer. The analytical laboratory project manager will be responsible for execution of the analytical testing program for the project. Enesco Wadsworth Alert Laboratories, Inc. (EWAL) has been selected to perform analytical testing for the project. Mr. Jay Bernading will be the Laboratory Project Manager and Ms. Kathie Teuscher is the Laboratory Quality Assurance Officer.

3.8 TECHNICAL AND SUPPORT STAFF

Individuals in this category will participate in the technical activities associated with the project. They will report to the Project Manager.

4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this Phase II Investigation is to provide procedures which, when followed properly, will assure that all decisions based on laboratory and field data generated during this investigation are technically sound, statistically valid, and properly documented. Specific procedures to be utilized for sampling, laboratory analyses, data reporting, and data validation, are presented in other sections of the QA/QC Plan. The primary purpose of this section is to define statistical acceptance criteria for chemical data generated by the analytical laboratory.

Due to the varying nature of the data required, there are several applicable levels of data quality for the Phase II Investigation. A primary component of data quality is selection of the appropriate analytical level for the intended data use. Analytical levels, as described in "Data Quality Objectives for Remedial Response Activities" (EPA, March 1987), are as follows:

- Level I - Field screening or analysis using portable instruments. Results are often not compound-specific and not quantitative, but are available in real-time.

- Level II - Field analysis using more sophisticated portable analytical instruments; in some cases, the instruments may be set up in a mobile laboratory on site. There is a wide range in the quality of data that can be generated that is dependent on the use of suitable calibration standards, reference materials and sample preparation equipment. Results are available in real-time or several hours.

- Level III - All analyses are performed in an off-site analytical laboratory. Level III analyses may or may not use Contract Laboratory Program (CLP) procedures, but not usually utilize the validation or documentation procedures required of CLP Level IV analyses. The laboratory may or may not be a CLP laboratory.

- Level IV - CLP routine analytical services. All analyses are performed in an off-site CLP analytical laboratory following CLP protocols. Level IV is characterized by rigorous QA/QC protocols and documentation.

- Level V - Analysis by nonstandard methods. All analyses are performed in an

off-site laboratory that may or may not be a CLP laboratory. Method development or method modification may be required for specific constituents or detection limits. CLP special analytical services are Level V.

Level I will be used for the screening activities required for Task 2 activities. All off-site (chemical) analytical services are Level IV.

The quality of data generated for this investigation can be characterized in terms of precision, accuracy, completeness, representativeness, and comparability. The QA objectives for accomplishing the investigation objectives for precision, accuracy, and completeness are indicated in Table 4-1. Further discussion on the specific routine procedures used to assure data precision, accuracy and completeness are presented in Section 13.0. These data characteristics are defined in the following subsections. Appendix D presents EWAL quality assurance procedures.

4.1 PRECISION

Precision is defined as a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision evaluation indicates whether the reproductibility of the analytical result is compromised due to analytical techniques, sample matrix interferences, or other factors.

4.2 ACCURACY

Accuracy is defined as the degree of agreement of a measurement (or an average of measurements of the same thing) with an accepted reference or true value, usually expressed as:

- The difference between the two values,
- The percentage of the difference relative to the reference or true value, or
- The ratio of the difference to the reference or true value.

4.3 COMPLETENESS

Completeness is defined as a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected under normal conditions. To determine completeness, the percentage of valid (*i.e.*, acceptable) data obtained, as judged by the precision and accuracy objectives, is compared to the total amount of data collection, resulting in a validation percent. Table 4-1 lists the completeness objectives for accomplishing the Phase I investigation objectives. Section 13.0 provides a discussion of procedures to assess data completeness.

4.4 REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent a measured characteristic of a population, parameter variations at the sampling point, a process condition, or an environmental condition.

The degree of representativeness is dependent upon the objectives of the measurement results. Thus, representativeness can be classified into the following four "objective levels":

- Level 1

  Only representative of the point of sampling. (Example: drum, storage tank, single point within a stream or a land area.)

- Level 2

  Sample is part of a set and represents a defined area or portion of land or water--not just the point of sampling. (Example: a stream transect, sampling grid of a land area, underground aquifer, etc.)

- Level 3

  Sample represents a relationship between the source of contamination and the location sampled. (Example: source and monitoring well, source and stream, etc.)

- Level 4

  Nonrepresentative sample. Sample is used for assessment purposes only. (Example: preliminary assessment, spot check.)

The objective of all sampling, monitoring, and analysis will be that results are representative of the medium investigated (soil, water, etc.) and its condition to a degree consistent with the desired objective level. Objective Levels 1 and 2 will be employed to accomplish the objectives of this study.

4.5 COMPARABILITY

A number of different organizations have collected data for the site in the past. Some of the data were collected by USEPA or their contractors and it is assumed that Contract Laboratory Procedures were used for these studies. The analysis of USEPA samples by CLP methods in use at the time of sampling and these methods may be slightly different than those listed herein.

Despite the qualifications mentioned above, past data from the USEPA will be used as supplementary information in the determination of the presence and levels of contaminants.

5.0 SAMPLING PROCEDURES

Sampling procedures are provide in this section. The discussion in this section includes general documentation requirements as well as specific data forms required for the various types of samples. Sample preparation and handling are addressed, as well as decontamination of sampling equipment. Sample locations are described in the Phase II Site Operations Plan and the Phase II Site Investigation Work Plan.

5.1 GENERAL DOCUMENTATION REQUIREMENTS

Each day work is performed at the site, a Field Activity Daily log will be completed by the field staff. It will be the responsibility of the site Field Supervisor to insure that this record is completed. Information to be provided on the log includes, as appropriate:

- Field activity subject
- General work activity
- Unusual events
- Changes to plans and specifications
- Visitors on site
- Subcontractor progress or problems
- Communication with PPG or others
- Weather conditions
- Personnel on site
- Field reagents used

The Field Activity Data Log will be signed by the individual who prepares it. Field Activity Daily Logs will be submitted on a weekly basis to the Project Manager. Following review, the logs will be placed in the project file. A blank Field Activity Daily Log is provided in Appendix B.

A photographic record of site operations will be prepared. Photographs of the following are normally obtained:

- General site layout,
- Geologic features,
- Streams or impoundments,
- Test pits,
- Geoprobe operation,
- Drilling,
- Well installation,
- Sampling,
- Health and safety monitoring, and
- Other items of interest.

Photographs will be identified with the project number, date, and a brief description. The photographs will be placed in the project file.

Other data forms used for documenting boring data, sample data, and field tests are described in the subsections which follow. Chain-of-custody documentation is described in Section 6.0.

5.2 SAMPLING

Sampling of borings, monitoring wells, and stream sediments is discussed in the following subsections. In general, polyethylene sheet will be used at sampling sites to prevent the spread of contamination.

5.2.1 Geoprobe Samples

McLaren/Hart will utilize a Geoprobe® for the collection of soil samples. The Geoprobe® is a van mounted, hydraulically operated sampling unit designed to collect soil, soil gas and

groundwater samples at discrete depths. The use of the Geoprobe® during this phase of the soil investigation will provide an initial determination of the extent of affected soil.

Soil samples will be obtained by driving a two foot long, acetate-lined, stainless steel sampling tube equipped with an internal, moveable piston to a position just above the desired sampling depth. After the sampling tube is properly positioned, the internal piston will be released and the tube drive an additional 24 inches, allowing the soil to enter the tube. The sampling tube will then be retrieved to the surface and the soil sample will be removed from the tube within the acetate liner. Soil samples will be handled using clean decontaminated stainless steel spoons and bowls.

Approximately 199 soil samples obtained will be immediately field screened utilizing a HNu® photoionization detector (PID) and then transferred into sterile laboratory supplied bottles, labelled and preserved with ice to 4°C for delivery to a qualified analytical laboratory or for subsequent field screening. It is anticipated that 40 samples will be sent to EWAL for analysis of Table 8-1 parameters. Prior to placement into containers, sample aliquots will be homogenized (except for VOCs) using coming and quartering techniques. Ten composite samples will be analyzed for dioxins.

In the case of soil samples to be analyzed for 2,3,7,8-TCDD, individual soil samples will be composited so that the 40' sampling points will be combined to form 10 composite soil samples. Compositing will be accomplished by mixing equal volumes of the individual grab samples in stainless steel pans with stainless steel spoons. Once homogenization is complete, the composite sample is transferred to the sample jar to be shipped to the laboratory for analysis. Appropriate QA/QC samples in accordance to Region II SOPs will also be submitted.

All sampling equipment will be decontaminated prior to sampling at each location to minimize the potential for cross contamination. Decontamination procedures will include a soapy water scrub with a non-phosphate detergent, a distilled water rinse, a methanol spray rinse to remove

any remaining organic residues, and a final distilled water rinse and air dry.

The decontamination procedure for dioxin samples is the same as that described above, however samplers will use trichloroethylene in the solvent rinse step.

5.2.2 Test Pit Samples

Test Pits will be excavated in Areas A, B, and C using a backhoe. Excavated materials will be placed on polyethylene sheeting adjacent to the trench area or in a dump truck upon completion of trenching activities, the exploratory trenches will be lined with polyethylene sheeting, and the material will be placed back into the excavation. The trench location will be covered with polyethylene sheeting.

Soil samples will be collected below the limits of waste disposal, and at a depth of approximately five feet below grade from the area between the disposal trenches and near the outside extent of the exploratory trenches. It is anticipated that a total of 10 to 25 samples will be collected for field screening. All soil samples will be analyzed in the field using the field screening test kits; a minimum of five samples will be sent to the laboratory for analysis for the specified parameters (Table 8-1). Photographic documentation will also be collected for each excavation.

5.2.3 Samples From Borings

The investigation will include a number of borings from which samples for analytical testing will be obtained. Samples of soil and unconsolidated materials will be obtained from Standard Penetration Tests (SPTs). The procedures which will be used for drilling and sampling are provided in Appendix A.

Soil samples will be obtained from the stainless-steel split-barrel sampler used for SPTs. The samples will be examined and classified, and the data, including visual observations, will be

entered onto the boring log. Samples will then be placed in a clear glass jar provided by the driller. Pertinent sample data such as project number, boring number, sample number, depth, and SPT blow counts will be marked on the jar labels and lids. The jars will be placed in boxes, and the boxes will be stored in a secure area which is protected from the weather and from temperature extremes. Sampling equipment will include clean decontaminated stainless-steel spoons and bowls.

Split-barrel samples receiving analytical testing will be placed in appropriate containers supplied by the laboratory, and the containers will be labeled and handled as described in Section 5.3. Soil samples (except those to be analyzed for volatile organics) will be thoroughly homogenized.

5.2.4 Sediment Samples

Sediment samples (seven) will be grab samples and will be collected manually in a downstream to upstream sequence if sampling occurs at more than one location. All sample locations will be approached from downstream. Sediment samples will be obtained with a stainless steel trowel. The portion of the sample for volatile organics analysis shall be placed directly in two 40 ml VOA vials. The remaining sample will be placed in a stainless steel pan and thoroughly mixed with the trowel. The sediment in the pan will be scraped from the sides, corners, and bottom of the pan, rolled to the middle of the pan and initially mixed. The sample will then be quartered and each quarter moved to a corner of the pan. The quarters will be mixed individually, then recombined in the center of the pan and mixed again. The sediment will then be placed in the appropriate sample containers. The lids will be secured tightly after any sediment is removed form the threads of the containers. Sampling locations will be marked with a numbered stake which will be surveyed to record the sample location. The labeling and handling of sample containers is discussed in Section 5.3.

Two additional sediment samples will be obtained from the storm water catch basins, if sedim is determined to be present in the catch basins. Analytical parameters are as specified in Table 8-1.

5.2.5 Groundwater Sampling

A minimum of four monitoring wells will be installed at the site. The procedures for well installation and development are provided in Appendix A.

The primary consideration for sampling monitoring wells will be to obtain a representative sample of the groundwater in the vicinity of the well. To safeguard against collecting nonrepresentative stagnant water from a well, the following guidelines and techniques will be observed for purging monitoring wells prior to sample withdrawal:

- Water levels will be measured with an accuracy of ± 0.01 foot. The reference point for measurements will be the top of the monitoring well riser pipe and this will be indicated on the Water Sample Field collection Report (Appendix B).
- The submerged casing volume (standing water column) in the well will be determined from the following formula:

  $V = 3.14 \backslash f^2 \backslash h \backslash 7.48$

  where,

  V = volume (gallons)

  f = riser pipe radius (feet)

  h = standing water height as determined from water level measurements and monitoring well construction logs (feet).

  This quantity constitutes one well volume of water. A minimum of three well volumes will be purged prior to sampling.

- Purging shall be accomplished by using an air lift pump or by hand bailing water from the monitoring well. Purge water for each well will be collected in drums or a tank. The stored purge water will be analyzed to assess proper disposal methods.

- A well stabilization test will be performed during the purging of the well. Temperature, pH, and specific conductance will be measured after fractional increments of the purge volume have been removed (*e.g.*, after each third of the purge volume). If the last three sets of readings are approximately constant, the purging will be considered complete. If the readings have not stabilized (within < 10% of successive recordings), additional water will be purged until the desired results are obtained.

- Wells will be sampled within three hours of purging. If a well purges dry (*i.e.*, all standing water is removed) prior to removal of three well volumes, a well-stabilization test will not be required. Such wells will be recorded at time of sampling. If the well recharges too slowly to fill all the required containers within three hours, the containers will be filled in the following order:

  - VOAs,
  - Semivolatile Organics,
  - PCBs/Pesticides,
  - Total Recoverable Petroleum Hydrocarbons,
  - Metals, and
  - Cyanide.
  - 2,3,7,8-TCDD

  The water level, pH, temperature, and specific conductance will be recorded when samples are taken.

Wells will be sampled with a bottom-loading stainless-steel hand bailer, which has been decontaminated prior to usage (see Section 5.4). The bailers will be lowered into the well by hand and allowed to fill completely. The bailer will be removed and the sample containers will be filled by discharging water through the check valve at the bottom of the bailer.

Bailer lines shall be of polypropylene monofilament, stainless steel wire, or teflon-coated wire, or alternatively a nylon line with a ten-foot leader of one of these materials.

If a pump is used to evacuate a well, the tubing which comes into contact with the water will be polyethylene or teflon and will be dedicated to a single well. The intake will be kept just below the water surface as water is withdrawn. No equipment with neoprene, Tygon, silicon rubber, or viton parts shall be lowered into monitoring wells.

The labeling and handling of sample containers is described in Section 5.3.

5.2.6 Surface Water Sampling

Surface water samples will be collected (at locations proximal to the sediment sampling locations) using a shallow (8-inch long, 2-inch diameter) stainless-steel bailer. These samples will be placed directly into the respective sample bottles. Analysis of these samples will according to the parameters listed in Table 8-1. Equipment decontamination procedures will be consistent with those stipulated in Section 5.4.

5.2.7 Underground Storage Tank Sampling

UST samples will be collected based on the field conditions encountered. It is anticipated soil samples will be collected adjacent and slightly downgradient to each UST. If the USTs are accessible, then a sample of the contents will be obtained. UST sampling procedures will be consistent with those specified in the Compendium of ERT Waste Sampling Procedures,

EPA/540/p-91/008, January 1991. If determined necessary, additional soil and/or other sampling will be conducted with the concurrence with EPA on-site representative. UST-related samples will be analyzed for TRPH.

5.2.8 Sample Collection

The following order of sample collection will be followed for all samples matrices:

- TCL VOCs;
- TCL SVOCs;
- TCL Pesticides, Malathion, Sevin, Rotenone, PCNB;
- Dioxins/Furans;
- Metals; and,
- General Chemistry and TOX.

5.3 SAMPLE PREPARATION AND HANDLING

5.3.1 Sample Containers and Cleaning Requirements

Sample containers will be supplied by the analytical laboratory. This will insure that sufficient sample volume is obtained for laboratory analysis and associated laboratory QA/QC procedures. The containers which will be used for various sample types and analyses are indicated in Table 5-1.

Sample containers will be laboratory-supplies. Lot numbers will be recorded. All sample containers will be prepared in accordance with the requirements of OWSER Directive #9240.0-05A, "Specifications and Guidance for Contamination - Free Sample Containers." December, 1992.

5.3.2 Sample Preservation and Holding Times

Since certain constituents in water can change chemically with time, it will be necessary to preserve individual samples to maintain the integrity of time-dependant constituents. The preservation requirements for various types of samples are indicated in Table 5-1.

Laboratory personnel will send an aliquot of the preservative in a separate container. Field personnel will follow the preservation procedures listed below.

When collecting TCL volatile samples, the amount of 1:1 HCL required to achieve a pH <2 will be determined on a separate sample aliquot of equal volume to the actual sample.
The predetermined amount of acid will then be added to two fresh VOA vials almost completely filled with sample. The vials will then be filled completely, capped, and checked for air bubbles. Samples found to have air bubbles will be discarded and resampled as described above.

Note: If effervescence occurs upon addition of acid, the samples will be collected without preservation. This will be documented and the sample holding time will be 7 days.

When collecting non - volatile samples, preservatives will be added drop wise until the proper pH is achieved. The pH will be checked by pouring an aliquot of sample over pH paper.

Preservation includes maintaining the samples in a chilled condition (4°C) once they have been obtained. Preservation is generally intended to:

- Retard biological action,
- Retard hydrolysis of chemical compounds and complexes,
- Reduce volatility of constituents, and
- Reduce absorption effects.

The maximum recommended holding time for properly preserved samples are provided in Table

5-1. Most samples will require storage at 4°C prior to analysis to prevent deterioration. The laboratory analysis will be performed within specified holding times to ensure the validity of the analytical results.

5.3.3 Sample Filtration

Monitoring well water samples designed for metals analysis will be obtained as two separate samples: one unfiltered and one filtered. Approximately one liter of the filtered sample will be filtered immediately after collection and prior to preservation.

When filtering aqueous samples, a device made of polyethylene, polypropylene, or borosilicate glass will be used. The apparatus will be pre-cleaned by rinsing with 10% HNO3 solution, followed by a demonstrated analyte free de-ionized water rinse. This cleaning procedure will be used initially and between each sampled collected.

A field rinse blank will be collected daily for each apparatus filtration used.

A filter designed to pass only liquid and solid particles less than 45 microns will be used. Turbid samples may be prefiltered prior to filtering through the 0.45-micron cellulose-based membrane filter.

Samples will be preserved immediately with ultra pure HNO3, and the pH checked to ensure that the proper pH has been attained. All accompanying paper work will clearly state that samples have been field filtered. When filtration systems are used, care will be taken not to contaminate the filter or the container receiving filtrate prior to and during filtration.

5.3.4 Sample Labeling and Handling

All sample containers will have blank labels, as supplied by the laboratory. The labels will be

filled out at the time of sample collection by the field personnel performing the sampling. Information marked on the label will include:

- Sample Identification Number
- Collector's Name
- Date of Collection
- Type of Sample
- Preservatives Used
- Analysis to be Performed
- Number of Bottles in Sample Set

All samples will be transported to the laboratory in durable, secured metal or plastic coolers or laboratory-supplied, secured waterproof metal or plastic containers. Containers will be shipped via common overnight carrier (*e.g.*, Airborne or Federal Express) or hand carried within 24 hours of collection. Samples will be shipped in accordance with State and Federal DOT Regulations. Chain-of-custody documentation (Section 6.0) will always accompany the samples.

5.4 DECONTAMINATION

Decontamination of equipment used for sampling will be carefully performed to minimize any possibility of cross contamination through the use of tools and equipment. All equipment will be decontaminated prior to initial use.

5.4.1 General

A personnel decontamination station will be located immediately adjacent to the pavement near the northeast corner of Building 29. The station boundaries will be marked with flagging. The personnel decontamination station will have the following components:

- Plastic drop cloths for depositing heavily-contaminated equipment or outer protective clothing.

- Containers for storing contaminated equipment or protective clothing that is to be discarded.
- Tubs for wash and rinse solutions.
- Long handled brushes for washing and rinsing.
- Containers for storage of decontaminated clothing and equipment.
- Disposable wiping cloths and towels.

Personnel passing through the decontamination zone from the exclusion zone will go to a changing trailer located nearby where personal items and clean clothing will be stored. Decontamination and disposal clothing and equipment liquids from the decontamination station will be containerized for storage on site.

Small equipment will be decontaminated in the area where it is used. Fluids and waste materials will be collected for storage on site. Additional details are provided in Section 5.4.2.

Drilling rigs and other large equipment (backhoe for example), if appropriate, will be decontaminated on a pad constructed by the drilling contractor. The pad shall be graded and covered with plastic such that decontamination fluids can be collected in a sump which is integral with the pad. Soil and decontamination liquids will be collected for storage on site. Additional details of the decontamination process are provided in Section 5.4.3.

5.4.2 Small Tools and Equipment

This category includes small tools and other apparatus used for sampling, such as split-spoon samplers, bailers, and trowels. The following procedures will be employed for decontamination of this equipment:

- Wash with low-phosphate detergent solution (Alconox or Liquinox);
- Rinse with tap water;

- Rinse with one percent nitric acid, ultra pure;
- Rinse with tap water;
- Acetone rinse;
- Thorough rinse with deionized water;
- Air dry; and
- Wrap in aluminum foil.

The deionized water shall meet the following criteria:

- Purgeable organics < 10 ppb,
- Semivolatile organics < CRQL,
- Pesticides < CRQL,
- PCBs < CRQL, and
- Inorganics < CRQL.

The deionized water shall have been tested and documented no longer than three months prior to use. The documentation will be transmitted to the USEPA prior to usage of the deionized water in the field.

5.4.3 Large Equipment

Large equipment such as a drilling rig and backhoe will be decontaminated on site between sampling locations and prior to leaving the site. Soil or waste present will be scraped or brushed off. The drill rod and other parts of the rig which could be involved in cross contamination will be steam cleaned. If the drilling rig leaves the site, the boom will also be steam cleaned. All equipment decontamination shall be subject to the approval of the field engineer/geologist observing the work.

5.4.4 Other Equipment

Other equipment used in the development of monitoring wells (such as submersible pumps) shall be decontaminated as described in Section 5.4.2 or discarded and replaced (such as dedicated tubing and bailer cord). The field engineer shall determine if equipment is to be decontaminated or replaced.

6.0 SAMPLE CUSTODY

6.1 FIELD PROCEDURES

A chain-of-custody record will be established and maintained to document sample possession from the time of collection to delivery to the laboratory. Once samples are received by the laboratory, they will be handled under the laboratory internal chain-of-custody procedures. Personnel obtaining samples in the field will initiate a chain-of custody record by reporting the following minimum data as the samples are collected:

- Name of Sampler
- Sample Identification
- Data and Time Collected
- Amount Collected
- Containers Used

Other information such as preservatives used and analyses required shall be indicated as appropriate. Sample chain of custody forms and traffic reports will be placed in waterproof plastic bags and taped to the underside of the cooler lids. In addition to the chain-of-custody record, a sample-collection form will be completed at the time of sampling to record sample location and other parameters associated with sample acquisition. A typical chain-of-custody record and sample collection form are provided in Appendix B.

Subsequently, at each change of possession, the chain-of-custody record will be signed by the person relinquishing the samples and the person receiving the samples. This could occur as the samples are transferred from the contaminated portion of the site to a designated clean area, as the samples are transported to the lab, or when the samples are received at the analytical laboratory.

Once the sample containers have been decontaminated and preservatives have been added,

custody seals will be placed around each individual sample container and cap, so that any disturbance to the sample prior to analysis can be detected. Samples will then be carefully packed in coolers made of waterproof metal or plastic. Cushioning material such as vermiculite will be added to the cooler to prevent breakage. Sufficient amounts of ice packs will be added to maintain a sample temperature of 4 degrees C. The shipping container (e.g., cooler, styrofoam box) will be sealed with custody seals. At a minimum, a custody seal will be placed on diagonal corners of the container lid to seal the lid to the main portion of the shipping container. Depending upon the shipping container, additional custody seals will be used as necessary to prevent unnoticed tampering with the samples. Coolers will be secured with nylon or fiber strapping tape in addition to custody seals and will be shipped within 24 hours of collection via an overnight carrier service.

If for any reason samples on a chain-of-custody record are split and sent to separate destinations, multiple copies of the records will be generated with a clear indication of which sample went to which destination.

When the samples are received at the analytical laboratory, the person receiving the samples will note the condition of the custody seals found on both the coolers and sample containers. The person assuming custody of the samples will do so by signing the chain of custody record after accounting for all samples indicated on the record. At this point, the chain-of-custody records will be placed in the laboratory project file and copies will be transmitted to the Project Manager.

6.2 LABORATORY PROCEDURES

The laboratory will designate a sample custodian for the project. This individual will be authorized to sign chain-of-custody records upon sample receipt and will be responsible for verifying that the custody seals are intact.

The project samples will be stored at the laboratory for a period of time related to the type and

nature of the samples. Maximum laboratory holding times for various parameters are provided.in Table 5-1. When the storage times have expired, the laboratory will dispose of the samples in accordance with applicable regulations.

7.0 CALIBRATION PROCEDURES

Calibration procedures which will be followed for sample analysis are discussed in this section. Calibration information is recorded in a laboratory log book.

7.1 INORGANIC PARAMETERS

Initial calibration for inorganic parameters will be performed in accordance with procedures and frequency described in the reference methods for the analyses to be performed. In general, an analytical curve will be established for each parameter. The analytical curve for inductively Coupled Plasma (ICP) analysis will be established on the basis of a calibration blank and at least one laboratory standard. A calibration blank and at least three laboratory standards will be used for established analytical curves for cyanide and atomic absorption spectroscopy (AAS) analyses. A calibration blank will be analyzed each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10 percent during the run. The results for the calibration blank solution shall be recorded. The results for the blank analysis will be reported down a the instrument detection limit. If this blank result is greater than the practical quantitation limit (PQL), the analysis will be terminated, the problem will be corrected, and recalibration will occur.

7.2 ORGANIC PARAMETERS

7.2.1 GC/MS Parameters

GC/MS procedures will be used for the analysis of volatile and semivolatile compounds and 2,3,7,8-TCDD and PCNB. Calibration of the GC/MS will comprise the following:

- Instrument tuning;
- Initial calibration;

- System performance; and
- Continuing calibration.

7.2.1.1 CC/MS Tuning: The GC/MS will be tuned a minimum of once every 12 hours to determine if the instrument is operating property. The tuning specifications for extractables and volatiles are provided in the reference methods for the parameters to be analyzed. The tuning criteria must be met before standards and samples are analyzed.

7.2.1.2 Initial Calibration: The initial CC/MS calibration will be performed with a blank and five levels of laboratory standards, as described in the reference methods for the parameters to be analyzed. Frequency of calibration will be as described in the reference method. From each calibration standard, the area corresponding to the primary characteristic ion will be tabulated against concentration for each compound. The relative response factor (RRF) will be calculated for each compound at each concentration using equations provided in the reference methods. Based on the RRF from the initial calibration, the percent relative standard deviation (%RSD) for the calibration check compounds (CCC) will then be calculated. Calibration check comps are described in Section 7.2.1.4.

7.2.1.3 System Performance: After initial calibration, a system performance check will be carried out to ensure that minimum average relative response factors are met before the calibration curve is used. A system performance check will be made each twelve hours. The system performance check will enable the identification of problems with the analytical system, including fast purge rate, degradation caused by contaminated lines or active sites in the system, or standard mixture degradation. The volatile system performance calibration compounds (SPCC) and the minimum acceptable average response factors are chloromethane-0.3, 1,1 - dichloroethane-0.3, 1,1,2,2-tetrachloroethane-0.3, chlorobenzene-0.3, and bromoform-0.25. The semivolatile SPCC for CLP methods and SW-846 Method 8270 are N-nitroso-di-n2-propylamine, hexachlorocyclopentadiene, 2,4 dinitrophenol, and 4-nitrophenol; each compound must have an average response factor greater than 0.05. If the average response factor for any

SPCC is below the minimum value specified in the reference method for the parameter being analyzed, the system will be considered to be not operating properly, because the SPCC are usually the first compounds to show poor performance. No samples will be analyzed until the system is once again operating properly and the SPCC average response factors are acceptable.

7.2.1.4 Continuing Calibration: If the SPCC criteria are met, calibration check compounds will be used to check the validity of the initial calibration. A CCC standard will be analyzed every 12 hours after the SPCC criteria are met. If the percent difference for each CCC is less than or equal to 25 percent, the initial calibration is valid. If the criteria are not met (>25 percent difference) for any one CCC, corrective action will be taken. If no source of the problem can be determined after corrective action has been taken, the initial five-point calibration will be repeated. The volatile CCC are vinyl chloride, ethylbenzene, toluene, 1,2, dichloroproprane, chloroform, and 1,1-dichloroethene. Semivolatiles can be divided into two fractions: base/neutral and acid. The CCC for the base/neutral fraction are acenaphthene, 1,4 - dichlorobenzene, hexachlorobutadiene, n-nitroso-di-n-phenylamine, di-n-octylphthalate, fluoranthene, and benzo(a)pyrene. The CCC for the acid fraction are 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, and 2,4,6-trichlorophenol.

7.2.2 GC Parameters

Gas chromatographic systems will be used for PCBs/pesticides and will be calibrated using the external standard technique. This technique is described in detail in the reference methods for the analyses to be performed. A calibration curve for each compound will be prepared based on a minimum of three concentration levels. The sequence of standard analysis and evaluation of standard will be as described in the reference methods. Pesticides tentatively identified in the primary analysis will be confirmed in accordance with CLP and SW-846 procedures.

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7.3 FIELD PARAMETERS

Specific conductance, pH, and temperature will be determined in the field when water samples are collected. Calibration procedures and frequency are provided in the reference methods for specific conductance and temperature. The pH calibration procedures will follow the instrument manufacturer's instructions. The reference methods are indicated in Section 8.4. OVA and HNu will be calibrated according to manufacturer's instructions.

7.4 CALIBRATION STANDARDS

Calibration standards will be obtained from chemical vendors who document the treatability of their material. If dilutions of the vendor-supplied standards are made, the dilution volumes and procedures will be documented. The dilution calculations will be verified by an individual not involved in the dilution.

7.5 DOCUMENTATION

7.5.1 Field Documentation

Personnel performing calibration of field instruments will record the following information:

- Date,
- Instrument,
- Name,
- Calibration standard,
- Calibration results, and
- Corrective action taken.

For water samples, calibration data for pH, specific conductance, and temperature will be recorded on the Water Sample Field Collection Report (Appendix B) for each water sample collected. OVA/HNu calibration will be documented on the Field Activity Daily Log (Appendix B).

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7.5.2 Laboratory Documentation

Laboratory personnel will record the appropriate calibration information in bound notebooks. A notebook will be kept with each instrument. Information recorded will include the applicable information listed in Section 7.5.1.

8.0 ANALYTICAL PROCEDURES

Enseco-Wadsworth/Alert Laboratories located in North Canton, Ohio and Pittsburgh, Pennsylvania will perform the analytical procedures for this investigation.

Most of the analytical procedures for this project will be "USEPA Contract Laboratory Procedures" (CLP) procedures. For parameters for which CLP methods are not available, other EPA-approved methods will be used. The USEPA Contract Laboratory Program identifies two lists of compounds which can be analyzed by their methodology. The Target Compound List (TCL) includes organic analytes and the Target Analyte List (TAL) includes inorganic analytes of interest. TCL and TAL compounds are indicated in Appendix C. Several samples will be analyzed for the compounds on the TCL and TAL. Most samples will be analyzed for TCL pesticides and three additional pesticides (sevin, pentachloronitrobenzene (PCNB), and malathion) in accordance with the Order.

Other parameters for which analyses will be performed include field parameters associated with water samples (specific conductivity, pH, and temperature). Analyses for these field parameters will be in accordance with USEPA-approved methods. Table 8-1 lists the Phase II analytical program.

Table 8-2 lists the preparation and analytical procedures to be followed for all laboratory parameters, including those on the TCL and TAL. Changes in methodology due to higher or lower than anticipated concentrations being detected may change the preparation or analytical method used.

8.1 TAL AND TCL PARAMETERS

The TCL and TAL compounds for this project can be divided into five groups: volatiles, semivolatiles, PCBs/pesticides, metals, and cyanide. The analytical methods for TCL Volatiles,

TCL Semivolatiles, TCL Pesticide/PCBs will be as described in the USEPA CLP SOW for Organic Analysis OLM01.9. The analytical methods for TAL Metals and Cyanide will be described in the USEPA CLP SOW for Inorganic Analysis ILM03.0.

8.2 NON-TCL PARAMETERS

Non-TCL parameters for which analyses will be performed are the pesticides sevin, PCNB, and malathion. Table 8-2 lists the analytical methods for these pesticides. Non-TCL parameters will be analyzed by the following methods:

A.	Sevin and Rotenone	- SW846 8318 Carbamate Pesticides
B.	PCNB	- Added to TCL BNA CLP SOW OLM01.9
C.	Malathion	- SW846 8141 Organophosphorus Pesticides
D.	TRPH	- (soil) SW846 3550/USEPA MCAWW 418.1
	TRPH	- (water) USEPA MCAWW 418.1

8.3 GEOTECHNICAL PARAMETERS

Geotechnical analysis will be performed on select test pit and soil boring samples. These analysis will be performed in accordance with applicable ASTM methods include:

- Grain Size Analysis;
- Atterberg Limits;
- Plasticity
- Permeability.

8.4 TCDD

2,3,7,8-TCDD will be analyzed via the procedures outlined in the USEPA CLP SOW DFIMO1.1 (9/91)

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9.0 DATA REDUCTION, VALIDATION, AND REPORTING

9.1 DATA REDUCTION

The analytical laboratory will utilize computer software to calculate sample concentrations for many parameters. A response factor or standard curve will be generated from the calibration standards and applied to either the absorbance, peak height, or area measured by the laboratory instrument to calculate the sample concentration. This value will be corrected for any dilutions or preparation. Calculations that are not performed by computer programs will be performed manually by the analyst. The analyst will employ the same methodology used in the computer software. Corrections for dilutions and other preparations will be done manually. The data reduction methods are summarized in the following subsections.

9.1.1 GC/MS Analyses

Sample concentrations for volatile and semivolatile parameters analyzed by GC/MS will be calculated as follows:

$$\text{Reported Results} = \frac{(Q)\,(D)}{C}$$

where: Q = Amount reported on software-generated quantitation report (ppm or ppb)

D = Dilution factor (unitless)

C = Concentration factor (unitless)

C for volatiles = I

C for semivolatiles:

$$C\ (\text{Soils}) = \frac{\text{gm sample extracted}}{\text{ml } CH_2Cl_2} \times \frac{1}{[\%\ \text{Solids}/100]}$$

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$$C\ (\text{Water}) = \frac{\text{Volume of Sample Extracted (ml)}}{\text{Final Volume of Extract (ml)}}$$

Soil results will be reported on a dry-weight basis.

9.1.2 GC Analyses

Concentrations of PCBs and pesticides analyzed by GC will be calculated by computer software based on the following equations:

$$\text{Water (ug/1)} = \frac{(A)(E)(C)(D)}{(B)(F)(G)}$$

$$\text{Soil (ug/Kg)} = \frac{(A)(E)(C)(D)}{(B)(F)(H)(S)}$$

where:

A = Peak Height for Parameter of Interest

B = Peak Height of External Standard

C = Volume of Extract (ul)

D = Dilution Factor

E = Amount of Standard Injected (ng)

F = Volume of Extract Injected (ul)

G = Volume of Water Extracted (ml)

H = Weight of Soil Extracted (gm)

$S = \frac{\%\ \text{Solid}}{100}$

9.1.3 Metals

Concentrations will be calculated directly by the instrument (AAS or ICP). This concentration will then be corrected for dilutions and preparation by the analyst.

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Water

$$\text{Concentration (mg/1)} = (A)(D)\frac{E}{F}$$

Soil

$$\text{Concentration (mg/kg)} = (A)(D)\frac{E}{(G)(S)}$$

Where:

A = Instrument Reading (mg/1)

D = Dilution Factor (unitless)

E = Final Sample Volume (ml)

F = Original Sample Volume (ml)

G = Original Sample Amount (gm)

S = Percent Solids/100

9.1.4 Cyanide

A standard curve will be prepared by plotting absorbance of the laboratory standard versus cyanide concentration, as described in the reference method for cyanide. The concentration of cyanide in the sample will be calculated as follows:

Water

$$\text{CN (ug/l)} = \frac{A \times 1{,}000}{B} \times \frac{50\text{ ml}}{C}$$

where: A = ug Cn from Standard Curve

B = g or ml of Original Sample

C = ml Taken for Colorimetric Analysis

Soil

$$CN\ (ug/1) = \frac{(A)\ \left(\frac{50\ ml}{B}\right)}{(C)\ \left(\frac{\%\ Solids}{100}\right)}$$

where: A = ug CN from Standard Curve
B = ml used for Colorimetric Analyses
C = Wet Weight of Original Sample (g)

9.1.5 2,3,7,8-TCDD

The concentration of 2,3,7,8-TCDD is calculated using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times RRF_n \times W}$$

where:
C_x = 2,7,3,8-TCDD concentration in ug/kg or ug/l
A_x = The sum of integrated ion abundance detected for m/z 320 and 322
A_{is} = The sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of $^{13}C_{12}$- 2,3,7,8-TCDD the internal standard
Q_{is} = Quantity (in ng) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction
RRF_n = Calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD
W = The weight (in g) of soil/sediment extracted or volume of water extracted (in ml)

For samples in which unlabeled 2,3,7,8-TCDD was not detected the estimated maximum possible concentration (MPC) will be calculated. The MPC is the concentration required to produce a signal with a peak height of 2.5 times the background signal height and is calculated as follows:

$$MPC = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times RRF_n \times W}$$

where: MPC = Maximum possible concentration of unlabeled 2,3,7,8-TCDD required to produce H_x

H_x = Peak height for m/z 310 or 322 in the same group of >5 scans used to measure Ais .

H_{is} = Peak height for the appropriate ion characteristic of the internal standard, m/z 332 when 320 is used to determine A_x, and m/z 334 when 322 is used to determine A_x

Q_{is} = Quantity (in ng) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction

RRF_n = Calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD

W = Weight (in g) of wet soil/sediment sample or volume of water extracted (in ml)

9.2 DATA VALIDATION AND ASSESSMENT

The primary objective of this project is to provide data of sufficient quality for decision-making purposes concerning response action alternatives. It is anticipated that by using CLP and USEPA procedures the laboratory will provide reliable and acceptable data to adequately characterize the site for response actions.

The analytical laboratory will perform a review of the data prior to submittal to McLaren/Hart. After the laboratory review, QC data and sample results will be reviewed for validation purposes

by McLaren/Hart. Guidelines described in USEPA Standard Operating Procedures, in the reference methods, CLP guidelines, and this plan will be used in the results validation by the laboratory and McLaren/Hart Associates.

USEPA Standard Operating Procedures which will be utilized in the data validation include the following:

- SOP No. HW-2, Evaluation of Metals Data for the Contract Laboratory Program, Revision 11, (1/92).
- PCDD/PCDF Data Review, SOP No. 11 (Rev. 2, 6/93).
- SOP No. HW-6, CLP Organics Data Review Revision 8, (1/92).

McLaren/Hart will receive a data summary package from the laboratory. A data summary package will contain the following information when applicable:

- Analytical results,
- MS/MSD results,
- Surrogate recoveries,
- Method and trip blanks results,
- ICP interference results,
- Duplicate sample analysis,
- Spike sample analysis, and
- Holding times.

The information in the data summary package will be used by McLaren/Hart to validate the results. Validation will be performed using criteria and data flags defined in the corresponding Region II SOP to reach particular analysis. McLaren/Hart will provide validated analytical results in the Phase I report and will maintain this information in accordance with the Order. PPG and its subcontractors will make available to the USEPA results of all sampling and/or tests pursuant to the implementation of the Order.

Criteria for data validation include operational parameters (e.g., GC conditions), calibration data, and quality control procedures. Operational parameters are discussed in the reference methods for specific parameters. Calibration data and the limits controlling calibration to ensure valid results are described in Section 7.0. The following subsections provide a summary of guidelines for data validation relating to the quality control procedures discussed in Section 10.0.

9.3 DATA REPORTING

The analytical laboratory will follow CLP procedures for reporting of TCL compounds. Data related to non-TCL pesticides, pH, specific conductance, and temperature will be reported in accordance with procedures indicated in their respective reference methods. In general, soil sample data will be reported on a dry-weight basis and pH will be reported to 0.1 pH units. For volatiles and semivolatiles, one significant figure will be reported for values less than ten and two significant figures will be reported for values above ten. PCBs data will be reported to two significant figures. Reported concentrations will not be corrected for contaminants found in associated blanks.

9.3.1 Qualifiers

The analytical laboratory will use CLP-defined qualifiers in reporting data. The flags for organic analyses are as follows:

- U - Indicates analysis was performed for compound but was not detected.
- J - Indicates an estimated value.
- C - Applies to pesticide results where the identification has been confirmed by GC/MS.
- B - Used when the analyte is found in the associated blank as well as in the sample.

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- E - Identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for a specific analysis. This flag will not apply to PCBs analyzed by GC/EC methods.

- D - Identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is re-analyzed at a higher dilution factor, the "DL" suffix will be appended to the sample number and all reported concentration values will be flagged with the "D".

In addition to the preceding qualifiers, the following qualifiers will be used as applicable:

- Each surrogate recovery outside the QC limits will be flagged with an asterisk (*). If the surrogates are diluted out, the recoveries will be flagged with a "D." Surrogates diluted out will not be counted as outliers in the determination of completeness.

  Each percent recovery for matrix spikes which is outside the QC limits will be flagged with an asterisk (*). Also, if the RPD is outside the QC -limits for MS/MSD, the data will be flagged with an asterisk

Additional information, including examples of qualifiers, is provided in the reference methods.

9.3.2 Data Reporting System

McLaren/Hart will receive from the laboratory the sample results and a summary of the associated QC data. After the data have been validated (Section 9.2), the results will be reported in accordance with the guidelines previously discussed.

The results will be presented in the Phase I Site Investigation Report which will be submitted to the USEPA. Support data will be available at the laboratory and at McLaren/Harts' office for review. Records and documents will be maintained for 10 years from the effective date of the Order.

10.0 QUALITY CONTROL

A quality control (QC) program will be implemented so that consistent results of known and documented quality can be obtained. Both field and laboratory quality control measures will be followed to measure laboratory and total system variability. These measures are discussed in this section. Field QC involves the collection of split samples and field blanks. Laboratory QC includes internal procedures (blanks, spikes, surrogates, etc.) which are described in the reference methods for the parameters which will be analyzed. The analytical laboratory will implement stringent QC requirements that meet or exceed CLP requirements. Laboratory QC procedures which will be used for this project include:

- Initial calibration and calibration verification
- GC/MS instrument tuning requirements
- Surrogates
- Matrix spike analysis
- Duplicate analysis
- Matrix spike duplicate analysis
- Method blanks
- ICP interference check sample

The above-listed procedures are applicable to either organic or inorganic analyses or both. The first two items are described in Section 7.0, and the remainder are described in the following subsections. QC procedures for non-TCL parameters are described in the reference methods for the specific parameters.

10.1 FIELD DUPLICATE SAMPLES

A field duplicate sample is a sample prepared by dividing a sample into two aliquots. Duplicate samples will be handled as individual samples. The laboratory will not be made aware that a sample is a duplicate sample.

10.2 FIELD BLANKS

Field blanks are aliquots of analyte-free water brought to the field in sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two types of field blanks which will be used for this project.

Trip blanks will not be opened in the field. They are a check on sample contamination originating from sample transport, shipping and site conditions. Equipment blanks will be opened in the field and the contents will be poured over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample. Sufficient water will be taken to the field so that the equipment blank sample container is completely filled. The equipment blank will be handled in the same manner as aqueous samples, including preservation and cooling to 4 degrees C. For metal sample a separate equipment blank, consisting of analyte free de-ionized water poured over the filtration apparatus will also be collected. Equipment blanks are a check on sampling device cleanliness.

Trip blanks will accompany each batch of samples analyzed for volatiles. Equipment blanks will be collected at a frequency of one per type of equipment used each day a decontamination event takes place, not to exceed once per day.

10.3 LABORATORY ORGANIC QUALITY CONTROL

The following paragraphs pertain to QC procedures.related to the analysis of organic compounds.

10.3.1 Surrogates

Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds will be spiked into all blanks, standards, samples, and spiked samples

prior to analysis. Percent recoveries will be calculated for each surrogate. The surrogate compounds are listed in the reference methods.

The percent recoveries of the surrogates document laboratory performance on individual samples. Section 9.2.1 provides guidelines for reviewing and validation of data based on surrogates.

10.3.2 Method Blanks

A method blank is a volume of deionized, distilled laboratory water carried through the entire analytical scheme. For volatile analyses, a method blank will be performed once for each 12-hour time period. For semivolatiles, PCBs/pesticides, and inorganics, method blanks will be performed at the frequency described in the reference method for the parameter being analyzed. Section 9.2.2 describes criteria for the evaluation of method blanks.

10.3.3 Matrix Spike/Matrix Spike Duplicate Analysis

In matrix spike/matrix spike duplicate (MS/MSD) analysis, predetermined quantities of stock solutions of certain analytes will be added to a digestion and analysis. Samples will be split into duplicates, spiked, and analyzed. Percent recoveries will be calculated for each of the analytes detected. The relative percent difference between duplicate samples will be calculated and used to assess analytical precision.

Project-specific MS/MSD analysis will be performed at the frequency of 5 percent. Additional sample volume (3X) will be collected for samples designated for MS/MSD analysis.

10.3.4 Trip Blanks

Trip blanks consist of analyte-free water prepared by the laboratory which has been placed in VOA vials, preserved with 1:1HCL until a pH <2 is achieved, sealed and transported with the empty sample containers to the site. At the site, the vials remain sealed and then accompany the

sample containers back to the laboratory. The trip blank is analyzed for volatile organic compounds to determine the existence of contamination problems that have been introduced during sample shipment and handling. A trip blank will be prepared and analyzed with each sampling shipment containing samples that will be subjected to volatile organic analysis.

10.4 LABORATORY INORGANIC QUALITY CONTROL

All data reduction and validation procedures used to evaluate the quality control checks outlined in this section will be those specified in the USEPA Region II SOPs, referred to in section 9.2.

10.4.1 Method Blanks

Method blanks will be generated for other parameters with the same objectives as for volatile and semivolatile parameters (Section 10.3.2). Deionized, distilled water will be processed through the same preparation procedure at the frequency indicated in the reference method for the parameter being analyzed.

10.4.2 ICP Interference Check Sample

An ICP Interference Check Sample will be analyzed to verify interelement and background correction factors. This sample will be analyzed at the beginning and end of each sample analysis run or a minimum of twice per eight-hour time period, whichever is most frequent.

10.4.3 ICP Serial Dilution Analysis

The laboratory will perform ICP serial dilution analysis at the frequency described in the reference method for the parameter being analyzed. This will be done prior to reporting data.

10.4.4 Spike Sample Analysis

A known amount of the analyte of interest will be added to a sample at the frequency described in the reference method for the parameter being analyzed. Percent recovery will be calculated for each analyte. The spiked sample analysis will provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike will be added before the digestion and prior to any distillation steps.

10.4.5 Duplicate Sample Analysis

The laboratory will split a sample into two aliquots at the frequency described in the reference method for the parameter being analyzed. One aliquot will be the original sample and the other will be identified as the duplicate sample. Relative percent differences for each detected analyte will be calculated.

10.5 TCDD QUALITY CONTROL

The quality control samples listed below will be performed for every group of 24 dioxin samples collected or every 24 samples collected over a two day period, whichever comes first.

10.5.1 Performance Evaluation Sample

Two performance evaluation samples will be obtained from EMSL-LV, containing a known concentration of 2,3,7,8-TCDD. These samples will be submitted with field samples and will be unknown to the laboratory.

10.5.2 Field Duplicate

One environmental duplicate will be taken, and designated as such by the sampling team.

10.5.3 Field Blank (Blind Blank)

One uncontaminated sample soil, to be obtained from EMSL-LV, will be submitted to the lab.

10.5.4 Known Blank Sample

One uncontaminated soil sample, to be obtained from EMSL-LV, will be collected and designated as a known blank sample. This sample will be fortified by the lab with 1 ug/Kg 2,3,7,8-TCDD.

10.5.5 Rinsate Blank

Spectrophotometric grade trichloroethane (minimum 200 mls) will be poured over decontaminated sampling equipment as a check that decontamination has been adequately carried out and that no cross contamination has occurred.

11.0 AUDITS

11.1 FIELD OPERATIONS AUDIT

A field operations audit will be conducted during initial sampling/field activities to verify that field personnel are correctly following sampling procedures described in Sections 5.0 and 6.0. The field operations audit will involve an on-site visit by the Quality Assurance Officer or her designated representative and possibly by additional members of the project staff who are technically competent in the operations to be audited and who are not participating in the project. Items to be examined may, as appropriate, include the availability and implementation of approved work procedures; calibration and operation of equipment; labeling, packaging, storage, and shipping of samples obtained; performance documentation and checking; subcontractor performance; and variance documentation.

A field operations audit will be scheduled during the field activities. The Quality Assurance Officer will be notified by the Project Manager of the initiation of field work.

11.2 AUDIT PROCEDURES

Auditing will be initiated as early in the performance of an activity as practicable and will be conducted in accordance with the procedures described in the following paragraph.

Audits will be performed on the basis of a written checklist prepared prior to the audit. Checklists shall be developed to assure the review of necessary items and to document the results of the audit. The field operations audit will cover the applicable items listed in Section 11.1. During the audit and upon its completion, the auditor will discuss any findings with the individuals audited and will cite corrective actions to be initiated.

11.3 AUDIT REPORTS AND RESPONSES

Following completion of an audit, the auditor will prepare a report and submit it to the Project Director and Project Manager. This report will serve as notification to the appropriate management of audit results. The report will also be transmitted to pertinent individuals contacted during the audit. The report will be prepared as soon as possible (within 15 days) following the audit and will provide, as appropriate:

- Date and location of the audit;
- Identification of audit participants;
- Identification of activities audited;
- Audit results;
- Description of items requiring corrective action and, if possible, the means for correction; and
- Due date for completion of corrective actions, preventative actions, and/or audit response (maximum of 30 days from the issuance of the audit report).

The report will be signed by the auditor. Checklists may be provided with the audit report. The individuals audited will be required to respond in writing to the audit report. The response shall clearly state the corrective action taken or planned. Completion of corrective action shall be verified by the auditor through written communication, re-audit, or other appropriate means.

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11.4 AUDIT CLOSURE

After acceptance and verification of all corrective actions, the auditor will issue an audit closure to the same individuals receiving the audit report.

12.0 PREVENTIVE MAINTENANCE

The analytical laboratory will keep instrument log books which document maintenance schedules, dates, maintenance performed, and details of each maintenance action. Routine maintenance will be performed by laboratory personnel in accordance with manufacturer recommendations. Service agreements and preventive maintenance contracts are routinely secured by the laboratory for all critical instruments and equipment. These agreements provide for regular checks by qualified service personnel. Critical spare parts are maintained in accordance with manufacturer recommendations.

13.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

Precision and accuracy for this project will be assessed through the analysis of duplicates, surrogates, matrix spike/matrix spike duplicate and spike samples. The appropriate QC procedure is specified in the analytical method. Section 10.0 summarizes the QC analyses.
Completeness will be determined after validation of the data.

13.1 PRECISION

To determine the precision of the analytical methods, a program of replicate analyses will be followed. The laboratory will split a sample into two subsamples and analyze them independently at the frequency listed in the appropriate method.
The results of the replicate analysis will be used to calculate the quality control parameter (relative percent difference) for precision evaluation. The following equation is used to calculate relative percent difference (RPD):

$$RPD = \frac{|D_2 - D_1|}{(D_1 + D_2)/2} \times 100$$

where: D_1 - First subsample value
D_2 - Second subsample value

In addition to evaluation of the method precision, duplicate or split samples will be collected in the field and analyzed independently. The results will be used to evaluate the total systems variability, including sampling variations. The identity of field splits will not be known by the laboratory personnel.
The analytical precision produced by laboratory duplicate analyses will be evaluated by both the laboratory and McLaren/Hart, while field splits will be evaluated only by McLaren/Hart. Evaluation of both types of data will be in accordance with the reference methods and this plan.

13.2 ACCURACY

Accuracy is qualitatively discussed in Section 4.2. To determine the accuracy of an analytical method, a program of sample spiking will be followed. The spiking frequency will be as stated in the reference methods. The results of sample spiking will be used to calculate the quality control parameter (percent recovery) for accuracy evaluation. The following equation will be used:

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

where: SSR - Spiked sample result
SR - Sample result
SA - Spike added

Section 9.2.7 discusses the evaluation of spike sample analysis.

In addition to a spiking program, all samples, standards, and blanks subject to organic analyses will be spiked with surrogate compounds. Laboratory performance on individual samples will be established by the recovery of surrogate compounds. Additional discussion on surrogates is provided in Section 9.2.1.

13.3 COMPLETENESS

The objective of the Phase I sampling and analysis program is to determine site-specific characteristic parameters for possible additional investigations. The completeness goals to meet this objective are listed in Table 4-1. Completeness for each parameter is calculated as follows:

$$\frac{\text{Valid Data}}{\text{Total Data Generated}} \times 100 = \%\ \text{Complete}$$

Valid data will be determined in accordance with the appropriate reference method and this plan (Section 9.0). The percent complete will be used to determine whether the data quality meets the objectives for the Phase I investigation.

If the completeness objectives are not met for individual parameters, the reasons for the invalid data will be reviewed by McLaren/Hart. Depending on the reasons (e.g., holding time exceeded) and the effect of the incomplete data on the accomplishment of the objectives, additional samples may be collected and analyzed. This subjective evaluation will also be performed if a sample does not generate data for a parameter category (e.g., pesticides, PCBS). Such a data gap could result from sample container breakage or loss or sample custody not being maintained. If it is determined by McLaren/Hart that the missing results are critical to accomplishing the project objectives, additional sampling will be performed to obtain the missing data.

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14.0 NONCONFORMANCE/CORRECTIVE ACTION

14.1 NONCONFORMANCE

Nonconforming items and activities are those which do not meet the project requirements specified in the Order, Phase I Site Operations Plan, or the QA/QC Plan. Nonconformances may be detected and identified by:

- Project Staff
  During investigation and testing, supervision of subcontractors, and preparation and verification of analyses.

- Laboratory Staff
  During analysis, internal quality control, and data validation activities.

- Quality Assurance Officer
  As the result of audits (Section 11.0) and other quality assurance activities.

Each nonconformance shall be documented by the personnel identifying or originating it. Documentation will, as

appropriate, include:

- Identification of the individual(s) identifying or originating the nonconformance.

- Description of the nonconformance.

- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted.

- Schedule for completing corrective action.

The nonconformance documentation shall be submitted to the Project Manager, who will be responsible for notification of other staff, PPG, and regulatory agencies, as appropriate.

14.2 CORRECTIVE ACTION

Corrective actions pertaining to field activities will be defined when specific nonconformances are identified and documented.

Corrective actions pertaining to analytical procedures are directly related to the calibration, internal quality control, and data validation. Planned corrective actions and the predetermined limits for data acceptability, beyond which corrective action is required, are presented in Sections 7.0 through 10.0.

The authority to implement planned corrective action when nonconformances are detected or control limits are exceeded rests with the Project Manager for field and overall project operations and with the laboratory Quality Assurance Director for laboratory operations.

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15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Reports detailing the results of audits and the performance of data measurement systems will be submitted to project management. These results will include:

- Periodic assessments of measurement data accuracy, precision, and completeness.
- Results of system audits.
- Results of performance audits.
- Significant QA problems and recommended solutions.

A description of these reports is provided in the following paragraphs.

15.1 MEASUREMENT DATA REPORTS

An assessment of measurement data precision, accuracy, and completeness will be summarized in a report. This report will be prepared by the analytical laboratory Quality Assurance Director and Project Quality Assurance Officer and will be submitted to the Project Manager and Quality Assurance Officer. At the completion of the project, the results of all previous reports will be summarized in a final report.

15.2 REPORTS OF SIGNIFICANT QA PROBLEMS AND RECOMMENDED SOLUTIONS

Significant quality assurance problems will be documented as findings in audit reports. Any such findings will require a written response from the Project Manager to the Quality Assurance Officer. In the event that these individuals cannot come to agreement, the Project Director will determine the resolution of the problem. All of the decisions and their bases will be documented.

TABLE 3-1
ADDRESS AND PHONE NUMBERS OF KEY PROJECT INDIVIDUALS

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

KEY INDIVIDUAL/POSITION	ADDRESS	TELEPHONE NUMBER
Mark Terril/Project Coordinator	PPG Industries, Inc. One Allison Park Pittsburgh, PA 15238	(412) 492-5532
Greg Peterson/Project Director David Kerschner/Project Manager Robert Anderson/QA/QC Officer Neale Misquitta/Task Manager	McLaren/Hart 8500 Brooktree Rd Suite 300 Wexford, PA 15090	(412) 934-3744
Jay Bernading/Director Kathy Teuscer/Project Manager	Enseco Wadsworth Alert Labs 4101 Shuffel Drive, NW North Canton, OH 44720	(216) 497-9396

NOTES: A description of project personnel responsibilities is presented in Section 3.0 of the QA/QC plan. Figure 3-1 presents a flow chart of project responsibilities.

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TABLE 4-1
PRECISION, ACCURACY, AND COMPLETENESS OBJECTIVES

DATE: 8/1/94
REVISION: 2

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

PARAMETER	MATRIX	PRECISION	ACCURACY	COMPLETENESS
Volatile Organics	Soil/Water	(1)	(1)	80%
Semivolatile Organics	Soil/Water	(1)	(1)	80%
PCBs/Pesticides (2)	Soil/Water	(1)	(1)	80%
Metals	Soil/Water	(1)	(1)	80%
Cyanide	Soil/Water	(1)	(1)	80%
pH	Water	+/-0.2 pH units	+/- 10%	80%
Specific Conductance	Water	<20%	+/- 10%	80%
Temperature	Water	+/-0.5 deg. C	+/- 10%	80%
TCDD	Soil/Water	(1)	(1)	80%
TRPH	Soil	62 RPD	24%-162%	80%
TRPH	Water	30 RPD	57%-141%	80%

NOTES: 1. Refer to the appropriate analytical method for precision and accuracy of individual parameters.
2. Includes PCNB, Sevin, Rotenone, and Malathion

TABLE 5-1
CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

DATE: 8/1/94
REVISION: 2

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

SOIL OR SEDIMENT			
ANALYSIS	BOTTLE TYPE	PRESERVATIVE	HOLDING TIME
Volatiles	2 120-ml glass (1) vials (including Teflon lined caps)	Cool, 4°C	10 days of collection
Semivolatiles (2)	1 pint glass	Cool, 4°C	Extraction 7 days of collection Analysis 40 days
PCBs/Pesticides	1 pint glass	Cool, 4°C	Extraction 7 daysof collection Analysis 40 days
Metals, Cyanide	1 pint glass	Cool, 4°C	6 months (metals) 28 days (Hg) of collection 14 days (Cn) of collection
Sevin	1 pint glass	Cool, 4°C	Extraction 7 days of collection 40 days to Analysis
Dioxin	1 pint glass	Cool, 4°C	6 months
Malithion	1 pint glass	Cool, 4°C	Extraction 7 days of collection 40 days to Analysis
TRPH	1 pint glass	Cool, 4°C	Extraction 7 days of collection 40 days to Analysis

Aqueous			
ANALYSIS	BOTTLE TYPE	PRESERVATIVE	HOLDING TIME
Volatiles	2 40-ml glass vials (including Teflon lined caps)	Cool, 4°C, 1:1 HCl (pH<2)	7 days of sampling (non-preserved) 14 days of sampling (HCl preservative)
Semivolatiles (2)	2 1,000-ml glass (amber)	Cool, 4°C	extraction 7 days of collection analysis 40 days
PCBs	2 1,000-ml glass (amber)	Cool, 4°C	extraction 7 days of collection analysis 40 days
Pesticides	2 1,000-ml glass (amber)	Cool, 4°C	extraction 7 days of collection analysis 40 days
Metals	1 1,000-ml polyethylene	HNO3 (pH<2)	6 months (Hg-28 days) of collection
Cyanide (3)	1 1,000-ml polyethylene	NaOH (pH>12)	14 days of collection
Dioxin	2 1,000-ml glass	Cool, 4°C	extraction 10 days of collection analysis 40 days
TRPH	2 1,000-ml glass (amber)	Cool, 4°C, 1:1 HCl	extraction 7 days of collection analysis 40 days
Sevin	2 1,000-ml glass	Cool, 4°C	extraction 7 days of collection analysis 40 days
Malithion	2 1,000-ml glass	Cool, 4°C	extraction 7 days of collection analysis 40 days

NOTES:
(1) Glass bottles are clear unless otherwise noted.
(2) Semivolatile analysis includes PCNB
(3) For the aqueous cyanide analysis, if a positive test for oxidizers is indicated by KI-starch paper, 0.6 gm of ascorbic acid will be added and a retest will be performed to ensure a negative result.
The sample will also be checked for the presence of sulfides and, if present, ascorbic acid will be added with NaOH to preserve the sample.

TABLE 8-1
PHASE II SAMPLE SUMMARY

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

DATE: 8/1/94
REVISION: 2

SAMPLE MEDIA	NO. OF SAMPLES	DUPLICATES	REPLICATES	TRIP BLANKS	FIELD BLANKS	ANALYTICAL PARAMETERS
SOIL	15(1)	2	2	1	1	Field Test Kit Screening - Total Chlorinated Pesticides Test Kit Analysis - Arsenic, Lead
	199	10	10	4	4	Field Test Kit Analysis - Total Chlorinated Pesticides - Arsenic, Lead
	40 (2)	5	5	1	1	TCL, VOCs, SVOCs, Pesticides, TOX(3), Sevin, PCNB Malathion, Arsenic, Lead, Chromium, Cadmium
	10 (4)					Dioxins (+5 QA/QC Per Region II SOPs)
	6					Geotechnical Analysis
SURFACE WATER	7	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion, Rotenone Metals: Arsenic, Lead, Chromium, Cadmium General Chemistry: Field pH, temp, and spec. cond.
SEDIMENT	7	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion Metals: Arsenic, Lead, Chromium, Cadmium General Chemistry:pH, TOC
GROUNDWATER	Approx 10	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion Total and Dissolved Metals: Arsenic, Lead - Chromium, Cadmium General Chemistry: Field pH, temp, and spec. cond.
	10					Dioxins (Based on Soil Sampling Results)
TEST PITS	10 to 25	2	2	1	1	Test Kit Field Survey Analysis
	5	1	1	1	1	TCL VOCs TCL SVOCs TCL Pesticides, Sevin, Malathion, Herbicides, PCNB Rotenone, Dioxins, TOX Metals: Arsenic, Lead, Chromium, Cadmium
AIR SAMPLING	3					TCL Pesticides
UST	3	1	1	1	1	Total Petroleum Hydrocarbons

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TABLE 8-1 (continued)

NOTES:

(1) Indicates that these samples will be collected to select the appropriate field screening test kit (*i.e.* Dexsil or Millipore)

(2) These samples are intended for fixed base laboratory confirmatory analysis, and comprise 20 % of the total number of samples collected for field screening..

(3) Chromium analysis indicates both total chromium and hexavalent chromium will be analyzed for.

(4) Dioxin analysis will be of composite samples from the 40 samples to be sent for confirmatory analysis. QA/QC for these samples will be as per Region II Guidance.

(5) Two additional sediment samples may be obtained from the storm sewer catchment basins (if sediment is present within the basins).

TABLE 8-2
ANALYTICAL METHODS

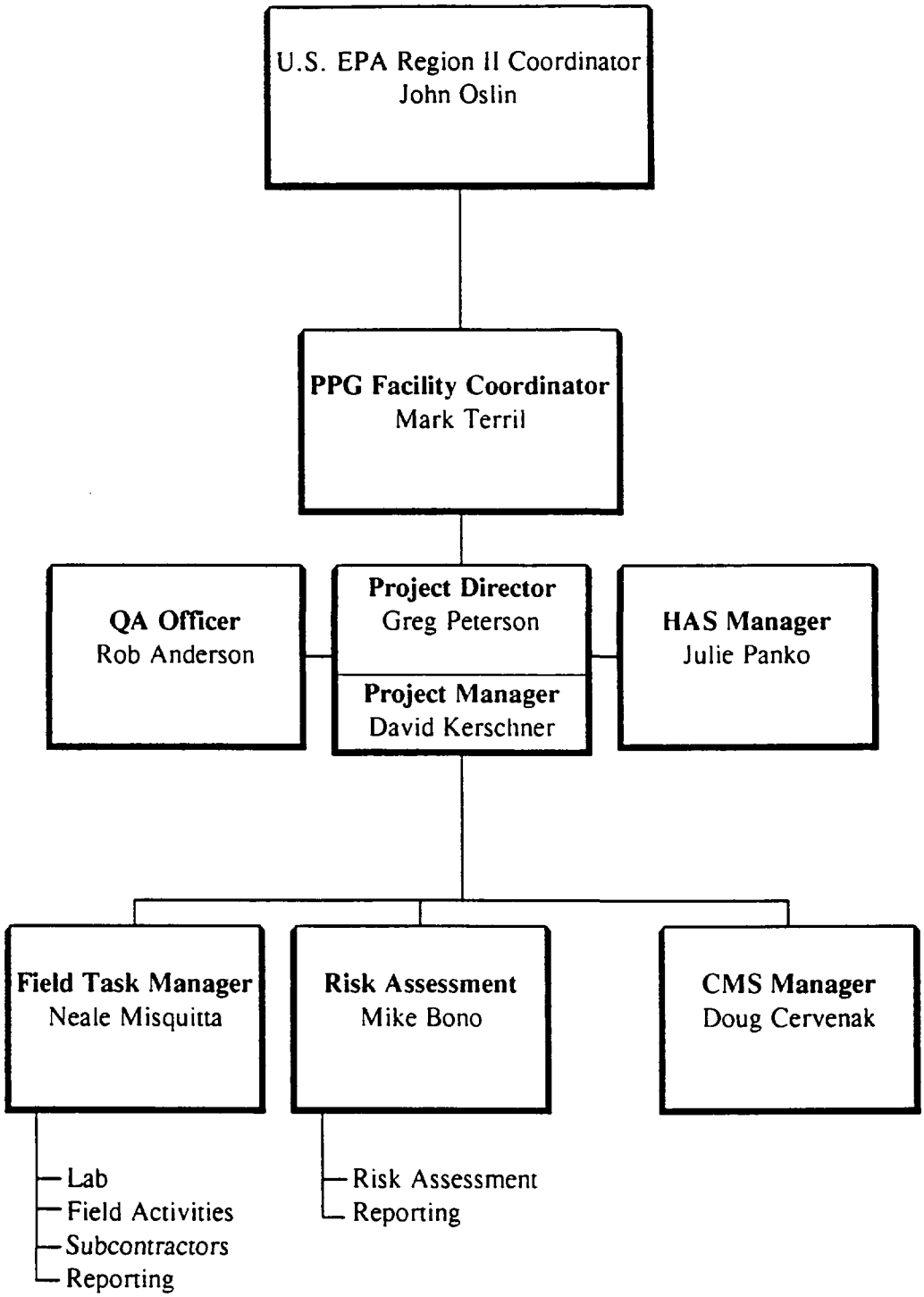
DATE: 8/1/94
REVISION:2

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

CATEGORY	MATRIX	EXPECTED CONCENTRATION (1)	SAMPLE PREPARATION	ANALYSIS	METHOD (2)
Volatiles	water	low	purge & trap	GC/MS	SOW OLM01.9
	soil	low	purge & trap	GC/MS	SOW OLM01.9
Semivolatiles	water	low	liquid/liquid extrtn.	GC/MS	SOW OLM01.9
	soil	low	ultrasonic extrtn.	GC/MS	SOW OLM01.9
PCBs/Pesticides	water	low - medium	liquid/liquid extrtn.	GC/EC	SOW OLM01.9
	soil	medium - high	ultrasonic extrtn.	GC/EC	SOW OLM01.9
Metals					
Mercury	water	low	digestion	cold vapor AA	SOW 1LM03.0
	soil	low	digestion	cold vapor AA	SOW 1LM03.0
Arsenic, Lead, and	water	low	acid digestion	Furnace AA	SOW 1LM03.0
Selenium	soil	low	acid digestion	Furnace AA	SOW 1LM03.0
Other TAL	water	low	acid digestion	ICP	SOW 1LM03.0
Metals	soil	low	acid digestion	ICP	SOW 1LM03.0
Cyanide	water	low	distillation	colorimetric	SOW 1LM03.0
	soil	low	distillation	colorimetric	SOW 1LM03.0
Sevin and Rotenone	water	low	sep. funnel extrtn.	HPLC	SW846 8318
	soil	medium	sep. funnel extrtn.	HPLC	SW846 8318
Malathion	water	low	3510/3520	GC	SW846 8141
	soil	medium	3540/3550	GC	SW846 8141
PCNB (3)	water	low	liquid/liquid extrtn.	GC/MS	SOW OLM01.9
	soil	medium	ultrasonic extrtn.	GC/MS	SOW OLM01.9
Dioxin	water	low	liquid/liquid extrtn.	GC/MS	SOW DFIM01.1
	soil	low	ultrasonic extrtn.	GC/MS	SOW DFIM01.1
TRPH	water	low	sep. funnel extrtn.	IR	418.1
	soil	medium	ultrasonic extrtn.	IR	3550/418.1

(1) The determination of expected concentrations is based on sensitivity of analytical methods and does not apply to the magnitude of the expected environmental concentrations.
(2) Refer to CLP methods for detailed procedures of sample preparation.
(3) PCNB will be determined with TCL semivolatiles. For samples which do not have semivolatile analysis, PCNB will be determined by USEPA Methods 3540/3550 and SE-846 8270.

FIGURE 3-1
PHASE II INVESTIGATION ORGANIZATION
PPG PULVERIZING SERVICE SITE
MOORESTOWN, NEW JERSEY



APPENDIX A

DRILLING, SAMPLING, AND MONITORING WELL INSTALLATION PROCEDURES

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APPENDIX A

DRILLING, SAMPLING, AND MONITORING WELL INSTALLATION PROCEDURES

A.1 TEST BORINGS

Test borings will be advanced using hollow-stem augers. A center plug or stinger will be utilized while advancing the boring. Standard penetration tests will be conducted in each test boring in accordance with ASTM D-1586.

A split-barrel sampler will be driven with a guided hammer into the unconsolidated soil. The inside of the split barrel will be flush with the inside of the drive shoe.

The bottom of the sampler shall be sharpened to form a cutting edge at its inside circumference. The beveled edge of the drive shoe will be maintained in good condition and, if excessively worn, will be replaced or reshaped to the satisfaction of the field engineer/geologist. The drive shoe of the sampler will be replaced if damaged in such a manner as to cause projections within the interior surface of the shoe. Each drill rig will be equipped with a minimum of two drive shoes which are in good condition. The sampler will be fastened to its drive pipe by a connection embodying a check valve arranged so as to permit the escape of fluid entrapped above the soil sample as the spoon is driven down into the soil, but which will close as the soil sample and sampler are withdrawn, thus preventing the development of hydraulic pressure on top of the soil sample.

The hammer or ram used to drive the sampler shall weigh 140 pounds and shall fall freely through a height of 30 inches. The number of blows required to drive the sampler each six inches for a total depth of 24 inches will be observed and recorded. The record will clearly show the

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number of blows for each six inches of penetration. Cumulative blows will not be accepted. In soil requiring 50 blows or more per six inches of penetration, the sampler will be driven 12 inches and the number of blows for each successive six inches of penetration will be observed and recorded. In hard materials requiring more than 100 blows per six inches of penetration, the blows for smaller amounts of penetration may be observed and recorded with special note of the amount of penetration actually obtained.

Immediately upon removal from the borehole, the split-barrel sampler will be carefully disassembled and the soil will be classified. The most representative and least disturbed portion of the sample, measuring about three inches in length, will be trimmed and placed immediately into a glass jar. When a change in strata is observed in the materials found within the split-barrel sampler, a sample of each material shall be taken, and the depth of the change will be recorded.

A.2 CLASSIFICATION OF SOIL SAMPLES

Soils will be classified in accordance with the following classification categories. In general, soil will be considered either as granular or cohesive.

- Texture
  A granular soil will be considered basically either a gravel or a sand. Soils in either category shall be described as coarse, medium, or fine. The supplementary texture of the granular material will be described through the use of one adjective only. A cohesive soil will be considered basically either a silt or a clay. The supplementary texture of the cohesive material will be described through use of one adjective only.

- State
  Granular soils will be defined in terms of density, as very loose, loose, medium dense, dense or very dense (on the basis of SPT blowcounts). Cohesive soils will be defined in terms of consistency, as very soft, soft, medium

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stiff, stiff, very stiff, or hard (on the basis of pocket penetrometer unconfined compressive strength).

- Moisture
  The amount of moisture present in a soil sample will be defined in terms of wet, moist, or dry.

- Color
  The basic color of a soil will be provided and the description will be amplified if necessary using adjectives such as light, dark, mottled, or mixed.

- Evidence of Contamination
  Any visual evidence of contamination will be noted. In addition, any odor that may indicate possible contamination will also be noted.

All information will be recorded on a boring log. Borings will be terminated at a depth of 20 feet, unless visible contamination is present below that depth. Typical boring log forms are provided in Appendix B.

A.3 GROUTING PROCEDURES

Upon completion of drilling, soil borings will be grouted from the bottom to the surface using the tremie method. A tremie pipe will be lowered to the bottom of the boring and a grout mix will be pumped through the pipe to the bottom of the boring. The grout mix will be in the following proportions:

- 1 94-pound bag Portland Type IA cement;
- 7 gallons water; and
- 5 pounds powdered bentonite.

A.4 MONITORING WELL INSTALLATION PROCEDURES

Six shallow monitoring wells will be installed as part of the Phase I Investigation. A deep monitoring well will be installed in a downgradient location as part of Phase II of the project. The deep monitoring well will be installed in the first groundwater-bearing zone

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beneath the clay layer located 20 to 30 feet below the surface. A pilot test boring will be drilled at each of the shallow monitoring well locations as described in Section A.1. The hollow-stem augers utilized will have an inside diameter of at least eight inches and an outside diameter of not less than 11 inches. Each of these borings will be terminated approximately ten feet below the groundwater surface in the uppermost water bearing unit encountered at the site.

The deep monitoring well will be drilled using bentonite mud rotary drilling techniques. Potable water will be used in making the bentonite mud. The boring will be advanced in two stages. First, the entire uppermost water bearing unit will be drilled, and split-barrel samples will be obtained on 2.5-foot centers. Upon encountering the clay stratum, the hole will be reamed to 16 inches in diameter and 12-inch diameter Schedule 40 PVC casing will be lowered to the bottom of the boring and tremie grouted in place using a cement-bentonite grout. The grout mixture will be as defined in Section A.3.

After installation of the 12-inch diameter casing, the drill rig and associated equipment will be moved to the decontamination pad and thoroughly steam cleaned.

Twenty-four hours after the 12-inch casing has been installed, fluid within the casing will be flushed out and the underlying clay will be drilled to the top of the next saturated unit. The second stage of drilling will be performed using a tri-cone roller bit with bentonite mud as the circulating fluid.

Upon encountering the saturated unit, an eight-inch diameter Schedule 40 PVC casing will be lowered to the bottom of the boring and will be tremie grouted in place using cement-bentonite grout. The grout mixture will be as defined in Section A.3. The drill rig and associated equipment will again be steam cleaned.

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Twenty-four hours after the eight-inch casing has been installed, fluid within the casing will be flushed out and the saturated unit will be drilled at an approximate diameter of eight inches to the desired depth.

A.5 WELL DESIGN AND CONSTRUCTION MATERIALS

All monitoring wells will be constructed in accordance with New Jersey regulations. Each will be constructed of four-inch inside diameter, Schedule 40 PVC flush joint threaded casing and screen. Only casing with water-tight joints will be used. Factory manufactured screen slots are required and shall be No. 10 size (.010 inch). Minimum screen length will be 10 feet. Joints will be wrapped with teflon tape and no PVC cementing agents will be used. All well construction materials (as well as drill rig and tools) will be thoroughly steam cleaned before use and between wells.

Once a boring has been drilled and all pertinent data obtained, the field engineer/geologist will determine the monitoring well design based on site-specific factors and the following general criteria:

1. All fluids shall be flushed from the temporary casing prior to well construction.

2. The annular space between the borehole wall and the well casing shall be backfilled with clean, bagged sand to a point at least two feet above the top of the screen. The sand filter should not be in contact with a water-bearing zone other than the one targeted for monitoring. The hollow-stem augers shall be withdrawn approximately one foot at a time while the sand is poured from the surface. The sand level should be frequently sounded and kept at the base of the augers or temporary casing until the desired length of sand filter pack is in place.

3. Approximately three feet of pelletized bentonite shall be placed above the sand-pack and allowed to hydrate. The bentonite seal shall then be tamped into a cohesive clay mass.

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DCR

4. The remainder of the annular space shall be grouted with a cement-bentonite grout from the top of the bentonite seal upward to ground surface with a side-discharge tremie pipe.

5. A concrete pad shall be constructed at the surface to secure a protective steel casing with locking cap over the monitoring well.

Typical construction details for shallow and deep monitoring wells are shown on Figures A-1 and A-2, respectively.

A.6 WELL DEVELOPMENT

Each well will be developed using a surge block and pump or bailer. Prior to surging, the wells will be pumped to remove debris, sediment, and grout from the inside of the casing. Wells will be developed for a minimum of two hours or until they produce low-turbidity, chemically stable water. Field measurements of pH and conductivity will be used to establish chemical stability and to verify that grout contamination is not present. Monitoring wells yielding high pH water indicative of grout contamination of the sand filter pack will be unacceptable.

A Ground Water Monitoring Well Certification, Forms A and B will be completed for each well and submitted to the State by the Contractor. In addition, the NJDEP Well Permit Number will be affixed to each well by the Contractor.

A.7 FIELD CLEANING

The well casing and well screens will be field cleaned. They will be scrubbed to remove foreign material and then steam cleaned inside and out until all traces of oil and grease are removed. Adequate cleaning of well casing an screens shall be determined by the field engineer/geologist.

All equipment used in drilling and well installation must be properly cleaned to remove all foreign material. This includes drilling tools

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and trailers used to haul equipment. Once cleaned, equipment must be stored in a manner to prevent contamination. All drilling equipment will be cleaned after work at one boring/well has been completed and prior utilization of the equipment at another boring/well.

A.8 GENERAL CONDITIONS

All such drilling, sampling, and other activities shall be conducted in accordance with the project Health and Safety Plan.

The Contractor shall supply all well construction materials, well development equipment, health and safety monitoring equipment, and all decontamination materials.

The Contractor shall be licensed to conduct the activities described above in the State of New Jersey and shall be responsible for obtaining and filing all appropriate permits for such work.

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GEOPROBE

GEOPROBE OPERATION METHODS

McLaren/Hart will utilize a Geoprobe® for the collection of soil samples. The Geoprobe® is a van mounted, hydraulically operated sampling unit designed to collect soil, soil gas and groundwater samples at discrete depths. The use of the Geoprobe® during this phase of the soil investigation will provide an initial determination of the extent of affected soil and will help in the placement of temporary well points.

Soil samples will be obtained by driving a two foot long, acetate-lined, stainless steel sampling tube equipped with an internal, moveable piston to a position just above the desired sampling depth. After the sampling tube is properly positioned, the internal piston will be released and the tube driven an additional 24 inches, allowing the soil to enter the tube. The sampling tube will then be retrieved to the surface and the soil sample will be removed from the tube within the acetate liner.

Soil samples will be immediately field screened utilizing a HNu® photoionization detector (PID) and then transferred into sterile laboratory supplied bottles, labelled and preserved with ice to 4°C for delivery to a qualified analytical laboratory.

Soil samples will be collected from the unsaturated zone at predetermined increments in accordance to the work plan requirements. Sample collection will also be in accordance to the work plan.

All sampling equipment will be decontaminated prior to sampling at each location to minimize the potential for cross contamination. Decontamination procedures will include a soapy water scrub with a non-phosphate detergent, a distilled water

rinse, a methanol spray rinse to remone any remaining organic residues, and a final distilled water rinse and air dry. An additional, nitric spray rinse followed by a distilled water rinse will be added to the decontamination procedure when sampling for metals. A new pair of latex gloves will be donned prior to sampling to minimize potential cross contamination.

All soil sample locations will be sealed with cement grout upon completion of the sampling.

FIGURES

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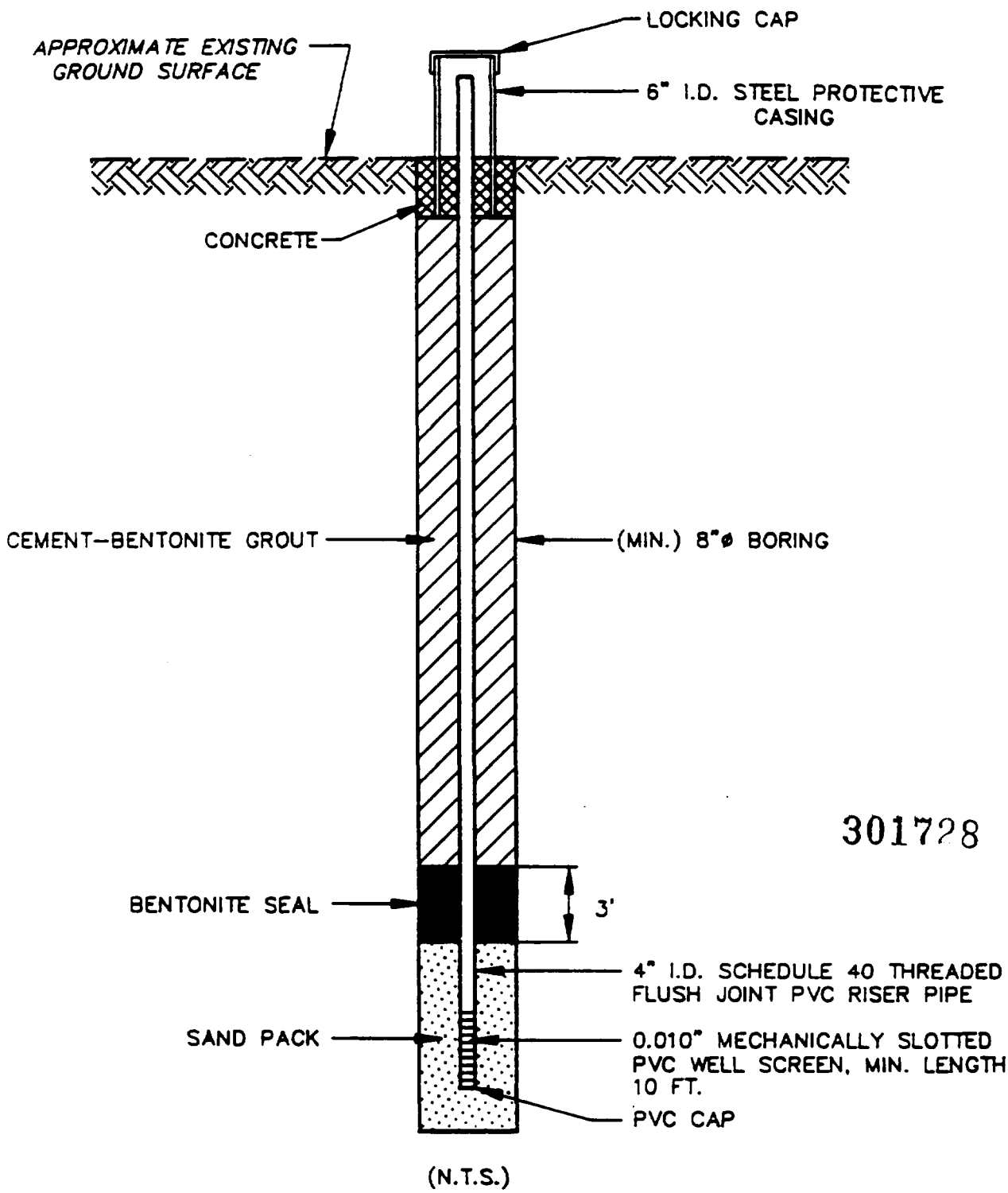

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FIGURE A-1

SHALLOW MONITORING WELL
PULVERIZING SERVICE SITE
MOORESTOWN, NEW JERSEY

PREPARED FOR

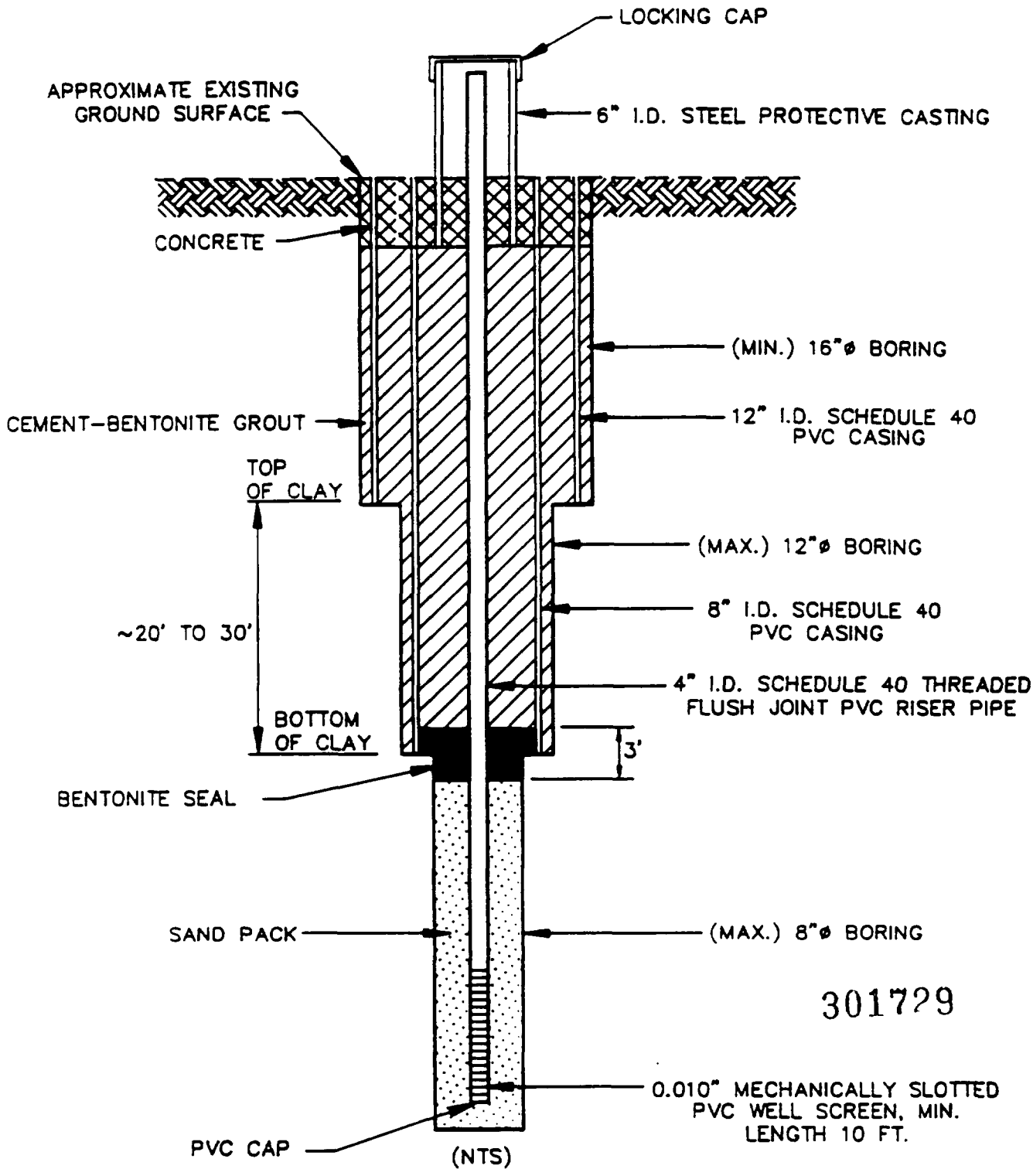
PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

Paul C. Rizzo Associates, Inc.
CONSULTANTS

DRAWN BY	D.J.D. 6-13-89	CHECKED BY JTO	APPROVED BY JTO	6-16-89	6-16-89	DRAWING NUMBER 89-595-A3 Δ

Δ GENERAL REVISIONS JTO 9-29-89

REVISIONS



301729

FIGURE A-2

DEEP MONITORING WELL
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

PREPARED FOR

PPG INDUSTRIES, INC.
PITTSBURGH, PENNSYLVANIA

Paul C. Rizzo Associates, Inc.
CONSULTANTS

DRAWN BY	T.Jor.	CHECKED BY	JTO	6-16-89	DRAWING NUMBER	89-59[illegible]-A4
	6-13-89	APPROVED BY	JTO	6-16-89		

GENERAL REVISIONS JTO 9-29-89

REVISIONS

APPENDIX B

FORMS AND RECORDS

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NOTE

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VISUAL CLASSIFICATION OF SOILS

PROJECT NO. _____ PROJECT NAME _____

BORING NUMBER _____ APPROX. EL. _____ PAGE ____ OF ____

COORD. (N) _____ GWL: DEPTH ____ DATE/TIME ____ DATE _____

(E) _____ ____ DATE/TIME ____ DATE STARTED _____

DRILLING METHODS _____ ENG/GEO ____ DATE COMPLETED _____

DEPTH ( )	SAMPLE NO. AND TYPE	BLOWS ON SAMPLER PER ( )	RECOVERY ( )	DESCRIPTION	U.S.C.S. SYMBOL	MEASURED CONSISTENCY (TSF)	REMARKS

NOTES:

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VISUAL CLASSIFICATION OF ROCK

PROJECT NO. _____ PROJECT NAME _____
BORING NUMBER _____ APPROX. EL. _____ PAGE ____ OF ____
COORD. (N) _____ ENG/GEO _____ DATE _____
(E) _____ DRILLING METHODS _____ CORE SIZE _____

CASING INFORMATION		GROUNDWATER LEVEL DATA			
SIZE	DEPTH	ACTUAL TIME	DEPTH	ACTUAL TIME	DEPTH

RUN NUMBER	DEPTH ( )	RECOVERY	% RECOVERED	RQD	PROFILE	DESCRIPTION	JOINT SPACING	REMARKS

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TEST PIT CLASSIFICATION

PROJECT NAME _____ PROJECT NO. _____ PAGE ____ OF ____
FIELD ENG/GEO _____ APPROX. EL. _____ TEST PIT NO. ____
COORD. (N) _____ DEPTH TO GWL _____ DATE _____
(E) _____

DEPTH ( )	SAMPLE NO. AND TYPE	SOIL PROFILE	DESCRIPTION	U.S.C.S. SYMBOL	REMARKS

NOTES:

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CONSULTANTS

SOIL SAMPLE FIELD COLLECTION REPORT

PROJECT NAME _____ PROJECT NUMBER _____

DATE COLLECTED _____ TIME COLLECTED _____

COLLECTED BY _____

SAMPLE(S) LOCATION SKETCH (USE BACK SIDE IF NECESSARY)

SAMPLE ID NUMBER	DEPTH OF SAMPLE	SOIL DESCRIPTION (COLOR, COMPOSITION, STAINING, ODOR, FIELD MEASUREMENTS (1))

SAMPLING METHOD _____

COMPOSITE SAMPLE ? Y ☐ N ☐ COMPOSITE SAMPLE ID NUMBER _____

DESCRIBE COMPOSITING _____

SAMPLE TYPES COLLECTED

TYPE (2)	VOLUME	PER SAMPLE ?	PER COMPOSITE ?
		Y ☐ N ☐	Y ☐ N ☐
		Y ☐ N ☐	Y ☐ N ☐
		Y ☐ N ☐	Y ☐ N ☐
		Y ☐ N ☐	Y ☐ N ☐

NUMBER OF CONTAINERS _____

DATE RECEIVED BY LAB _____ LABORATORY _____

WEATHER CONDITIONS: _____

REMARKS _____

(1) ORGANIC VAPOR ANALYSIS, POCKET PENETROMETER, ETC.
(2) METALS, VOA, ORGANICS, ETC.

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CONSULTANTS

WATER SAMPLE FIELD COLLECTION REPORT

PROJECT NAME _____ PROJECT NUMBER _____

DATE COLLECTED _____ TIME COLLECTED _____

COLLECTED BY _____

SAMPLE ID NUMBER _____ SAMPLE LOCATION _____

STATIC WATER LEVEL _____ (FT.). MEASURED FROM(1) _____

CASING STICK UP _____ (FT.)

WELL VOLUMES PURGED _____. PURGING METHOD(2) _____

TYPE OF SAMPLE(3) _____. SAMPLING METHOD(4) _____

DEPTH OF SAMPLE _____ (FT.). MEASURED FROM(1) _____

SAMPLE COLLECTION ORDER _____

FIELD MEASUREMENTS

WATER TEMPERATURE _____ pH _____

SPECIFIC CONDUCTANCE _____ µmho/cm AT _____ TEMPERATURE

OTHER _____

METER CALIBRATION					
pH STD	METER READING	SP. COND. STD	METER READING	_____ STD	METER READING

SAMPLE TYPES COLLECTED

TYPE(5)	VOLUME	FILTERED		PRESERVATION(6)	
		Y ☐	N ☐	Y _____	N ☐
		Y ☐	N ☐	Y _____	N ☐
		Y ☐	N ☐	Y _____	N ☐
		Y ☐	N ☐	Y _____	N ☐
		Y ☐	N ☐	Y _____	N ☐
		Y ☐	N ☐	Y _____	N ☐

NUMBER OF CONTAINERS _____

DATE RECEIVED BY LAB _____ LABORATORY _____

WEATHER CONDITIONS: _____

REMARKS _____

(1) T.O.C.=TOP OF PROTECTIVE CASING; T.O.W.=TOP OF WELL CASING; G.S.=GROUND SURFACE
(2) BAILED, PUMPED, AIR LIFT, ETC.
(3) STREAM, POND, SPRING, WELL, SEEP, SUPPLY, ETC.
(4) BAILER, KEMMERER, GRAB, PUMP, ETC.
(5) GENERAL CHEM., METAL, VOA, ORGANICS, ETC.
(6) HNO_3, NaOH, H_2SO_4, $Na_2O_3S_2$, ETC.

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CONSULTANTS

WELL DEVELOPMENT FORM

WELL NO. _____

PROJECT NAME: | PROJECT NO.

INITIAL WATER LEVEL: _____

WATER LEVEL AFTER DEVELOPMENT: _____

DATE: _____

TIME DEVELOPMENT STARTED: _____

TIME DEVELOPMENT CEASED: _____

WELL DEVELOPMENT TECHNIQUE(S) USED: _____

WAS WATER INJECTED INTO WELL DURING DEVELOPMENT YES ____ NO ____

IF YES GIVE APPROXIMATE VOLUME _____

APPROXIMATE VOLUME OF WATER REMOVED DURING DEVELOPMENT _____

VISUAL DESCRIPTION OF WATER: PRIOR TO DEVELOPMENT _____

AFTER DEVELOPMENT _____

WAS RECOVERY TEST RUN FOLLOWING DEVELOPMENT YES ____ NO ____

IF SO GIVE RESULTS: _____

ADDITIONAL REMARKS: _____

FIELD ENGINEER: _____ DATE: _____

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PAGE _____ of _____

Paul C. Rizzo Associates, Inc.
CONSULTANTS

CHAIN OF CUSTODY RECORD

PROJECT NO.:

PROJECT NAME:

SAMPLER(S) (SIGNATURE):

SAMPLE IDENTIFICATION	DATE	TIME	SAMPLE TYPE	SAMPLING LOCATION	VOLUME COLLECTED	NO. OF CON-TAINERS							COMMENTS

RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	REMARKS:		

Distribution–Original accompanies shipment, copy to project files.

SAMPLE TYPE:
W–WATER, S–SOLID, A–AIR, O–OTHER

LABORATORY:

NOTE: ALL SAMPLES ARE TO BE INSPECTED FOR PHYSICAL INTEGRITY UPON RECEIPT BY THE ANALYTICAL LABORATORY.

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DATE			
NO.			
SHEET		OF	

FIELD ACTIVITY DAILY LOG

PROJECT NAME	PROJECT NO.

FIELD ACTIVITY SUBJECT:

DESCRIPTION ON DAILY ACTIVITIES AND EVENTS:

VISITORS ON SITE:	CHANGES FROM PLANS AND SPECIFICATIONS, AND OTHER SPECIAL ORDERS AND IMPORTANT DECISIONS.
WEATHER CONDITIONS:	IMPORTANT TELEPHONE CALLS:

PERSONNEL ON SITE:

(FIELD ENGINEER) DATE

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APPENDIX C

LABORATORY QA/QC PLAN

FINAL QUALITY ASSURANCE/QUALITY CONTROL PLAN PHASE II SITE INVESTIGATION PULVERIZING SERVICES SITE MOORESTOWN, NEW JERSEY

Prepared For:

PPG INDUSTRIES, INC.

Prepared By

Paul C. Rizzo Associates, Inc.
300 Oxford Drive
Monroeville, Pennsylvania 15146

January 23, 1990

Modified By:

McLaren/Hart Environmental Engineering Corporation
Penn Center West
Building 3, Suite 106
Pittsburgh, Pennsylvania 15276

January 13, 1994

301741

EPA REGION II
SCANNING TRACKING SHEET

DOC ID # 38785

DOC TITLE/SUBJECT:

PHASE II SITE INVESTIGATION REPORT - WORK PLAN, QUALITY ASSURANCE / QUALITY CONTROL (QA/QC), HEALTH AND SAFETY PLAN (CONFIDENTIAL)
Pages 301742-301914

THIS DOCUMENT CONTAINS CONFIDENTIAL INFORMATION AND CAN BE LOCATED IN THE

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Enseco
A CORNING Company

ANALYTICAL SERVICES
QUALITY ASSURANCE PROJECT PLAN

FOR

USEPA - CONTRACT LABORATORY PROGRAM
POLYCHLORINATED DIBENZO-p-DIOXINS (PCDD) AND
POLYCHLORINATED DIBENZOFURANS (PCDF) BY
HIGH RESOLUTION GAS CHROMATOGRAPHY/LOW RESOLUTION
MASS SPECTROMETRY (HRGC/LRMS)

Prepared by:

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2.2 DISTRIBUTION LIST FOR QUALITY ASSURANCE PROJECT PLANS

Name	Number of Copies
USEPA Contract Laboratory Program Sample Management Office	1
USEPA Environmental Monitoring Systems Laboratory	1
USEPA National Enforcement Investigations Center	1
Michael J. Miille, Enseco, Inc.	1
Shelly Eyraud, Enseco-CAL	1
Gail Cho Celaschi, Enseco-CAL	1
Walter Berger, Enseco, Inc.	1

3.0 PROJECT DESCRIPTION

2,3,7,8-tetrachlorinated dibenzo-p-dioxin (2,3,7,8-TCDD), 2,3,7,8-tetrachlorinated dibenzofuran (2,3,7,8-TCDF), and the 2,3,7,8-substituted penta- hexa-, hepta-, and octachlorinated dibenzo-p-dioxins and dibenzofurans will be analyzed at Enseco Incorporated, California Analytical Laboratory (CAL). CAL is a part of the largest and most experienced environmental testing laboratories in the United States. The Enseco Incorporated laboratory network consists of:

- Enseco-Erco Laboratory in Cambridge, Massachusetts
- Enseco-East in Somerset, New Jersey
- Enseco-Rocky Mountain Analytical Laboratory in Denver, Colorado
- Enseco-Houston in Houston, Texas
- Enseco-California Analytical Laboratory in Sacramento, California
- Enseco-CRL in Garden Grove, California
- Enseco-El Monte in El Monte, California
- Enseco-Mobile Laboratories headquartered in Garden Grove, California
- Wadsworth/ALERT, North Canton, Ohio
- Wadsworth/ALERT, Cleveland, Ohio
- Wadsworth/ALERT, Pittsburgh, Pennsylvania
- Wadsworth/ALERT, Tampa, Florida

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The purpose of this Quality Assurance Project Plan (QAPP) is to describe the methods and procedures that will be used by Enseco-CAL to ensure quality, precision, accuracy, and completeness of the analytical data generated as part of the USEPA Contract Laboratory Program. The analytical data which will be generated by Enseco-CAL will come from chemical analysis of dioxins and furans in water, soil, fly ash and chemical waste samples collected by the ten EPA regions.

This QAPP is based on the USEPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80 and also reflects the requirements of CLP-SOW, Section E, Document Number DFLM01.0 including Revision DFLM01.1 (September, 1991).

This QAPP focuses on the acquisition of environmental data of defined and acceptable quality.

4.0 PROJECT ORGANIZATION AND AUTHORITIES

The Enseco Western Region hierarchy is shown in Figure 17-1 and the Enseco Cal Lab Low Resolution Team hierachy is showing in Figure 4-1. A schematic of the project work flow is shown in Figure 4-2. Responsibilities are outlined as follows:

DIVISIONAL QUALITY ASSURANCE DEPARTMENTS

Members

Each Divisional QA Department is managed by a QA Director. The QA Director reports directly to the Regional General Manager and indirectly to the Corporate QA Director. The QA Director is supported by a QA staff within the laboratory. The QA Director is the final authority within each laboratory on all issues dealing with data quality. He/she has the authority to require that procedures be amended or discontinued or analyses suspended or repeated. He/she can make recommendations to the Regional General Manager and the Corporate QA Director regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the Division QA Director comes directly from the Corporate QA Director.

DIVISIONAL MANAGEMENT

Members

The managers, supervisors, department directors, and program administrators who direct the analytical work at each laboratory are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.

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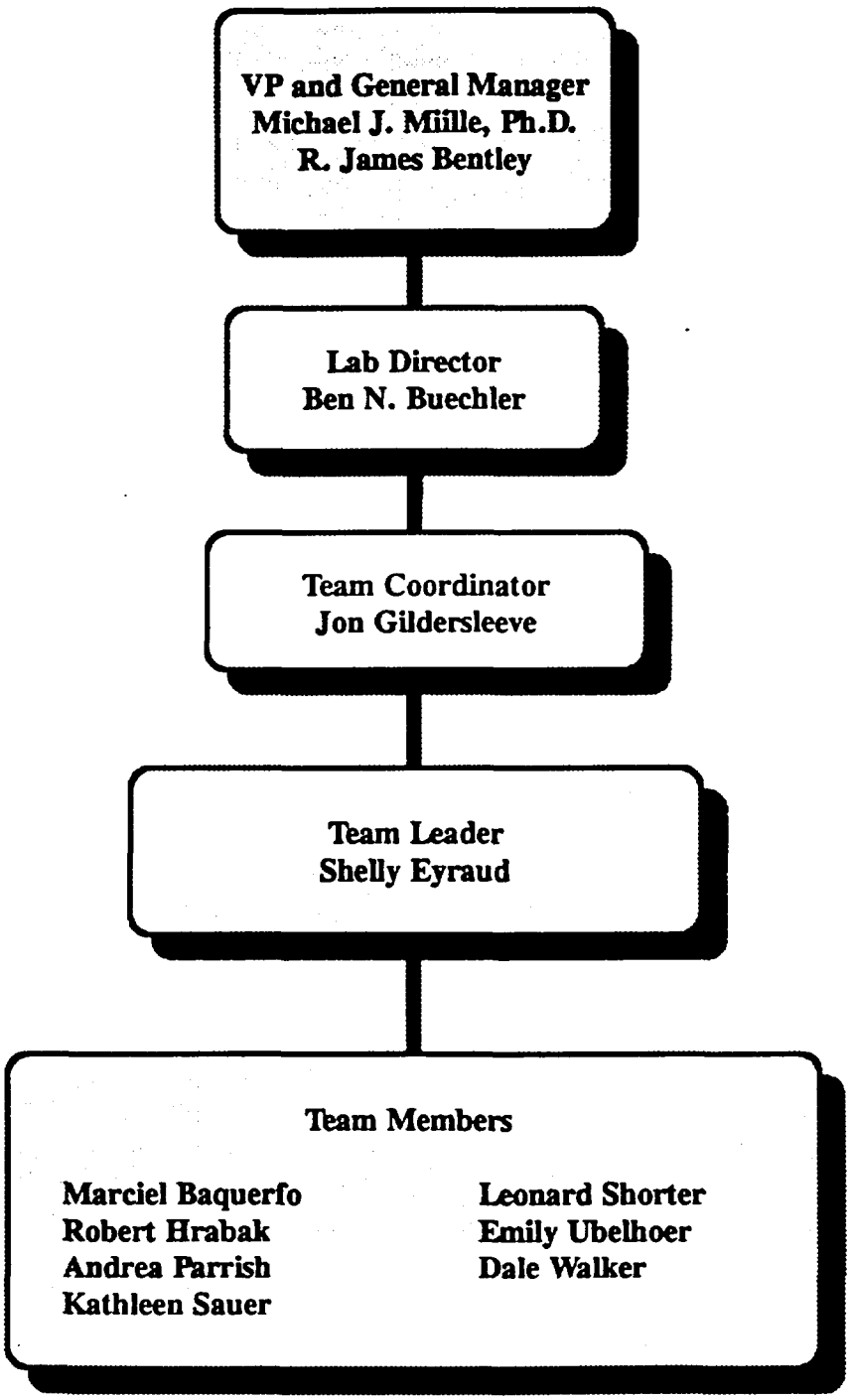
Figure 4.1

Low Resolution Team



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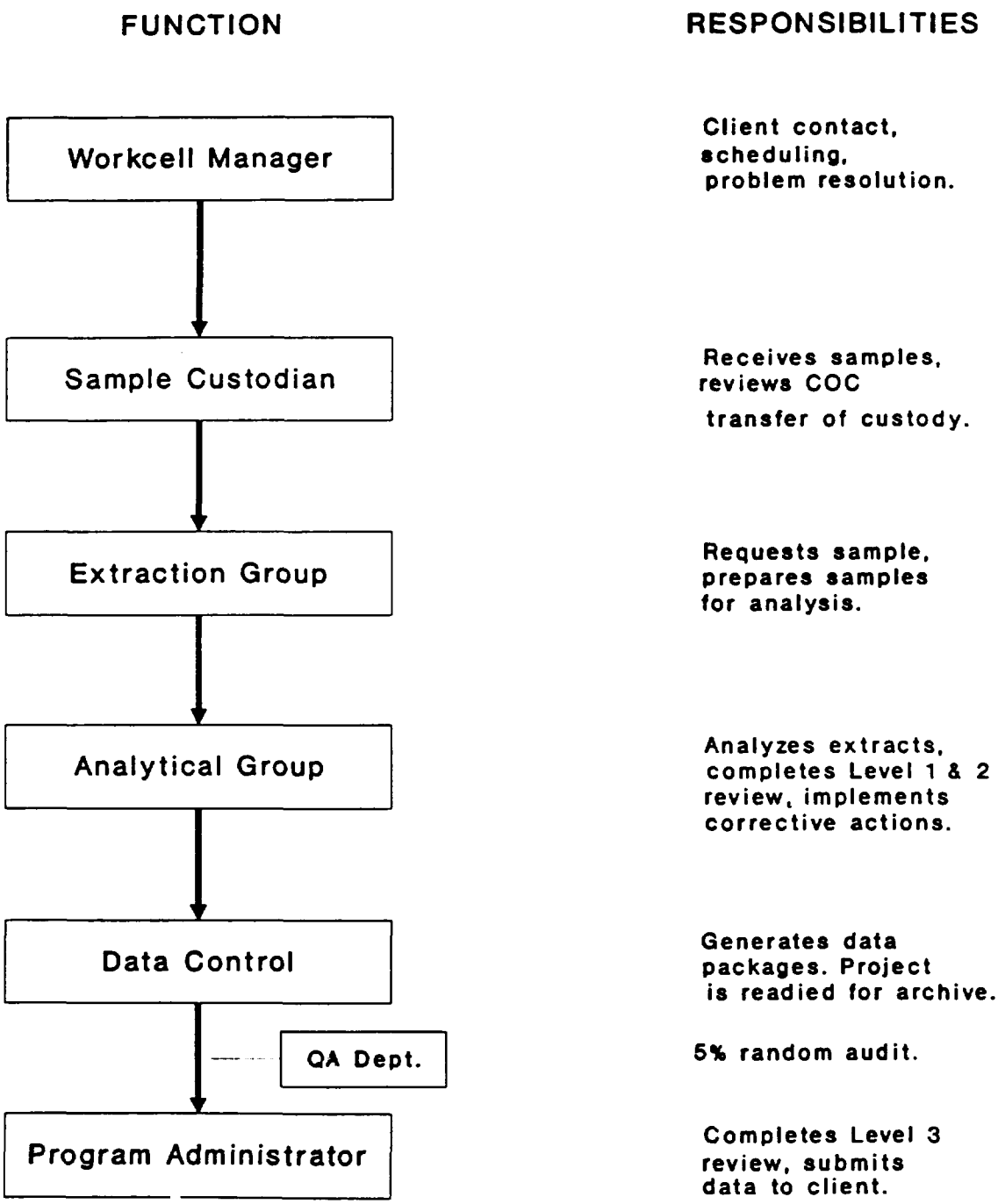
Project Responsibilities

Figure 4.2



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The managers and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgement, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the laboratory management comes directly from the President and the Regional General Manager.

DIVISIONAL PERSONNEL

Members

All laboratory personnel (including chemists, managers, etc.) involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan and any project-specific requirements in this quality assurance project plan.

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance or rejection of data that fall outside of established QC guidelines must be approved by laboratory management and the QA department. The authority of the laboratory personnel flows from the Regional General Manager.

5.0 QUALITY ASSURANCE OBJECTIVES

FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

DCS = Duplicate control samples, a pair of standard, control matrix that is spiked with a group of target compounds.

RPD = Relative percent difference.

RSD = Relative standard deviation.

CV = Coefficient of variation.

S = Standard deviation.

X = A measured value.

$\bar{X}$ = Average. Calculated as the sum of all measured values in a population divided by the number of values in the population.

n = Number of measurements or values in a population.

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with control limits established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.

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Standard deviation (S) is calculated as follows:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{N} (Xi - \bar{X})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$RSD = 100\ (s/\bar{X})$$

or

$$CV = 100\ (s/\bar{X})$$

where: RSD = relative standard deviation

CV = coefficient of variation

s = Standard deviation

$\bar{X}$ = mean

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In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

D_1 = first sample value

D_2 = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using DCS, standard reference materials, or spiked environmental samples. USEPA Performance Evaluation samples will be analyzed, if made available. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing DCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results, or accordance with program requirements (ie. internal standard recovery).

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} - \frac{X}{T} \times 100$$

where: X = the observed value of measurement

T = "true" value

Representativeness is 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. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

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

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

The completeness objective is 100%. Reanalysis will be performed in accordance with the procedures stated in Section 9.0, Analytical Procedures.

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

"Statement of Work for Analysis of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF)" USEPA Contract Laboratory Program, Multi-Media, Multi-Concentration, Document Number DFLM01.0, including Revision DFLM01.1 (September 1991) will be the protocol used for the analysis of dioxins and furans. Modifications to procedures, equipment, or reporting are listed in Table 5.2. Method specific accuracy and precision objectives are listed in Table 5.1.

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TABLE 5.1

ACCURACY AND PRECISION OBJECTIVES

COMPOUND	MATRIX SPIKE LEVEL (ng)	% RECOVERY	RPD
2,3,7,8-TCDF	25	50-150	50
1,2,3,7,8-PeCDF	62.5	50-150	50
1,2,3,6,7,8-HxCDF	62.5	50-150	50
1,2,3,4,6,7,8-HpCDF	62.5	50-150	50
OCDF	125	50-150	50
2,3,7,8-TCDD	25	50-150	50
1,2,3,7,8-PeCDD	62.5	50-150	50
1,2,3,6,7,8-HxCDD	62.5	50-150	50
1,2,3,4,6,7,8-HpCDD	62.5	50-150	50
OCDD	125	50-150	50

INTERNAL STANDARDS	INTERNAL STANDARD CONCENTRATION (ng)	% RECOVERY
$13C_{12}$-2,3,7,8-TCDD	50	25 - 150
$13C_{12}$-2,3,7,8-TCDF	50	25 - 150
$13C_{12}$-1,2,3,6,7,8-HxCDD	50	25 - 150
$13C_{12}$-1,2,3,4,6,7,8-HPCDF	100	25 - 150
$13C_{12}$-OCDD	100	25 - 150

TABLE 5.2

MODIFICATIONS TO THE STATEMENT OF WORK

SOW Section No.	SOW	CAL
D-20, 7.3.2.1	During initial calibration, the GC Resolution Criteria for DB-5 requires that the chromatographic peak separation between the 123478-HxCDD and 123678-HxCDD in the CC3 solution be resolved with a valley of $\leq$ 50 percent.	Replace DB-5 with SP-2331 to achieve isomer specificity for all 2378-substituted PCDDs/PCDFs

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6.0 SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters (see Table 6.1);
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization (see Table 6.1);
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed (see Table 6.1);
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met.

Sample Containers and Preservatives

Enseco can assist in the sample collection process by providing consultation and assistance to client designing sampling programs. Also, Enseco will make available to the client the Enseco "Sample Safe™", a set of sample containers that are properly cleaned and preserved for use in sample collection.

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Analysis and extraction holding times are not specified in the SOW DFLM01.0 PCDDs/PCDFs. Instead, the contract stipulates a data package turnaround time of 45 days from the verified time of sample receipt thereby ensuring that all PCDDs/PCDFs extractions, analyses, and reanalyses are completed within a maximum of 45 days. In lieu of analysis holding times, the 45 day turnaround time will be the analysis constraint.

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TABLE 6.1

CONTAINERS AND VOLUMES

Parameter	Contract Number	Matrix	Container	Preservative	Min. Sample Size
Dioxins/Furans	DFLM01.0	Water	One liter glass	4°C	1000 ml (a)
	PCDDs/ PCDFs	Soil/Waste	Core tube or glass jar	4°C	50 g (b)

(a) 2 1-liter glass required for analysis. 4 1-liter glass for samples requiring MS/MSD analysis.
(b) 2 core tubes or glass jars for samples requiring MS/MSD analysis.

7.0 SAMPLE CUSTODY

A sample is considered in custody if:

- It is in the sampler's or the transferee's actual possession;
- It is in the sampler's or the transferee's view, after being in his/her physical possession;
- It was in the sampler's or the transferee's physical possession and then he/she secured it to prevent tampering; and
- It is placed in a designated secure area.

Upon transfer of custody to Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

All samples are received by Enseco's Sample Control Group's designated sample custodian and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. Samples where the presence of PCDDs/PCDFs have been confirmed will be returned to the client; otherwise, sixty days after report submission, sample disposal will be in accordance with Federal, State, and local waste materials regulations. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

In the event that the laboratory sample custodian judges the sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), the work cell manager will be advised immediately and the samples will not be analyzed until the manager so authorizes. The manager or designated representative will immediately contact the sample management office. Any problem with a sample will be noted on the chain-of-custody form and telephone record log.

An example of the Chain-Of-Custody Record used to transmit samples from the client to the laboratory, or containers from the laboratory to field crews, is given in Figure 7-2.

Sample bottles will be provided to the client by Enseco and will be transmitted under custody using the Enseco "Sample Safe TM". Examples of sample labels and chain-of-custody seals are given in Figure 7-3.

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FIGURE 7-1

ENSECO SAMPLE PROCESSING FLOW CHART

Sample Control

* Check and document physical condition of sample
* Verify documentation and parameter assignment
* Log into LIMS
* Send acknowledgement letter to client

Proper Storage

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

Laboratories

* Document analytical work
* Return unused samples to Sample Control

Sample Control

* Return sample to client or arrange for sample disposal

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Figure 7-2

CHAIN-OF-CUSTODY RECORD

Enseco
A Corning Company

Section 7.0
Revision 0.0
July 1992

PAGE ___ OF ___

SAMPLER (Signature)		DATE SHIPPED	CARRIER
PHONE	AIRBILL NO.	CUSTODY SEAL NO'S	COOLER NO

SHIP TO:

- ENSECO EAST, 200 Colonial Lane, Somerset, NJ 08873 ☐
- CALIFORNIA ANALYTICAL LAB, 2544 Industrial Blvd, West Sacramento, CA 95691 ☐
- ERCO LAB, 205 Alewife Brook Pkwy, Cambridge, MA 02138 ☐
- ENSECO MARBLEHEAD, Doaks Lane at Little Harbor, Marblehead, MA 01945 ☐

SEND RESULTS TO

CLIENT NAME	
COMPANY	
ADDRESS	
PHONE NO.	

PROJECT NAME	PROJECT NO.	P.O. NO

RELINQUISHED BY (Signature)	RECEIVED BY (Signature)	DATE	TIME
RELINQUISHED BY (Signature)	RECEIVED BY (Signature)	DATE	TIME
RELINQUISHED BY (Signature)	RECEIVED AT LAB BY (Signature)	DATE	TIME
RELINQUISHED FROM LAB BY (Signature)	RECEIVED BY (Signature)	DATE	TIME

ANALYSIS REQUEST

SAMPLE ID NO.	SAMPLE DESCRIPTION	DATE / TIME SAMPLED	ANALYSIS REQUESTED	SAMPLE UPON

SPECIAL INSTRUCTIONS / COMMENTS:

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NOTE: UNUSED PORTIONS OF NON-AQUEOUS SAMPLES WILL BE RETURNED TO CLIENT.

EXPECTED ANALYTICAL T.A.T.'S	___ Immediate Attention (200% Surcharge)	___ RUSH (50-100% Surcharge)	___ Standard

ENSECO EAST LOG NUMBER (lab use only)

ENS—1045-B

Client Retains White Copy Only

FIGURE 7-3

Sample Labels and Custody Seals

CUSTODY SEAL CALIFORNIA ANALYTICAL LABS

Enseco

№ 24430

SIGNATURE_____ DATE_____

Enseco
A Corning Company

SITE NAME	DATE
ANALYSIS	TIME
	PRESERVATIVE

SPECIALTY CLEANED CONTAINER

ENS—1186

Enseco
A Corning Company

SITE NAME	DATE
ANALYSIS	TIME
	PRESERVATIVE

SPECIALTY CLEANED CONTAINER

ENS—1186

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8.0 ANALYTICAL CALIBRATION PROCEDURES AND FREQUENCY

Standard/Reagent Preparation

To ensure the highest purity possible, all primary reference standards and standard solutions used by Enseco are obtained from the National Institute of Standards and Technology, the EPA Repository or other reliable commercial sources. Dioxins/furans standards will be purchased from Cambridge Isotope Laboratories, Woburn, Massachusetts or from the USEPA, EMSL-Las Vegas (when available). All standards and standard solutions are logged into a data base that identifies the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

Balances/Ovens/Refrigerators

Balances are calibrated daily or as used with Class S or Class S traceable weights at specific weights of use with the results entered in a logbook kept near the balance.

Ovens and refrigerators are fitted with uniquely marked thermometers and monitored daily. Limits for refrigerators are 2°C to 6°C. If a temperature falls outside these limits, the appropriate laboratory manager is alerted and corrective action is taken. The readings are entered in a logbook kept near the thermometer. Annually the thermometer is calibrated vs. an NIST traceable thermometer.

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Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Specific procedures are described as follows:

Tuning

Mass calibration of the mass spectrometer is recommended prior to the analyzing the calibration solutions, blanks, samples, and quality control samples. The compound FC43 is used to tune the instrument for greater sensitivity in the high mass range to achieve better response in the later eluting compounds.

Window Defining Mix (WDM)

The window defining mix is analyzed to verify that the switching times between the descriptors have been appropriately set and is analyzed for the following conditions:

- Before initial calibration on each instrument and on each gas chromatography column used for analysis.
- Each time a new initial calibration is performed, regardless of reason.
- Each time that adjustments or instrument maintenance activities are performed that may affect retention times.
- Any time the retention time of either the $^{13}C_{12}$-1234-TCDD or $^{13}C_{12}$-123789-HxCDD recovery standards in <u>any</u> analysis varies by more than 10 seconds from its retention time in the most recent continuing calibration standard.

Initial Calibration

Five tridecane solutions (CC1-CC5) containing 10 unlabled and 7 carbon-labeled PCDDs/PCDFs at known concentrations are used to calibrate the instrument before sample analysis can commence. Analytes and concentrations are listed in Table 8.1. The relative ion abundance criteria for PCDDs/PCDFs listed in Table 8.2 must be met for all PCDD/PCDF peaks, including the labeled internal and recovery standards, in all solutions. In addition, all analytes must fall within the retention time windows as determined by the WDM and meet the mass spectrometer sensitivity criteria, i.e., the signal-to-noise ratio (S/N) must be greater than 2.5 for the unlabled PCDD/PCDF ions, and greater than 10 for the internal and recovery standard ions. Also, the absolute retention times of the recovery standards, $^{13}C_{12}$-1234-TCDD and $^{13}C_{12}$-123678-HxCDD, shall not change by more than 10 seconds between the initial CC3 analysis and the analysis of any other standard.

The GC resolution criteria requires chromatographic peak separation between the $^{13}C_{12}$-2378-TCDD peak and $^{13}C_{12}$-1234-TCDD isomer (resolution with a valley of $\leq$ 25%) in all calibration standards (see D-20, DFLM01.0). In addition, the chromatographic peak separation between the 123478-HxCDD and 123678 HxCDD in the CC3 solution shall be resolved with a valley of $\leq$ 50 percent. Since isomer specificity for all 2378-substituted PCDDs/PCDFs cannot be achieved on the 60 m DB-5 column, the above chromatographic resolution criteria will be met using a SP-2331 GC column.

The % RSD of the five RRFs (CC1 - CC5) for the unlabled PCDDs/PCDFs and the internal standards must not exceed 15.0 percent. Formulas used to calculate % RSD and RRF are listed in Section 14.0.

Continuing Calibration Standard (Daily Standard)

The continuing calibration (CC3) standard is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance. The RRF of the compounds in the continuing calibration standard must be within 30% of the average RRF determined from the Initial Calibration. If this criteria cannot be met, analysis is suspended, the problem investigated, corrective actions implemented, and a new 5 point calibration is performed, if needed.

As discussed in the initial calibration section, ion abundance ratios, retention times GC column resolution and instrument sensitivity criteria must also be met. If this criteria cannot be met, analysis is suspended, the problem investigated and a new initial calibration, beginning with the analysis of the window defining mixture, is performed.

In order to demonstrate that the GC/MS/DS system has retained adequate sensitivity during the course of sample analyses, the lowest of the standards (CC1) must be analyzed at the end of each 12 hour period. The CC1 solution must meet the retention time, ion abundance ratio and instrument sensitivity criteria discussed in the initial calibration section.

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TABLE 8.1

Concentration Calibration Solutions

Analyte	CC1	CC2	CC3	CC4	CC5
2378-TCDD	0.1	0.25	0.5	1.0	2.0
2378-TCDF	0.1	0.25	0.5	1.0	2.0
12378-PeCDF	0.1	0.25	0.5	1.0	2.0
12378-PeCDD	0.1	0.25	0.5	1.0	2.0
23478-PeCDF*	---	---	0.5	---	---
123478-HxCDF*	---	---	1.25	---	---
123678-HxCDF	0.25	0.625	1.25	2.5	5.0
123478-HxCDD*	---	---	1.25	---	---
123678-HxCDD	0.25	0.625	1.25	2.5	5.0
123789-HxCDD*	---	---	1.25	---	---
234678-HxCDF*	---	---	1.25	---	---
123789-HxCDF*	---	---	1.25	---	---
1234789-HpCDF*	---	---	1.25	---	---
1234678-HpCDF	0.25	0.625	1.25	2.5	5.0
1234678-HpCDD	0.25	0.625	1.25	2.5	5.0
OCDD	0.5	1.25	2.5	5.0	10.0
OCDF	0.5	1.25	2.5	5.0	10.0
$^{13}C_{12}$-2378-TCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-2378-TCDF	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-123678-HxCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-1234678-HpCDF	1.0	1.0	1.0	1.0	1.0
$^{13}C_{12}$-OCDD	1.0	1.0	1.0	1.0	1.0
$^{13}C_{12}$-1234-TCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-123789-HxCDD	0.5	0.5	0.5	0.5	0.5
$^{37}Cl_4$-2378-TCDD	---	---	0.25	---	---

All concentrations are in ng/ul.

*Supplemental commercial standard. Do not perform % RSD calculations on these analytes.

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TABLE 8.2

Criteria for Isotopic Ratio Measurements
for PCDDs and PCDFs

Analyte	Selected Ions	Theoretical Ion Abundance	Control Limits
TCDD	320/322	0.77	0.65-0.89
PeCDD	356/358	1.55	1.24-1.86
HxCDD	390/392	1.24	1.05-1.43
HpCDD	424/426	1.04	0.88-1.20
OCDD	458/460	0.89	0.76-1.02
TCDF	304/306	0.77	0.65-0.89
PeCDF	340/342	1.55	1.24-1.86
HxCDF	374/376	1.24	1.05-1.43
HpCDF	408/410	1.04	0.88-1.20
OCDF	442/444	0.89	0.76-1.02
Internal Standards			
$^{13}C_{12}$-1234-TCDD	332/334	0.77	0.65-0.89
$^{13}C_{12}$-123678-HxCDD	402/404	1.24	1.05-1.43
$^{13}C_{12}$-OCDD	470/472	0.89	0.76-1.01
$^{13}C_{12}$-2378-TCDF	316/318	0.77	0.65-0.89
$^{13}C_{12}$-1234678-HpCDF	420/422	1.04	0.88-1.20
Recovery Standards			
$^{13}C_{12}$-1234-TCDD	332/334	0.77	0.65-0.89
$^{13}C_{12}$-123789-HxCDD	402/404	1.24	1.05-1.43

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9.0 ANALYTICAL PROCEDURES

The method used is that specified by the USEPA, as provided in the following reference:

- "Statement of Work for Analysis of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF)" USEPA Contract Laboratory Program, Multi-Media, Multi-Concentration, Document Number DFLM01.0, including Revision DFLM01.0 (September 1991).

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. The method selected will detect and quantify all seventeen 2378-PCDD/PCDF isomers. Samples that initially do not meet quality assurance objectives will be reanalyzed once to demonstrate matrix effects or interferences. Specific analytical procedures are as follows:

Identification

For a gas chromatographic peak to be unambiguously identified as a PCDD/PCDF, it must meet the following criteria:

- The absolute retention times must be within method acceptance limits.
- All of the specified ions listed in Table 9.1 for each PCDD/PCDF must be present in the ion current profile. The ion current response for the two quantitation ions and the M-[COCl]+ ions for the analytes must maximize simultaneously.
- The integrated ion current for each analyte ion listed in Table 9.1 must be at least 2.5 times background noise with no detector saturation.
- Ion abundance ratios must meet the criteria listed in Table 8.2.
- The identification of a GC peak as a PCDF cannot be made if a signal having a S/N greater than 2.5 is detected at the same retention time in the corresponding PCDPE channel (see Table 9.1).

301946

TABLE 9.1

Ions Specified for Selected Ion Monitoring
for PCDDs/PCDFs

Analyte	Quantitation Ions		$M-[COCl]^+$
TCDD	320	322	259
PeCDD	356	358	293
HxCDD	390	392	327
HpCDD	424	426	361
OCDD	458	460	395
TCDF	304	306	243
PeCDF	340	342	277
HxCDF	374	376	311
HpCDF	408	410	345
OCDF	442	444	379
Internal Standards			
$^{13}C_{12}$-2378-TCDD	332	334	---
$^{13}C_{12}$-123678-HxCDD	402	404	---
$^{13}C_{12}$-OCDD	470	472	---
$^{13}C_{12}$-2378-TCDF	316	318	---
$^{13}C_{12}$-1234678-HpCDF	420	422	---
Recovery Standard			
$^{13}C_{12}$-1234-TCDD	332	334	---
$^{13}C_{12}$-123789-HxCDD	402	404	---

TABLE 9.1, continued

Ions Specified for Selected Ion Monitoring
for PCDDs/PCDFs

Analyte	Quantitation Ions		M-[COCl]+
Cleanup Standard			
$^{37}C_{14}$-2378-TCDD	328	(1)	265
Polychlorinated diphenyl ethers			
HxCDPE	376	---	---
HpCDPE	410	---	---
OCDPE	446	---	---
NCDPE	480	---	---
DCDPE	514	---	---

(1) There is only one quantitation ion monitored for the cleanup standard.

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TABLE 9.2

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

		Quantitation Limits[1]			
PCDD/PCDF	CAS Number	Water (ng/L)	Soil (ug/Kg)	Fly Ash (ug/Kg)	Chemical[2] Waste (ug/Kg)
2378-TCDD	1746-01-6	10	1.0	1.0	10
2378-TCDF	51207-31-9	10	1.0	1.0	10
12378-PeCDF	57117-41-6	25	2.5	2.5	25
12378-PeCDD	40321-76-4	25	2.5	2.5	25
23478-PeCDF	57117-31-4	25	2.5	2.5	25
123478-HxCDF	70648-26-9	25	2.5	2.5	25
123678-HxCDF	57117-44-9	25	2.5	2.5	25
123478-HxCDD	39227-28-6	25	2.5	2.5	25
123678-HxCDD	57653-85-7	25	2.5	2.5	25
123789-HxCDD	19408-74-3	25	2.5	2.5	25
234678-HxCDF	60851-34-5	25	2.5	2.5	25
123789-HxCDF	72918-21-9	25	2.5	2.5	25
1234678-HpCDF	67562-39-4	25	2.5	2.5	25
1234678-HpCDD	35822-46-9	25	2.5	2.5	25
1234789-HpCDF	55673-89-7	25	2.5	2.5	25
OCDD	3268-87-9	50	5.0	5.0	50
OCDF	39001-02-0	50	5.0	5.0	50

Note 1: All CRQL values listed here are based on the wet weight of the sample.

Note 2: Chemical waste includes the matrices of oils, stillbottoms, oily sludge, wet fuel oil, oil-laced soil, and surface water heavily contaminated with these matrices.

Confirmation

Samples are first analyzed on a GC/MS fitted with a 60 M DB-5 chromatographic column. If any 2,3,7,8-substituted tetra-, penta-, or hexa- PCDDs/PCDFs are detected, the sample extracts will be reanalyzed using a 60 M SP-2331 chromatographic column since specificity for all of the isomers cannot be achieved on a 60 M DB-5 chromatographic column. If data resulting from SP-2331 column does not confirm results from the DB-5 column, only data calculated from the SP-2331 column will be reported for the 2,3,7,8-substituted isomers. The total congeners will be reported from the DB-5 column data. All data will be corrected following proper error correction protocol.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

All analytical data generated within Enseco laboratories are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review, as described below (also see Figure 10-1). Data will be recorded in logbooks and loose, preprinted sheets and are archived in client files. Access to analytical data entered into a laboratory information management system (LIMS) is controlled by the use of passwords, and assigned security levels. The security level and accessibility is assigned based on the tier of review at the time of data validation.

Level 1 Review

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs.

Data will be reduced by an analyst in one of the following ways:

- Manually computing results directly on the data sheet, chromatogram, or on calculation pages attached to the raw data;
- Inputting raw data for computer processing.

If data are manually reduced by an analyst, all steps in the computation will be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and RFs, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than).

Finally, each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample identification is correct;
- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met;
- Congener identification is correct; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, Out-of-Control forms [if required] are complete; holding times are documented, etc.).

The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

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Level 2 Review

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Transcription errors during LIMS entry are corrected;
- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; Out-of-Control forms [if required] are complete; holding times are documented, etc.).
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

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Level 3 Review

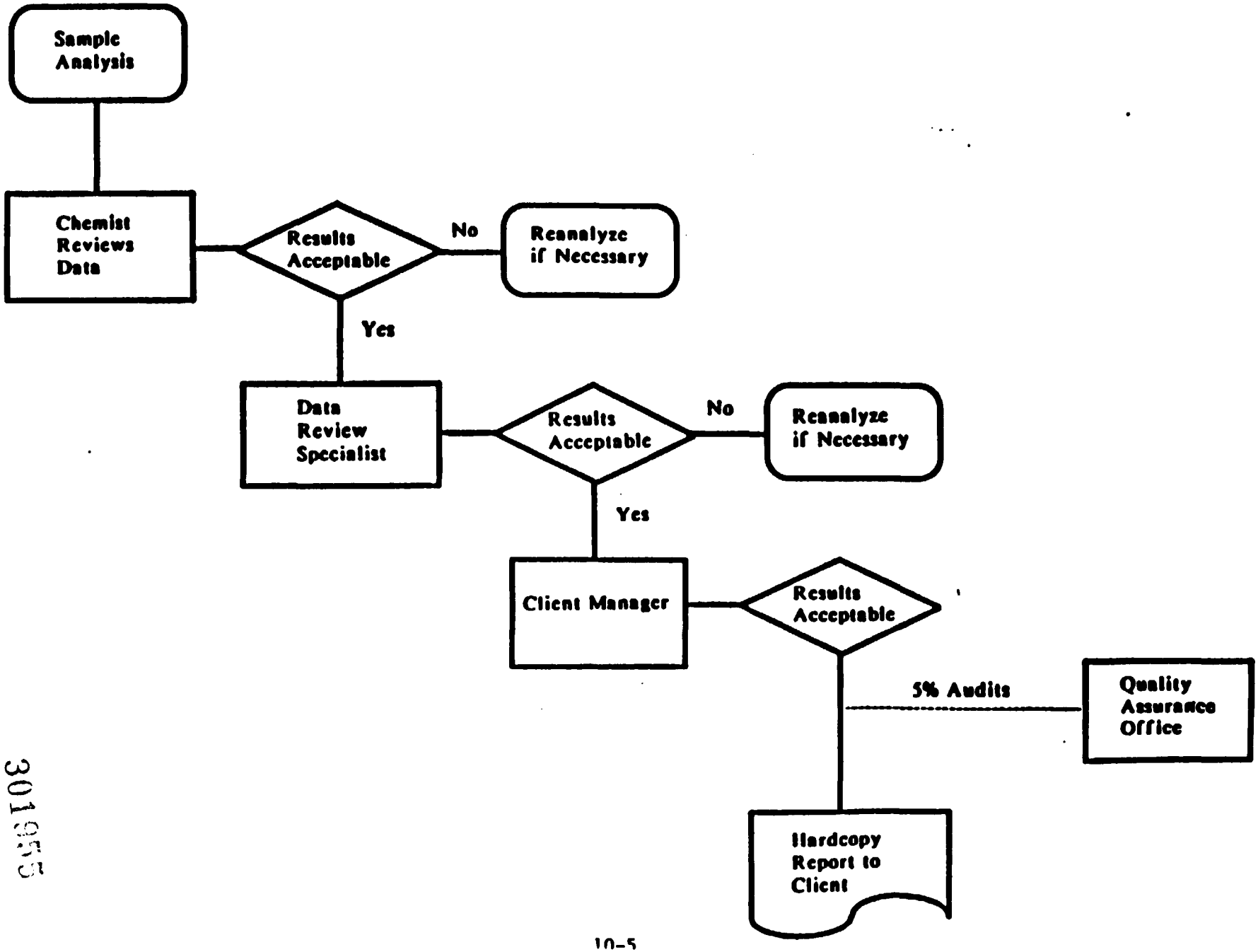
Before the report is released to the client, the Program Administrator who is responsible for interfacing directly with the client reviews the report to ensure that the data meet the overall objectives of the client, as understood by the Program Administrator. This review is labeled Level 3 review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgement of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. Errors that are found are documented and transmitted to the appropriate supervisors and managers. The cause of the errors is then addressed with additional training or clarification of procedures to that quality data is generated from the lab. The process continues until no errors are found or until the data package has been reviewed in its entirety.

FIGURE 10-1

Data Validation Scheme



301955

Detection Limits

An Estimated Detection Limit (EDL) will be calculated for each 2,3,7,8-substituted isomer that is not identified, regardless of whether or not non-2,3,7,8-substituted isomers in that homologue are present. The sample specific EDL is the estimate made by the laboratory of the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level and will be affected by sample size, dilutions, etc. EDL calculations are listed in Section 14.0

An Estimated maximum possible concentration (EMPC) is calculated for 2,3,7,8-substituted isomers that are characterized by a response with a signal-to-noise ratio of at least 2.5 for both the quantitation ions listed in Table 9.1, but that do not meet all of the identification criteria specified in the SOW DFLM01.0, Section D, pages D-42 to D-43. EMPC calculations are listed in Section 14.0.

Reporting

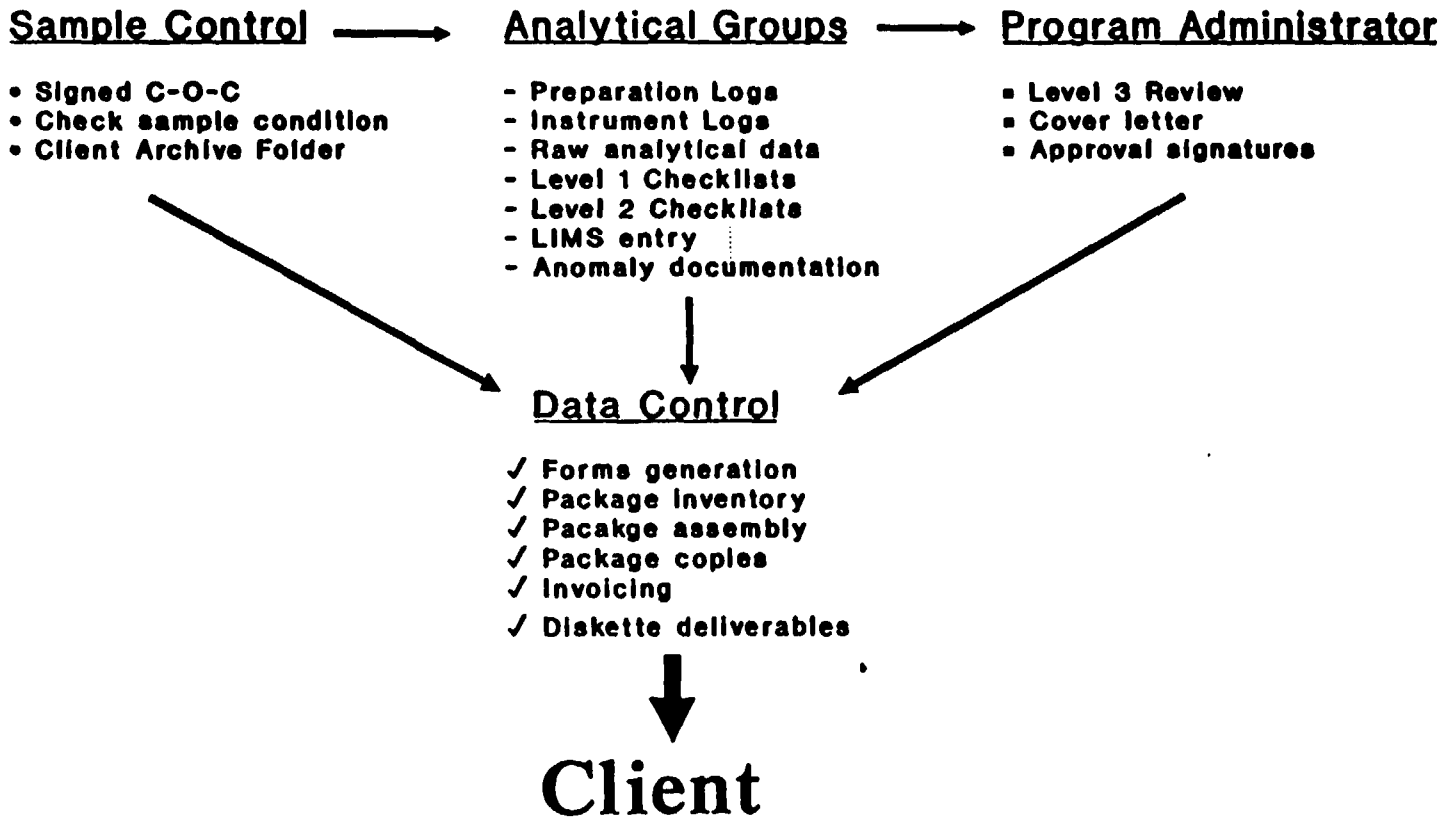
Results will be reported to the second decimal place in units of ng/L (ppt) for aqueous samples, and ug/kg (ppb) for solid samples.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. All dioxins/furans data will be reported in accordance with USEPA PCDD/PCDF SOW DFLM01.0 Section B, Forms 1DFA-DC-2. Examples of these forms and documentation requirements are delineated in Section B, SOW DFLM01.0

301956

Report Generation Flow
Figure 10.2



301957

Data Validation

Data validation involves reviewing sample results, quality control, and procedures for compliance with the data quality objectives outlined in this document or with client specified objectives. The scheme used for validation of data is shown in Figure 10.1.

11.0 INTERNAL QC CHECKS

Laboratory Performance QC samples will be added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration so that accuracy and precision can be determined and control charts can be prepared. Measures taken to control analytical data quality include use of specific acceptance criteria for instrument calibration, laboratory control samples, duplicate analyses, blank samples, and spiked samples.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks; and
- The generation of daily calibration data.

Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS consists of a standard, control matrix that is spiked with the internal standards and the surrogate representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference [RPD] between each analyte in the DCS pair) data are compared to control limits that have been established for each of the analytes contained in the DCS. Control limits for analytes spiked into the DCS are taken directly from the CLP program. Control limits for accuracy for each analyte are based on the SOW DFLM01.0 contract required limits. Control limits for precision for each analyte are based on the USEPA recommended RPD.

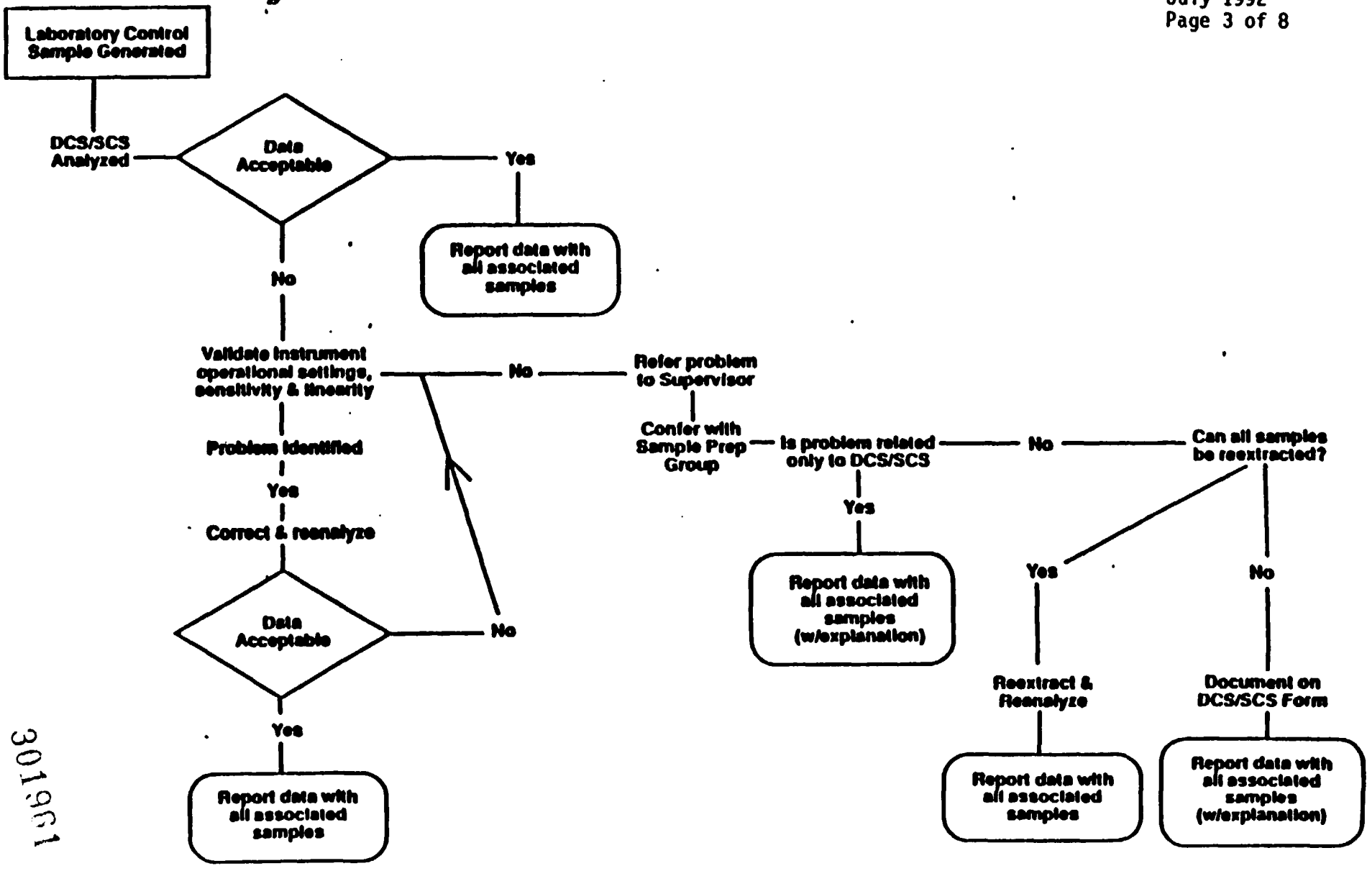
Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. The procedure used to evaluate data from control samples is given in Figure 11-1. The protocols include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

Method Blank

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

301960

Laboratory Performance QC Control Sample Evaluation



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As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

A method blank is prepared and analyzed with every analytical lot per matrix or for every 20 samples per matrix. Acceptable laboratory method blanks must not:

> contain any chemical interference or electronic noise at the m/z of the specified unlabled PCDD/PCDF ions that is greater than 5 percent of the signal of the appropriate internal standard quantitation ion, or
>
> exceed 2 percent of the signal of the appropriate internal standard if a peak meets the identification criteria as a PCDD/PCDF isomer.

If the method blank is deemed unacceptable, then the associated positive samples and any samples containing unidentified peaks must be reanalyzed.

Matrix-Specific QC

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, Enseco encourages its clients to include Matrix-Specific QC that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of 10 of the 2,3,7,8-substituted isomers have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

Matrix Spike/Matrix Spike Duplicate (MSD) are environmental samples that are divided into two separate aliquots, each of which is spiked with known concentrations of the analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery. Precision and accuracy objectives for matrix spikes are listed in Table 5.1.

Field Blank

Field blanks are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. Solvents such as trichloroethylene are the medium of choice for field blanks when sampling for dioxins and furans; however, if solvents are also parameters of interest, analyte-free water may be used. Due to the insolubility of dioxins and furans in water, the usefulness of a water blank is limited, therefore the use of water blanks is not expected for the Cranston project. One example of a field blank is an equipment blank. An equipment blank is blank water or solvent that is poured through the sample collection device to check the adequacy of the cleaning procedures for the sampling equipment. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks.

301963

Internal Standards

To quantitate and measure recovery of the analytes, labelled internal standards will be added to all pre-extracted samples, QC samples, blanks, and also to the calibration solutions. Calculated internal standard recoveries must be greater than 25% or less than 150% or reextraction and re-analysis of that sample is required. Associated analytes and internal standards are listed in Table 11.1.

Cleanup Standard

Prior to cleanup, all extracts are spiked with the $^{37}Cl_4$-2378-TCDD cleanup standard. The recovery of this standard is used to monitor the efficiency of the cleanup procedures. Calculated recoveries must be greater than 25% or less than 150% or reextraction and reanalysis are required.

Recovery Standard

To measure the % recovery of the labelled internal standards, the recovery standards $^{13}C_{12}$-1234-TCDD and $^{13}C_{12}$-123678-HxCDD will be added to all blanks, samples, and quality control sample extracts just prior to GC/MS analysis. If the absolute retention time of either recovery standard in a sample extract shifts by more than 10 seconds from the retention time of that standard in the continuing calibration standard, then the sample must be reextracted and reanalyzed to verify the presence of a matrix effect or the effectiveness of laboratory corrective actions.

TABLE 11.1

Relationship of Internal Standards to Analytes, and Relationship of Recovery Standards to Analytes, Internal Standards and Cleanup Standard

INTERNAL STANDARDS VS. ANALYTES

$^{13}C_{12}$-TCDD	$^{13}C_{12}$-HxCDD	$^{13}C_{12}$-OCDD	$^{13}C_{12}$-TCDF	$^{13}C_{12}$-HpCDF
TCDD	HxCDD	OCDD	TCDF	HxCDF
PeCDD	HpCDD	OCDF	PeCDF	HpCDF

RECOVERY STANDARDS VS. ANALYTES, INTERNAL STANDARDS AND CLEANUP STANDARD

$^{13}C_{-12}$-1234-TCDD	$^{13}C_{12}$-123789-HxCDD
TCDD	HxCDD
TCDF	HxCDF
PeCDD	HpCDD
PeCDF	HpCDF
	OCDD
	OCDF
$^{13}C_{12}$-2378-TCDD	$^{13}C_{12}$-123678-HxCDD
$^{13}C_{12}$-2378-TCDF	$^{13}C_{12}$-1234678-HpCDF
$^{37}Cl_4$-2378-TCDD	$^{13}C_{12}$-OCDD

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Additional Quality Control

In addition, the following quality control samples will be analyzed:

- Method blanks
- Duplicate control sample (DCS)

Definitions, frequency, and corrective action are discussed in Section 11.0, Internal QC Checks and Frequency. Acceptability criteria are listed in Table 5.1, Accuracy and Precision Objectives.

Calculations

A summary of calculations used to determine PCDF/PCDD recoveries and concentrations are listed in Section 14.0, Specific Procedures Used to Assess Data Precision, Accuracy, and Completeness.

12.0 PERFORMANCE AND SYSTEMS AUDIT

Enseco laboratories participate in a variety of federal and state certification programs, (including the USEPA CLP), that subject each of the laboratories to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A compliance audit is a periodic, random, review of in-progress laboratory procedures to ve rify that the staff and the procedures in place are conducted in accordance with contract or program requirements. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the USEPA, US Army, US Navy, and several states. The most current list of Enseco certification is available upon request.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

A summary of the types and frequency of systems and performance audits is summarized in Table 12.1.

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TABLE 12.1

SUMMARY OF PERFORMANCE AND SYSTEM AUDITS

PROGRAM	NAME	TYPE (P OR S)	FREQUENCY
Drinking water	WS	Performance	Semi-Annual
Waste water	WP	Performance	Semi-Annual
EPA-CLP	QB	Performance	Quarterly*
U.S. Navy	PE	Performance	Every 18 mos.*
U.S. Army	PE	Performance	Every 18 mos.*
Calif. Dept. Food and Agriculture	PE	Performance	Quarterly
NPDES	DMR-QA	Performance	Annually
EPA-CLP		System	Annually*
Calif. ELAP		System	Biannually
U.S. Navy		System	Annually
Utah		System	Annually
U.S. Army		System	Every 18 mos.*
Divisional QA		System	Quarterly (approx.)
ENSECO Corporate		System	Annually
Enseco-CAL		Compliance	Within 2 months of Scheduled Sample Delivery at a Minimum

Clients may request performance of specific performance and systems audits as a requirement of contract award.

* Contract award required.

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13.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. In addition, multiple instruments are maintained for each analysis to minimize the impact on the project.

Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and schedules.

All aspects of routine and non-routine instrument maintenance are recorded in logbooks, and a log book is dedicated to each instrument.

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14.0 SPECIFIC PROCEDURES USED TO ASSESS DATA PRECISION ACCURACY, AND COMPLETENESS

Calculations for accuracy (% R) and precision (RPD, RSD) were discussed and defined in Section 5.0 Data Quality Objectives and in Sections B, D, and E of the SOW DFLM01.0 PCDDs/PCDFs. Formulas used to calculate relative response factors (RRF), concentration, and detection limits are as follows:

Definitions

A_n^1 and A_n^2	=	integrated areas of the two quantitation ions of the isomer of interest (Table 9.1).
A_{is}^1 and A_{is}^2	=	integrated areas of the two quantitation ions of the appropriate internal standard (Table 9.1).
A_{rs}^1 and A_{rs}^2	=	integrated areas of the two quantitation ions of the appropriate recovery standard (Table 9.1).
Q_n	=	quantity of unlabled PCDD/PCDF analyte injected (ng).
Q_{is}	=	quantity of appropriate internal standard injected (ng).
Q_{rs}	=	quantity of appropriate recovery standard injected (ng).
D	=	dilution factor
W	=	weight of sample extracted, in grams
V	=	volume of sample extracted, in liters
Cn	=	concentration of native PCDD/PCDF found in the sample
R_{is}	=	percent recovery for each internal standard and the cleanup standard in the sample extract
RRF_n		is the response factor of the quantitation ion of the isomer of interest relative to that of the appropriate internal standard
RRF_{is}		is the response factor of the internal standard relative to that of the appropriate recovery standards

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Relative Response Factors (RRF)

$$RRF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

$$RRF_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

Relative Standard Deviation (%RSD)

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

Detection Limits

Estimated Maximum Possible Concentration (EMPC)

ALL MATRICES OTHER THAN WATER

$$EMPC\ (ug/kg) = \frac{(A_n^1 + A_n^2) \times Q_{is} \times D}{(A_{is}^1 + A_{is}^2) \times RRF_n \times W}$$

WATER

$$EMPC\ (ng/L) = \frac{(A_n^1 + A_n^2) \times Q_{is} \times D}{(A_{is}^1 + A_{is}^2) \times RRF_n \times V}$$

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Sample Specific Estimated Detection Limit (EDL)

ALL MATRICES OTHER THAN WATER

$$EDL\ (ug/kg) = \frac{2.5 \times Q_{is} \times (H_x^1 + H_x^2) \times D}{W \times (H_{is}^1 + H_{is}^2) \times RRF_n}$$

WATER

$$EDL\ (ng/L) = \frac{2.5 \times Q_{is} \times (H_x^1 + H_x^2) \times D}{V \times (H_{is}^1 + H_{is}^2) \times RRF_n}$$

Where:

H_x^1 and H_x^2 = Peak heights of the noise for both of the quantitation ions of the 2,3,7,8-substituted isomer of interest.

H_{is}^1 and H_{is}^2 = Peak heights of both the quantitation ions of the appropriate internal standards.

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Concentration

ALL MATRICES OTHER THAN WATER

$$C_n \text{ (ug/kg)} = \frac{Q_{is} \times (A_n^1 + A_n^2)}{W \times (A_{is}^1 + A_{is}^2) \times RRF_n}$$

WATER

$$C_n \text{ (ng/L)} = \frac{Q_{is} \times (A_n^1 + A_n^2)}{V \times (A_{is}^1 + A_{is}^2) \times RRF_n}$$

If the area of any internal standard in a diluted sample is less than 10 percent of the area of that internal standard in the continuing calibration standard, then the unlabled PCDD/PCDF concentrations in the sample shall be estimated using the recovery standard, using the formulae that follow. The purpose is to ensure that there is an adequate MS response for quantitation in a diluted sample. While use of a smaller aliquot of the sample might require smaller dilutions and therefore yield a larger area for the internal standard in the diluted extract, this practice leads to other concerns about the homogeneity of the sample and the representativeness of the aliquot taken for extraction.

ALL MATRICES OTHER THAN WATER

$$C_n \text{ (ug/kg)} = \frac{Q_{rs} \times (A_n^1 + A_n^2) \times D}{W \times (A_{rs}^1 + A_{rs}^2) \times RRF_{rs}}$$

WATER

$$C_n \text{ (ng/L)} = \frac{Q_{rs} \times (A_n^1 + A_n^2) \times D}{V \times (A_{rs}^1 + A_{rs}^2) \times RRF_{rs}}$$

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Recovery

Calculate the percent recovery for each internal standard and the cleanup standard in the sample extract, R_{is}, using the formulae:

$$R_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times RRF_{is} \times Q_{is}} \times 100\%$$

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15.0 CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

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

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, 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 supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department. Corrective action documentation is routinely reviewed by the QA department.

Control limits and required corrective actions for anomalies are delineated in Sections B and E of the SOW document number DFLM01.0 PCDDs/PCDFs.

The Enseco-CAL Program Administrator will notify the TPO, EMSL-LV and NEIC of any issues affecting the QAPP such as EPA modifications to the SOW or changes in key laboratory personnel. Enseco-CAL will revise this document, and redistribute the approved, modified plan. Revisions will be summarized in Section 18.0.

16.0 QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Regional QA Directors submit extensive monthly reports to the Corporate QA Director and the Regional General Manager. These reports include:

- The results of internal systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the 5% QA data audits conducted.

The Corporate Director of QA submits regularly reports on the status of the QA program to the President and Regional General Manager. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

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17.0 KEY PERSONNEL

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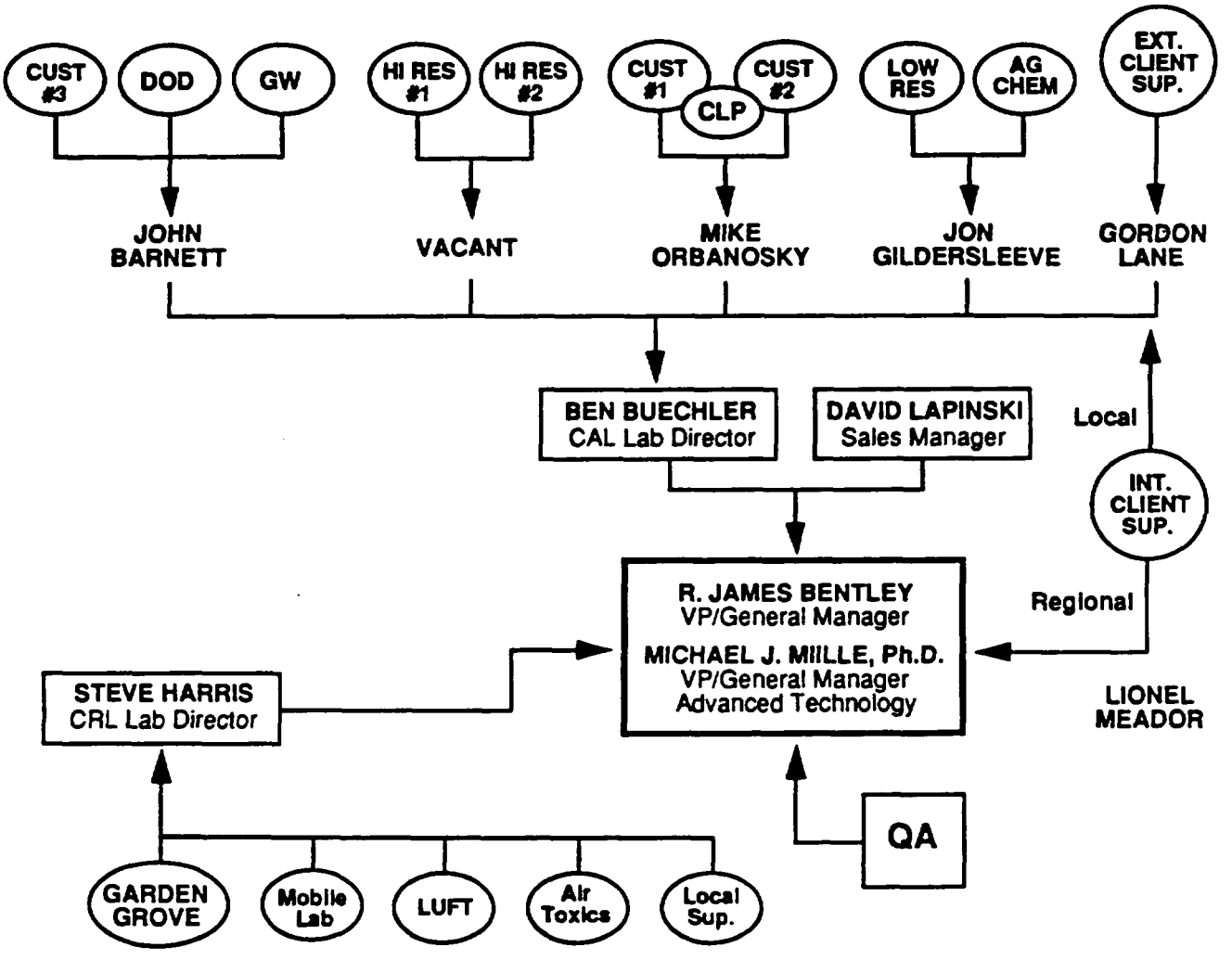
Figure 17-1

Enseco Western Region

Organization Chart



Enseco
A Corning Company

Revised 6/92

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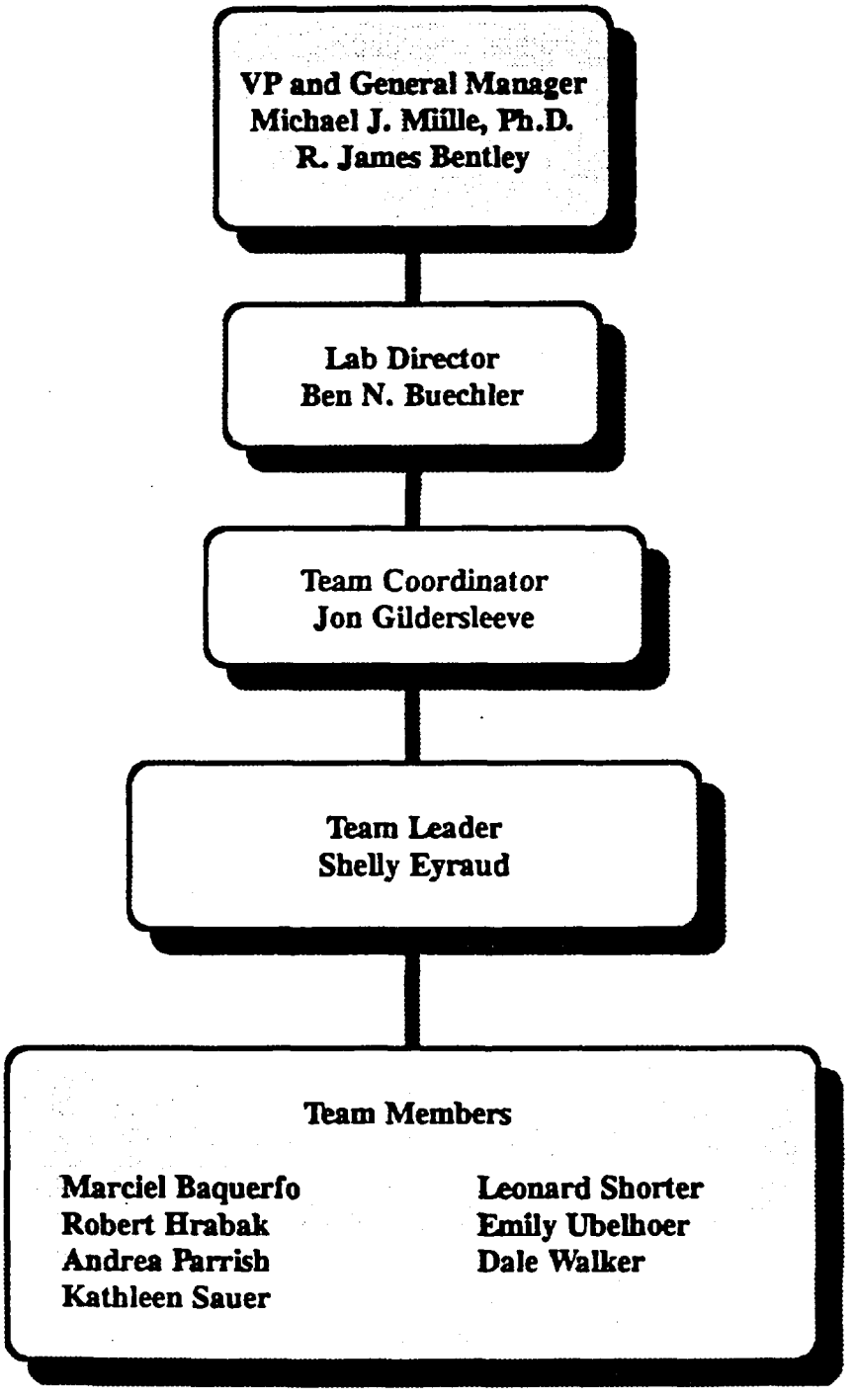
Figure 17-2

Low Resolution Team



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18.0 Changes to the Quality Assurance Project Plan

EXHIBIT A

SUMMARY OF REQUIREMENTS

301982

DFLM01.0

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SECTION I

GENERAL REQUIREMENTS

A. Purpose of the Statement of Work

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs) are among the pollutants that are of concern to EPA at such sites. PCDDs/PCDFs are believed to be among the most toxic organic compounds ever released into the environment.

With the advent of the Superfund program in 1980, EPA required the analyses of many more environmental samples than could possibly be handled through its own laboratories. Therefore, EPA elected to procure analytical services through commercial laboratories and established the Contract Laboratory Program (CLP) as a means of obtaining standardized analyses on a long-term firm, fixed-price basis. This Statement of Work (SOW) provides a technical and contractual framework for laboratories to apply EPA analytical methods to the analysis of PCDDs/PCDFs in environmental samples. The SOW provides not only the analytical methods to be applied, but also the specific technical and contractual requirements by which EPA will evaluate the data.

B. General Requirements

This SOW provides an analytical method for the isolation, detection and quantitative measurement of PCDDs and PCDFs in water, soil, fly ash, and chemical waste samples such as oil, sludge, and stillbottoms. There are 210 possible PCDD/PCDF isomers, and the methods were developed for the analysis of the 17 PCDDs/PCDFs that bear chlorine atoms in the 2,3,7 and 8 positions of their respective structures. These 17 compounds, termed the "2,3,7,8-substituted PCDDs/PCDFs," are those PCDDs/PCDFs that, based on structure activity relationships, are believed to pose the greatest risks to human health and the environment. The SOW also requires determination of the total concentrations of all PCDDs or PCDFs in a given level of chlorination (i.e., Total TCDD, Total PecDD, etc.), although complete chromatographic separation of all 210 PCDDs/PCDFs is not possible under the instrumental conditions described in the method.

The SOW requires the calculation of the 2378-TCDD toxicity equivalence using the procedures described in the "Update of Toxicity Equivalency Factors (TEFs) for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs)," March 1989, (EPA 625/3-89/016). To aid in the assessment

of risks associated with exposure to these compounds, a factor is assigned to each of the 17 2,3,7,8-substituted PCDDs and PCDFs that relates the toxicity of that isomer to a concentration of the most toxic isomer, 2378-TCDD. The concentrations of any isomers that are detected in an environmental sample can then be adjusted by the toxicity equivalency factor (TEF) and summed, yielding a concentration of 2378-TCDD with an equivalent toxicity.

Because isomer specificity for all 17 2378-substituted PCDDs/PCDFs may not be achieved using a single gas chromatographic column, the SOW requires analysis of sample extracts on a second column when the TEF-adjusted concentration exceeds a specified level. This level varies by sample matrix.

The sample preparation procedures in the SOW use matrix-specific extraction techniques and a single set of cleanup techniques. The sensitivity of this method is dependent upon the level of interferents within a given sample. Interferents co-extracted from the sample may vary considerably from source to source, depending on the origin of the sample and the matrix type. PCDDs and PCDFs are often associated with other chlorinated compounds such as PCBs and polychlorinated diphenyl ethers which may occur at concentrations several orders of magnitude higher than that of the analytes of interest and may cause interference problems.

The samples to be analyzed by the Contractor are from known or suspected hazardous waste sites and may contain hazardous organic and/or inorganic materials at high concentration levels. The Contractor should be aware of the hazards associated with the handling and analysis of these samples. The Contractor is responsible for taking all necessary measures to ensure the health and safety of its employees.

The Contractor must be aware of the importance of maintaining the integrity of the data generated under the contract, as data may be used to make decisions regarding public health and environmental welfare. In addition, the data may be used in litigation against potentially responsible parties in the enforcement of Superfund legislation.

C. Applications and Limitations of the Statement of Work

This SOW is designed as part of the documentation for a contract between EPA and a commercial laboratory performing analyses in support of EPA Superfund programs. The resulting data may be used by EPA for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assigning administrative priority to such sites based on the risk of exposure, determining appropriate cleanup actions, and determining when remedial actions are complete.

The methods described in this SOW are designed for the analysis of specific analytes in specific environmental matrices and over a limited concentration range. However, this SOW is not suitable for all analytical situations and should not be applied to matrices, analytes,

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SECTION II

SPECIFIC REQUIREMENTS

A. Sample shipments to the Contractor's facility will be scheduled and coordinated by the EPA CLP Sample Management Office (SMO) acting on behalf of the Administrative Project Officer. The Contractor shall communicate with SMO personnel by telephone, as necessary throughout the process of sample scheduling, shipment, analysis and data reporting, to ensure that samples are properly processed.

If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation/paperwork (e.g., Traffic Reports not with shipment, sample and Traffic Report numbers do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

B. Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period and includes one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

A Case consists of one or more Sample Delivery Group(s). A Sample Delivery Group (SDG) is defined by the following, whichever is most frequent:

- Each Case of field samples received, OR
- Each 20 field samples within a Case, OR
- Each 14 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

Samples may be assigned to SDGs by matrix (e.g., all soil samples in one SDG, all water samples a second SDG, and all fly ash samples in a third SDG), at the discretion of the laboratory. Such assignment must be made at the time the samples are received and may <u>not</u> be made retroactively.

All data for all samples in a SDG are due concurrently to all data recipients as stipulated in the Delivery Schedule in Exhibit B, Section I. Data for all samples in a SDG must be submitted together (in one package) in the order specified in Exhibit B. The SDG number is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number

shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms.

The SDG receipt date is the day the last sample in the SDG is received. Data for all samples in the SDG are due as stipulated in the Delivery Schedule in Exhibit B.

The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

C. Each sample received by the Contractor will be labeled with an EPA sample number and will be accompanied by a Traffic Report bearing the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the Traffic Report, recording the date of sample receipt and sample condition for each sample container.

The Contractor shall submit signed copies of Traffic Reports for all samples in a SDG to SMO within three calendar days following receipt of the last sample in the SDG. Traffic Reports shall be submitted in SDG sets (i.e., all Traffic Reports for a SDG shall be clipped together) with a SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

D. The Contractor shall use EPA Case numbers (including SDG numbers) and EPA sample numbers to identify samples received under this contract both verbally and in reports/correspondence.

E. Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station or other carrier-service within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays.

F. The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

G. The Contractor shall prepare, extract, cleanup extracts, and analyze samples according to the analytical procedures outlined in Exhibit D. The Contractor shall also adhere to the QA/QC requirements specified in Exhibit D, including the analyses of calibration standards, blanks, spiked samples, duplicate analyses, etc., as specified in the exhibit.

H. EPA has provided the Contractor with forms for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in the Contract Performance/Delivery Schedule.

Use of formats other than those designated by EPA will be deemed as noncompliance. Such data are unacceptable. Resubmission in the specified format will be required at no additional cost to the Government.

I. The Contractor shall have sufficient gas chromatograph/mass spectrometer/data system (GC/MS/DS) capability to meet all the terms and conditions of the EPA contract. The Contractor shall maintain, at a minimum, all analytical equipment allocated for this contract at the time of contract award. (See Section III for instrumentation requirements.)

J. Certain samples may require sample reruns (reextraction and/or reanalysis) due to either problems with the sample matrix or Contractor insufficiencies. Sample reruns may be considered either as billable or nonbillable as defined in Exhibit D. For the purposes of this contract, the term "automatic rerun" shall signify only billable rerun analyses.

K. EPA may provide standards for use in analyses performed under the contract, subject to availability. However, the SOW identifies specific solutions that must be purchased from commercial sources, and will <u>not</u> be provided by EPA. When provided, EPA-supplied materials are intended for use only on EPA samples, and the Contractor may be asked to demonstrate during EPA on-site evaluations that separate standards are maintained for non-EPA work. The Contractor will be instructed how and where to request EPA standards at time of contract award. The Contractor is responsible for ensuring that all required standards are available at the Contractor's facility before accepting any samples from EPA.

L. The Contractor shall respond within seven days to requests from data recipients for additional information or explanations that result from the Government's inspection activities.

M. The Contractor shall preserve all sample extracts after analysis in bottles/vials with Teflon-lined septa and shall maintain stored extracts in the dark at room temperature. The Contractor is required to retain the sample extracts for 365 days after data submission. During that time, the Contractor shall submit the extracts within seven days after request, as specified in the Contract Performance/Delivery Schedule.

N. The Contractor shall adhere to chain-of-custody procedures described in Exhibit F. Documentation, as described therein, shall show that all procedures are being strictly followed. This documentation shall be reported as the Complete SDG File (see Exhibit B).

SECTION III

DETAILED TECHNICAL AND MANAGEMENT REQUIREMENTS

The Contractor shall have the following technical and management capabilities. For those technical functions that require a minimum educational degree and experience, an advanced degree in chemistry or any scientific/engineering discipline (e.g., Master's or Doctorate) does not substitute for the minimum experience requirements.

The Contractor shall notify in writing the Technical Project Officer and the Administrative Project Officer of any changes affecting key personnel listed in this section within 14 days of the change. The Contractor shall provide a detailed resume to the Technical Project Officer, Administrative Project Officer, and EMSL-LV for the replacement personnel within 14 days of the Contractor's assignment of the personnel. The resume shall include position description of titles, education (pertinent to this contract), number of years of experience (pertinent to this contract), month and year hired, previous experience and publications.

A. TECHNICAL CAPABILITY

1. Technical Functions

a. GC/MS Laboratory Supervisor

(1) Responsible for all technical efforts of the GC/MS laboratory to meet all terms and conditions of the EPA contract.

(2) Qualifications:

(a) Education:

Minimum of a Bachelor's degree in chemistry or any scientific/engineering discipline.

(b) Experience:

Minimum of three years of laboratory experience with dioxin and furan analyses, including at least one year of supervisory experience.

b. Sample Preparation Laboratory Supervisor

(1) Responsible for all technical efforts of sample preparations to meet all terms and conditions of the EPA contract.

(2) Qualifications:

(a) Education:

Minimum of a Bachelor's degree in chemistry or any scientific/engineering discipline.

(b) Experience:

Minimum of three years of laboratory experience, including at least one year of supervisory experience.

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c. Quality Assurance Officer

(1) Responsible for overseeing the QA aspects of data and reporting directly to upper management to meet all terms and conditions of the EPA contract.

(2) Qualifications:

(a) Education:

Minimum of a Bachelor's degree in chemistry or any scientific/engineering discipline.

(b) Experience:

Minimum of three years of laboratory experience, including at least one year of applied experience with QA principles and practices in an analytical laboratory.

d. GC/MS Operator Qualifications

One year of experience in operating and maintaining GC/MS/DS used for selected ion monitoring (SIM) with a Bachelor's degree in chemistry or any scientific/engineering discipline, <u>or</u> in lieu of the Bachelor's degree, three years of experience in operating and maintaining the GC/MS and interpreting GC/MS SIM data.

e. Extract Cleanup Expert Qualifications

One year of experience in extract cleanup with a Bachelor's degree in chemistry or any scientific/engineering discipline, <u>or</u> in lieu of the Bachelor's degree, three years of experience in sample extraction and cleanup.

f. Extraction/Concentration Expert Qualifications

(1) Education:

Minimum of high school diploma and a college-level course in general chemistry.

(2) Experience:

Minimum of one year of experience in extraction/concentration.

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g. Technical Staff Redundancy

The bidder shall have a minimum of one chemist available at any one time as a back-up technical person with the following qualifications to ensure continuous operations to accomplish the required work as specified by the EPA contract.

(1) Education:

Minimum of a Bachelor's degree in chemistry or any scientific/engineering discipline.

(2) Experience: Minimum of one year in each of the following areas -

- o GC/MS operation and maintenance using selected ion monitoring.
- o Dioxin/furan analysis.
- o Sample extraction and cleanup.

2. Facilities

The adequacy of the facilities and equipment is of equal importance as the technical staff to accomplish the required work as specified by the EPA contract.

a. Sample Receipt Area

Adequate, contamination-free, well-ventilated work space provided with chemical resistant bench top for receipt and safe handling of EPA samples.

b. Storage Area

Sufficient space to maintain unused EPA sample volume for 60 days after data submission and sample extracts for 365 days after data submission. Samples must be stored in an atmosphere demonstrated to be free from all potential contaminants.

c. Sample Preparation Area

Adequate, contamination-free, well-ventilated work space provided with:

(1) Benches with chemical resistant tops, exhaust hoods.

NOTE: Standards must be prepared in a glove box or isolated area.

(2) Source of distilled or demineralized organic-free water.

(3) Analytical balance(s) located away from draft and rapid change in temperature.

3. Instrumentation

At a minimum, the Contractor shall have the following instruments operative and committed for the full duration of the contract.

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a. Primary Instrument Requirements

(1) GC/MS equipped with GC to MS interface capable of extending a 60 meter by 0.32 mm ID, bonded DB-5 (or equivalent), fused silica capillary column into the MS ion source.

(2) GC/MS computer interfaced by hardware to the MS and capable of monitoring at least 18 selected ions for the duration of the chromatographic analysis.

(3) GC/MS computer equipped with mass storage device for saving all data from GC/MS analyses.

(4) GC/MS computer software capable of searching GC/MS analyses for specific ions and plotting the intensity of the ions with respect to time or scan run.

(5) Magnetic tape storage device capable of recording data for long-term, off-line storage.

b. Secondary Instrument Requirements

The Contractor shall have one back-up instrument, identical to the requirements above, in place and operational at any time. This instrument must be included in the bidder's inventory of equipment. In addition, the Contractor shall have an in-house stock of instrument parts and circuit boards to ensure continuous operation to meet contract-specified holding and turnaround times.

4. Data Handling and Packaging

The Contractor shall have reasonable capacity to submit reports and data packages as specified in Exhibit B. To complete this task, the Contractor shall be required to:

a. Provide space, tables and copy machines to meet the contract requirements.

b. Designate personnel.

B. LABORATORY MANAGEMENT CAPABILITY

The Contractor must have an organization with well-defined responsibilities for each individual in the management system to ensure sufficient resources for EPA contract(s) and to maintain a successful operation. To establish this capability, the Contractor shall designate personnel to carry out the following responsibilities for the EPA contract. Functions include, but are not limited to, the following:

1. Technical Staff

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Responsible for all technical efforts for the EPA contract.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

SECTION I

CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

The following table reiterates the contract reporting and deliverables requirements specified in the Contract Schedule and specifies the distribution that is required for each deliverable. NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The Administrative Project Officer (APO) will notify the Contractor in writing of such changes when they occur.

Item	No. Copies	Delivery Schedule	Distribution (1)	(2)	(3)	(4)
A. Updated SOPs	3	60 days after contract award.		X	X	X
*B. Sample Traffic Reports (original)	1	3 days after receipt of last sample in Sample Delivery Group (SDG).**	X			
C. Sample Data Summary Package	1	45 days after receipt of last sample in SDG.	X			
***D. Sample Data Package	2	45 days after receipt of last sample in SDG.	X		X	
****E. Complete SDG File	1	45 days after receipt of last sample in SDG.		X		
*****F. Quality Assurance Plan	3	60 days after contract award and as required in Exhibit E.			X	X
G. GC/MS Tapes	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO and/or EMSL-LV.	As Directed			

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Item	No. Copies	Delivery Schedule	Distribution (1)	(2)	(3)
H. Extracts	Lot	Retain for 365 days after data submission, or submit within 7 days after receipt of written request by APO or SMO.	As Directed		

Distribution:
(1) Sample Management Office (SMO)
(2) Region-Client (Technical Project Officer (TPO))
(3) Environmental Monitoring Systems Laboratory (EMSL-LV)
(4) National Enforcement Investigations Center (NEIC)

* Copy also required in the Sample Data Summary Package.

** Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 14 days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that all samples have been delivered. (See Exhibit A for further description.)

*** Concurrent delivery required. Delivery shall be made such that all designated recipients receive the item on the same calendar day.

**** Complete SDG File will contain the original sample data package plus all of the original documents described under Section II, Part E.

***** See Exhibit E for a more detailed description.

NOTE: As specified in the Contract Schedule (G.2 Government Furnished Supplies and Materials), unless otherwise instructed by SMO, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of analytical data.

Distribution Addresses:

(1) USEPA Contract Laboratory Program
Sample Management Office
P.O. Box 818
Alexandria, VA 22314

For overnight delivery service, use street address:
300 North Lee Street
Alexandria, VA 22314

(2) USEPA Regions:

SMO, acting on behalf of the APO, will provide the Contractor with the list of addressees for the 10 EPA Regions. SMO will provide the

Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

(3) USEPA Environmental Monitoring Systems Laboratory
P.O. Box 93478
Las Vegas, NV 89193-3478
ATTN: Data Audit Staff

For overnight delivery service, use street address:
944 E. Harmon, Executive Center
Las Vegas, NV 89109
ATTN: Data Audit Staff

(4) USEPA National Enforcement Investigations Center (NEIC)
Attn: CLP Audit Program
Denver Federal Center Building 53
P. O. Box 25227
Denver, CO 80225

SECTION II

REPORT DESCRIPTIONS AND ORDER OF DATA DELIVERABLES

The Contractor shall provide reports and other deliverables as specified in the Contract Schedule (Reporting Requirements and Deliverables, F.2). The required content and form of each deliverable are described in this exhibit.

All reports and documentation MUST BE:

- Legible.
- Clearly labeled and completed in accordance with instructions in this exhibit.
- Arranged in the order specified in this section.
- Paginated consecutively in ascending order starting from the SDG Narrative.

If submitted documentation does not conform to the above criteria, the Contractor shall be required to resubmit such documentation with deficiencies corrected, at no additional cost to the Government.

Whenever the Contractor is required to submit or resubmit data as a result of an onsite laboratory evaluation or through an APO/TPO action, the data must be clearly marked as ADDITIONAL DATA and must be sent to all three contractual data recipients (SMO, EMSL-LV and the Region). A cover letter shall be included which describes what data are being delivered, to which EPA Case(s) the data pertain, and <u>who requested the data</u>.

Whenever the Contractor is required to submit or resubmit data as a result of contract compliance screening by SMO, the data must be sent to all three contractual data recipients (SMO, EMSL-LV and the Region). In all three instances the data must be accompanied by a color-coded Cover Sheet (Laboratory Response To Results of Contract Compliance Screening) provided by SMO.

Section III of this exhibit contains forms instructions to assist the Contractor in accurately providing EPA with all required data. Section IV contains copies of the required data reporting forms in EPA-specified formats.

Descriptions of the requirements for each deliverable item cited in Reporting Requirements and Deliverables (Contract Schedule, Section F) are specified in this section. Items submitted concurrently MUST BE arranged in the order listed. Additionally, the components of each item MUST BE arranged in the order presented in this section when the item is submitted. Examples of specific data deliverables not included herein may be obtained by submitting a written request to the APO, stating the information requested and signed by the Laboratory Manager.

A. Quality Assurance Plan and Standard Operating Procedures

See Exhibits E and F for requirements.

B. Sample Traffic Reports

The original Sample Traffic Report (TR) page marked "Lab Copy for Return to SMO" shall be delivered with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG. TRs shall be submitted in SDG sets (i.e., TRs for all samples in a SDG shall be clipped together) with a SDG Cover Sheet attached.

The SDG Cover Sheet shall contain the following items:

- Laboratory name.
- Contract number.
- Sample analysis price - full sample price from the EPA contract.
- Case number.
- List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received and their dates of receipt (LRDs).

A. Updated Standard Operating Procedures

The Contractor shall submit updated copies of all required standard operating procedures (SOPs) that were submitted with the preaward performance evaluation sample results. The updated SOPs must address any and all issues of laboratory performance and operation identified through the review of the performance evaluation sample data and the evaluation of bidder-supplied documentation.

The Contractor must supply SOPs for:

1. Evidentiary SOPs.

2. Sample Receipt and Storage.

3. Sample Preparation.

4. Glassware Cleaning.

5. Calibration (balances, GPCs).

6. Analytical Procedures (for each analytical system).

7. Maintenance Activities (for each analytical system).

8. Analytical Standards.

9. Data Reduction Procedures.

10. Documentation Policy/Procedures.

11. Data Validation/Self-Inspection Procedures.

12. Data Management and Handling.

B. Sample Traffic Reports

The original Sample Traffic Report (TR) page marked "Lab Copy for Return to SMO" shall be delivered with laboratory receipt information and signed in original Contractor signature, for each sample in the SDG. TRs shall be submitted in SDG sets (i.e., TRs for all samples in a SDG shall be clipped together) with a SDG Cover Sheet attached.

The SDG Cover Sheet shall contain the following items:

- Laboratory name.
- Contract number.
- Sample analysis price - full sample price from the EPA contract.
- Case number.
- List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received and their dates of receipt (LRDs).

The Contractor shall retain a copy of the Sample Data Package for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

1. SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Case number; sample numbers in the SDG, differentiating between initial analyses and reanalyses; SDG number; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.

Whenever data from sample reanalyses are submitted, the Contractor shall state in the SDG Narrative for <u>each</u> reanalysis, whether it considers the reanalysis to be billable, and if so, why.

The Contractor must also include any problems encountered, both technical and administrative, the corrective actions taken and the resolutions, and an explanation for all flagged edits (i.e., manual edits) on quantitation lists.

NOTE: If a column is used that has different first and last eluting isomers than the DB-5 column, the Contractor shall fully document, in the SDG Narrative, the order of elution of the isomers and identify the first and last eluting isomers for that particular column for the window defining mix and CC3 solution.

The SDG Narrative shall contain the following statement, <u>verbatim</u>: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or his designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature. Additionally, the SDG Narrative itself must be signed in original signature by the Laboratory Manager or his designee and dated. All copies of the SDG Narrative shall be signed in original signature.

2. Traffic Reports

A copy of the TRs submitted in Part A for all of the samples in the SDG shall be delivered. The TRs shall be arranged in increasing EPA sample numbering order, considering <u>both</u> letters and numbers in ordering samples. Copies of the SDG Cover Sheet shall be included with the copies of the TRs.

If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the

302001

(6) SICP for each analysis above [b.(1) - (5)]. SICPs must contain the header information described in a. (4) above.

c. Calibration Data

(1) Initial Calibration Data (Form VI PCDD-1 and Form VI PCDD-2) - in order by instrument, if more than one instrument used.

(a) PCDD/PCDF standard(s) SICPs for the initial (five-point) calibration shall be labeled as stated above.

(b) When more than one initial calibration is performed, the data must be arranged in chronological order by instrument.

(2) Continuing Calibration Data (Form VII PCDD-1 and Form VII PCDD-2) - in order by instrument, if more than one instrument is used.

(a) PCDD/PCDF standard(s) SICPs for all continuing calibrations shall be labeled as stated above.

(b) When more than one continuing calibration is performed, the data must be arranged in chronological order, by instrument.

d. Raw Quality Control Data

(1) Blank Data - in order by EPA sample number assigned to the blank. SICPs shall be submitted for each blank analyzed and labeled as above.

(2) Spiked Sample Data - in order by EPA sample number. SICPs shall be submitted for each spiked sample analyzed and labeled as above.

E. Complete SDG File

One Complete SDG File (CSF), including the original Sample Data Package, shall be delivered to the Region concurrently with delivery of the Sample Data Package to SMO and EMSL-LV. The contents of the CSF will be numbered according to the specifications described in Sections III and IV. The Document Inventory Sheet, Form DC-2, is contained in Section IV. The CSF will contain all original documents where possible. No copies will be placed in the CSF unless the originals are bound in a logbook which is maintained by the laboratory. The CSF will contain all original documents specified in Sections III and IV, and Form DC-2.

The CSF will consist of the following original documents in addition to the documents in the Sample Data Package:

1. Original Sample Data Package.

302002

If the laboratory does submit SDG-specific documents to EPA after submission of the CSF, the documents should be numbered as an addendum to the CSF, and a revised DC-2 Form should be submitted, or the documents should be numbered as a new CSF, and a new DC-2 Form should be submitted to the Regions only.

F. GC/MS Tapes

The Contractor must store all raw and processed GC/MS data on magnetic tape in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, initial calibrations and continuing calibrations, as well as all laboratory-generated quantitation reports and SICPs required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards and blanks. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case and SDG.

The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the APO or EMSL-LV.

When submitting GC/MS tapes to EPA, the following materials must be delivered in response to the request:

1. All associated raw data files for samples, blanks, matrix spikes, initial and continuing calibration standards, and window defining mix solutions.

2. All processed data files and quantitation output files associated with the raw data files described above.

3. All associated identifications and calculation files used to generate the data submitted in the data package.

4. A copy of the Contractor's written reference logbook relating tape files to EPA sample number, calibration data, standards, blanks and matrix spikes. The logbook must include EPA sample numbers and lab file identifiers for all samples, blanks and standards, identified by Case and SDG.

The laboratory must also provide a statement attesting to the completeness of the GC/MS data tape submission, signed and dated by the Laboratory Manager. This statement must be part of a cover sheet that includes the following information relevant to the data tape submission:

1. Laboratory name.

2. Date of submission.

3. Case number.

4. SDG number.

302003

If the laboratory does submit SDG-specific documents to EPA after submission of the CSF, the documents should be numbered as an addendum to the CSF, and a revised DC-2 Form should be submitted, or the documents should be numbered as a new CSF, and a new DC-2 Form should be submitted to the Regions only.

F. GC/MS Tapes

The Contractor must store all raw and processed GC/MS data on magnetic tape in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, initial calibrations and continuing calibrations, as well as all laboratory-generated quantitation reports and SICPs required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards and blanks. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case and SDG.

The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the APO.

When submitting GC/MS tapes to EPA, the following materials must be delivered in response to the request:

1. All associated raw data files for samples, blanks, matrix spikes, initial and continuing calibration standards, and window defining mix solutions.

2. All processed data files and quantitation output files associated with the raw data files described above.

3. All associated identifications and calculation files used to generate the data submitted in the data package.

4. A copy of the Contractor's written reference logbook relating tape files to EPA sample number, calibration data, standards, blanks and matrix spikes. The logbook must include EPA sample numbers and lab file identifiers for all samples, blanks and standards, identified by Case and SDG.

The laboratory must also provide a statement attesting to the completeness of the GC/MS data tape submission, signed and dated by the Laboratory Manager. This statement must be part of a cover sheet that includes the following information relevant to the data tape submission:

1. Laboratory name.

2. Date of submission.

3. Case number.

4. SDG number.

302004

SECTION III

FORM INSTRUCTION GUIDE

This section includes specific instructions for the completion of all required forms. These instructions are arranged in the following order:

A. General Information and Header Information

B. PCDD/PCDF Sample Data (Form I PCDD-1, PCDD-2 and PCDD-3)

C. PCDD/PCDF Total Congener Concentration Summary (Form II)

D. PCDD/PCDF Spiked Sample and Duplicate Sample Results (Form III PCDD-1 and PCDD-2)

E. PCDD/PCDF Method Blank Summary (Form IV)

F. PCDD/PCDF Window Defining Mix Summary, Chromatographic Resolution Summary, and Analytical Sequence (Form V PCDD-1, PCDD-2 and PCDD-3)

G. PCDD/PCDF Initial Calibration Data Summary (Form VI PCDD-1 and PCDD-2)

H. PCDD/PCDF Continuing Calibration Data Summary (Form VII PCDD-1 and PCDD-2)

I. Sample Log-In Sheet (Form DC-1)

J. Document Inventory Sheet (Form DC-2)

302005

The "Contract" is the number of the EPA contract under which the analyses were performed.

The "SDG No." is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter "SAS No.," and leave "Case No." blank. If samples are analyzed according to the Routine Analytical Services (IFB) protocols and have additional SAS requirements, enter both "Case No." and "SAS No." on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. NOTE: Some samples in a SDG may have a SAS No., while others do not.

The other information common to most of the forms is the "EPA Sample No." This number appears either in the upper right-hand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of Form I, III or IV, it should be entered on the middle line of the three lines that comprise the box.

All samples, spiked samples, duplicate samples, blanks and standards shall be identified with an EPA sample number. For field samples, spiked samples, and duplicates samples, the EPA sample number is based on the unique identifying number given in the Traffic Report or sample shipping records for that sample.

In order to facilitate data assessment, the following sample suffixes must be used:

XXXXX	-	EPA sample number
XXXXXS	-	Spiked aliquot of sample "XXXXX"
XXXXXD	-	Duplicate aliquot of sample "XXXXX"
XXXXXRE	-	Reextracted and reanalyzed aliquot of sample "XXXXX"
XXXXXDL	-	Diluted analysis of sample "XXXXX"

Form V PCDD-3 requires that all samples analyzed in a given 12-hour analytical sequence be listed, regardless of whether or not they are part of the SDG being reported, and regardless of whether or not they are EPA samples. Therefore, use "ZZZZZ" as the EPA sample number for any sample analysis not associated with the SDG being reported.

For blanks and standards, the following identification scheme must be used as the "EPA Sample No."

1. Method blanks shall be identified as DFBLK**.

302006

The "Contract" is the number of the EPA contract under which the analyses were performed. In the case of multiple laboratories operating under a corporate-wide contract, the contract number entered shall be that of the corporate contract, regardless of the facility performing the analyses.

The "SDG No." is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services (SAS). If samples are to be analyzed under SAS only and reported on these forms, then enter "SAS No.," and leave "Case No." blank. If samples are analyzed according to the Routine Analytical Services (IFB) protocols <u>and</u> have additional SAS requirements, enter <u>both</u> "Case No." and "SAS No." on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. NOTE: Some samples in a SDG may have a SAS No., while others do not.

The other information common to most of the forms is the "EPA Sample No." This number appears either in the upper right-hand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of Form I, III or IV, it should be entered on the middle line of the three lines that comprise the box.

All samples, spiked samples, duplicate samples, blanks and standards shall be identified with an EPA sample number. For field samples, spiked samples, and duplicates samples, the EPA sample number is based on the unique identifying number given in the Traffic Report or sample shipping records for that sample.

In order to facilitate data assessment, the following sample suffixes <u>must</u> be used:

XXXXX	-	EPA sample number
XXXXXS	-	Spiked aliquot of sample "XXXXX"
XXXXXD	-	Duplicate aliquot of sample "XXXXX"
XXXXXRE	-	Reextracted and reanalyzed aliquot of sample "XXXXX"
XXXXXDL	-	Diluted analysis of sample "XXXXX"

Form V PCDD-3 requires that <u>all</u> samples analyzed in a given 12-hour analytical sequence be listed, regardless of whether or not they are part of the SDG being reported, and regardless of whether or not they are EPA samples. Therefore, use "ZZZZZ" as the EPA sample number for any sample analysis <u>not</u> associated with the SDG being reported.

For blanks and standards, the following identification scheme <u>must</u> be used as the "EPA Sample No."

1. Method blanks shall be identified as DFBLK##.

302007

up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

B. PCDD/PCDF Sample Data

1. Form I PCDD-1

This form is used for tabulating and reporting the sample analysis results for target analytes. It is related to Form I PCDD-2, and for each sample for which there is a Form I PCDD-1, there must be a corresponding Form I PCDD-2.

Complete all header information according to the instructions in Part A and as follows:

Enter the "Matrix" of the sample being analyzed. The designation of matrix must reflect which one of the matrix-specific extraction procedures in Exhibit D was used for extraction of the sample.

For "Sample wt/vol," enter the number of grams (for soil) or milliliters (for water) of sample used in the first blank line, and the units, either "G" or "ML," in the second blank.

For water samples, indicate the extraction procedure used by entering "SEPF" for separatory funnel extraction or "CONT" for continuous liquid-liquid extraction in the field labeled "Water Sample Prep."

Enter the actual volume of the most concentrated sample extract, in microliters, under "Conc. Extract Volume:" This volume will typically be 100 microliters, although this volume is split into two aliquots before analysis.

Enter "GC Column," "Instrument," "Lab Sample ID," and "Lab File ID" as described in Part A.

"Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the validated time of sample receipt, VTSR) for that sample. It must be entered as MM/DD/YY.

"Date Extracted" and "Date Analyzed" must also be entered as MM/DD/YY. If continuous liquid-liquid extraction procedures are used for water samples, enter the date on which the procedure was started as the "Date Extracted." If separatory funnel procedures are used for water samples, enter the date on which the procedure was completed. The "Date Analyzed" must be the date of the analysis for which the results are reported on Form I. (If the sample requires a second column confirmation and is reported on Form I PCDD-3, the "Date Analyzed" on Form I PCDD-3 must be the date of the second analysis, while the date on Form I PCDD-1 and PCDD-2 will be the date of the first analysis.)

302008

calculation takes into account the sample weight/volume extracted, the volume of the most concentrated extract, the injection volume, and dilution of the most concentrated extract prior to analysis. The calculation does not consider the percent solids content of the sample, as all results are reported on a wet weight basis.

J - Indicates an estimated value. This flag is used when the mass spectral data indicate presence of an analyte meeting all the identification criteria in Exhibit D, but the result is less than the sample quantitation limit, but greater than zero.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.

E - This flag identifies analytes whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample extract must be diluted and reanalyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged "E" on the Form I for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, the results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have the "DL" suffix appended to the EPA sample number.

D - This flag indicates all compounds identified in an analysis at a secondary dilution factor. If a sample extract is reanalyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the EPA sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample extract.

S - This flag indicates that the analyte in question is, in the opinion of the GC/MS Interpretation Specialist, a PCDD/PCDF, even though the M-[COCl]+ ion did not meet the requirement of 2.5 times signal-to-noise (see Exhibit D, Section 11.3).

H - This flag indicates that the analyte in question was quantitated using peak heights rather than peak areas for both the analyte and its internal standard (see Exhibit D, Section 11.4).

X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description must be attached to the Sample Data Summary

equivalents determines whether or not the sample needs to be analyzed on a second GC column to more completely separate the 2378-TCDF from all other TCDD and TCDF isomers (see Exhibit D).

Complete the header information as above. The header of Form I PCDD-2 must match the header of Form I PCDD-1 for the same sample.

For each 2,3,7,8-substituted isomer positively identified in the sample, enter the concentration found in the column labeled "CONCENTRATION." If an isomer was not detected, i.e., flagged "U" on Form I PCDD-1, for the purposes of this calculation, enter 0.0 (zero) as the concentration. EMPC values are not included in the TEF calculations under this SOW.

Multiply each concentration times the TEF listed on the form for that isomer, and enter the product of the two in the column labeled "TEF-ADJUSTED CONCENTRATION." Add all 17 TEF-adjusted concentrations together, including any zeros, and enter the total on the line at the bottom of the form.

If the total TEF-adjusted concentration is greater than the values listed at the bottom of the form and in Exhibit D, then a second column confirmation analysis is required (see Exhibit D).

3. Form I PCDD-3, Second Column Confirmation Results

This page of Form I is used to report the results of all second column confirmation analyses performed. The requirements for second column confirmation are discussed above and in Exhibit D. Each time a second column confirmation is performed, the results are reported on Form I PCDD-3.

Complete the header information as above, except note that the fields for "GC Column" and "Date Analyzed" must correspond to the second column confirmation analysis, i.e., they must not match those fields in the header of Form I PCDD-1 or PCDD-2. Other fields such as "Instrument," "Dilution Factor," and "Lab File ID" may also differ and must correspond to the second column confirmation analysis.

Complete the information in the lower portion of the form in a fashion similar to that for Form I PCDD-1, but entering the results of the second column confirmation.

Enter the data on recovery of the internal standards and cleanup standard from the second column confirmation analysis in a fashion similar to that for the original analysis.

C. PCDD/PCDF Total Congener Concentration Summary (Form II)

This form is used to report the total concentration of all PCDD/PCDF isomers in a given homologue that are detected in the sample, including those isomers that do not represent the 2,3,7,8-substituted isomers of greatest toxicological concern. Because there are many isomers in each

difference (RPD) between the results of the original and duplicate analyses of one sample of each matrix in each SDG. In order to allow direct comparison of the results of both the analyses, the concentration results from the original and duplicate analyses are reported on a single copy of Form III PCDD-2.

Complete the header information as described in Part A above, but enter the EPA sample number, lab sample ID, and lab file ID of the duplicate aliquot in these fields on Form III PCDD-2. Enter the concentration units.

For each target analyte, enter the results from both the analyses under the columns "Sample Concentration" and "Duplicate Concentration." These values must match those on Form I for these aliquots, <u>except</u> that undetected analytes (flagged "U" on Form I) are reported as zero on Form III PCDD-2. If either or both the analyses resulted in an EMPC for any analyte, enter the EMPC as the concentration, and use that value in the calculations.

Calculate the relative percent difference between the two concentrations or EMPCs, as described in Exhibit D, using zero for undetected analytes, and report this value to the nearest whole percentage point under "RPD." If the analyte was not detected in <u>either</u> aliquot, enter zero for both concentrations, and report the RPD as zero as well. Flag all values outside the quality control limits listed on the forms by entering an asterisk (*) under the number (#) symbol.

E. <u>PCDD/PCDF Method Blank Summary (Form IV)</u>

This form summarizes the samples associated with each method blank analysis. A copy of Form IV is required for each blank.

Complete the header information as described in Part A. The EPA sample number entered in the box at the top of the form shall be the number assigned to the method blank. The matrix entered on this form refers to the matrix of the associated samples, as one blank is required each time that samples of a similar matrix are extracted together. Therefore, samples of differing matrices cannot be mixed together on a single Form IV.

Summarize the samples associated with a given method blank in the box in the lower portion of the form, entering the EPA sample number, lab sample ID, lab file ID, and date of analysis of each sample. Include spiked samples and duplicate samples as well.

F. <u>PCDD/PCDF Window Defining Mix Summary, Chromatographic Resolution Summary, and Analytical Sequence (Form V)</u>

1. PCDD/PCDF Window Defining Mix Summary (Form V PCDD-1)

This page of Form V is used to report the results of the analysis of the window defining mixture that precedes each initial

3. PCDD/PCDF Analytical Sequence (Form V PCDD-3)

This page of Form V is used to report the sequence of analyses, including the analysis of the window defining mixture, the calibration standards, blanks, samples, duplicates, and spiked samples. One copy of Form V PCDD-2 is required for each 12-hour period during which samples, blanks, standards, etc. associated with the SDG are analyzed.

Complete the header information as described in Part A. Enter the inclusive dates and times of the analyses of the first and last initial calibration standards in the fields for "Init. Calib. Date(s)" and "Init. Calib. Times." Dates must be in the format MM/DD/YY, and all times are expressed as HHMM, in military time (i.e., a 24-hour clock).

In the box in the lower portion of the form, enter the EPA sample number, lab sample ID, lab file ID, and date and time of analysis of all standards, samples, blanks, duplicates, spiked samples, dilutions, reanalyses, etc. All analyses in the 12-hour period must be listed on Form V. If analysis is not associated with the SDG being reported, enter the EPA sample number as "ZZZZZ," as described in Part A. The 12-hour sequence must end with the analysis of the appropriate calibration standard, as described in Exhibit D. In order to meet the requirements of the 12-hour sequence, the standard must be injected within 12 hours of the injection of the standard that began the sequence (CC3 on the DB-5, and the column performance solution on the SP-2331).

If the analytical sequence includes the analysis of the initial calibration standards, these standards and the window defining mix must be included on that copy of Form V, identified by the EPA sample numbers described in Part A. A copy of the analytical sequence that includes these initial calibration standards and the window defining mix must be submitted with each data package to which the initial calibration applies, but the Case number and SAS number must match those of each data package in which these initial calibration data are reported.

G. PCDD/PCDF Initial Calibration Data Summary (Form VI)

1. PCDD/PCDF Initial Calibration Response Factor Summary (Form VI PCDD-1)

This form is used to summarize the response factors for each target analyte, internal standard and cleanup standard calculated from the initial calibration. Complete the header information as described in Part A. Enter the inclusive initial calibration date(s) and times, as described for Form V PCDD-2. One copy of Form VI PCDD-1 must be completed for each initial calibration, for each instrument and GC column used for analysis of samples, and must be accompanied by a corresponding Form VI PCDD-2.

Form VI. One copy of Form VII PCDD-1 must be completed for each continuing calibration performed, and must be accompanied by a corresponding copy of Form VII PCDD-2.

Complete the header information as described in Part A. The date and time of analysis and lab file ID in the header must correspond to the analysis of the CC3 standard. Enter the date of the associated initial calibration in the field for "Init. Calib. Date(s):" If the calendar date changed during the initial calibration, enter the inclusive dates of the first and last standards in the associated initial calibration in the fields for "Init. Calib. Date(s)."

For each of the native analytes, internal standards, and the cleanup standard, enter the relative response factor (RRF) determined from the analysis of the continuing calibration standard in the column labeled "RRF." Enter the mean RRF for each analyte from the associated initial calibration, in the column labeled "MEAN RRF." For the seven native analytes and the cleanup standard that undergo only a single-point calibration, enter the CC3 RRF from the initial calibration, which is also entered as the mean RRF on Form VI. The values reported in this column must match those reported on the Form VI for the associated initial calibration. Calculate the percent difference (%D) between the RRF and the mean RRF for each analyte, and report under "%D." If the percent difference exceeds the quality control limits shown on the form (±30%), flag that analyte by placing an asterisk (*) in the "RRF FLAG" column. Report the ion abundance ratio of each analyte under the "ION RATIO" column. Flag any ion ratio that fails outside the quality control limits shown on the form by placing as asterisk (*) in the "ION FLAG" column.

Note that because only one ion is monitored for the cleanup standard, no ion ratio is determined for this analyte. For the recovery standards, relative response factors are not calculated or reported on Form VII, but the ion abundance ratios for these standards must be reported on Form VII.

2. PCDD/PCDF Continuing Calibration Summary (Form VII PCDD-2)

This page of Form VII is used to summarize the absolute and relative retention times of the analytes in the continuing calibration standard that must be analyzed in each 12-hour analytical sequence. Absolute retention times and relative retention times are critical to the identification of PCDDs/PCDFs by this method. One copy of Form VII PCDD-2 must be completed for each continuing calibration performed and must be accompanied by a corresponding copy of Form VII PCDD-1.

Complete the header information as described in Part A. The date and time of analysis and lab file ID in the header must correspond to the analysis of the CC3 standard. Enter the date of the associated initial calibration in the field for "Init. Calib. Date(s):" If the calendar date changed during the initial

SECTION IV

DATA REPORTING FORMS

302014

1DFB
PCDD/PCDF TOXICITY EQUIVALENCE SUMMARY

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: _____ (Soil/Water/Waste/Ash) Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Water Sample Prep.: _____ (Sepf/Cont) Date Received: _____

Concentrated Extract Volume: ____ (uL) Date Extracted: _____

Injection Volume: ___ (uL) % Solids: _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm) Dilution Factor: _____

CONCENTRATION UNITS: (ng/L or ug/Kg) _____

ANALYTE	CONCENTRATION	TEF	TEF-ADJUSTED CONCENTRATION
2378-TCDD		x 1.0 =	
2378-TCDF		x 0.1 =	
12378-PeCDF		x 0.05 =	
12378-PeCDD		x 0.5 =	
23478-PeCDF		x 0.5 =	
123478-HxCDF		x 0.1 =	
123678-HxCDF		x 0.1 =	
123478-HxCDD		x 0.1 =	
123678-HxCDD		x 0.1 =	
123789-HxCDD		x 0.1 =	
234678-HxCDF		x 0.1 =	
123789-HxCDF		x 0.1 =	
1234678-HpCDF		x 0.01 =	
1234678-HpCDD		x 0.01 =	
1234789-HpCDF		x 0.01 =	
OCDD		x 0.001 =	
OCDF		x 0.001 =	
		Total =	

NOTE: Do <u>not</u> include EMPC or EDL values in the TEF-adjusted Concentration.

If the Total Toxic Equivalent Concentration of the sample is greater than 7 ng/L for an aqueous sample, greater than 0.7 ug/Kg for any solid matrix, or greater than 7 ug/Kg for a chemical waste sample, then second column confirmation of the results may be required.

302015

2DF

PCDD/PCDF TOTAL HOMOLOGUE CONCENTRATION SUMMARY

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: _____ (Soil/Water/Waste/Ash) Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Water Sample Prep.: _____ (Sepf/Cont) Date Received: _____

Concentrated Extract Volume: ____ (uL) Date Extracted: _____

Injection Volume: ___ (uL) % Solids: _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm) Dilution Factor: _____

CONCENTRATION UNITS: (ng/L or ug/Kg) _____

HOMOLOGUE	PEAKS	CONCENTRATION	Q	EMPC/EDL
DIOXINS				
Total TCDD____	____	_____	____	_____
Total PeCDD____	____	_____	____	_____
Total HxCDD____	____	_____	____	_____
Total HpCDD____	____	_____	____	_____
FURANS				
Total TCDF____	____	_____	____	_____
Total PeCDF____	____	_____	____	_____
Total HxCDF____	____	_____	____	_____
Total HpCDF____	____	_____	____	_____

NOTE: Concentrations, EMPCs, and EDLs are calculated on a wet weight basis. The total congener concentrations do not affect the TEF calculations.

3DFB
PCDD/PCDF DUPLICATE SAMPLE SUMMARY

EPA SAMPLE NO.

Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: _____ (Soil/Water/Waste/Ash)

CONCENTRATION UNITS: (ng/L or ug/Kg) _____

ANALYTE	SAMPLE CONCENTRATION	DUPLICATE CONCENTRATION	RPD	#	QC LIMITS
2378-TCDD					50
2378-TCDF					50
12378-PeCDF					50
12378-PeCDD					50
23478-PeCDF					50
123478-HxCDF					50
123678-HxCDF					50
123478-HxCDD					50
123678-HxCDD					50
123789-HxCDD					50
234678-HxCDF					50
123789-HxCDF					50
1234678-HpCDF					50
1234678-HpCDD					50
1234789-HpCDF					50
OCDD					50
OCDF					50

If an analyte is not detected in either analysis, enter 0 (zero) as the concentration.

Column to be used to flag values outside QC limits.

QC limits are advisory

5DFA
PCDD/PCDF WINDOW DEFINING MIX SUMMARY

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Lab File ID: _____

Instrument ID: _____ Date Analyzed: _____

Time Analyzed: _____

CONGENER	RT FIRST ELUTING	RT LAST ELUTING
TCDD_____	_____	_____
TCDF_____	_____	_____
PeCDD_____	_____	_____
PeCDF_____	_____	_____
HxCDD_____	_____	_____
HxCDF_____	_____	_____
HpCDD_____	_____	_____
HpCDF_____	_____	_____

302018

5DFC
PCDD/PCDF ANALYTICAL SEQUENCE SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Instrument ID: _____

Init. Calib. Date(s): _____ _____

Init. Calib. Times: _____ _____

THE ANALYTICAL SEQUENCE OF STANDARDS, SAMPLES, BLANKS, SPIKES, AND DUPLICATES IS AS FOLLOWS:

EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED

302019

6DFB

PCDD/PCDF INITIAL CALIBRATION ION ABUNDANCE RATIO SUMMARY

Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Instrument ID: _____

Init. Calib. Date(s): _____ _____

Init. Calib. Times: _____ _____

NATIVE ANALYTES	SELECTED IONS	ION ABUNDANCE RATIO CC1	CC2	CC3	CC4	CC5	FLAG	QC LIMITS
2378-TCDD	320/322							0.65-0.89
2378-TCDF	304/306							0.65-0.89
12378-PeCDF	340/342							1.24-1.86
12378-PeCDD	356/358							1.24-1.86
23478-PeCDF	340/342							1.24-1.86
123478-HxCDF	374/376							1.05-1.43
123678-HxCDF	374/376							1.05-1.43
123478-HxCDD	390/392							1.05-1.43
123678-HxCDD	390/392							1.05-1.43
123789-HxCDD	390/392							1.05-1.43
234678-HxCDF	374/376							1.05-1.43
123789-HxCDF	374/376							1.05-1.43
1234678-HpCDF	408/410							0.88-1.20
1234678-HpCDD	424/426							0.88-1.20
1234789-HpCDF	408/410							0.88-1.20
OCDD	458/460							0.76-1.02
OCDF	442/444							0.76-1.02
INTERNAL STANDARDS								
13C-2378-TCDD	332/334							0.65-0.89
13C-2378-TCDF	316/318							0.65-0.89
13C-123678-HxCDD	402/404							1.05-1.43
13C-1234678-HpCDF	420/422							0.88-1.20
13C-OCDD	470/472							0.76-1.02
RECOVERY STANDARDS								
13C-1234-TCDD	332/334							0.65-0.89
13C-123789-HxCDD	402/404							1.05-1.43

QC limits represent ± 15% window around the theoretical ion abundance ratio.

single point calibration is performed for seven of the native analytes and the cleanup standard.

ne laboratory must flag any analyte in any calibration solution which does not meet the ion abundance ratio QC limit by placing an asterisk in the flag column.

7DFB

PCDD/PCDF CONTINUING CALIBRATION RETENTION TIME SUMMARY

Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Instrument ID: _____

Date Analyzed: _____ Time Analyzed: _____

Lab File ID: _____ Init. Calib. Date(s): _____ _____

NATIVE ANALYTES	RRT	RT
2378-TCDD		
2378-TCDF		
12378-PeCDF		
12378-PeCDD		
23478-PeCDF		
123478-HxCDF		
123678-HxCDF		
123478-HxCDD		
123678-HxCDD		
123789-HxCDD		
234678-HxCDF		
123789-HxCDF		
1234678-HpCDF		
1234678-HpCDD		
1234789-HpCDF		
OCDD		
OCDF		
INTERNAL STANDARDS VS. RECOVERY STDS.		
13C-2378-TCDD	NA	
13C-2378-TCDF	NA	
13C-123678-HxCDD	NA	
13C-1234678-HpCDF	NA	
13C-OCDD	NA	
37Cl-2378-TCDD		
RECOVERY STANDARDS		
13C-1234-TCDD	NA	
13C-123789-HxCDD	NA	

RRT = (RT of analyte)/(RT of appropriate internal standard)

PCDD/PCDF COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____ CITY/STATE _____

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ _____ _____ SAS NO. _____

CONTRACT NO. _____ SOW NO. _____

All documents delivered in the complete SDG file must be original documents where possible. (REFERENCE EXHIBIT B, SECTION II and SECTION III.)

	PAGE NOs FROM	TO	CHECK LAB	EPA
1. Inventory Sheet (Form DC-2) (Do not number)	___	___	___	___
2. SDG Narrative	___	___	___	___
3. Traffic Report	___	___	___	___
4. PCDD/PCDF Data				
a. Sample Data				
TCL Results (Form I PCDD-1)	___	___	___	___
Calculation of the Toxicity Equivalence (Form I PCDD-2)	___	___	___	___
Second Column Confirmation Summary (Form I PCDD-3)	___	___	___	___
Selected Ion Current Profile (SICP) for each sample and each analysis of each sample	___	___	___	___
Total Congener Concentration Results (Form II PCDD)	___	___	___	___
b. Quality Control Data				
Spiked Sample Results (Form III PCDD-1)	___	___	___	___
Duplicate Sample Results (Form III PCDD-2)	___	___	___	___
Method Blank Summary (Form IV PCDD)	___	___	___	___
Window Defining Mix Summary (Form V PCDD-1)	___	___	___	___
Chromatographic Resolution Summary (Form V PCDD-2)	___	___	___	___
SICP for each QC analysis	___	___	___	___
c. Calibration Data				
Initial Calibration Data (Form VI PCDD-1 and Form VI PCDD-2) and PCDD/PCDF standard(s) SICPs for the initial (five-point) calibration	___	___	___	___
Continuing Calibration Data (Form VII PCDD-1 and Form VII PCDD-2) and PCDD/PCDF standard(s) SICPs for all continuing calibrations	___	___	___	___
d. Raw Quality Control Data				
Blank Data and SICPs for each blank analyzed	___	___	___	___
Spiked Sample Data and SICPs for each spiked sample analyzed	___	___	___	___

302022

PCDD/PCDF COMPLETE SDG FILE (CSF) INVENTORY SHEET (Cont.)

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____ _____ _____ SAS NO. _____

9. Comments: _____

Completed by: _____ _____ _____
(CLP Lab) (Signature) (Printed Name/Title) (Date)

ted by: _____ _____ _____
(EPA) (Signature) (Printed Name/Title) (Date)

EXHIBIT C

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

NOTE: The values in these tables are quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to be simply detected above the background noise. The quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

The CRQL values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D. All CRQL values are reported on a wet weight basis, as are sample data produced using the specifications in Exhibit D.

EXHIBIT C

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

NOTE: The values in these tables are quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to be simply detected above the background noise. The quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

The CRQL values listed on the following pages are based on the analysis of samples according the specifications given in Exhibit D. All CRQL values are reported on a wet weight basis, as are sample data produced using the specifications in Exhibit D.

In addition, data are reported for the total concentration of all detected PCDDs or PCDFs in the following homologues. However, because the number of non-2,3,7,8-substituted isomers that might be detected in a sample is unpredictable, it is not possible to assign CRQL values to the total homologue concentrations.

Homologue	CAS Number	Number of Possible Isomers	Number of 2,3,7,8-Substituted Isomers
Total TCDD	41903-57-5	22	1
Total TCDF	55722-27-5	38	1
Total PeCDD	36088-22-9	14	1
Total PeCDF	30402-15-4	28	2
Total HxCDD	34465-4608	10	3
Total HxCDF	55684-94-1	16	4
Total HpCDD	37871-00-4	2	1
Total HpCDF	38998-75-3	4	2

There is only one isomer in both the OCDD and OCDF homologues, hence the total concentration is the same as the 2,3,7,8-substituted concentration listed on the previous page.

TCDD - Tetrachlorinated dibenzo-*p*-dioxin
TCDF - Tetrachlorinated dibenzofuran
PeCDD - Pentachlorinated dibenzo-*p*-dioxin
PeCDF - Pentachlorinated dibenzofuran
HxCDD - Hexachlorinated dibenzo-*p*-dioxin
HxCDF - Hexachlorinated dibenzofuran
HpCDD - Heptachlorinated dibenzo-*p*-dioxin
HpCDF - Heptachlorinated dibenzofuran
OCDD - Octachlorinated dibenzo-*p*-dioxin
OCDF - Octachlorinated dibenzofuran

302026

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302027

1. Scope and Application

1.1 This method is appropriate for the detection and quantitative measurement of 2378-tetrachlorinated dibenzo-*p*-dioxin (2378-TCDD), 2378-tetrachlorinated dibenzofuran (2378-TCDF), and the 2,3,7,8-substituted penta-, hexa-, hepta- and octachlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) in water, soil, fly ash, and chemical waste samples including stillbottom, fuel oil, and sludge matrices. The analytical method requires the use of high resolution gas chromatography and low resolution mass spectrometry (HRGC/LRMS) on sample extracts that have been subjected to specified cleanup procedures. The calibration range is dependent on the compound and the sample size. The sample size varies by sample matrix. The Contract Required Quantitation Limits (CRQLs) for each matrix and compound are listed in Exhibit C. The upper limit of the calibration range for each compound is 20 times the CRQL. Samples in which any target compound is found above the calibration range must be diluted and reanalyzed.

1.2 The protocol requires the calculation of the 2378-TCDD toxicity equivalence according to the procedures given in the U.S. Environmental Protection Agency "Update of Toxicity Equivalency Factors (TEFs) for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-*p*-Dioxins and Dibenzofurans (CDDs/CDFs)" March 1989 (EPA 625/3-89/016). This procedure recognized that structure-activity relationships exist between the chemical structure of a particular PCDD/PCDF "and its ability to elicit a biological/toxic response in various in vivo and in vitro test systems." Of the 210 possible chlorinated dibenzo-*p*-dioxins and chlorinated dibenzofurans, the 17 isomers that bear chlorine atoms in the 2,3,7 and 8 positions of their respective structures are the compounds of greatest concern. To aid in the assessment of risks to human health and the environment, a factor is assigned to each of these 17 2,3,7,8-substituted PCDDs and PCDFs that relates the toxicity of that isomer to a concentration of the most toxic isomer, 2378-TCDD. These factors are called TEFs. The concentrations of any of the 17 isomers that are detected in an environmental sample can then be adjusted by the TEF and summed, yielding a concentration of 2378-TCDD with an equivalent toxicity.

1.3 If the toxicity equivalence is less than 0.7 parts per billion (ppb) for a soil or fly ash sample, less than 7 parts-per-trillion (ppt) for an aqueous sample, or less than 7 ppb for a chemical waste, no further analysis is required. If the toxicity equivalence is greater than or equal to 0.7 ppb (soil or fly ash), 7 ppt (aqueous), or 7 ppb (chemical waste), analysis on a column capable of resolving all 2,3,7,8-substituted PCDDs/PCDFs is required.

For any sample analyzed on a DB-5 (or equivalent) column in which either 2378-TCDD or 2378-TCDF is reported as an "Estimated Maximum Possible Concentration" (see Section 15.7), regardless of TEF-adjusted concentration or matrix, analysis of the extract is required on a second GC column which provides better specificity for these two isomers.

amounts of floating, suspended and settled particulate matter. Multiple phases should not be present (see Section 8.4). Approximately 1 L of the water sample is spiked with the internal standard solution and filtered to separate the aqueous and particulate fractions. The filtered aqueous fraction is extracted with methylene chloride using a separatory funnel or continuous liquid-liquid extractor. The particulate fraction is extracted with toluene in a SDS extractor. The extracts of the two fractions are then combined for cleanup.

2.3 Fly Ash Extraction

For the purposes of this method, a fly ash sample is defined as a solid matrix from an incineration or other combustion process which may contain water and other solids. It should not contain an obvious liquid phase. A 10 g aliquot of the fly ash is washed with dilute hydrochloric acid, spiked with the internal standard solution, and extracted with toluene in a SDS extractor.

2.4 Chemical Waste Sample Extraction

For the purposes of this method, a chemical waste sample includes sample matrices of oils, stillbottoms, oily sludge, oil-laced soil, and surface water heavily contaminated with the matrices listed above (see Section 8.2). Internal standards are added in the concentrations listed in Table 4 to a 1 or 10 g aliquot of chemical waste. Wet fuel oil and oily sludge samples, showing signs of water, are spiked with the internal standard solution, fitted with a reflux condenser and a Dean Stark water separator to remove the water, and extracted with toluene. Stillbottom samples are spiked with the internal standard solution, refluxed with toluene, and filtered.

2.5 Cleanup and Analysis

Immediately prior to cleanup, all extracts are spiked with a ^{37}Cl-2378-TCDD standard. Because it is added after extraction, the recovery of this standard may be used to differentiate between losses of analytes or internal standards during extraction and losses that occur during the various cleanup procedures. The extracts are subjected to an acid-base washing treatment and dried. Following a solvent exchange step, the extract is cleaned up by column chromatographic procedures, including silica gel, acid alumina, and carbon on celite columns, to eliminate sample components that may interfere with the detection and measurement of PCDDs/PCDFs. The extracts are concentrated and the solvent is exchanged to tridecane. The recovery standards are added to an aliquot (50 uL) of the extract and the aliquot is reduced to the final volume of 50 uL. The remaining 50 uL of extract is retained in the event that dilutions or reanalyses are required. One or two uL of the concentrated aliquot containing the recovery standards are injected onto a fused silica capillary column in a gas chromatograph (GC) interfaced to a mass spectrometer (MS) (see Paragraph 4.1.1).

The identification of PCDD/PCDF isomers is based on the simultaneous detection of the two most abundant ions in the molecular ion regions and the M-COCl ion. In addition, the identification of OCDD and five

it with the analytes at concentrations specified in Section 13. This aliquot is analyzed and the recovery of the spiked analytes is determined.

2.9 In order to provide information on the precision of the analysis in the sample matrix, the laboratory must perform a duplicate analysis on one sample of each matrix in each SDG. The samples to be analyzed in duplicate may be specified by the Region in advance; however, if no samples are so specified, the laboratory must select a sample of each matrix for duplicate analyses. The precision of the analysis is determined as the relative percent difference of the concentrations as specified in Section 14.

2.10 Due to a variety of situations that may occur during contract performance, the laboratory shall be required to reextract and reanalyze certain samples or groups of samples. As used hereafter, except in the case of dilutions, the term "rerun" shall indicate sample reextraction, cleanup and reanalysis. When dilutions are required, the original extract shall be diluted and reanalyzed (see Section 10.4).

When the rerun is required due to matrix effects, interferences or other problems encountered, the Government will pay the Contractor for the reruns. Such reruns shall be billable and accountable under the specified contract allotment of automatic reruns. When the rerun is required due to Contractor materials, equipment or instrumentation problems or lack of Contractor adherence to specified contract procedures, the rerun shall not be billable nor accountable under the terms of this contract. The Contractor's failure to perform any of the sample reruns specified herein, either billable or nonbillable, shall be construed as Contractor nonperformance and may result in the termination of the contract for default. Specific requirements for reextraction and reanalysis are given in Section 17.

NOTE: A contaminated method blank is the only circumstance that may require more than one rerun per sample.

3. <u>Interferences</u>

3.1 Any compound that yields ions listed in Table 5 and also elutes within the retention time window of the corresponding homologue is a potential interference. PCDDs/PCDFs are often associated with other chlorinated compounds such as polychlorinated biphenyls (PCBs) and polychlorinated diphenyl ethers (PCDPEs). These compounds may be found at concentrations several orders of magnitude higher than that of the analytes of interest and may otherwise interfere with the analysis of PCDDs/PCDFs. Therefore, the retention time of the target analytes must be verified using reference standards and compared to retention time windows established during the calibration. While the cleanup procedures specified in this method are designed to minimize these interferences, some samples may ultimately require additional cleanup steps to achieve the detection limits.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines which may cause

4.1.4 GC/MS interfaces constructed of all glass or glass-lined materials are required. Glass can be deactivated by silanizing with dichlorodimethylsilane. Inserting a fused silica column directly into the MS source is recommended; care must be taken not to expose the end of the column to the electron beam.

4.1.5 The Contractor shall use a magnetic media storage device capable of recording data suitable for long-term off-line storage. The Contractor shall record all raw GC/MS data acquired during the entire contract period on magnetic media in appropriate instrument manufacturer format.

4.2 GC Column

Fused silica capillary columns are required. The columns shall demonstrate the required separation of all 2378-specific isomers whether a dual column or a single column analysis is chosen. Column operating conditions shall be evaluated at the beginning and end of each 12-hour period during which samples or concentration calibration solutions are analyzed (see Section 7.4).

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 m DB-5 column. In order to determine the concentration of the individual 2,3,7,8-substituted isomers, if the toxicity equivalence is greater than 0.7 ppb (solids), 7 ppt (aqueous), or 7 ppb (chemical waste), the sample extract shall be reanalyzed on a 60 m SP-2330 or SP-2331 (or equivalent) GC column.

For any sample analyzed on a DB-5 (or equivalent) column in which either 2378-TCDD or 2378-TCDF is reported as an Estimated Maximum Possible Concentration (see Section 15.7), regardless of TEF-adjusted concentration or matrix, analysis of the extract is required on a second GC column which provides better specificity for these two isomers.

Analysis on a single column is acceptable if the required separation of all the 2378-specific isomers is demonstrated and the minimum acceptance criteria outlined in Sections 7.1, 7.2 and 7.3 are met. See Section 11 for the specifications for the analysis of the 2378-specific isomers using both dual columns and single columns.

4.3 Miscellaneous Equipment

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

4.3.1 Nitrogen evaporation apparatus (N-Evap* Analytical Evaporator Model 111. Organomation Association Inc., Northborough, MA, or equivalent).

4.3.2 Balance capable of accurately weighing ± 0.01 g.

NOTE: Extraction thimbles must be of sufficient size to hold 100 g of sand, 5 g of silica gel, and at least 10 g of solid sample, with room to mix the sand and sample in the thimble.

4.4.9 Dean Stark Water Separator Apparatus, with a Teflon stopcock. Must fit between Soxhlet extractor body and condenser.

4.4.10 Concentrator tubes. 15 mL conical centrifuge tubes.

4.4.11 Separatory funnels. 125 mL and 2 L separatory funnels with a Teflon stopcock.

4.4.12 Continuous Liquid-Liquid Extractor. 1 L sample capacity, suitable for use with heavier than water solvents.

4.4.13 Boiling chips. Teflon boiling chips washed with hexane prior to use.

4.4.14 Buchner funnel. 15 cm.

4.4.15 Filtration flask. For use with Buchner funnel, 1 L capacity.

4.5 Glassware Cleaning Procedures

Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused shall be scrupulously cleaned as soon as possible after use, applying the following procedure.

4.5.1 Rinse glassware with the last solvent used in it.

4.5.2 Wash with hot water containing detergent.

4.5.3 Rinse with copious amounts of tap water and several portions of distilled water. Drain dry.

4.5.4 Rinse with high purity acetone and hexane.

4.5.5 After glassware is dry, store inverted or capped with aluminum foil in a clean environment.

Do <u>not</u> bake reusable glassware as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated baking may cause active sites on the glass surface that will irreversibly adsorb PCDDs/PCDFs.

CAUTION: The analysis for PCDDs/PCDFs in water samples is for much lower concentrations than in soil/sediment, fly ash, or chemical waste samples. Extreme care must be taken to prevent cross-contamination between soil/sediment, fly ash, chemical waste and water samples. Therefore, it is strongly recommended that separate glassware be reserved for analyzing water samples.

4.6 Preextraction of Glassware

It is required that all glassware be rinsed or preextracted with solvent immediately before use. The SDS apparatus and continuous liquid-liquid extractors must be preextracted for approximately three hours immediately prior to use. The pooled waste solvent for a set of extractions may be concentrated and analyzed as a method of demonstrating that the glassware was free of contamination.

It is recommended that each piece of reusable glassware be numbered in such a fashion that the laboratory can associate all reusable glassware with the processing of a particular sample. This procedure will assist the laboratory in tracking down possible sources of contamination for individual samples, identifying glassware associated with highly contaminated samples that may require extra cleaning, and determining when glassware should be discarded.

5. Reagents and Consumable Materials

Brand names and catalog numbers are for illustrative purposes only and do not imply an endorsement by EPA. Equivalency of materials from other suppliers may be demonstrated by performing analyses that meet the specifications of this method.

5.1 Solvents. High purity, distilled-in-glass: hexane, methanol, methylene chloride, toluene, isooctane, cyclohexane, acetone, tridecane (or nonane).

5.2 Filters

5.2.1 Filter paper. Whatman No. 1 or equivalent.

5.2.2 Glass fiber filter. 15 cm, for use with Buchner funnel.

5.2.3 0.45 micron, Millipore or equivalent, PTFE or other material compatible with toluene. Rinse with toluene.

5.3 White quartz sand. 60/70 mesh, for use in the SDS extractor. Bake at 450°C for 4 hours minimum.

5.4 Glass wool, silanized. Extract with methylene chloride and hexane before use.

5.5 Sodium Sulfate. Granular, anhydrous. Before use, heat to 400°C in a shallow tray for approximately 4 hours, cool in a desiccator, and store in a glass jar.

5.6 Potassium Hydroxide. ACS grade, prepare a 20% (w/v) solution in distilled water.

5.7 Sulfuric Acid, concentrated. ACS grade, specific gravity 1.84.

5.8 Sodium Chloride. ACS grade, prepare a 5% (w/v) solution in distilled water.

5.13 Recovery Standard Solution

The hexane solution contains the recovery standards, $^{13}C_{12}$-1234-TCDD and $^{13}C_{12}$-123789-HxCDD, at concentrations of 5.0 ng/uL, in a solvent other than tridecane or nonane (see Section 10.2). Depending upon the availability of materials, EMSL-LV will provide the solution.

5.14 Continuing Calibration Solution

This solution contains standards to be used for identification and quantitation of target analytes. In order to have all 2,3,7,8-substituted isomers and the cleanup standard present for quantitation purposes, a commercially supplied supplemental standard and the cleanup standard solution are combined with the EPA-supplied CC4 solution to produce the CC3 solution (see Paragraph 7.4.1). This solution is identified in Table 3.

5.15 Window Defining Mix

This solution is to be obtained by the laboratory through commercial vendors. The solution contains the first and last eluting isomer of each homologue (see Table 9) and is used to verify that the switching times between the descriptors have been appropriately set.

The window defining mix need not contain any of the labeled internal or recovery standards, as no quantitative measurements are based on this mixture. However, these standards and other isomers may be added to the mixture listed in Table 7 at the discretion of the laboratory, so long as the additional contents of the mixture are clearly specified in every SDG Narrative.

If the laboratory employs a GC column that has a different elution order than those specified here, the laboratory must ensure that the first and last eluting isomers in each homologue are represented in the window defining mix used to evaluate that column. The concentrations of any additional isomers should be approximately the same as those listed in Table 9.

EMSL-LV does not supply the window defining mix (see Table 9).

5.16 Supplemental Calibration Solution

This solution contains seven 2,3,7,8-substituted PCDD/PCDF isomers to be added to the CC4 solution to produce the CC3 solution that is used for identification and quantitation of target analytes. EMSL-LV does not supply this solution (see Table 10).

5.17 Cleanup Standard

This solution contains $^{37}Cl_4$-2378-TCDD at a concentration of 5 ng/uL (5 ug/mL) in tridecane (or nonane) and is added to all sample extracts prior to cleanup. The solution may be added at this concentration or

analysis, as dictated by the results of the continuing calibration (see Section 7.4). The continuing calibration is required at the beginning of each 12-hour time period during which samples are analyzed.

Samples shall not be analyzed until acceptable descriptor switching times, chromatographic resolution, and calibrations, as described in Sections 7.1, 7.2, 7.3 and 7.4, are achieved and documented. The sequence of analyses is shown in Table 13.

7.1 Window Defining Mix

The window defining mix shall be analyzed before any calibration standards in order to evaluate the descriptor switching times. The commercially available mix (see Section 5.15) contains the first and last eluting isomers in each homologue. Mixes are available for various columns. The mix for the DB-5 (or equivalent) column may not be appropriate for the SP-2331 or other columns.

The ions in each of the four recommended descriptors are arranged so that there is overlap between the descriptors. The ions for the TCDD, TCDF, PeCDD and PeCDF isomers are in the first descriptor, the ions for the PeCDD, PeCDF, HxCDD and HxCDF isomers are in the second descriptor, the ions for the HxCDD, HxCDF, HpCDD and HpCDF isomers are in the third descriptor, and the ions for the HpCDD, HpCDF, OCDD and OCDF isomers are in the fourth descriptor.

The descriptor switching times are set such that the isomers that elute from the GC during a given retention time window will also be those isomers for which the ions are monitored. For the homologues that overlap between descriptors, the laboratory may use discretion in setting the switching times. However, do not set descriptor switching times such that a change in descriptors occurs at or near the expected retention time of any of the 2,3,7,8-substituted isomers.

The window defining mix need not contain any of the labeled internal or recovery standards, as no quantitative measurements are based on this mixture. However, these standards and other isomers may be added to the mixture listed in Table 7 at the discretion of the laboratory, so long as the additional contents of the mixture are clearly specified in every SDG Narrative.

7.1.1 Analyze a 2 uL aliquot of the window defining mix, using the GC column conditions in Table 1.

7.1.2 Adjust the descriptor switching times and the GC column conditions as needed to ensure that the isomers elute in the appropriate ion descriptors (see Table 7).

7.1.3 The window defining mix must be analyzed at the following frequency:

7.1.3.1 Before initial calibration on each instrument and GC column used for analysis.

7.2.3 GC Resolution Criteria for SP-2331 or Equivalent Column. The chromatographic peak separation between unlabeled 2378-TCDD and the peaks representing all other unlabeled TCDD isomers shall be resolved with a valley of $\leq$ 25 percent, where:

Valley = (x/y)(100).
y = the peak height of any TCDD isomer.
x = the distinction from the baseline to the bottom of the valley between adjacent peaks, measured as shown in Figure 5.

The resolution criteria must be evaluated using measurements made on the selected ion current profile (SICP) for the appropriate ions for each isomer. Measurements are not made from total ion current profiles.

Further analyses may not proceed until the GC resolution criteria have been met.

7.2.4 If the laboratory uses a GC column other than those specified here, the laboratory must ensure that the isomers eluting closest to 2378-TCDD on that column are used to evaluate GC column resolution. The chromatographic peak separation between unlabeled 2378-TCDD and the peaks representing all other unlabeled TCDD isomers shall be resolved with a valley of $\leq$ 25 percent.

7.2.5 Analysis on a single GC column (as opposed to situations requiring a second column confirmation) is acceptable if the required separation of all of the 2,3,7,8-substituted isomers is demonstrated and the resolution criteria for <u>both</u> the DB-5 and SP-2331 (or equivalent) columns are met (see Paragraphs 7.3.2.1 and 7.2.3).

7.3 Initial Calibration

Once the window defining mix has been analyzed and the descriptor switching times have been verified (and after the analysis of the column performance solution if using a GC column other than DB-5), the five concentration calibration solutions (CC1-CC5), described in Table 3, shall be analyzed prior to any sample analysis. The CC1, CC2, CC4 and CC5 solutions shall be used as provided by EPA. The CC3 solution is prepared by combining CC4 solution, the supplemental calibration solution, and the internal, cleanup, and recovery standard solutions as described in Paragraph 7.4.1.

7.3.1 Analyze a 2 uL (see Paragraph 4.1.1) aliquot of each of the five concentration calibration solutions, beginning with CC3 solution (see Paragraph 7.4.1). The following MS/DS conditions shall be used:

1234-TCDD and $^{13}C_{12}$-123678-HxCDD, shall not change by more than 10 seconds between the initial CC3 analysis and the analysis of any other standard.

7.3.2.4 MS Sensitivity. For all calibration solutions, including the CC1 solution, the signal-to-noise ratio (S/N) must be greater than 2.5 for the unlabeled PCDD/PCDF ions, and greater than 10 for the internal standard and recovery standard ions.

7.3.3 Calculate the relative response factors (RRFs) for the 17 unlabeled target analytes relative to their appropriate internal standards (RRF_n) (see Table 8), according to the formulae below. For the seven unlabeled analytes and the $^{37}Cl_4$-2378-TCDD cleanup standard that are found only in the CC3 solution, only one RRF is calculated for each analyte. For the other 10 unlabeled analytes, calculate the RRF of each analyte in each calibration standard.

Calculate the RRFs for the five labeled internal standards and the cleanup standard relative to the appropriate recovery standard (RRF_{is}) (see Table 8), in each calibration standard, according to the following formulae:

$$RRF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

$$RRF_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

where:

A_n^1 and A_n^2 = integrated areas of the two quantitation ions of the isomer of interest (Table 5).

A_{is}^1 and A_{is}^2 = integrated areas of the two quantitation ions of the appropriate internal standard (Table 5).

A_{rs}^1 and A_{rs}^2 = integrated areas of the two quantitation ions of the appropriate recovery standard (Table 5).

Q_n = quantity of unlabeled PCDD/PCDF analyte injected (ng).

Q_{is} = quantity of appropriate internal standard injected (ng).

Q_{rs} = quantity of appropriate recovery standard injected (ng).

For quantitations involving the use of peak heights instead of peak areas, see Section 11.4.

7.3.7 If any of the requirements listed in Paragraphs 7.3.2 or 7.3.5 are not met, the Contractor is responsible for taking corrective action before sample analyses are performed. The following suggestions may be useful.

7.3.7.1 Check and adjust the GC and/or MS operating conditions.

7.3.7.2 Replace the GC column.

7.3.7.3 Adjust the MS for greater or lesser resolution using FC-43 (see Section 6).

7.3.7.4 Recalibrate the mass scale.

Once the corrective actions have been completed, the Contractor must perform a new initial calibration that does meet all the QC requirements, beginning with analysis of the window defining mix, before sample analyses may proceed.

7.4 Continuing Calibration

The continuing calibration consists of two parts: evaluation of the chromatographic resolution and verification of the RRF values to be used for quantitation. At the beginning of each 12-hour period, the chromatographic resolution is verified in the same fashion as in the initial calibration: through the analysis of the CC3 solution on the DB-5 (or equivalent) column or through the analysis of the column performance solution on the SP-2331 (or equivalent) column.

NOTE: The 12-hour time period is defined as beginning with the injection of the CC3 solution on the DB-5 (or equivalent) column or the injection of the column performance solution on the SP-2331 (or equivalent) column. The 12-hour period continues until 12:00 hours have elapsed according to the system clock. To be included in a given 12-hour time period, a sample or standard must be <u>injected</u> within 12:00 hours of the CC3 solution or the column performance solution.

7.4.1 Prepare the CC3 solution by combining the following volumes of the solutions listed in Section 5:

500 uL	CC4 Solution
125 uL	Supplemental Calibration Solution
50 uL	Internal Standard Solution
50 uL	Recovery Standard Solution
50 uL	Cleanup Standard Solution
225 uL	Tridecane (or nonane)

to yield a final volume of 1.0 mL at the concentrations specified for the CC3 solution in Table 3.

calibration has been performed and documented according to th following criteria: GC resolution, ion abundance ratios, retention times, instrument sensitivity, and response factors.

7.4.6.1 GC Column Resolution Criteria. The chromatographic resolution on the DB-5 (or equivalent) column must meet the QC criteria in Paragraph 7.3.2.1. The chromatographic resolution on the SP-2331 (or equivalent) column must meet the QC criteria in Paragraph 7.2.3. In addition, the chromatographic peak separation between the 123478-HxCDD and the 123678-HxCDD in the CC3 solution shall be resolved with a valley of ≤ 50 percent.

7.4.6.2 Ion Abundance Criteria. The relative ion abundances listed in Table 6 shall be met for all PCDD/PCDF peaks, including the labeled internal and recovery standards.

7.4.6.3 Instrument Sensitivity Criteria. For the CC3 solution, the S/N ratio shall be greater than 2.5 for the unlabeled PCDD/PCDF ions, and greater than 10.0 for the labeled internal and recovery standards.

7.4.6.4 Response Factor Criteria. The measured RRFs of each analyte and internal standard in the CC3 solution must be within ± 30.0 percent of the mean RRFs established during initial calibration for the EPA-supplied standards and within ± 30.0 percent of the single point RRFs established during initial calibration for the supplemental calibration standards and the cleanup standards.

$$\% \text{ Difference} = \frac{(RRF_i - RRF_c)}{RRF_i} \times 100$$

where:

RRF_i = Relative response factor established during initial calibration.

RRF_c = Relative response factor established during continuing calibration.

7.4.7 If any of the criteria listed in Paragraph 7.4.6 are not met, the Contractor must take corrective actions and reanalyze the continuing calibration standard (CC3). If the criteria in Paragraph 7.4.6 <u>are</u> met after the corrective action, then sample analysis may begin, as described in Section 10.

If the criteria in Paragraph 7.4.6 are <u>not</u> met after the corrective action, then the Contractor must perform a new initial calibration, beginning with the analysis of the window

7.5.4.3.2 Neither 2378-TCDD or 2378-TCDF were detected, even if other PCDDs or PCDFs were detected.

7.5.4.3.3 Any 2,3,7,8-substituted PCDD or PCDF is reported as an Estimated Maximum Possible Concentration (see Section 15.7).

These reanalyses are necessary because poor S/N ratios indicate a loss of sensitivity that could lead to false negative results, underestimation of concentrations, or could cause ion abundance ratios to fall outside the QC limits.

8. Sample Homogenization, Preservation and Handling

8.1 Homogenization

Although sampling personnel will attempt to collect homogeneous samples, the Contractor shall examine each sample and determine if the sample needs phase separation or mixing. The extent to which phase separation or mixing is required will depend on the sample type.

The Contractor is responsible for taking a representative sample aliquot from the phase or phases to be analyzed. This responsibility entails efforts to make the sample phase as homogeneous as possible. Stirring is recommended when possible.

8.2 Sample Types

8.2.1 For the purpose of this method, a chemical waste sample includes the sample matrices of oils, oily sludge, stillbottom, oil-laced soil, and surface water heavily contaminated with any of the above matrices. The sample may contain particulates and an obvious non-aqueous liquid phase.

8.2.2 For the purpose of this method, a soil/sediment sample is defined as a single phase solid system composed of soil or sediment. The sample may contain stones and vegetation, but should not contain an obvious aqueous or non-aqueous liquid phase.

CAUTION: Finely divided soils contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing the analytes. Such samples should be handled in a confined environment (e.g., a closed hood or a glove box).

8.2.3 For the purpose of this method, a water sample is defined as a single phase system, the primary component of which is water.

8.4.6 If a water sample does not contain significant amounts of suspended particulates, homogenize the sample by carefully shaking the capped sample bottle.

8.4.7 Centrifugation. If centrifugation of a sample is necessary, place the entire sample in a suitable centrifuge bottle(s) with a 250-300 mL capacity, and centrifuge for 30 minutes at 400 x G. Decant the liquid phase into a clean container. Remove the solid phase by careful pouring or using a clean spatula or glass rod. Proceed with the analysis of the appropriate phase or phases.

CAUTION: A phase that is not analyzed may contain PCDDs/PCDFs and should be handled and disposed of appropriately.

9. Extraction Procedures

Four types of extraction procedures are employed in these analyses depending on the sample matrix. Chemical waste samples are extracted by refluxing with a Dean Stark water separator. Fly ash samples and soil/sediment samples are extracted in a combination of a Soxhlet extractor and a Dean Stark water separator. Water samples are filtered and then the filtrate is extracted using either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. The filtered particulates are extracted in a combination of a Soxhlet extractor and a Dean Stark water separator.

9.1 Chemical Waste Sample Extraction

9.1.1 Assemble a flask (50 mL or 125 mL, see below), a Dean Stark trap, and a condenser, and preextract with toluene for three hours. Preextraction will ensure that the glassware is as clean as possible and minimize cross-contamination problems. Discard the used toluene, or pool it for later analysis to verify the cleanliness of the glassware.

9.1.2 Oily Sludge/Wet Fuel Oil. Weigh about 1 g of sample to two decimal places into a tared preextracted 125-mL flask. Add 1 mL of the acetone-diluted internal standard solution (see Section 5.12) to the sample in the flask. Attach the preextracted Dean Stark water separator and condenser to the flask, and extract the sample by refluxing it with 50 mL of toluene for at least three hours.

Continue refluxing the sample until all the water has been removed. Cool the sample, and filter the toluene extract through a rinsed glass fiber filter into a 100 mL round bottom flask. Rinse the filter with 10 mL of toluene, and combine the extract and rinsate. Concentrate the combined solution to approximately 10 mL using a rotary evaporator as described in Section 9.6.

9.1.3 Stillbottom/Oil. Weigh about 1 g of sample to two decimal places into a tared preextracted 50-mL flask. Add 1 mL of the

The following procedures apply to all uses of the SDS apparatus for extracting matrices covered by this protocol.

NOTE: It may be necessary to wrap portions of the SDS apparatus with aluminum foil or glass wool to obtain proper operation.

9.2.1 Refer to Section 4.5 for detailed instructions on cleaning glassware such as the SDS apparatus. In particular, do <u>not</u> bake the components of the SDS apparatus as part of routine c ing, as repeated baking of glassware can cause active s on the glass surface that will adsorb PCDDs/PCDFs and other analytes. All glass parts of the SDS apparatus, including the thimbles, <u>must</u> be preextracted with toluene for approximately three hours immediately prior to use. Preextraction will ensure that the glassware is as clean as possible and minimize cross-contamination problems. Discard the used toluene, or pool it for later analysis to verify the cleanliness of the glassware.

9.2.2 The extraction of soil/sediment, fly ash, and particulates from water samples will require the use of a Soxhlet thimble. Prior to preextraction, prepare the thimble by adding 5 g of 70/230 mesh silica gel to the thimble to produce a thin layer in the bottom of the thimble. This layer will trap fine particles in the thimble. Add 80-100 g of quartz sand on top of the silica gel, and place the thimble in the extractor.

9.2.3 After preextraction for three hours, allow the apparatus to cool and remove the thimble. Mix the appropriate weight of sample with the sand in the thimble, being careful not to disturb the silica gel layer.

If the sample aliquot to be extracted contains large lumps or is otherwise not easily mixed in the thimble, the sand and sample may be mixed in another container. Transfer approximately 2/3 of the sand from the thimble to a clean container, being careful not to disturb the silica gel layer when transferring the sand. Thoroughly mix the sand and the sample with a clean spatula, and transfer the sand/sample mixture to the thimble.

If a sample with particularly high moisture content is to be extracted, it may be helpful to leave a small conical depression in the material in the thimble. This procedure will allow the water to drain through the thimble more quickly during the early hours of the extraction. As the moisture is removed during the first few hours of extraction, the depression will collapse, and the sample will be uniformly extracted.

The following procedures apply to all uses of the SDS apparatus for extracting matrices covered by this protocol.

NOTE: It may be necessary to wrap portions of the SDS apparatus with aluminum foil or glass wool to obtain proper operation.

9.2.1 Refer to Section 4.5 for detailed instructions on cleaning glassware such as the SDS apparatus. In particular, do <u>not</u> bake the components of the SDS apparatus as part of routine cleaning, as repeated baking of glassware can cause active sites on the glass surface that will adsorb PCDDs/PCDFs and other analytes. All glass parts of the SDS apparatus, including the thimbles, <u>must</u> be preextracted with toluene for approximately three hours immediately prior to use. Preextraction will ensure that the glassware is as clean as possible and minimize cross-contamination problems. Discard the used toluene, or pool it for later analysis to verify the cleanliness of the glassware.

9.2.2 The extraction of soil/sediment, fly ash, and particulates from water samples will require the use of a Soxhlet thimble. Prior to preextraction, prepare the thimble by adding 5 g of 70/230 mesh silica gel to the thimble to produce a thin layer in the bottom of the thimble. This layer will trap fine particles in the thimble. Add 80-100 g of quartz sand on top of the silica gel, and place the thimble in the extractor.

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If the sample aliquot to be extracted contains large lumps or is otherwise not easily mixed in the thimble, the sand and sample may be mixed in another container. Transfer approximately 2/3 of the sand from the thimble to a clean container, being careful not to disturb the silica gel layer when transferring the sand. Thoroughly mix the sand and the sample and a clean spatula, and transfer the sand/sample mixture to the thimble.

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extraction procedure. For extremely wet samples, the Dean Stark trap may need to be drained one or more times during the 16-hour extraction. Collect the water from the trap, and measure its volume to the nearest 0.1 mL. Assume a density of 1.0 g/mL, and calculate the percent solids content according to the formula below:

$$\text{Percent Solids} = \frac{(\text{Wet weight of sample} - \text{Weight of water})}{\text{Wet weight of sample}} \times 100$$

9.4.4 Concentrate this extract as described in Section 9.6.

9.4.5 Prepare a sample aliquot for the duplicate sample analysis and a sample aliquot for the spiked sample analysis, using the procedures in Sections 13 and 14 and at the frequency given in those sections.

9.5 Water Sample Extraction

9.5.1 Allow the sample to come to ambient temperature, then mark the water meniscus on the side of the 1-L sample bottle for determination of the exact sample volume. Add 1 mL of the acetone-diluted internal standard solution (see Section 5.12) to the sample bottle. Cap the bottle, and mix the sample by gently shaking for 30 seconds. Filter the sample through a 0.45 micron filter that has been rinsed with toluene.

NOTE: Reagent water used as a blank must also be filtered in a similar fashion and subjected to the same cleanup and analysis as the water samples.

If the total dissolved and suspended solids contents are too much to filter through the 0.45 micron filter, centrifuge the sample, decant, and then filter the aqueous phase (see Paragraph 8.4.7). Combine the solids from the centrifuge bottle(s), the particulate on the filter and the filter itself, and proceed with the SDS extraction in Paragraph 9.5.4.

9.5.2 The filtered aqueous sample is poured into a 2-L separatory funnel. Add 60 mL methylene chloride to the sample bottle, seal, and shake for 60 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the Contractor shall employ mechanical techniques to complete the phase separation (i.e., glass stirring rod). Drain the methylene chloride extract into a 500-mL KD concentrator (mounted with a 10-mL concentrator tube) by passing the extract through a funnel packed with a glass wool plug and half-filled with anhydrous sodium sulfate. Extract the water sample two more times using 60 mL of fresh methylene

aqueous filtrate and the filtered particulates must be combined prior to cleanup. Two procedures may be used for macro-concentration, Kuderna-Danish (K-D) or rotary evaporator. Concentration of toluene by K-D requires the use of a heating mantle, as toluene boils above the temperature of a water bath. The two procedures are described in general terms below.

9.6.1 Concentration by K-D

9.6.1.1 Add one or two clean boiling chips to the round bottom flask from the SDS extractor or the reflux flask. Attach a three-ball macro Snyder column.

9.6.1.2 Pre-wet the column by adding approximately 1 mL of toluene through the top. Place the round bottom flask in a heating mantle and apply heat as required to complete the concentration in 15-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood.

9.6.2 Concentration by Rotary Evaporator

9.6.2.1 Assemble the rotary evaporator according to manufacturer's instructions, and warm the water bath to 45°C. On a daily basis, preclean the rotary evaporator by concentrating 100 mL of clean extraction solvent through the system. Archive both the concentrated solvent and the solvent in the catch flask for contamination check if necessary. Between samples, three 2-3 mL aliquots of toluene should be rinsed down the feed tube into a waste beaker.

9.6.2.2 Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

9.6.2.3 Lower the flask into the water bath and adjust the speed of rotation and the temperature as required to complete the concentration in 15-20 minutes. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

9.6.2.4 When the liquid in the concentration flask has reached an apparent volume of 2 mL, remove the flask from the water bath, and stop the rotation. Slowly and carefully, admit air into the system. Be sure

9.6.5.3 Concentrate the combined extract to approximately 10 mL (the volume of the toluene) by either K-D or rotary evaporator.

9.6.5.4 Transfer the concentrated combined extract to a 125 mL separatory funnel. Rinse the concentrator with three 5 mL volumes of hexane, and add each rinse to the separatory funnel. Proceed with acid-base washing treatment per Section 9.7.

9.7 Extract Cleanup Procedures (All Matrices)

9.7.1 Prior to cleanup, all extracts are spiked with the $^{37}Cl_4$-2378-TCDD cleanup standard (Section 5.17). The recovery of this standard is used to monitor the efficiency of the cleanup procedures. Spike 5 uL of the cleanup standard (or a larger volume of diluted solution containing 25 ng of $^{37}Cl_4$-2378-TCDD) into each separatory funnel containing an extract, resulting in a concentration of 0.25 ng/uL in the final extract analyzed by GC/MS.

9.7.2 Partition the concentrated extract against 40 mL of concentrated sulfuric acid. Shake for two minutes. Remove and discard the acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer. (Perform acid washings a maximum of four times.)

CAUTION: Concentrated sulfuric acid is hazardous and should be handled with care.

9.7.3 Partition the concentrated extract against 40 mL of 5 percent (w/v) sodium chloride. Shake for two minutes. Remove and discard the aqueous layer (bottom).

9.7.4 Partition the concentrated extract against 40 mL of 20 percent (w/v) potassium hydroxide (KOH). Shake for two minutes. Remove and discard the base layer (bottom). Repeat the base washes until color is not visible in the bottom layer (perform base washes a maximum of four times). Strong base (KOH) is known to degrade certain PCDDs/PCDFs; therefore, contact time should be minimized.

9.7.5 Partition the concentrated extract against 40 mL of 5 percent (w/v) sodium chloride. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the organic layer by pouring it through a funnel containing a rinsed filter half-filled with anhydrous sodium sulfate. Collect the extract in an appropriate size (100-250 mL) round bottom flask. Wash the separatory funnel with two 15-mL portions of hexane, pour through the funnel and combine the extracts. Concentrate the extracts to 1.0 mL using the procedures described in Section 9.8.

9.9.3 Add hexane to each column until the packing is free of air bubbles. A small positive pressure (5 psi) of clean dry nitrogen may be used if needed. Check the columns for channeling. If channeling is present, discard the column.

CAUTION: Do not tap a wetted column.

9.9.4 Assemble the two columns such that the eluate from Column 1 (silica gel) drains directly into Column 2 (alumina).

9.9.5 Apply the hexane solution from Paragraph 9.8.4 to the top of the silica gel column. Rinse the vial with enough hexane (1-2 mL) to complete the quantitative transfer of the sample to the surface of the silica.

9.9.6 Using 90 mL of hexane, elute the extract from Column 1 directly onto Column 2 which contains the alumina.

CAUTION: Do not allow the alumina column to run dry.

9.9.7 Add 20 mL of hexane to Column 2, and elute until the hexane level is just below the top of the alumina. Do not discard the eluted hexane, but collect in a separate flask and store it for later use, as it may be useful in determining where the labeled analytes are being lost if recoveries are less than 50 percent.

9.9.8 Add 20 mL of 20% methylene chloride/80% hexane (v/v) to Column 2 and collect the eluate.

9.9.9 Concentrate the extract to approximately 2 to 3 mL using the procedures in Section 9.8.

CAUTION: Do not concentrate the eluate to dryness. The sample is now ready to be transferred to the carbon column.

9.10 Carbon Column Chromatographic Procedure

9.10.1 Thoroughly mix 5.35 g active carbon AX-21 and 62.0 g Celite 545 to produce a 7.9% w/w mixture. Activate the mixture at 130°C for six hours, and store in a desiccator.

Check each new batch of the Carbon/Celite and maintain the results from the analyses for examination during EPA on-site evaluations. To accomplish this, add 50 uL of the continuing calibration solution to 950 uL of hexane. Process the spiked solution in the same manner as a sample extract (Paragraphs 9.10.2 through 9.10.6). Concentrate the continuing calibration solution to 50 uL and proceed with Section 9.10. If the recovery of any of the analytes is less than 80%, this batch of Carbon/Celite mixture may not be used.

9.10.2 Prepare a 4-inch glass column by cutting off each end of a 10-mL disposable serological pipet. Fire polish both ends and

The final extract volume should be 100 uL of tridecane (or nonane). Seal the vial and store the sample extract in the dark at ambient temperature until just prior to GC/MS analysis.

10. GC/MS Analysis

10.1 Remove the extract of the sample or blank from storage. Gently swirl the solvent on the lower portion of the vial to ensure complete dissolution of the PCDDs/PCDFs.

10.2 Transfer a 50 uL aliquot of the extract to a 0.3 mL vial, and add sufficient recovery standard solution to yield a concentration of 0.5 ng/uL in a 50 uL volume. Reduce the volume of the extract back down to 50 uL using a gentle stream of dry nitrogen.

Inject a 2 uL aliquot of the extract into the GC/MS instrument (see Paragraph 4.1.1). Reseal the vial from Paragraph 9.11.2, containing the original concentrated extract.

10.3 Analyze the extract by GC/MS, and monitor all of the ions listed in Table 7. The same MS parameters used to analyze the calibration solutions shall be used for the sample extracts.

10.4 Dilutions

10.4.1 If the concentration of any PCDD/PCDF in the sample has exceeded the calibration range or the detector has been saturated, a dilution shall be performed.

An appropriate dilution will result in the largest peak in the diluted sample falling between the mid-point and high-point of the calibration range.

10.4.2 Dilutions are performed using an aliquot of the original extract, of which approximately 50 uL remain from Paragraph 9.11.2. Remove an appropriate size aliquot from the vial and add it to a sufficient volume of tridecane (or nonane) in a clean 0.3 mL conical vial. Add sufficient recovery standard solution to yield a concentration of 0.5 ng/uL (1.0 ng/uL ^{13}C-OCDD). Reduce the volume of the extract back down to 50 uL using a gentle stream of dry nitrogen.

10.4.3 The dilution factor is defined as the total volume of the sample aliquot and clean solvent divided by the volume of the sample aliquot that was diluted.

10.4.4 Inject 2 uL of the diluted sample extract into the GC/MS, and analyze according to Section 10.3.

10.4.5 Diluted samples in which the MS response of any internal standard is $\geq$ 10% of the MS response of that internal standard in the most recent continuing calibration standard are quantified using the internal standards.

for the analytes must maximize simultaneously (± 2 seconds). This requirement also applies to the internal standards and recovery standards. For the cleanup standard, only one ion is monitored.

11.3 Signal-To-Noise Ratio

The integrated ion current for each analyte ion listed in Table 5 must be at least 2.5 times background noise and must not have saturated the detector. The internal standard ions must be at least 10.0 times background noise and must not have saturated the detector. However, if the M-[COCl]+ ion does not meet the 2.5 times S/N requirement but meets all the other criteria listed in Section 11 and, in the judgement of the GC/MS Interpretation Specialist the peak is a PCDD/PCDF, the peak may be reported as positive and the data flagged on Form I. See the instructions in Exhibit B for Form I.

11.4 Ion Abundance Ratios

The relative ion abundance criteria listed in Table 6 for native analytes and internal standards must be met using peak areas to calculate ratios.

11.4.1 If interferences are present and ion abundance ratios are not met using peaks areas, but all other qualitative identification criteria <u>are</u> met (RT, S/N, presence of all three ions), then the Contractor may use peak heights to evaluate the ion ratio.

11.4.2 If, in the judgement of the GC/MS Interpretation Specialist the peak is a PCDD/PCDF, then report the ion abundance ratios determined using peak heights, <u>quantitate</u> the peaks using peak heights rather than areas for both the target analyte and the internal standard, and flag the data on Form I.

11.5 Polychlorinated Diphenyl Ether (PCDPE) Interferences

The identification of a GC peak as a PCDF cannot be made if a signal having S/N greater than 2.5 is detected at the same retention time (± 2 seconds) in the corresponding PCDPE channel (see Table 5). If a PCDPE is detected, it shall be documented in the SDG Narrative, and an Estimated Maximum Possible Concentration (EMPC) shall be calculated for this GC peak according to Section 15.7, regardless of the ion abundance ratio, and reported on Form I.

12. <u>Method Blanks</u>

12.1 A minimum of one blank per matrix shall be analyzed with each SDG. If samples of the same matrix are extracted in different episodes (i.e., different shifts or days), one blank per matrix must be prepared for each episode. When water samples in a SDG are extracted using both the separatory funnel and continuous liquid-liquid extraction procedures, at least one blank must be prepared by each procedure.

13.1 Prepare the spiked sample aliquot by taking the same weight (or volume) of the representative matrix as is indicated in Sections 9.1 to 9.5 and placing it in a clean container of suitable size.

13.2 Add 1.0 mL of the spiking solution in Section 5.18 and Table 11 to the aliquot. Manually mix the sample to distribute the spiking solution, and let the aliquot equilibrate for one hour.

13.3 Prepare and extract the spiked sample aliquot in the same fashion as is used for field samples, and carry the aliquot through the entire analytical procedure including cleanup.

13.4 Calculate the concentration of each analyte according to the procedures in Section 15.

13.5 Calculate the recovery of each spiked analyte, using the following equation:

$$R_{spike} = \frac{\text{Amount found - Amount in unspiked sample}}{\text{Amount spiked}} \times 100$$

where the recovery (R) is expressed as a percentage

13.6 The recovery of each spiked analyte must be in the range of 50-150 percent. If the recovery of any analyte falls outside this range, the laboratory must recheck all calculations, and confirm that the spiking solutions were added and were at the correct concentrations, but no further action is necessary by the laboratory at this time. Recovery limits for these analytes will be developed at a later date.

14. Duplicate Sample Analysis

In order to provide data on the precision of the analytical method, the laboratory is required to prepare and analyze a duplicate of one sample for each matrix being analyzed. For each SDG, the laboratory must prepare a duplicate sample for all of the following matrix types that occur in the SDG:

- Water
- Soil/Sediment
- Chemical Waste
- Fly Ash

If a matrix is not represented in a SDG, then no duplicate sample is required for that matrix. If the Region or samplers have identified a particular sample to be used for the duplicate, the laboratory must use an aliquot of that sample. If the Region or samplers have not identified a specific sample for use as the duplicate, then the laboratory may choose a sample from the SDG; however, the sample chosen must not be a sample identified by the Region as a field or trip blank.

W = weight of sample extracted, in grams.

V = volume of sample extracted, in liters.

Q_{is} = quantity (ng) of the appropriate internal standard added to the sample prior to extraction.

RRF_n = calculated relative response factor from continuing calibration (see Section 7.3).

For solids matrices, the units of ng/g that result from the formula above are equivalent to ug/Kg. Using isotope dilution techniques for quantitation, the concentration data are recovery corrected, and therefore, the volume of the final extract and the injection volume are implicit in the value of Q_{is}.

15.1.1 For homologues that contain only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD and TCDF), the RRF of the 2,3,7,8-substituted isomer from the continuing calibration (see Paragraph 7.3.2.3) will be used to quantitate both the 2,3,7,8-substituted isomers and the non-2,3,7,8-substituted isomers.

15.1.2 For homologues that contain more than one 2,3,7,8-substituted isomer (HxCDD, PeCDF, HxCDF and HpCDF), the RRF used to calculate the concentration of each 2,3,7,8-substituted isomers will be the RRF determined for that isomer during the continuing calibration (see Paragraph 7.3.2.3).

15.1.3 For homologues that contain one or more non-2,3,7,8-substituted isomers, the RRF used to calculate the concentration of these isomers will be the lowest of the RRFs determined during the continuing calibration (see Paragraph 7.3.2.3) for the 2,3,7,8-substituted isomers in that homologue. This RRF will yield the highest possible concentration for the non-2,3,7,8-substituted isomers.

NOTE: The relative response factors of given isomers within any homologue may be different. However, for the purposes of these calculations, it will be assumed that every non-2,3,7,8-substituted isomer for a given homologue has the same relative response factor. In order to minimize the effect of this assumption on risk assessment, the 2,3,7,8-substituted isomer with the lowest RRF was chosen as representative of each homologue. All relative response factor calculations for the non-2378-substituted isomers in a given homologue are based on that isomer.

15.2 In addition to the concentrations of specific isomers, the total homologue concentrations are also reported. Calculate the total concentration of each homologue of PCDDs/PCDFs as follows:

Total concentration = sum of the concentrations of every positively identified isomer of each PCDD/PCDF homologue.

therefore, only one peak area will be used in the numerator of this formula. Use both peak areas of the $^{13}C_{12}$-1234-TCDD recovery standard in the denominator.

15.5.1 The $^{13}C_{12}$-1234-TCDD is used to quantitate the tetra internal standards and the cleanup standard, and $^{13}C_{12}$-123789-HxCDD is used to quantitate the HxCDD, HpCDF and OCDD internal standards (see Table 8).

15.5.2 If the original sample, prior to any dilutions, has any internal standard with a percent recovery of less than 25% or greater than 150%, reextraction and reanalysis of that sample is required (see Section 17).

15.6 Sample Specific Estimated Detection Limit

The sample specific Estimated Detection Limit (EDL) is the estimate made by the laboratory of the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, etc.

An EDL is calculated for each 2,3,7,8-substituted isomer that is not identified, regardless of whether or not non-2,3,7,8-substituted isomers in that homologue are present. The EDL is also calculated for 2,3,7,8-substituted isomers giving responses for both the quantitation ions that are less than 2.5 times the background level.

Use the formulae below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF. The background level (H_x) is determined by measuring the height of the noise at the expected retention times of both the quantitation ions of the particular 2,3,7,8-substituted isomer. The expected retention time is determined from the most recent analysis of the CC3 standard on the same GC/MS system.

ALL MATRICES OTHER THAN WATER

$$\text{EDL (ug/kg)} = \frac{2.5 \times Q_{is} \times (H_x^1 + H_x^2) \times D}{W \times (H_{is}^1 + H_{is}^2) \times RRF_n}$$

WATER

$$\text{EDL (ng/L)} = \frac{2.5 \times Q_{is} \times (H_x^1 + H_x^2) \times D}{V \times (H_{is}^1 + H_{is}^2) \times RRF_n}$$

Where:

H_x^1 and H_x^2 = Peak heights of the noise for both of the quantitation ions of the 2,3,7,8-substituted isomer of interest.

The exclusion of homologues such as mono-, di-, tri- and the non-2,3,7,8-substituted isomers in the higher homologues does not mea they are not toxic. Their toxicity, as estimated at this time, : less than the toxicity of the compounds listed in Table 11. Hence, only the 2,3,7,8-substituted isomers are included in the TEF calculations. The procedure for calculating the 2378-TCDD toxic equivalence cited above is not claimed by the CDWG to be based on a thoroughly established scientific foundation. Rather, the procedure represents a "Consensus Recommendation on Science Policy."

When calculating the 2378-TCDD toxicity equivalence of a sample, the Contractor shall include only those 2,3,7,8-substituted isomers that were detected in the sample <u>and</u> met all of the qualitative identification criteria in Section 11. Do <u>not</u> include EMPC or EDL values in the TEF calculations. Further instructions regarding the calculation of the 2378-TCDD toxicity equivalence may be found in Exhibit B.

The 2378-TCDD toxicity equivalence of a sample is used in Sections 16 and 17 of this procedure to determine when second column confirmation or reextractions and reanalyses may be required.

16. <u>Isomer Specificity</u>

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60 m DB-5 column alone. Historically, problems have been associated with the separation of 2378-TCDD from 1237-TCDD and 1268-TCDD, and separation of 2378-TCDF from 2347-TCDF. Because of the toxicologic concern associated with 2378-TCDD and 2378-TCDF, additional analyses may be required for some samples, as described below.

16.1 If the toxicity equivalence calculated in Section 15 is greater than 0.7 ppb (soil/sediment or fly ash), 7 ppb (chemical waste), or 7 ppt (aqueous), better isomer specificity is required than can be achieved on the DB-5 column. The Contractor may utilize either of the two options listed below to achieve adequate isomer specificity.

16.1.1 The sample extract may be reanalyzed on a 60 m SP-2330 or SP-2331 (or equivalent) GC column in order to achieve better GC resolution, and therefore, better identification and quantitation of the individual 2,3,7,8-substituted isomers.

16.1.2 The sample extract may be analyzed on a single GC column capable of resolving all 2,3,7,8-substituted PCDDs/PCDFs from other isomers, but not necessarily resolving all the non-2,3,7,8-substituted isomers from one another.

Regardless of GC column used, for a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF isomer, it must meet the ion abundance, signal-to-noise, and retention time criteria listed in Section 11. In addition, when using any GC column other than those specified here (DB-5, SP-2330 or SP-2331), the Contractor shall clearly document, in the SDG Narrative, the elution order of all the analytes of interest on any such column.

limits, the Contractor must reanalyze the sample extract on a second GC column with different elution characteristics, as discussed in Section 16. No reextraction is required for such an analysis. This reanalysis is only billable if the same internal standard ion abundance ratios are outside the control limits on the second column, indicating matrix effects may have occurred.

17.1.4 If the absolute retention time of either the $^{13}C_{12}$-1234-TCDD or $^{13}C_{12}$-123789-HxCDD recovery standard in a sample extract shifts by greater than 10 seconds from the retention time of that standard in the continuing calibration standard, then the sample extract must be reanalyzed after the Contractor has investigated the cause of the retention time shift and taken corrective action. No reextraction is required for such an analysis. This reanalysis is only billable if the same recovery standard retention time shifts by greater than 10 seconds in the second analysis, indicating matrix effects may have occurred.

17.2 If the calculated concentration of the unlabeled PCDDs/PCDFs exceeded the initial calibration range, the sample extract shall be diluted and reanalyzed (see Section 10.4). Such sample dilutions are billable under the contract.

NOTE: Only one dilution shall be billable per sample and <u>only</u> as an additional analysis with no extraction.

17.3 The following sample reruns shall be performed at the Contractor's expense and shall <u>not</u> be billable under the terms of the contract.

17.3.1 All positive samples associated with a contaminated method blank <u>and</u> any samples which contain peaks that do not meet all of the qualitative identification criteria in Section 11 associated with a contaminated method blank must be reextracted and reanalyzed. Acceptable laboratory method blanks must not contain any chemical interference or electronic noise at the m/z of the specified unlabeled PCDD/PCDF ions that is greater than five percent of the signal of the appropriate internal standard quantitation ion. A peak that meets identification criteria in the method blank must not exceed two percent of the signal of the appropriate internal standard.

17.3.2 If the chromatographic peak separation between $^{13}C_{12}$-2378-TCDD and $^{13}C_{12}$-1234-TCDD is <u>not</u> resolved with a valley of $\leq$ 25% on the DB-5 (or equivalent) column, or 2378-TCDD is <u>not</u> resolved from the closest eluting isomer with a valley of $\leq$ 25% on the SP-2331 (or equivalent) column, then the Contractor shall adjust the GC/MS operating conditions and rerun the affected sample. This criterion applies to sample analyses. If this criterion is not met for a <u>calibration standard</u>, all associated samples must be rerun.

TABLE 1. SUGGESTED OPERATING CONDITIONS FOR A DB-5 (OR EQUIVALENT) COLUMN

Stationary Phase	DB-5 (or equivalent)
Film Thickness	0.25 um
Column Dimensions	60 m x 0.32 mm
Helium Linear Velocity	35 - 40 cm/sec at 240°C
Initial Temperature	170°C
Initial Time	10 minutes
Temperature Program	increase to 320°C at 8°/minute
Hold Time	until OCDF elutes
Total Time	40-45 minutes

TABLE 3. CONCENTRATION CALIBRATION SOLUTIONS

Analyte	CC1	CC2	CC3	CC4	CC5
2378-TCDD	0.1	0.25	0.5	1.0	2.0
2378-TCDF	0.1	0.25	0.5	1.0	2.0
12378-PeCDF	0.1	0.25	0.5	1.0	2.0
12378-PeCDD	0.1	0.25	0.5	1.0	2.0
*23478-PeCDF	---	---	0.5	---	---
*123478-HxCDF	---	---	1.25	---	---
123678-HxCDF	0.25	0.625	1.25	2.5	5.0
*123478-HxCDD	---	---	1.25	---	---
123678-HxCDD	0.25	0.625	1.25	2.5	5.0
*123789-HxCDD	---	---	1.25	---	---
*234678-HxCDF	---	---	1.25	---	---
*123789-HxCDF	---	---	1.25	---	---
*1234789-HpCDF	---	---	1.25	---	---
1234678-HpCDF	0.25	0.625	1.25	2.5	5.0
1234678-HpCDD	0.25	0.625	1.25	2.5	5.0
OCDD	0.5	1.25	2.5	5.0	10.0
OCDF	0.5	1.25	2.5	5.0	10.0
$^{13}C_{12}$-2378-TCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-2378-TCDF	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-123678-HxCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-1234678-HpCDF	1.0	1.0	1.0	1.0	1.0
$^{13}C_{12}$-OCDD	1.0	1.0	1.0	1.0	1.0
$^{13}C_{12}$-1234-TCDD	0.5	0.5	0.5	0.5	0.5
$^{13}C_{12}$-123789-HxCDD	0.5	0.5	0.5	0.5	0.5
$^{37}Cl_4$-2378-TCDD	---	---	0.25	---	---

All concentrations are in ng/uL.

*Supplemental commercial standard. Do not perform %RSD calculations on these analytes. (See Paragraph 7.4.1 for CC3 standard preparation.)

TABLE 5. IONS SPECIFIED FOR SELECTED ION MONITORING FOR PCDDs/PCDFs

Analyte	Quantitation Ions		$M-[COCl]^+$
TCDD	320	322	259
PeCDD	356	358	293
HxCDD	390	392	327
HpCDD	424	426	361
OCDD	458	460	395
TCDF	304	306	243
PeCDF	340	342	277
HxCDF	374	376	311
HpCDF	408	410	345
OCDF	442	444	379
Internal Standards			
$^{13}C_{12}$-2378-TCDD	332	334	---
$^{13}C_{12}$-123678-HxCDD	402	404	---
$^{13}C_{12}$-OCDD	470	472	---
$^{13}C_{12}$-2378-TCDF	316	318	---
$^{13}C_{12}$-1234678-HPCDF	420	422	---
Recovery Standards			
$^{13}C_{12}$-1234-TCDD	332	334	---
$^{13}C_{12}$-123789-HxCDD	402	404	---
Cleanup Standard			
^{37}C14-2378-TCDD	328	(1)	263
Polychlorinated diphenyl ethers			
HxCDPE	376	---	---
HpCDPE	410	---	---
OCDPE	446	---	---
NCDPE	480	---	---
DCDPE	514	---	---

(1) There is only one quantitation ion monitored for the cleanup standard.

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TABLE 5. IONS SPECIFIED FOR SELECTED ION MONITORING FOR PCDDs/PCDFs

Analyte	Quantitation Ions		$M-[COCl]^+$
TCDD	320	322	259
PeCDD	356	358	293
HxCDD	390	392	327
HpCDD	424	426	361
OCDD	458	460	395
TCDF	304	306	243
PeCDF	340	342	277
HxCDF	374	376	311
HpCDF	408	410	345
OCDF	442	444	379
Internal Standards			
$^{13}C_{12}$-2378-TCDD	332	334	---
$^{13}C_{12}$-123678-HxCDD	402	404	---
$^{13}C_{12}$-OCDD	470	472	---
$^{13}C_{12}$-2378-TCDF	316	318	---
$^{13}C_{12}$-1234678-HPCDF	420	422	---
Recovery Standards			
$^{13}C_{12}$-1234-TCDD	332	334	---
$^{13}C_{12}$-123789-HxCDD	402	404	---
Cleanup Standard			
^{37}C14-2378-TCDD	328	(1)	265
Polychlorinated diphenyl ethers			
HxCDPE	376	---	---
HpCDPE	410	---	---
OCDPE	446	---	---
NCDPE	480	---	---
DCDPE	514	---	---

(1) There is only one quantitation ion monitored for the cleanup standard.

TABLE 7. RECOMMENDED SELECTED ION MONITORING DESCRIPTORS

Descriptor 1	Descriptor 2	Descriptor 3	Descriptor 4
243	277	311	345
259	293	327	361
277	311	345	379
293	327	361	395
304	338	374	408
306	340	376	410
316	342	390	420
318	354	392	422
320	356	402	424
322	358	404	426
328	374	408	442
332	376	410	444
334	390	420	458
340	392	422	460
342	402	424	470
356	404	426	472
358	410	446	480
376	446	480	514

The ions at m/z 376 (HxCDPE), 410 (HpCDPE), 446 (OCDPE), 480 (NCDPE) and 514 (DCDPE) represent the polychlorinated diphenyl ethers.

The ions in each of the four recommended descriptors are arranged so that there is overlap between the descriptors. The ions for the TCDD, TCDF, PeCDD and PeCDF isomers are in the first descriptor, the ions for the PeCDD, PeCDF, HxCDD and HxCDF isomers are in the second descriptor, the ions for the HxCDD, HxCDF, HpCDD and HpCDF isomers are in the third descriptor, and the ions for the HpCDD, HpCDF, OCDD and OCDF isomers are in the fourth descriptor.

NOTE: The descriptors used by the laboratory must be documented, and this information must be available for examination during the EPA on-site evaluations.

TABLE 9. PCDD/PCDF ISOMERS IN THE WINDOW DEFINING MIX FOR A 60 M DB-5 (OR EQUIVALENT) COLUMN

Homologue	First Eluted	Last Eluted	Approximate Concentration
TCDD	1368-	1289-	0.5
TCDF	1368-	1289-	0.5
PeCDD	12479-	12389-	0.5
PeCDF	13468-	12389-	0.5
HxCDD	124679-	123467-	1.25
HxCDF	123468-	123489-	1.25
HpCDD	1234679-	1234678-	1.25
HpCDF	1234678-	1234789-	1.25

TABLE 11. MATRIX SPIKING SOLUTION

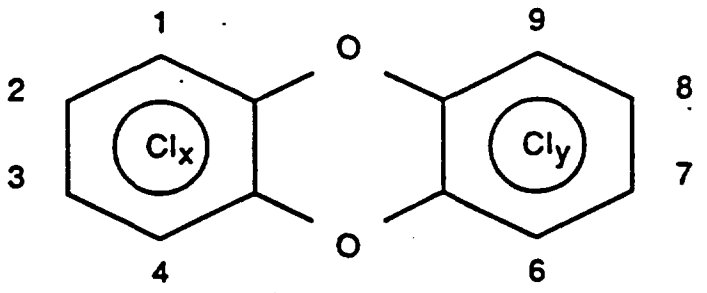
Analyte	Concentration (ng/uL)
2378-TCDD	2.5
2378-TCDF	2.5
12378-PeCDF	6.25
12378-PeCDD	6.25
123678-HxCDF	6.25
123678-HxCDD	6.25
1234678-HpCDF	6.25
1234678-HpCDD	6.25
OCDD	12.5
OCDF	12.5

This solution is prepared in tridecane (or nonane) and diluted with acetone prior to use (see Section 5.18).

TABLE 13. EXAMPLE ANALYTICAL SEQUENCES

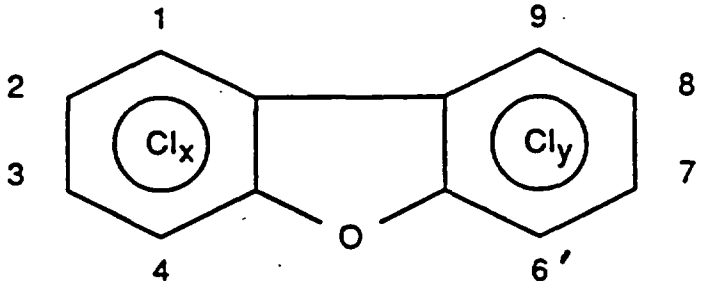
Time	Analysis
	Window Defining Mix
	Column Performance Solution (SP-2331)
Hour 0	CC3
	CC1 (Initial Calibration)
	CC2
	CC4
	CC5
	Blanks and Samples
	o
	o
	o
	o
Hour 12	CC1
	Column Performance Solution (SP-2331)
Hour 0	CC3
	Blanks and Samples
	o
	o
	o
	o
Hour 12	CC1
	Column Performance Solution (SP-2331)
Hour 0	CC3
	Blanks and Samples
	o
	o
	o
	etc.
	CC1 (whenever the sequence does end)

NOTE: Matrix spike and duplicate samples may be analyzed in place of any "sample" listed above.



Polychlorinated Dibenzo-*p*-Dioxin

where $x + y \leq 8$



Polychlorinated Dibenzofuran

Figure 2: General Structures of PCDDs and PCDFs

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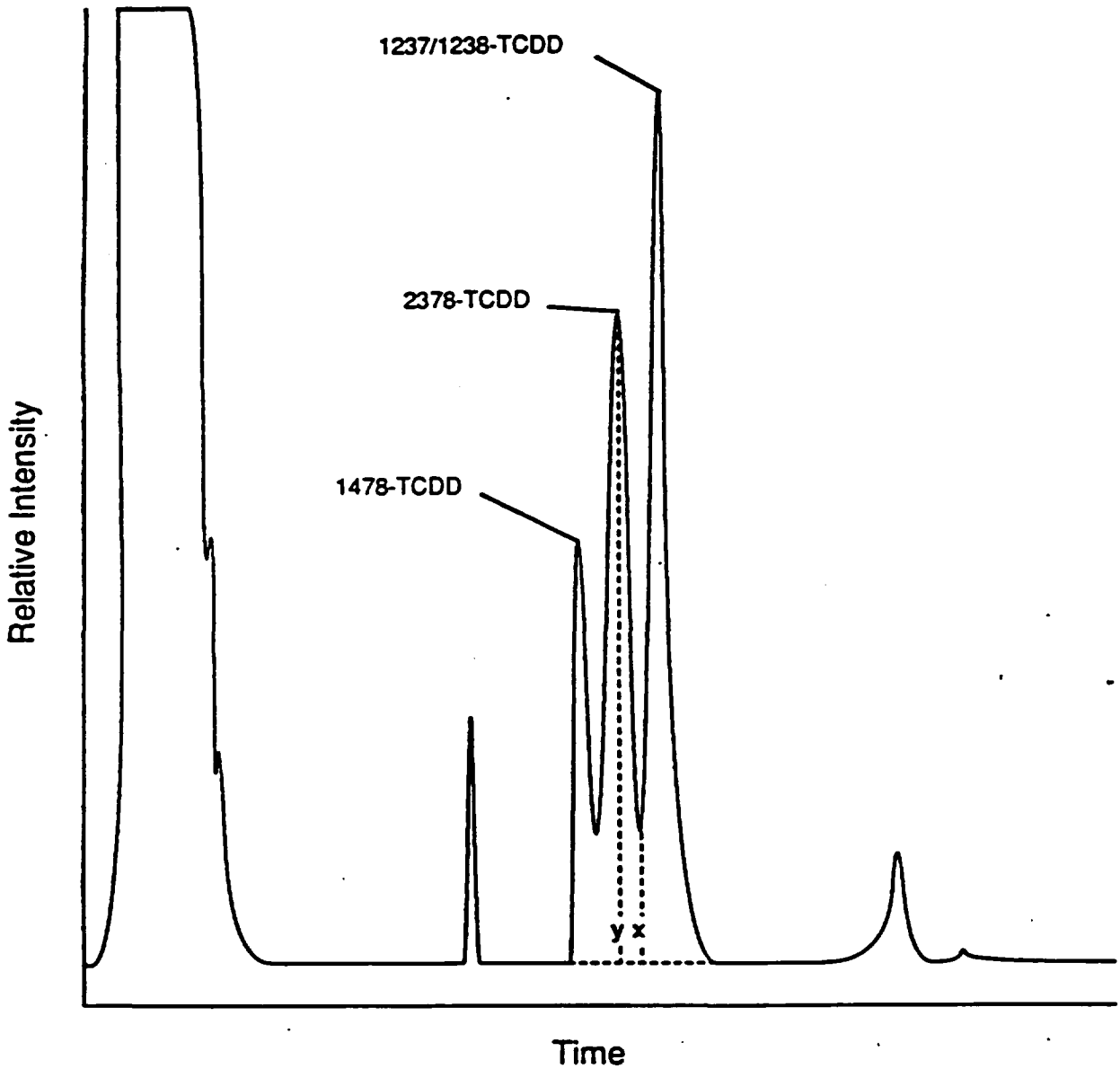

Figure 5: Valley Between 2378-TCDD and Other Closely Eluting Isomers on an SP-2331 (or Equivalent) Column

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

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OVERVIEW

Quality assurance (QA) and quality control (QC) are integral parts of the CLP.[1,2,4,5,6] The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity to ensure that data provided are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.[1]

During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.[1,2,3]

This exhibit describes the overall QA/QC operations and the processes by which the CLP meets the QA/QC objective defined above. This contract requires a variety of QA/QC activities. These contract requirements are the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing the EPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

- A written QA Plan, the elements of which are designated in Section II.
- Written preparation of and adherence to QA/QC standard operating procedures (SOPs) as described in Section III.
- Adherence to the analytical methods and associated QC requirements specified in the contract.
- Verification of an analytical standard and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical supply houses.
- Submission of all raw data and pertinent documentation for Regional review.
- Participation in the analysis of laboratory evaluation samples, including adherence to corrective action procedures.
- Submission, upon request, of GC/MS tapes and applicable documentation for tape audits.
- Participation in on-site laboratory evaluations, including adherence to corrective action procedures.
- Submission of all original documentation generated during sample analyses for EPA review.

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SECTION II

QUALITY ASSURANCE PLAN

The Contractor shall establish a QA program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

As evidence of such a program, the Contractor shall prepare a written QA Plan (QAP) which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and usability.
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility.
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable.
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The QAP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during an on-site laboratory evaluation. Additional information relevant to the preparation of a QAP can be found in EPA and ASTM publications.[2,4]

The elements of a QAP are as follows:

A. Organization and Personnel
   1. QA Policy and Objectives
   2. QA Management
      a. Organization
      b. Assignment of QC and QA Responsibilities
      c. Reporting Relationships
      d. QA Document Control Procedures
      e. QA Program Assessment Procedures
   3. Personnel
      a. Resumes
      b. Education and Experience Pertinent to this Contract
      c. Training Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives
2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy
2. Samples Tracking/Custody Procedures
3. Logbook Maintenance and Archiving Procedures
4. Case File Organization, Preparation and Review Procedures
5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs
6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency
2. Sample Preparation/Extraction Procedures
3. Sample Analysis Procedures
4. Standards Preparation Procedures
5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures
2. Data Reduction Procedures
3. Data Validation Procedures
4. Data Reporting and Authorization Procedures

F. Quality Control

1. Solvent, Reagent and Adsorbent Check Analysis
2. Reference Material Analysis
3. Internal Quality Control Checks
4. Corrective Action and Determination of QC Limit Procedures
5. Responsibility Designation

G. Quality Assurance

1. Data Quality Assurance
2. Systems/Internal Audits
3. Performance/External Audits
4. Corrective Action Procedures
5. Quality Assurance Reporting Procedures
6. Responsibility Designation

Updating and Submission of the QAP:

Within 60 days of contract award:

During the contract solicitation process, the Contractor was required to submit their QAP to EMSL-LV and NEIC. Within sixty (60) days after contract award, the Contractor shall send a revised QAP, fully compliant with the requirements of this contract, to the TPO, EMSL-LV and NEIC. The revised QAP will become the official QAP under the contract. The revised QAP must include:

1. Changes resulting from the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures and the Contractor's implementation of the requirements of the contract; and

2. Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder-supplied documentation, and recommendations made during the preaward laboratory site evaluation.

Subsequent submissions:

During the term of contract, the Contractor shall amend the QAP when the following circumstances occur:

1. The Agency modifies the contract,

2. The Agency notifies the Contractor of deficiencies in the QAP,

3. The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,

4. The Contractor identifies deficiencies resulting from the internal review of the QAP,

5. The Contractor's organization, personnel, facility, equipment, policy or procedures change, or

6. The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy or procedures.

The Contractor shall amend the QAP within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP must be clearly marked (i.e., indicating where the change is in the document with a bar in the margin, underlining the change, printing the change in bold, or using a different print font). The amended pages must have the date on which the changes were implemented.

The Contractor shall incorporate all amendments to the current QAP. The Contractor shall archive all amendments to the QAP for future reference by the Agency. The Contractor shall send a copy of the current QAP within 14 days of a request by the TPO or APO to the designated recipients.

Corrective action:

If the Contractor fails to adhere to these requirements, the Contractor may expect, but the Agency is not limited to, the following actions: reduction of numbers of samples sent under this contract, suspension of sample shipment to the Contractor, GC/MS tape audit, data package audit, on-site laboratory evaluation, remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives
2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy
2. Samples Tracking/Custody Procedures
3. Logbook Maintenance and Archiving Procedures
4. Case File Organization, Preparation and Review Procedures
5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs
6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency
2. Sample Preparation/Extraction Procedures
3. Sample Analysis Procedures
4. Standards Preparation Procedures
5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures
2. Data Reduction Procedures
3. Data Validation Procedures
4. Data Reporting and Authorization Procedures

F. Quality Control

1. Solvent, Reagent and Adsorbent Check Analysis
2. Reference Material Analysis
3. Internal Quality Control Checks
4. Corrective Action and Determination of QC Limit Procedures
5. Responsibility Designation

G. Quality Assurance

1. Data Quality Assurance
2. Systems/Internal Audits
3. Performance/External Audits
4. Corrective Action Procedures
5. Quality Assurance Reporting Procedures
6. Responsibility Designation

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- Title page.
- Scope and application.
- Definitions.
- Procedures.
- QC limits.
- Corrective action procedures, including procedures for secondary review of information being generated.
- Documentation description and example forms.
- Miscellaneous notes and precautions.
- References.

B. Required SOPs

The following SOPs are required by EPA:

1. Evidentiary SOPs (see Exhibit F).
2. Sample receipt and storage.
3. Sample preparation.
4. Calibration.
5. Standards purity/preparation.
6. Maintaining instrument records and logbooks.
7. Sample analysis and data control systems.
8. Glassware cleaning.
9. Technical and managerial review of laboratory operation and data package preparation.
10. Internal review of contractually required QA/QC data for each individual data package.
11. Chain-of-custody procedures and document control including Complete Sample Delivery Group (SDG) File preparation.
12. Laboratory data validation/laboratory self-inspection.
    a. Data flow and chain-of-command for data review.
    b. Procedures for measuring precision and accuracy.
    c. Evaluation parameters for identifying systematic errors.[9]
    d. Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in Exhibit B.

e. Demonstration of internal QA inspection procedures (demonstrated by supervisory sign-off on personal notebooks, internal PE samples, etc.).

f. Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

g. Demonstration of problem identification-corrective actions and resumption of analytical processing and sequence resulting from internal audit (i.e., QA feedback).

h. Documentation of audit reports (internal and external), response, corrective action, etc.

13. Data management and handling.

a. Procedures for controlling and estimating data entry errors.

b. Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

c. Lifecycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

d. Database security, backup and archival procedures including recovery from system failures.

e. System maintenance procedures and response time.

f. Individuals(s) responsible for system operation, maintenance, data integrity and security.

g. Specifications for staff training procedures.

C. <u>SOP Delivery Requirements</u>

Updating and submission of SOPs:

During the contract solicitation process, the Contractor was required to submit their SOPs to EMSL-LV and NEIC. Within sixty (60) days after contract award, the Contractor shall send a complete revised set of SOPs, fully compliant with the requirements of this contract, to the TPO, EMSL-LV and NEIC. The revised SOPs will become the official SOPs under the contract. The revised SOPs must include:

1. Changes resulting from the Contractor's internal review of their procedures and the Contractor's implementation of the requirements of the contract;

2. Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder-supplied documentation, and recommendations made during the preaward laboratory site evaluation.

Subsequent submissions:

During the term of contract, the Contractor shall amend the SOPs when the following circumstances occur:

1. The Agency modifies the contract,

2. The Agency notifies the Contractor of deficiencies in their SOPs documentation,

3. The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,

4. The Contractor's procedures change,

5. The Contractor identifies deficiencies resulting from the internal review of their SOPs, or

6. The Contractor identifies deficiencies resulting from the internal review of their procedures.

The SOPs must be amended or new SOPs must be written within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs must be clearly marked (i.e, indicating where the change is in the document with a bar in the margin, underlining the change, printing the change in bold, or using a different print font). The amended/new SOPs must have the date on which the changes were implemented.

When the SOPs are amended or new SOPs are written, the Contractor shall document in a letter the reasons for the changes, and submit the amended SOPs or new SOPs to the TPO, EMSL-LV (quality assurance/technical SOPs) and NEIC (evidentiary SOPs). The Contractor shall send the letter and the amended sections of the SOPs or new SOPs within 14 days of the change. An alternate delivery schedule for the submittal of the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the TPO or APO, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the TPO, APO, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The TPO/APO will not grant an extension for greater than 30 days for amending/writing new SOPs. The TPO/APO will not grant an extension for greater than 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

The Contractor shall send a complete set of current SOPs within 14 days of a request by the TPO or APO to the recipients he/she designates.

Corrective action:

If the Contractor fails to adhere to these requirements, the Contractor may expect, but the Agency is not limited to, the following action: reduction of number of samples sent under this contract, suspension of sample shipment to the Contractor, GC/MS tape audit, data package audit, on-site laboratory evaluation, remedial laboratory evaluation sample, and/or contract sanction, such as a Cure Notice.

SECTION IV

QA/QC REQUIREMENTS

This section outlines the minimum QC operations necessary to satisfy the analytical requirements associated with the detection and quantitative measurement of 2378-tetrachlorinated dibenzo-*p*-dioxin and total tetra-, penta-, hexa-, hepta- and octachlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs), by using the procedures outlined in Exhibit D. This section is not intended as a comprehensive QC document, but rather as a guide to the specific QC operations that must be considered for PCDD/PCDF analysis.

The QC operations that must be considered include the following:

- Mass Calibration.
- Window Defining Mix.
- Chromatographic Resolution.
- GC/MS Initial Calibration.
- GC/MS Continuing Calibration.
- Instrument Sensitivity.
- Identification Criteria.
- Method Blank Analysis.
- Spiked Sample Analysis.
- Duplicate Sample Analysis.
- Toxicity Equivalency Factor and Isomer Specificity.
- Dilutions.
- Reanalyses.

1. <u>Mass Calibration</u>

1.1 Mass calibration of the mass spectrometer is recommended prior to analyzing the calibration solutions, blanks, samples and QC samples. It is recommended that the instrument be tuned to greater sensitivity in the high mass range in order to achieve better response for the later eluting compounds.

1.2 Optimum results using FC-43 for mass calibration can be achieved by scanning from 222-510 amu every one second or less, utilizing 70 volts (nominal) electron energy in the electron ionization mode (see Exhibit D, Section 6).

1.3 m/z 414 and m/z 502 should be 30-50 percent of m/z 264 base peak (see Exhibit D, Section 6).

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the chromatographic peak separation between the 123478-HxCDD and 123678-HxCDD in the CC3 solution must be resolved with a valley of ≤ 50 percent (see Exhibit D, Paragraph 7.3.2.1).

4.6 The relative ion abundance criteria for PCDDs/PCDFs must be met for all PCDD/PCDF peaks, including the labeled internal and recovery standards, in all solutions (see Exhibit D, Table 6).

4.7 For all calibration solutions, the retention times of the isomers must fall within the appropriate retention time windows established by the window defining mix (see Exhibit D, Section 7.1).

4.8 For all calibration solutions, the signal-to-noise ratio must meet the criteria specified in Exhibit D, Paragraph 7.3.2.4.

4.9 The relative response factors for the 17 unlabeled target analytes relative to their appropriate internal standards, and the relative response of the five labeled internal standard standards relative to the appropriate recovery standard are determined according to the procedures in Exhibit D, Paragraph 7.3.3.

4.10 Calculate the mean RRF and percent relative standard deviation (%RSD) of the five RRFs (CC1 to CC5) for each unlabeled PCDD/PCDF, and labeled internal and recovery standards, present in all five concentration calibration solutions as described in Exhibit D, Paragraph 7.3.5. As indicated in the referenced paragraph, no %RSD calculation is possible for the 2,3,7,8-substituted isomers in the CC3 supplemental calibration solution, because they are only present in the one solution.

4.11 The %RSD is calculated for the EPA-supplied unlabeled and labeled analytes only. To establish linearity, the %RSD of the five RRFs (CC1-CC5) for the unlabeled PCDDs/PCDFs and the internal standards must not exceed 15.0% (see Exhibit D, Paragraph 7.3.5).

4.12 If the initial calibration criteria for GC resolution, ion abundance ratios, retention times, instrument sensitivity and relative response factors are not met, the Contractor must take the corrective actions as explained in Exhibit D, Paragraph 7.3.7.

4.13 The response factors to be used for determining the total homologue concentrations are described in Exhibit D, Section 15.2.

5. <u>GC/MS Continuing Calibration</u>

5.1 Once the GC/MS system has been calibrated, the calibration must be verified for each 12-hour time period for each GC/MS system.

5.2 The continuing calibration standard is prepared by mixing the commercially supplied supplemental standard with the EPA supplied CC4 solution (see Exhibit D, Paragraph 7.4.1).

5.3 The continuing calibration consists of two parts: evaluation of the chromatographic resolution and verification of the RRF values to be used for quantitation. At the beginning of each 12-hour period, the

6.5 For this analysis, the signal-to-noise ratio shall be greater than 2.5 for the unlabeled PCDD/PCDF ions, and greater than 10.0 for the labeled internal and recovery standards.

7. Identification Criteria

7.1 For a gas chromatographic peak to be unambiguously identified as a PCDD/PCDF, the peak must meet all of the following criteria.

7.2 The identification of the PCDD/PCDF isomers is based on simultaneous detection of the two most abundant ions in the molecular ion regions and the M-COCl ion. In order to make a positive identification, the relative retention time criteria specified in Exhibit D, Section 11.1 must be met.

7.3 All of the ions specified for each PCDD/PCDF homologue and labeled standards must be present in the selected ion current profile. The ion current response for the analytes and labeled standards must meet the QC criteria (see Exhibit D, Section 11.2).

7.4 The integrated ion current for each analyte ion listed in Exhibit D, Table 5 must be at least 2.5 times background noise and must not have saturated the detector. The internal standard ions must be at least 10 times background noise and must not have saturated the detector (see Exhibit D, Section 11.3).

7.5 The relative ion abundance criteria for the native analytes and internal standard must be met (see Exhibit D, Table 6).

7.6 The identification of a GC peak as a PCDF cannot be made if a signal having a signal-to-noise ratio greater than 2.5 is detected in the corresponding PCDPE channel (see Exhibit D, Section 11.5).

8. Method Blank Analysis

8.1 A method blank is a volume of clean reference matrix that is carried through the entire analytical sequence.

8.2 A minimum of one blank per matrix must be analyzed with each SDG at a frequency described in Exhibit D, Section 12.1.

8.3 An acceptable method blank must not contain any chemical interferences or electronic noise at the m/z of the specified unlabeled PCDD/PCDF ions which is greater than 5 percent of the signal of the appropriate internal standard, or any peak that meets the identifications criteria as a PCDD/PCDF which is greater than 2 percent of the appropriate internal standard (see Exhibit D, Section 15.2).

8.4 If the blank is contaminated, the associated positive samples and any samples containing peaks that do not meet all the identification criteria must be rerun (see Exhibit D, Paragraph 12.2.3).

on the DB-5 column. The Contractor may utilize either of the two options listed in Exhibit D, Paragraphs 16.1.1 or 16.1.2 to achieve adequate isomer specificity.

12. Dilutions

If the concentration of any PCDD/PCDF in the sample exceeds the calibration range or the detector is saturated, a dilution must be performed using the procedures given in Exhibit D, Section 10.4.

13. Reanalyses

The requirements for reextraction and for reanalysis of samples are given in Exhibit D, Section 17.

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SECTION V

ANALYTICAL STANDARDS REQUIREMENTS

A. Overview

EPA will not supply all the analytical reference standards required for performance of this contract. See Exhibit D, Section 5 for the standards that may be provided by EPA, subject to availability. Contractors will be required to prepare from neat materials or purchase from private chemical supply houses the standards not supplied by EPA but necessary to successfully and accurately perform the analyses required in this contract.

B. Preparation of Chemical Standards from the Neat High Purity Bulk Material

The Contractor may prepare chemical standards from neat materials. Commercial sources for neat chemical standards pertaining to compounds listed on the Target Compound List are given in Appendix C of the "Quality Assurance Materials Bank: Analytical Reference Standards," Seventh Edition, January 1988. Laboratories should obtain the highest purity possible when purchasing neat chemical standards; standards purchased at less than 97% purity must be documented as to why a higher purity could not be obtained.

1. Neat chemical standards must be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of neat chemicals is essential in order to safeguard them from decomposition.

2. The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, the Contractor is responsible for having analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use either differential scanning calorimetry, gas chromatography with flame ionization detection, high performance liquid chromatography, infrared spectrometry, or other appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

   Equation 1

$$\text{weight of impure compound} = \frac{\text{weight of pure compound}}{(\text{percent purity}/100)}$$

   where "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

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3. Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. The Contractor is responsible for having analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified. Identification confirmation, when performed, should use gas chromatographic/mass spectrometry analysis on at least two different analytical columns, or other appropriate techniques.

4. Calculate the weight of material to be weighed out for a specified volume taking into account the purity of the compound and the desired concentration. A second person must verify the accuracy of the calculations. Check balances for accuracy with a set of standard weights. All weighing should be performed on an analytical balance to the nearest 0.1 mg and verified by a second person. The solvent used to dissolve the solute should be compatible with the protocol in which the standard is to be used; the solute should be soluble, stable, and nonreactive with the solvent. In the case of a multicomponent solution, the components must not react with each other.

5. Transfer the solute to a volumetric flask, and dilute to the specified solution volume with solvent after ensuring dissolution of the solute in the solvent. Sonication or warming may be performed to promote dissolution of the solute. This solution is to be called the primary standard, and all subsequent dilutions must be traceable back to the primary standard.

6. Log notebooks are to be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be recorded and verified by a second person. All solution standards are to be refrigerated when not in use. All solution standards are to be clearly labeled as to the identity of the compound(s), concentration, date prepared, solvent, and initials of the preparer.

C. Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by the Contractor provided they meet the following criteria:

1. Laboratories must maintain the following documentation to verify the integrity of the standard solutions they purchase:

   a. Mass spectral identification confirmation of the neat material.

   b. Purity confirmation of the neat material.

   c. Chromatographic and quantitative documentation that the solution standard was QC-checked according to the following section.

2. The Contractor must purchase standards for which the quality is demonstrated statistically and analytically by a method of the supplier's choice. One way quality can be demonstrated is to prepare and analyze three solutions; a high standard, a low

d. The test statistic must be calculated:

Equation 5
TEST STATISTIC = $|(M_3 /1.1) - (M_1 /0.9)|/(V_p /3)^{0.5}$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate a twenty percent difference between the high and low standards. In such a case, the standards are not acceptable.

e. The test statistic must be calculated:

Equation 6
TEST STATISTIC = $|M_2 - (M_1 /1.8) - (M_3 /2.2)|/(V_p /4)^{0.5}$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

f. The 95 percent confidence intervals for the mean result of each standard must be calculated:

Equation 7
Interval for Low Standard = $M_1 \pm (2.13)(V_p /6)^{0.5}$

Equation 8
Interval for Target Standard = $M_2 \pm (2.13)(V_p /6)^{0.5}$

Equation 9
Interval for High Standard = $M_3 \pm (2.13)(V_p /6)^{0.5}$

These intervals must not overlap. If overlap is observed, the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable.

In any event, the Contractor is responsible for the quality of the standards employed for analyses under this contract.

D. Requesting Standards From the EPA Standards Repository

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can place an order for standards only after demonstrating that these standards are not available from commercial vendors either in solution or as a neat material.

E. Documentation of the Verification and Preparation of Chemical Standards

Each laboratory is responsible for maintaining the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, chromatograms, mass spectra,

SECTION VI

CONTRACT COMPLIANCE SCREENING

Contract Compliance Screening (CCS) is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the sample data package delivered to EPA.

CCS is performed by SMO under the direction of the EPA. To assure a uniform review, a set of standardized procedures have been developed to evaluate the sample data package submitted by a Contractor against the technical and completeness requirements of the contract.

CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor must send all corrections to the Regional client, EMSL-LV and SMO.

CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

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SECTION VIII

LABORATORY EVALUATION SAMPLES

Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of laboratory evaluation samples will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

Sample sets may be provided to participating Contractors as frequently as on a SDG-by-SDG basis as a recognizable QC sample of known composition, as a recognizable QC sample of unknown composition, or not recognizable as a QC material. Laboratory evaluation samples may be sent either by the Regional client or the NPO and may be used for contract action.

Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

At a minimum, the results are evaluated for compound identification, quantification, and sample contamination. Confidence intervals for the quantification of target compounds are based on reported values using population statistics. EPA may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample. Normally, a fraction of the compounds spiked into the sample are not specifically listed in the contract.

A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

1. Acceptable, No Response Required (Score greater than or equal to 90 percent):

   Data meets most or all of the scoring criteria. No response is required.

2. Acceptable, Response Explaining Deficiency(ies) Required (Score greater than or equal to 75 percent but less than 90 percent):

   Deficiencies exist in the Contractor's performance.

   Within 14 days of receipt of notification from EPA, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the APO, the TPO and EMSL-LV.

SECTION IX

GC/MS TAPE AUDITS

Periodically, EPA requests from Contractors the GC/MS magnetic tapes corresponding to a specific Case in order to accomplish tape audits. Generally, tape submissions and audits are requested for the following reasons:

- Program overview.
- Indication of data quality problems from EMSL-LV, SMO, or Regional data reviews.
- Support for on-site audits.
- Specific Regional requests.

Depending upon the reason for an audit, the tapes from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Tape audits provide a mechanism to assess adherence to contractual requirements and ensure the consistency of data reported on the hardcopy forms with that generated on the GC/MS tapes. This function provides external monitoring of CLP QC requirements and checks adherence of the Contractor to internal QA procedures. In addition, tape audits enable EPA to evaluate the utility, precision and accuracy of the analytical methods.

The GC/MS tape shall include raw data and quantitation reports for samples, blanks, laboratory evaluation samples, initial calibrations, and continuing calibrations associated with the SDG requested. The specific requirements for submissions of GC/MS tapes are discussed in Exhibit B.

Upon request of the APO or EMSL-LV, the required tapes and all necessary documentation shall be submitted to EPA within seven days of notification.

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1. Procedural Audit

The procedural audit consists of review and examination of actual SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

2. Written SOPs Audit

The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

3. Analytical Project File Evidence Audit

The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine:

- Accuracy of the document inventory.
- Completeness of the file.
- Adequacy and accuracy of the document numbering system.
- Traceability of sample activity.
- Identification of activity recorded on the documents.
- Error correction methods.

C. Discussion of the On-Site Team's Findings

The QA and evidentiary auditors discuss their findings with the APO/TPO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

D. Corrective Action Reports For Follow-Through to Quality Assurance and Evidentiary Audit Reports

Following an on-site evaluation, QA and evidentiary audit reports which discuss deficiencies found during the on-site evaluation will be forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the on-site visit and discussed in the on-site reports in a letter to the APO, TPO, EMSL-LV (response to the QA report) and NEIC (response to the evidentiary report) within 14 days of receipt of the finding or within the time agreed upon between the APO/TPO and the Contractor. If SOPs are required to be written or amended, the Contractor must provide

SECTION XI

QUALITY ASSURANCE AND DATA TREND ANALYSIS

Data submitted by laboratories are subject to review from several aspects: compliance with contract-required QC, usability, and full data package evaluation. Problems resulting from any of these reviews may determine the need for a GC/MS tape audit, an on-site laboratory evaluation and/or a remedial laboratory evaluation sample. In addition, QC prescribed in the methods provides information that is continually used by EPA to assess sample data quality, Contractor data quality and CLP data quality via data trend analysis. Trend analysis is accomplished by entering data into a computerized database. Statistical reports that evaluate specific anomalies or disclose trends in many areas, including the following, are generated from this database:

- Internal standard recovery.
- Laboratory evaluation sample.
- Blanks.
- Gas chromatographic resolution of analytes.
- Initial and continuing calibration data.
- Other QC and method parameters.

Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each Contractor using a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trends analyses are included in overall evaluation of a Contractor's performance, and are reviewed to determine if corrective action or an on-site laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

Contractor performance over time is monitored using these trend analysis techniques to detect departures of Contractor output from required or desired levels of QC, and to provide an early warning of Contractor QA/QC problems which may not be apparent from the results of an individual case.

As a further benefit to the CLP, the database provides the information needed to establish performance-based criteria in updated analytical protocols, where advisory criteria has been previously used. The vast empirical data set produced by contract laboratories is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of QC and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis of environmental samples. This information, in turn, assists EPA in meeting its objectives of obtaining data of known and documented quality.

- Each version of the production system will be given an identification number, a date of installation, and a date of last operation, and will be archived.
- System and operations documentation must be developed and maintained for each system. Documentation must include a user's manual and an operations and maintenance manual.

Individual(s) responsible for the following functions must be identified:

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and quality control.
- Data and system security, backup and archiving.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL,
AND STANDARD OPERATING PROCEDURES

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1. Sample Chain-of-Custody

A sample is physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To accomplish this task, Contractors are required to develop and implement the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures.

1.1 Sample Identification

To assure traceability of the samples while in the possession of the Contractor, the Contractor shall have a specified method for maintaining identification of samples throughout the laboratory. Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

1.2 Chain-of-Custody Procedures

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if:

- It is in your possession, or
- It is in your view after being in your possession, or
- It was in your possession and you locked it up, or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

1.3 Sample Receiving Procedures

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available.

1.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

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2. Document Control Procedures

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analysis. The following document control procedures have been established to ensure that all laboratory records are assembled and stored for delivery to EPA or are available upon request from EPA prior to the delivery schedule.

2.1 Preprinted Laboratory Forms and Logbooks

2.1.1 All documents produced by the Contractor that are directly related to the preparation and analysis of EPA samples shall become the property of EPA and shall be placed in the Complete SDG File (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG are compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents that are directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to EPA, the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.5 Shipment of Deliverables

The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that the containers cannot be opened without damaging or breaking the seal. The Contractor shall document what was sent, to whom, the date, and the method (carrier) used. A copy of the transmittal letter for the CSF shall be sent to the National Enforcement Investigations Center and SMO.

3. Specifications for Written Standard Operating Procedures

The Contractor shall have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, sample tracking, and assembly of completed data. A SOP is defined as a written narrative stepwise description of laboratory operating procedures including examples of laboratory documents. The SOPs shall accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1 The Contractor shall have written SOPs describing the sample custodian's duties and responsibilities.

3.2 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include, but not be limited to, documenting the following information:

3.2.1 Presence or absence of EPA chain-of-custody forms.

3.2.2 Presence or absence of airbills or airbill stickers.

3.2.3 Presence or absence of Traffic Reports or SAS Packing Lists.

3.2.4 Presence or absence of custody seals on shipping and/or sample containers and their condition.

3.2.5 Custody seal numbers, when present.

3.2.6 Airbill or airbill sticker numbers.

3.2.7 Presence or absence of sample tags.

3.2.8 Sample tag ID numbers.

3.2.9 Condition of the shipping container.

3.2.10 Condition of the sample bottles.

- A description of the method used by the laboratory to verify consistency and completeness of the CSF.
- Procedures for the shipment of deliverables packages using custody seals.

4. Handling of Confidential Information

A Contractor conducting work under this contract may receive confidential information from EPA. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

4.1 All confidential documents shall be under the supervision of a designated DCO.

4.2 Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO will log these documents into a Confidential Inventory Log. The information will then be available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the Technical Project Officer and Administrative Project Officer. The DCO will enter all copies into the document control system described above. In addition, this information may not be disposed of except upon approval by the Technical Project Officer and Administrative Project Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record on the disposition in the Confidential Inventory Log.

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GLOSSARY

ALIQUOT - a measured portion of a sample taken for analysis.

ANALYSIS DATE/TIME - the date and military time of the injection of the sample, standard or blank into the GC/MS or GC system.

BLANK - see Method Blank.

CASE - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

CONCENTRATION CALIBRATION SOLUTION (Table 3) - solutions (tridecane) containing known amounts of selected analytes, five internal standards and two recovery standards that are analyzed prior to sample analysis. The solutions are used to determine the ratio of the instrument response of the analytes to that of the appropriate internal standard and the internal standards to that of the recovery standards.

CONTINUING CALIBRATION SOLUTION - a mixture of known amounts of analytes that is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance and establish the retention time windows for each homologue. The same solution is used for the mid-level concentration calibration solution, CC3.

DAY - unless otherwise specified, day shall refer to calendar day.

ESTIMATED DETECTION LIMIT (EDL) - the concentration of a analyte required to produce a signal with peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8-substituted isomer for which the response of the quantitation and confirmation ions is less than 2.5 times the background level.

ESTIMATED MAXIMUM POSSIBLE CONCENTRATION (EMPC) - the concentration of a given analyte that would produce a signal with a given peak area. The EMPC is calculated for 2,3,7,8-substituted isomers for which the quantitation and/or the confirmation ion(s) has signal-to-noise in excess of 2.5 but does not meet identification criteria.

FIELD BLANK - a portion of chemical waste, soil or water that is not contaminated with PCDDs/PCDFs and is submitted with the samples. The field blank is used to check for contamination from the time of sample collection through the time of sample analysis.

HOMOLOGUE - a member or members of a particular homologous series that has the same molecular weight but not necessarily the same structural arrangement. For example, the 28 pentachlorinated dibenzofurans are homologues.

POLYCHLORINATED DIBENZO-P-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) - compounds (Figure 2) that contain from one to eight chlorine atoms. The 15 2,3,7,8-substituted PCDDs (total PCDDs is 75) and PCDFs (total PCDFs is 135) are shown in Table 13. The number of isomers at different chlorination levels is shown in Table 12.

PROTOCOL - describes the exact procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Synonymous with Statement of Work (SOW).

REAGENT WATER - water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest.

RECOVERY - a determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\%\text{Recovery} = \frac{\text{measured value}}{\text{known value}} \times 100\%$$

RECOVERY STANDARD (Table 9) - $^{13}C_{12}$-1234-TCDD and $^{13}C_{12}$-123789-HxCDD are added to every blank, quality control sample, and sample extract aliquot just prior to analysis and are present in all solutions except the internal standards solutions. Recovery standards are used to measure the recovery of the internal standards. When a dilution is required (see Exhibit D, Paragraph 13.2.5), recovery standards are used to quantitate the native PCDDs/PCDFs; the TCDD recovery standard is used to quantitate the tetra- and penta- isomers and the HxCDD recovery standard is used to quantitate the hexa- through octa- isomers.

RELATIVE RESPONSE FACTOR (RRF) - the ratio of the response of the mass spectrometer to a known amount of an analyte relative to that of a known amount of an internal standard as measured in the initial and continuing calibrations. The RRF is used to determine instrument performance and is used in the quantitation calculations.

RESOLUTION (also termed separation) - the separation between peaks on a chromatogram. Resolution is calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multipled by 100.

RINSATE - a portion of the solvent that is used to rinse sampling equipment. The rinsate is later analyzed to demonstrate that samples were not contaminated during collection.

SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in a SDG are due concurrently. A SDG is defined by one of the following, whichever occurs first:

TWELVE-HOUR TIME PERIOD - the 12-hour time period begins with the injection of the CC3 solution on the DB-5 (or equivalent) column or the injection of the column performance solution on the SP-2331 (or equivalent) column. The 12-hour period continues until 12:00 hours have elapsed according to the system clock. To be included in a given 12-hour time period, a sample or standard must be injected within 12:00 hours of the CC3 solution or the column performance solution.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report.

APPENDIX B

HEALTH AND SAFETY PLAN AND ADDENDUM

PHASE II SITE INVESTIGATION
PPG INDUSTRIES, INC
MOORESTOWN, NEW JERSEY

SITE SAFETY AND HEALTH PLAN
ADDENDUM I

CLIENT: PPG Industries, Inc.

SITE NAME: Pulverizing Services Site

PROJECT/TASK ID#: 09.0801460.001

SITE ADDRESS: Moorestown, New Jersey

DATE: August 1, 1994

PLAN EXPIRATION DATE: November 1, 1994

SSHP APPROVALS:

	Name	Signature	Date
PROJECT MANAGER	Dave Kerschner		8/1/94
IH REVIEW	Julie Panko	Julie M Panko	8/1/94
FIELD SUPERVISOR/ SITE SAFETY OFFICER	Neale Misquitta	N Misquitta	8/01/94
REGIONAL HEALTH AND SAFETY COORDINATOR	Jim Less	(on-file)	

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SITE PERSONNEL ACKNOWLEDGEMENT SHEET

This Health and Safety Plan Addendum I, dated August 1, 1994, has been read and understood by the following personnel who will be on site during the project.

NAME (Print)	SIGNATURE	DATE

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PPG INDUSTRIES, INC.
PULVERIZING SERVICES SITE

HEALTH AND SAFETY PLAN

ADDENDUM I
August 1, 1994

INTRODUCTION

The purpose of this plan is to update the existing Health and Safety Plan (HASP) originally prepared for this site by Paul C. Rizzo and Associates. This addendum is intended to supplement, not replace the original HASP. Changes described below are the result of different site activities to be performed and clarifications made with respect to McLaren/Hart Health and Safety Policies and Procedures.

All site employees are required to read both the original plan and this addendum and sign the acknowledgement page indicating an understanding of the Health and Safety requirements for work on this site.

This addendum incorporates the following into the HASP.

DESCRIPTIONS OF ACTIVITIES

A Phase II Site Investigation will be conducted and will consist of the following tasks:

1. Conceptual Site Model - This task will not be conducted in the field. It will involve review of previously collected site data to develop a conceptual site model which describes the current understanding of chemical releases which occurred at the facility, and the movement of those chemical constituents in soils and groundwater at the site.

2. Soil Sampling - Soil samples will be collected throughout the site using the Geoprobe unit. Samples will be collected at intervals 0-1 and 1-2 feet depths, with a 3-4 feet depth collected if field screening of the 1-2 foot depth sample indicates greater than 100 mg/kg chlorinated compounds. Areas A, B and C as shown on Figure 1 are included in the soil sampling program.

3. Surface Water and Sediment Sampling - Three surface water and sediment samples will be collected from the drainage ditch flowing from Area A to Area C. In addition, two surface water and sediment samples may be collected from the ditch along the railroad track in Area B. Sediment samples will be field screened.

4. Literature Search - This task will not be conducted in the field. It will involve a review of available literature to supplement the current domestic/public well inventory and to gather existing information regarding the geology, hydrogeology, and physical/environmental setting of the study area. Any wells not previously identified will be located on a map which also shows the site.

5. Groundwater Monitoring Program - This task will involve production well sampling, installation of twenty piezometers, groundwater sampling of all newly installed wells, groundwater level measurement and potentially off-site well sampling. Additional monitoring wells may be installed if the groundwater level measurement indicates a need for them.

6. Test Pit Excavations

6.1 Area A - Two to three exploratory trenches will be excavated in the area behind Building 29, in an orientation perpendicular to the presumed orientation of the historic disposal trenches. The trenches will be excavated to a depth of eight to ten feet below grade, or to the extent of buried waste if that extent is deeper. In the event that buried waste occurs at a depth greater than the reach of the excavator, a larger excavator will be mobilized to complete the trenching activities.

Trenches will be dug in a step-wise pattern in 10 to 20 feet segments, by moving outward from the center of the target area until the disposal trench is encountered or it becomes evident that there is no disposal trench. No trenching will take place during periods of heavy rain or high winds. Any drums encountered will be left in place, and a soil sample will be collected adjacent to the drum. Excavated materials will be placed on sheeting adjacent to the trench area. Upon completion of trenching activities, the exploratory trenches will be lined with polyethylene sheeting and the material will be placed back into the trench. The trench location will be covered with polyethylene sheeting.

Soil sampling will be collected below the limits of waste disposal, and at a depth of approximately five feet below grade from the area between the disposal trenches and near the outside extent of the exploratory trenches. It is anticipated that a total of 10 to 20 samples will be collected.

A third trench will be excavated at the edge of the buried drum location to assess the integrity of the buried drums. The test pit will be excavated working from the outer area toward the disposal area. Materials around the first drum encountered will be removed to allow visual assessment. If the drum is open or rusted, a sample of the contents will be collected, and a second sample will be collected from the soil beneath the drum. If the drum is intact, only a sample of soil from beneath the drum will be collected.

An additional test pit will be excavated between B-11 and B-12 to assess the reported electromagnetic (EM) survey anomaly.

6.2 Areas B & C - Four exploratory test pits will be excavated in each of the Areas B and C to evaluate the potential for waste disposal in these areas (eight test pits total). If no waste is encountered, excavations will stop at five to six feet below grade. If waste is encountered, the excavation will extend to the base of the buried waste, or to the limit of the backhoe reach, whichever is less. If waste is encountered, a sample will be collected of the waste material and one from below the limit of waste disposal. Additional excavation activities will be conducted to define the limits of the disposal area.

7. Building 5 Trench Sampling - The location of the reported (concrete-lined) trench behind former Building 5 will be assessed by visual inspection of the area; means for access to this trench will also be assessed. If residual materials are found in the trench, they will be sampled, field screened and submitted for laboratory analysis.

8. Underground Tank Sampling - Three underground storage tanks will be visually located, and accessed by opening the fill port or manway. The contents of the tanks will be assessed visually (level, type of material, etc.) and grab-samples from outside the tank. If residual materials are present in a tank or tanks, it will be removed and the tank will be cleaned.

9. Storm Sewer Sampling - Two catch basins will be located and evaluated for the presence of sediments. If sediments are present, they will be sampled and submitted for laboratory analysis for the specified parameters. Samples will be obtained without entering the basins.

10. Potential Migration - Information regarding environmental quality in the vicinity of New Albany Road will be incorporated into the evaluation of site conditions. This task will not be conducted on site.

11. Feasibility Study - A focused feasibility study will be performed based on the information presented in the Phase I Report and Phase I and Phase II data sets. This task will not be conducted on site.

12. Risk Assessment - A qualitative Risk Assessment to address potential human health and/or ecological issues at the site. This task will not be conducted on site.

13. Site Investigation Report - This task will not be conducted on site.

14. Project Management - This is not an on-site work task.

15. Access Agreements - This is not an on-site work task.

PHYSICAL HAZARDS

Table I summarizes the physical hazards that may be encountered during work on this site. Of those indicated as potential site hazards, on-site utilities, excavation hazards and heat exposure are the most serious. Prior to any intrusive activies utility clearance must be conducted as described under Drilling Operations. Identification of all overhead and underground utilities will mimimize the potential for contact with the lines during drilling and excavation. The field work described above will likely be conducted during the summer/fall months and therefore, a potential for heat stress exists. There are four basic types of heat stress. Each type and associated symptoms include:

HEAT RASH - Heat rash is characterized by numerous tiny red bumps on the skin surface. It is caused by continuous contact of the skin with wet clothing, as often occurs when wearing chemical protective clothing. Heat rash can cause severe irritation if not treated and allowed to heal. Heat rash can be prevented by allowing the skin to dry.

HEAT CRAMPS - Heat cramps are characterized by severe cramping of the large muscle groups such as those in the legs and abdomen. Heat cramps are caused by strenuous physical work and excessive sweating which leads to loss of water and salts from the body. Heat cramps can be prevented by drinking adequate amounts of water before, during and after heavy work in hot environments. Normal salt intake during meals should be sufficient to replace lost electrolytes. SALT TABLETS SHOULD NOT BE TAKEN.

HEAT EXHAUSTION - Someone suffering from heat exhaustion will exhibit symptoms such as profuse sweating, cold/clammy skin, nausea, dizziness and fainting. A person suffering from heat exhaustion should be immediately moved to a shady area or indoors and given cool liquids to drink. Any protective clothing should be removed. Heat exhaustion is a serious condition which may lead to Heat Stroke. It should not be ignored.

HEAT STROKE - This is the most serious of the heat stresses and requires immediate medical attention. Heat stroke is the result of the failure of the body's cooling mechanism causing the internal body temperature to be elevated. Someone with heat stroke will have red, dry, skin; and may become incoherent. Heat stroke if left untreated will lead to coma and death. A person showing signs of heat stroke should be immediately removed from protective clothing and cooled down with cool but not ice cold water. The person **MUST** receive immediate medical attention!

All heat stresses can be prevented. All persons working in hot environments should be acclimatized (slowly get used to) to the heat by working only a few hours a. a time initially in the hot conditions. Additionally, adequate amounts of water should be consumed , frequent breaks taken during the work day and refrain from alcohol consumption.

DRILLING OPERATIONS

Follow McLaren/Hart Policy and Procedure 16.0 for Drilling Safety during all drilling and soil sampling using the Geoprobe. A copy of it is provided in Attachment 1. Utility clearance must be conducted prior to any drilling activities. The Utility Clearance documentation form must be forwarded to Julie Panko, Pittsburgh Health and Safety Manager prior to drilling.

EXCAVATION OPERATIONS

Follow McLaren/Hart Policy and Procedure 15.0 for Excavation Operations during excavation of soil and buried drums. A copy of it is provided in Attachment 1. As stated above, utility clearance must be conducted prior to any excavation activities. In accordance with HS 15.0, a competent/qualified person must inspect the excavation (s) daily. The Site Foreman will be designated as this person. Results of the excavation inspection should be noted in the construction daily log.

CHEMICAL HAZARDS

Previous site investigations have indicated the presence of 10 pesticides/insecticides compounds detected in the surface soils. A summary of the chemical hazards is provided on Table 1. Skin absorption is the major exposure pathway for the chemical contaminants at this site. Strict adherence to the PPE requirements will reduce the potential for exposure via skin absorption.

SITE ZONES/DELINEATION

Exclusion Zone:

____ Areas within barricades, cones and/or caution tape
_X_ Within 40-ft radius of drill rig or Geoprobe operations
_X_ Within 40-ft radius of excavation operations
____ Within 10-ft radius of hand auguring location
_X_ Within 10-ft radius of groundwater monitoring well locations
____ Other (describe): Within 20 feet of Building 5 trench, storm sewer and underground Tank sampling operations.

Contamination Reduction Zone: At perimeter of Exclusion Zone.

Support Zone: Upwind and outside of Contamination Reduction Zone.

All personnel must utilize the buddy system, or trained escort, and check-in with the Field Supervisor.

SITE COMMUNICATION

____ By two way radio
_X_ By telephone
____ By pager
_X_ By other means (describe): verbal and hand signals as necessary.

CONFINED SPACE ENTRY

Examples of confined spaces include trenches greater than 4 feet deep underground storage tanks, and sewer manholes. In accordance with McLaren/Hart Policy and Procedure 15.0 (Excavation), no site worker may enter the trench unless it has

been inspected by a Registered Civil Engineer. Therefore, no confined space entry is anticipated for this project. If, however, it becomes necessary, the Pittsburgh Health and Safety Manager must be contacted prior to work in the confined space. Appropriate air monitoring equipment such as an oxygen meter/explosimeter must be available on site in order to perform confined space work. All confined space entries must be made in accordance with the requirements specified in McLaren/Hart Health and Safety Policy HS 14.0 "Confined Space Entry".

PERSONAL PROTECTIVE EQUIPMENT (PPE)

The PPE required for this site work is specified on Table I. The SSO will be responsible for upgrading/downgrading PPE. All personnel must use tape to seal between the chemical protective coveralls and gloves/boots.

AIR MONITORING

Air monitoring will be conducted in the exclusion zone, decontamination zone and at the downwind perimeter of the site. Air monitoring in the exclusion zone is specified on Table I and is to be conducted continuously at 15 to 30 minute intervals. Air monitoring frequency may be decreased if contaminant concentrations remain steady below the action levels. All air monitoring data must be recorded on the Direct Reading Report forms included as Attachment II. Any changes in the air monitoring procedures must be documented on this form. The Direct Reading Report forms should be forwarded to the Julie Panko at the Pittsburgh office on a weekly basis. The Pittsburgh office fax number is (412) 787-7218.

All direct reading instruments must be calibrated daily, or in accordance with manufacturer's instructions. All calibration data should be recorded on the Instrument Calibration Log included in Attachment II.

DECONTAMINATION PROCEDURES

The decontamination procedures for Modified Level D (D+) and Level C are outlined in Attachment III. All personnel must conduct thorough decontamination upon exiting the exclusion zone. The SSO will be responsible for verifying that proper personnel and equipment decontamination is conducted.

INCIDENT REPORTING

An incident/injury report and McLaren/Hart Health and Safety Policy 8.0 - Accident Reporting and Investigation are provided in Attachment IV. This form should be filled out by the injured employee (if possible) and site supervisor for any injury sustained on site. A copy of this form must be forwarded to Julie Panko, or Russ Palchak, Corporate Director of Health and Safety within 24 hours of the incident. Pittsburgh fax number is (412) 787-7218.

All injuries, regardless of their magnitude must be reported. Additionally, any incident which does not necessarily result in an injury (underground utility rupture, equipment failure etc.) but under other circumstances could have, is considered a "near miss" should be reported.

TRAINING

All site personnel must have completed the OSHA 40-hr HAZWOPER training and hold current 8-hr refresher certification. Site specific training will be given to all personnel prior to work activities on site and will include a review of the this Addendum, and potential chemical and physical hazards that may be encountered. Additionally, at least two on-site personnel must have current First Aid and CPR certification.

Daily tailgate safety meetings should be conducted by the Field Supervisor or SSO and documented in the Form in Attachment II.

RECORDKEEPING

All site personnel must provide the SSO with a copy of their OSHA training certificates (both 40-hr and 8-hr update), medical surveillance cards, and respirator fit-test record prior to work on this site. The SSO will maintain these copies on site for the duration of the project and then archive them with the project files.

EMERGENCY RESPONSE

A list of Emergency Phone numbers, along with a map to the nearest hospital must be posted in the support zone near all telephones.

TABLE I

ASSESSMENT OF NON-CHEMICAL HAZARDS

Non-Chemical Hazard	Yes	No	Task No.(s)	Non-Chemical Hazard	Yes	No	Task No.(s)
1. Electrical (overhead lines)	X		2,5,6,7	16. Shoring		X	
2. Electrical (underground lines)	X		2,5,6,7	17. Biologic		X	
3. Gas/Water lines	X		2,5,6,7	18. Holes/Ditches	X		2,3,5,6,7,9
4. Drilling Equipment	X		2,5	19. Steep Grades		X	
5. Excavation Equipment	X		6,7	20. Slippery Surfaces	X		2,3,5,6,7,9
6. Machinery		X		21. Uneven Terrain	X		2,3,5,6,7,9
7. Heat Exposure	X		ALL	22. Unstable Surfaces		X	
8. Cold Exposure		X		23. Elevated Surfaces (scaffolding)		X	
9. Oxygen Deficiency				24. Poor Lighting		X	
10. Confined Spaces		X		25. Vehicle Traffic		X	
11. Noise	X		2,5	26. Insects/vermin	X		2,3,5,6,7,8,9
12. Ionizing Radiation		X		27. Poisonous Plants	X		2,3,5,6,7,8,9

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13. Non-ionizing Radiation		X		28.			
14. Fire	X		2,5,6,7	29.			
15. Explosive Atmospheres	X		2,5,6,7	30.			

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TABLE II

ASSESSMENT OF CHEMICAL HAZARDS

Task No.(s)	Chemical Name (or class)	PEL/TLV *	Other Pertinent Limits *	Potential Exposure Pathways	Acute Health Effects	Chronic Health Effects
	Arsenic	C 01/0.2 mg/m^3	None Cited	Inhalation; ingestion; skin	Eye, skin & respiratory irritation	Lung, skin & lymphatic cancer; GI & skin disorders; peripheral neuropathy
	Botran (2,6-Dichloro, 4-nitroaniline)	none	none	Inhalation; ingestion; skin	unknown	unknown
	DDE	none	none	Inhalation; ingestion; skin	unknown	unknown
	DDD	none	none	Inhalation; ingestion; skin	Similar to DDT	Similar to DDT
	DDT	1.0/1.0 mg/m^3	none	Inhalation; ingestion; skin	Paresthesia tongue, lips, face; apprehension; tremors; headache; fatigue; convulsions	Liver and kidney disorders, peripheral nervous system damage, animal carcinogen
	Dieldrin	0.25/ 0.25 mg/m^3	450 mg/m^3	Inhalation; ingestion; skin	Headache, vertigo; nausea, vomiting; fatigue; muscle twitching, convulsions	carcinogen
	Endrin Ketone	0.1 mg/m^3 (as Endrin)	2000 mg/m^3 (as Endrin)	Inhalation; ingestion; skin	Headache, dizziness, nausea, vomiting, convulsions	CNS and liver damage

* PEL — OSHA Permissible Exposure Limit, represents the maximum allowable 8-hr time weighted average (TWA) exposure concentration.

TLV - ACGIH Threshold Limit Value; represents the maximum recommended 8 hr. TWA exposure concentration

STEL - OSHA Short-term Exposure Limit; represents the maximum allowable 15 minute TWA exposure concentration.

TLV-STEL — ACGIH Short-term Exposure Limit, represents the maximum recommended 15 minute TWA exposure concentration

C - OSHA Ceiling Limit, represents the maximum exposure concentration above which an employee shall not be exposed during any period without respiratory protection.

IDLH - Immediately Dangerous to Life and Health; represents the concentration at which one could be exposed for 30 minutes without experiencing escape impairing or irreversible health effects.

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TABLE II

ASSESSMENT OF CHEMICAL HAZARDS

Task No.(s)	Chemical Name (or class)	PEL/TLV *	Other Pertinent Limits *	Potential Exposure Pathways	Acute Health Effects	Chronic Health Effects
	Endosulfan Sulfate	0.1 mg/m^3 (as Endosulfan)	none	Inhalation; ingestion; skin	Unknown	Unknown
	Lindane (BHC-gamma)	0.5/0.5 mg/m^3	1000 mg/m^3	Inhalation; ingestion; skin	Convulsions, vomiting, pulmonary edema, mental/motor retardation	Potential blood disorders
	Quintozene (pentachloro-nitrobenzene)	none	none	Inhalation; ingestion; skin	Unknown	Unknown
	Naphthalene	10	STEL = 15 ppm IDLH = 500 PPM	Inhalation; ingestion; dermal	Eye irritation; headache; nausea; abdominal pain	Damage to liver, kidneys, and CNS; dermatitis
	2- Methyl Naphthalene	None cited	None cited	Inhalation; ingestion; dermal	Eye irritation; headache; nausea; abdominal pain	Damage to liver, kidneys, and CNS; dermatitis
	Nitrobenzene	1/1 ppm	IDLH = 200 ppm	Inhalation; ingestion; dermal	Eye irritation, anemia	Possible damage to blood system, liver & kidney
	Malathion	10/10mg/m^3	IDLH = 5000 mg/m^3	Inhalation; ingestion; dermal	Blurred vision, aching eyes, skin irritation, vomiting	Damage to liver, respiratory system, and CNS

* PEL = OSHA Permissible Exposure Limit; represents the maximum allowable 8-hr. time weighted average (TWA) exposure concentration.

TLV = ACGIH Threshold Limit Value; represents the maximum recommended 8-hr. TWA exposure concentration.

STEL = OSHA Short-term Exposure Limit; represents the maximum allowable 15 minute TWA exposure concentration

TLV-STEL = ACGIH Short-term Exposure Limit; represents the maximum recommended 15 minute TWA exposure concentration.

C = OSHA Ceiling Limit; represents the maximum exposure concentration above which an employee shall not be exposed during any period without respiratory protection

IDLH = Immediately Dangerous to Life and Health; represents the concentration at which one could be exposed for 30 minutes without experiencing escape-impairing or irreversible health effects.

302110

TABLE II

ASSESSMENT OF CHEMICAL HAZARDS

Task No.(s)	Chemical Name (or class)	PEL/TLV *	Other Pertinent Limits *	Potential Exposure Pathways	Acute Health Effects	Chronic Health Effects
	Sevin	5/5 mg/m^3	None cited	Inhalation; ingestion; dermal	Eye & skin irritation, blurred vision, headache, vomiting	Suspect carcinogen, mutagen & teratogen
	1,1,1 - Trichloroethane	350/350 ppm	STEL = 450 ppm C = 800 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Possible liver damage, dermatitis
	1,1,2 - Trichloroethane	10/10 ppm	None cited	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Possible liver & kidney damage; dermatitis; suspected human carcinogen
	1,2,4 - Trichlorobenzene	TLV-C 5ppm	None cited	Inhalation; ingestion; dermal	Eye & upper respiratory irritation, headache	Possible liver and kidney damage
	Tetrachloroethane	25/50 ppm	C = 300 ppm TLV-STEL = 200 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression; skin burns	Liver damage; peripheral neuropathy; suspected human carcinogen
	Phenol	5/5 ppm	IDLH = 250 ppm	Inhalation; ingestion; dermal	Eye, nose & throat irritation; weak muscle ache	Damage to kidney & liver; dermatitis

* PEL - OSHA Permissible Exposure Limit; represents the maximum allowable 8-hr time weighted average (TWA) exposure concentration.

TLV - ACGIH Threshold Limit Value, represents the maximum recommended 8-hr TWA exposure concentration

STEL - OSHA Short-term Exposure Limit; represents the maximum allowable 15 minute TWA exposure concentration.

TLV-STEL ACGIH Short-term Exposure Limit; represents the maximum recommended 15 minute TWA exposure concentration

C OSHA Ceiling Limit, represents the maximum exposure concentration above which an employee shall not be exposed during any period without respiratory protection

IDLH Immediately Dangerous to Life and Health, represents the concentration at which one could be exposed for 30 minutes without experiencing escape-impairing or irreversible health effects

302111

TABLE II

ASSESSMENT OF CHEMICAL HAZARDS

Task No.(s)	Chemical Name (or class)	PEL/TLV *	Other Pertinent Limits *	Potential Exposure Pathways	Acute Health Effects	Chronic Health Effects
	1,1 - Dichloroethene	1/5 ppm	TLV-STEL = 20 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Possible liver & kidney damage; suspected human carcinogen
	Methylene Chloride	100/50 ppm	STEL = 1000 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Possible liver & kidney damage suspected human carcinogen
	4 - Methyl 2 - Pentanone	25/25 ppm	None cited	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation, CNS depression	None cited
	Ethyl Benzene	100/100 ppm	STEL = 125 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Dermatitis
	Xylenes	100/100 ppm	STEL = 150 ppm IDLH = 1000 ppm	Inhalation; ingestion; dermal	Eye, nose & throat irritation; dizziness; nausea, vomiting; staggering gait	Damage to liver, kidneys and blood forming system; dermatitis
	Chlorobenzene	75/10 ppm	None cited	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression; skin burns	Possible liver, kidney and lung damage; dermatitis

* PEL — OSHA Permissible Exposure Limit; represents the maximum allowable 8-hr time weighted average (TWA) exposure concentration.

TLV - ACGIH Threshold Limit Value; represents the maximum recommended 8 hr. TWA exposure concentration

STEL - OSHA Short-term Exposure Limit; represents the maximum allowable 15 minute TWA exposure concentration

TLV-STEL ACGIH Short-term Exposure Limit; represents the maximum recommended 15 minute TWA exposure concentration.

C - OSHA Ceiling Limit; represents the maximum exposure concentration above which an employee shall not be exposed during any period without respiratory protection.

IDLH Immediately Dangerous to Life and Health; represents the concentration at which one could be exposed for 30 minutes without experiencing escape-impairing or irreversible health effects.

302112

TABLE II

ASSESSMENT OF CHEMICAL HAZARDS

Task No.(s)	Chemical Name (or class)	PEL/TLV *	Other Pertinent Limits *	Potential Exposure Pathways	Acute Health Effects	Chronic Health Effects
	Acetone	750/750 ppm	STEL = 1000 ppm C = 30 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Dermatitis
	Carbon Disulfide	4/10 ppm	STEL = 12 ppm C = 30 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS & peripheral nervous system depression	Possible liver, kidney & cardiovascular injury; peripheral neuropathy
	Benzene	1/10 ppm	STEL = 5 ppm TLV-STEL = 0.3 ppm	Inhalation; ingestion; dermal	Eye, skin & respiratory irritation; CNS depression	Leukemia; dermatitis
	Diethyl phthalate	TLV = 5 mg/m^3	TLV-STEL = 20 mg/m^3	Inhalation; dermal	Slight eye & skin irritation	None cited

* PEL — OSHA Permissible Exposure Limit, represents the maximum allowable 8-hr. time weighted average (TWA) exposure concentration.

TLV = ACGIH Threshold Limit Value; represents the maximum recommended 8-hr. TWA exposure concentration.

STEL — OSHA Short-term Exposure Limit; represents the maximum allowable 15 minute TWA exposure concentration

TLV-STEL — ACGIH Short-term Exposure Limit, represents the maximum recommended 15 minute TWA exposure concentration

C = OSHA Ceiling Limit; represents the maximum exposure concentration above which an employee shall not be exposed during any period without respiratory protection.

IDLH — Immediately Dangerous to Life and Health; represents the concentration at which one could be exposed for 30 minutes without experiencing escape-impairing or irreversible health effects.

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TABLE III

PERSONAL PROTECTIVE EQUIPMENT (PPE) REQUIREMENTS

Task No.(s)	Initial Level of Protection	Level if Upgrade	Air Monitoring Equipment	Monitoring Protocol	Monitored Level for PPE Upgrade *	Monitored Level For Mandatory Work Stoppage **	Additional PPE[a] for Upgrade
1,4,10-15	N/A	N/A	N/A	N/A	N/A	N/A	N/A
2,3,5,9	D+	C	PID (11.7 ev bulb), Mini-ram Dust Meter, Benzene CT	Monitor breathing zone every 30 minutes. Frequency can be decreased if contaminant concentrations remain steady below the action levels	> 5 ppm total VOCs; > 0.5 ppm benzene > 0.05 mg/m³ total dust	250 ppm total VOCs; 25 ppm benzene 10 mg/m³	APR with OV/HEPA cartridges
6,7,8	D+	C	PID (11.7 ev bulb), Mini-ram Dust Meter, Benzene CT O./LEL Meter	Monitor breathing zone every 30 minutes. Frequency can be decreased if contaminant concentrations remain steady below the action levels	> 5 ppm total VOCs; >0.5 ppm benzene > 0.05 mg/m³	250 ppm total VOCs; 25 ppm benzene 10 mg/m³ dust > 10% LEL	APR with OV/HEPA cartridges

N/A -- Not applicable because these tasks will not be conducted on site.
* Monitoring performed in worker's breathing zone.
** Call Pittsburgh Health and Safety Manager or Regional Health and Safety Manager for consultation.
*** Upgrading/downgrading of PPE is the responsibility of the SSO. No one else is permitted to make this decision unless specifically authorized by the SSO or Pittsburgh Health and Safety Manager.
**** If LEL readings exceed 10%, all work must stop, ignition sources isolated and area allowed to vent. Operations may restart when LEL is reduced to below 10%.
CT = Colorimetric Detector Tube

LEVELS OF PROTECTION :
Level D+: Hard Hats, Steel Toe PVC boots, safety glasses, latex inner gloves, neoprene outer gloves, saranex coveralls.
Level C: Level D+ and air purifying respirator with combination organic vapor (OV) and high efficiency particular air filters (HEPA) cartridges

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TO BE POSTED

TITLE	NAME	TELEPHONE NUMBER
Police Department	Police	(609) 235-0130
Fire Department	Fire	(609) 234-1111
Local Hospital	Memorial Hospital	(609) 267-0700
Local Ambulance/Rescue	Paramedics	(609) 234-1111
Pittsburgh Health & Safety Manager	Julie Panko	(800) 457-7414 or (412) 833-7833 (home)
Central Region Health and Safety Manager	Jim Less	(800) 966-8113 or (419) 536-7614 (home)
Client Contact	Mark Terril	(412) 495-5532
Site Contact	N/A	N/A
Project Manager	Dave Kerschner	(800) 457-7414 or (412) 787-7144
Site Safety Officer	Neale Misquitta	N/A
Subcontractor Contact	N/A	N/A
Corporate Human Resources Dept.	M. L. Hollingsworth	(800) 366-3696 or (916) 638-3696

6.4 HOSPITAL NAME/ADDRESS/ROUTE

Name: Memorial Hospital

Address: 175 Madison Avenue, Moorestown, NJ 08060

Route: TO BE DETERMINED

ATTACHMENT I

MCLAREN/HART POLICY AND PROCEDURES

15.0 EXCAVATION OPERATIONS

AND

16.0 DRILLING SAFETY

TITLE: EXCAVATION PROGRAM		NO: HS 15
APPROVED: Russell B. Palchak	DATE: 06-01-93 SUPERSEDES: 11-05-90	PAGE: 1 OF 3

15.1 GENERAL POLICY STATEMENT

It is McLaren/Hart's Policy to conduct all excavation/trenching operations in a manner that minimizes risk of accidental injury to McLaren/Hart and other site personnel or damage to client and McLaren/Hart property.

15.2 DEFINITIONS

Competent Person: A person capable of identifying existing and predictable excavation hazards, unsanitary or otherwise dangerous conditions and is authorized to take prompt corrective measures to eliminate them.

15.3 PROCEDURES

15.3.1 Utility Clearance

15.3.1.1 A utility clearance must be conducted prior to the commencement of any excavation/trenching operations (Form HS 15-1).

15.3.1.2 The checklist outlined in the Excavation Safety Checklist form (Form HS 15-2) must be utilized in accomplishing utility clearance and for planning excavation activities.

15.3.1.3 The locations of all utilities must be clearly identified and precautions taken to ensure utilities are not damaged by equipment/machinery utilized in the excavation activity.

15.3.1.4 Excavations in landfills will require use of ground penetrating radar or equivalent device to properly characterize soil conditions in addition to the normal utility clearance approved by the HSM prior to the beginning of any excavation activity.

15.3.2 Excavation Entry

15.3.2.1 Employees entering an excavation shall be protected from cave-ins by adequate protective systems except when:

A. Excavations are made entirely in stable rock and excavation has been approved by a competent person prior to entry;

B. Excavations are less than five (5) feet in depth and examination of the ground by a competent person provides no indication of a potential cave-in.

15.3.2.2 One, or a combination of, adequate protective systems will be provided for excavations in excess of five (5) feet in depth including:

A. Sloping or benching in accordance with applicable OSHA requirements or design by a registered professional engineer.

15.3.4 Confined Space Operations

15.3.4.1 Excavations greater than four (4) feet in depth are considered to be confined spaces if oxygen deficiencies or a hazardous atmosphere exists or could reasonably be expected to exist such as in landfill areas or excavations in nearby areas where hazardous substances are stored or where internal combustion engines are utilized.

15.3.4.2 All requirements of McLaren/Hart Health and Safety Policy HS 14, Confined Space Safety, must be implemented for all confined space operations.

15.3.5 Miscellaneous

15.3.5.1 Suitable barriers must be established to re-route traffic patterns and prevent unauthorized or accidental entry into excavation areas.

15.4 RECORDKEEPING REQUIREMENTS

The original copies of Utility Clearance Request (HS 15-1) and Excavation Safety Checklist (HS 15-2) and where applicable, Permit Required Confined Space records, should be maintained with the project file. Copies must also be forwarded to Health and Safety Managers.

McLaren Hart

Date:_____ Ticket No._____
Dig Alert Expiration Date:_____
(List Utilities Present on Back of Sheet)

ONTACT UNDERGROUND SERVICE ALERT (USA) BEFORE YOU DIG

UTILITY CLEARANCE REQUEST

(Completed by Project Manager)

Work/Clearance Previously at this location? Y ☐ N ☐

Today's Date:_____ Dig-In Start Date:_____

Project Name:_____ Task No.:_____

Exact Location of Utility Clearance (address, etc.):_____

Facility Contact Name:_____ Telephone No.:( )_____

Facility Engineer Name:_____ Telephone No.:( )_____

Site Description (Include Operational Constraints/Hazards):_____

What Facility Utility Drawings are Available?_____ If available, please provide.

Project Manager Signature _____ Assigned Field Supervisor Signature _____

(Completed by Project Manager) **CHECKLIST** (Completed by Clearance Engineer)

1. Site/Facility Drawings Available? Y ☐ N ☐
2. Checked with (Fac. Engr., Facility Contract, Other)? Y ☐ N ☐
   Emergency/Safety Shut-Off Switches Located on Utility Plan? Y ☐ N ☐

Identify Known Utility

			Field Verification (Completed by Clearance Engineer)	
			INITIAL/DATE	NOTES
A.	Water Lines		A.	
B.	Sanitary/Industrial Sewer		B.	
C.	Storm Drains		C.	
D.	Electrical Lines or Vaults		D.	
E.	Natural Gas		E.	
F.	Liquid Fuel		F.	
G.	Steam		G.	
H.	Compressor Air		H.	
I.	Telephone/Cable		I.	
J.	Overhead Lines or Pipes		J.	
K.	Others (List)		K.	

Clearance Engineer:_____
(Name - Print) (Initial) (Date)

Clearance Review:_____
(Name - Print) (Initial) (Date)

Attachments: Must be included!
- Safety Plan (By P.M.)
- Sketch (By Clearance Engineer)

Others (List):_____

Distribution:
Project Manager_____
Field Supervisor_____
Project File_____
Health and Safety Manager_____

(J:FORMS\HS15-1:6/93)

HS 15-1

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McLaren Hart

EXCAVATION SAFETY CHECKLIST

DATE:_____ PERSON COMPLETING FORM:_____

	YES	NO	DATE
I. PRE-EXCAVATION ACTIVITIES			
1. Underground installations (utilities, etc.) verified?	☐	☐	_____
2. Facility Owners or Those Affected Contacted?	☐	☐	
Company Contacted / Name of Individual			
_____ / _____	☐	☐	_____
_____ / _____	☐	☐	_____
_____ / _____	☐	☐	_____
_____ / _____	☐	☐	_____
3. Surfaces encumbrances removed?	☐	☐	_____
EXCAVATION ACTIVITIES			
1. Competent person present?	☐	☐	
NAME_____			
2. Sloping, benching, shoring or protective systems utilized?	☐	☐	
3. Assessment made by competent person daily?	☐	☐	
4. Assessment made after rainstorm or other change in soil or excavation?	☐	☐	
5. Spoils stored a minimum of two (2) feet from edge of excavation?	☐	☐	
6. All access/egress means, ladders, ramps, etc. securely fastened with a maximum distance between egress points of 25 feet?	☐	☐	
7. Barriers set up around excavation area to prevent accidental entry?	☐	☐	
III. EXCAVATION CLOSE			
1. Excavation backfilled or rendered safe by retainer wall or other means?	☐	☐	_____

IV. APPROVALS

Site Supervisor_____ Date:_____

Project Manager_____ Date:_____

HSM Review_____ Date:_____

Copy to Health and Safety Manager and Project File

(I:\FORMS\HS15-2\6/95)

302121

16.1 GENERAL POLICY STATEMENT

It is the policy of McLaren/Hart to provide a safe and healthy working environment for field employees engaged in drilling operations. McLaren/Hart requires its employees to work in a safe manner while operating or working around drilling equipment.

16.2 PROCEDURE

16.2.1 Site Supervisor (McLaren/Hart or Contractor)

The responsibility of the Site Supervisor shall include, but is not limited to, the following:

16.2.1.1 Ensure the proper use and maintenance of tools, equipment and promote good general "housekeeping" on and around the drill rig.

16.2.1.2 Ensure that the drill operator has had adequate training, including applicable state certification, and is thoroughly familiar with the drill rig, its controls and capabilities. The employer may attest to the competence of a rig operator if the Site Supervisor is not qualified to make the assessment.

16.2.1.3 Inspect the rig daily for:

A. structural damage

B. loose nuts and bolts

C. proper tension in chain drives

D. loose or missing guards or protective covers

E. fluid leaks

F. damaged hoses

G. damaged pressure gauges and relief valves

16.2.1.4 Require repair to any damaged items, including safety devices, which might compromise operational safety of the drill rig prior to the start of drilling activities.

16.2.1.5 Check and test all safety devices.

16.2.1.6 Check all gauges, warning lights and control levers.

16.2.1.7 Ensure that all new drill workers are familiar with safe operating practices on or around the rig.

16.2.1.8 Ensure that there is a properly maintained first-aid kit and fire extinguisher on each rig and on each additional vehicle on the site.

B. Remove or tag ignition key while working on engine.

C. Before working under rig:

1. block wheels
2. lower leveling jacks
3. set hand brakes

D. The rig should be reduced to a "zero energy state" by releasing pressure on appropriate hydraulic drilling fluid and air pressure systems prior to performing maintenance.

E. Extreme caution must be used when opening:

1. drain caps
2. radiator caps
3. other pressurized plugs and caps

F. Adequate time should be allowed for the engine and exhaust systems to cool before work is performed on or near those systems.

G. All caps, filler plugs, protective guards or panels, high pressure hose clamps and chains or cables that have been removed or loosened must be replaced prior to returning the drill to service.

16.2.1.12 Safe Use of Hand Tools

A. Tools should only be used for their intended purposes.

B. If a tool becomes damaged, it should be repaired or replaced.

C. All tools should be kept clean and be properly stored when not in use.

D. Maintain sound footing while using wrenches and other hand tools to avoid slips and falls.

E. Protect hands from crushing and other injuries.

16.2.1.13 Preparation of Work Area

A. Prior to drilling, the Health and Safety Plan (HASP) should be referred to for a determination of potential hazards and the site should be cleared and adequately leveled to accommodate the drill rig supplies and to provide a safe work area.

1. Biological hazards, such as poison oak and ivy, snakes, etc., should be considered during clearing process.

B. Before raising the mast, field personnel must be informed that the mast is about to be raised and asked to clear the area.

C. There should be a minimum clearance of 20 feet between mast and overhead utility wires. The HASP should list minimum distances required by local regulations, if different.

D. The leveling jacks should be positioned prior to raising the mast and should remain in position until the mast is lowered.

E. If the rig settles after initial leveling, it should be re-leveled.

F. Secure or lock the mast according to manufacturer's recommendations prior to commencement of drilling activities.

16.2.4.3 **Drill Safety Activities**

A. The operator should be at the controls when the drill rig is in operation. All controls should be in neutral, or the rig should be shut down, when operator leaves control area.

B. All tools should be hand-passed between personnel or a hoist line should be used.

C. When using a ladder on a drill rig, face the ladder and maintain 3 point contact (two legs and one arm or two arms, one leg) while ascending or descending. Use a safety belt and life line, if necessary.

D. Personnel should not touch or stand within five (5) feet of the drill rig during drilling. If the rig become energized, an employee in simultaneous contact with the ground and the rig will serve as a conductor of current between the rig and the ground. The employee would most likely suffer severe injury or death.

16.2.5 **Safe Work Under Wet or Cold Conditions**

16.2.5.1 Clean mud and grease from your boots before mounting a drill platform and use hand holds and railings. Watch for slippery ground when dismounting from the platform.

16.2.5.2 During freezing weather, do not touch any metal parts of the drill rig with exposed flesh. Freezing of moist skin to metal can occur almost instantaneously.

16.2.5.3 All air and water lines and pumps should be drained when not in use if freezing weather is expected.

16.2.7.4 All incident/emergency responses should be handled in accordance with McLaren/Hart Health and Safety Policy HS 13 and reported according to HS 8.

16.2.8 **Miscellaneous**

16.2.8.1 All unattended boreholes must be adequately barricaded and/or covered or otherwise protected to prevent drill rig personnel, site visitors, or animals from stepping or falling into the hole. All open boreholes should be covered, protected, or backfilled adequately and according to state or local regulations upon completion of the drilling project.

16.2.8.2 "Horsing around" within the vicinity of the drill rig and tool and supply storage areas should never be allowed, even when the drill rig is shut down.

16.2.9 **Personal Protective Equipment (PPE) Requirements**

16.2.9.1 It is McLaren/Hart's policy to require personnel working on or around drilling rigs to wear the proper protective equipment.

A. Steel-toed boots with minimum six (6) inch high ankle support should be worn at all times when working on or near the drilling rig.

B. Safety glasses should be worn at all times when working with hand held tools or when drilling causes dirt/debris to become airborne.

C. Specific PPE will be assigned to each work site pursuant to the project HASP.

D. Cover-alls or protective outer clothing may be required for work in areas containing hazardous contaminants.

E. Gloves appropriate to the work site should be worn.

1. Cloth/leather in cold weather

2. Vinyl/polyethylene for contaminants

F. Hard hats should be worn at all times on the work site where drilling is taking place.

G. Respirators should be worn in accordance with the McLaren/Hart Respiratory Protection Program and site-specific HASP.

ATTACHMENT II

DIRECT READING REPORT,
INSTRUMENTATION CALIBRATION LOG
AND
TAILGATE SAFETY LOG

McLaren Hart
ENVIRONMENTAL ENGINEERING CORPORATION

DIRECT READING AIR MONITORING LOG

DATE: _____

PROJECT NAME _____ PROJECT NO.: _____

Time	Location	Activity	Instrument	Substance/ Agent	Concentration	Initials

Weather Conditions: _____ **Wind Speed:** _____ **Wind Dir.:** _____ **Temp.:** _____

Comments: _____

Copy to Health and Safety Manager and project file.

H&S Manager Review _____ Date _____

(J:\FORMS\HSS-3(12/97)

HS 5-3

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McLaren Hart
ENVIRONMENTAL ENGINEERING CORPORATION

INSTRUMENT CALIBRATION LOG

Page ____ of ____

Client Name and Site:	Project Manager:	Task Number:

Calibration Event:

Person Calibrating:	Date:
Instrument Type:	Calibration Gas:
Model:	Calibration Gas Concentration (ppm):
Serial #:	Reading (ppm):
Calibrator Model:	Adjusted Reading (If Necessary):
Comments:	

Person Calibrating:	Date:
Instrument Type:	Calibration Gas:
Model:	Calibration Gas Concentration (ppm):
Serial #:	Reading (ppm):
Calibrator Model:	Adjusted Reading (If Necessary):
Comments:	

Person Calibrating:	Date:
Instrument Type:	Calibration Gas:
Model:	Calibration Gas Concentration (ppm):
Serial #:	Reading (ppm):
Calibrator Model:	Adjusted Reading (If Necessary):
Comments:	

Person Calibrating:	Date:
Instrument Type:	Calibration Gas:
Model:	Calibration Gas Concentration (ppm):
Serial #:	Reading (ppm):
Calibrator Model:	Adjusted Reading (If Necessary):
Comments:	

Person Calibrating:	Date:
Instrument Type:	Calibration Gas:
Model:	Calibration Gas Concentration (ppm):
Serial #:	Reading (ppm):
Calibrator Model:	Adjusted Reading (If Necessary):
Comments:	

Comments:

NOTE: Return to HSM Upon Completion of Site Work.

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(HIO:CALIBRAT.FRM;4/15/93)

McLaren Hart
ENVIRONMENTAL ENGINEERING CORPORATION

TAILGATE SAFETY MEETING

DATE_____ TIME_____ PROJECT NO._____

SITE LOCATION_____

_____

TYPE OF TRAINING

_____ **Technical Transfer/H&S Meeting** _____ **Tailgate Safety Meeting**

_____ **HASP Reading/Review** _____ **Other:**_____

_____

TRAINING PRESENTED BY:_____

TOPICS COVERED:_____

_____

_____

ATTENDEES

NAME PRINT	SIGNATURE

SITE SUPERVISOR:_____ **DATE:**_____

(Copy to Health and Safety Manager and project file.)
H&S Manager Review_____ Date_____

HS 5-2

302129

ATTACHMENT III

DECONTAMINATION PROCEDURES

DECONTAMINATION EQUIPMENT

Station 1: Equipment Drop

a. Various Size Containers
b. Plastic Liners
c. Plastic Drop Cloths

Station 2: Outer Garment, Boots, and Gloves Wash and Rinse

a. Containers (20-30 Gallons)
b. Decon Solution
c. Rinse Water
d. Long-Handled, Scrub Brushes (2-3)

Station 3: Outer Boot and Glove Removal

a. Containers (20-30 Gallons)
b. Plastic Liners
c. Bench or Stools

Station 4: SCBA Cylinder Change

a. SCBA Cylinders or Masks and Cartridges Depending Upon Level
b. Tape
c. Boot Covers
d. Gloves

Station 5: Boot, Gloves, and Outer Garment Removal

a. Containers (20-30 Gallons)
b. Plastic Liners
c. Bench or Stools

Station 6: SCBA Removal

a. Plastic Sheets
b. Basin or Bucket
c. Soap and Towels
d. Bench or Stools

Station 7: Field Wash

a. Water
b. Soap
c. Tables
d. Wash Basin or Bucket

MODIFIED LEVEL D AND LEVEL C PERSONNEL DECONTAMINATION

Station 1:	Equipment Drop	1. Deposit equipment used on-site (tools, sampling devices and containers, monitoring instruments, radios, clipboards, etc.) on plastic drop cloths. Segregation at the drop reduces the probability of cross contamination. During hot weather operations, cool down station may be set up within this area.
Station 2:	Outer Garment, boots, and Gloves	2. Scrub outer boots, outer gloves and splash suit with decon solution or detergent water. Rinse off using copious amounts of water.
Station 3:	Outer Boot and Glove Removal	3. Remove outer boots and gloves. Deposit in container with plastic liner.
Station 4:	Respirator Cartridge or Mask Change	4. If worker leaves Exclusion Zone to change respirator cartridge (or mask), this is the last step in the decontamination procedure. Worker's respirator cartridge is exchanged, new outer gloves and boot covers donned, joints taped, and worker returns to duty.
Station 5:	Boot, Gloves, and Outer Garment Removal	5. Remove boots, chemical-resistant splash suit, and gloves and deposit in separate containers lined with plastic.
Station 6:	Respirator Facepiece	(Level C only) 6. Remove respirator facepiece. Avoid touching face with fingers Deposit respirator facepiece on plastic sheet.
Station 7:	Field Wash	7. Thoroughly wash hands and face. Shower as soon as possible.

ATTACHMENT IV

ACCIDENT/INJURY REPORT

AND

HS8 - ACCIDENT REPORTING AND INVESTIGATION

McLaren/Hart Health and Safety Policy

TITLE: ACCIDENT REPORTING AND INVESTIGATION		NO: HS 8
APPROVED: Russell B. Palchak	DATE: 06-01-93 SUPERSEDES: 11-07-90	PAGE: 1 OF 3

8.1 GENERAL POLICY STATEMENT

It is the Policy of McLaren/Hart to establish requirements and responsibilities for the reporting and investigation of accidents/incidents. All accidents/incidents must be reported to Corporate Health and Safety within 24 hours of the event. Major accidents/incidents require special procedures pursuant to Sandoz Directive #8, including immediate reporting. See Section 8.4.5.

8.2 DEFINITIONS

8.2.1 Accident/Injury is an unexpected incident or event that causes injury, illness or death to personnel or damage to property or the environment.

8.2.2 Accident, for the purpose of this policy, includes both accidents and incidents.

8.2.3 Major accident/incident (See Sandoz Directive #8).

8.3 RESPONSIBILITIES

8.3.1 **Corporate Health and Safety Director (CHSD)** shall appoint Accident review Boards for accident cases when necessary and maintain records.

8.3.2 **Regional Health and Safety Manager (RHSM)** will coordinate investigations of accidents in their region and participate in investigations in other regions as requested by the CHSD.

8.3.3 **Health and Safety Manager (HSM)** is responsible for immediate investigation of accidents.

8.3.4 **Project Managers (PM)** directly responsible for the company or client property or personnel involved, assure the prompt reporting of accidents to the HSM and **Corporate Health and Safety.**

8.3.5 **Field Supervisor/Site Safety Officer** is responsible for the immediate reporting of accidents involving employees to the Project Managers.

8.3.6 **Employees** are responsible for reporting all accidents/incidents to their immediate supervisors immediately, whenever practical, but in no case later than 24 hours of occurrence. In the event of serious injury or fatality, reporting must be immediate.

8.4 PROCEDURES

8.4.1 Accidents are to be reported and investigated for the purpose of identifying causative factors and corrective measures to avoid similar occurrences and to comply with governmental requirements.

8.4.2 All accidents are reported regardless of the severity of the injury or the lack thereof. Accident/Injury Report (Form HS 8-1), must be completed by the Project Manager or designee within 24 hours of occurrence. All accidents involving motor vehicles must be reported on the Motor Vehicle Accident Report (Form HS-8-2).

8.4.3 The Human Resources Representative at each office, or designee, is the central reporting point for accidents. The RHSM, CHSD and HSM must also receive a copy within 24 hours.

8.4.4 The Project Manager shall complete and file the Accident/Injury Report with HSM within 24 hours of the accident or on the first work day following the accident; the CHSD will receive copies of the Accident/Injury Report within 24 hours of the accident, or on the first work day following the accident.

8.4.5 If an accident involves **hospitalization of five (5) or more employees or a fatality**, the HSM must notify the appropriate local OSHA Office, the RHSM and CHSD within 24 hours of the accident. This includes weekend days-24 hour emergency reporting. Numbers are available from the Regional Manager, Regional Director or Corporate Executives. The report to the agency must include the following ten (10) items:

8.4.5.1 Time and date of accident.
8.4.5.2 Employer's name, address and telephone number.
8.4.5.3 Name and job title, or employee number of person reporting the accident.
8.4.5.4 Address of site of accident or event.
8.4.5.5 Name of person to contact at site of accident.
8.4.5.6 Name and address of injured employee(s).
8.4.5.7 Nature of injury.
8.4.5.8 Location where injured employee(s) was (were) moved to.
8.4.5.9 List and identify of other law enforcement agencies present at the site of accident.
8.4.5.10 Description of accident and whether the accident scene or instrumentality has been alerted.

Additionally, reporting to Sandoz is required according to Sandoz Directive #8. ONLY the Vice President of Corporate Programs or the CHSD is authorized to complete such reporting to Sandoz.

8.4.6 The CHSD, RHSM and HSM shall review all Accident/Injury Reports and accident investigation reports. All regional investigative material must be forwarded to the CHSD.

8.4.6.1 A review of incidents will be made to determine the need for procedural and safety training improvements.

8.4.6.2 A determination shall be made by the CHSD, in conjunction with the RHSM and the attending physician, as to the recordability of the accident. The classification and recordability shall be in compliance with OSHA 29 CFR 1904.

8.4.6.3 Major accident/injuries require special procedures, see Sandoz Directive #8. Mary Lynn Hollingsworth is the designated spokesperson for all media and Sandoz correspondence.

8.4.6.4 Motor Vehicle Accident Reports will be forwarded to the Contracts Department for insurance reporting.

8.4.6.5 Periodic reports summarizing accident descriptions and associated statistics will be made by the CHSD to company management.

8.5 RECORDKEEPING REQUIREMENT

8.5.1 Corporate Health and Safety shall maintain copies of the Accident/Injury Reports.

8.5.2 The log and summary OSHA No. 200 shall be maintained and updated by CHSD as necessary for five (5) years. (See McLaren/Hart's Health and Safety Policy HS 9, OSHA Recordkeeping and Workers' Compensation).

8.5.3 Corporate Health and Safety will provide office administrators/Human Resource Representative with the previous year's log and summary OSHA No. 200 to be posted on employee notice bulletin boards from February 1 through March 1 as required by 29 CFR 1904.

McLaren Hart

ACCIDENT/INJURY REPORT

(Submit a separate report for each incident, attach additional pages if needed)

Employee/Subcontractor Name:_____ SS#____-____-____
Sex: M ___ F ___ Date of Hire: _____ Date of Birth: _____
Title:_____ M/H Office:_____
Project Name and Exact Location: _____
_____ Project #:_____
M/H Office Managing Project:_____ Length of Time on Project:_____

Date of Incident ____-____-____ Time: _____ a.m. or p.m. Report Date___-___-___
Lost Time? Yes___ No____ Estimated Date of Return: ___-___-___
Employee Sent to: MD ____ Hospital____ Home____ Remained working_____
Medical Care Received: Yes____ No____ Where? On-site ____ Off-site ____
Treatment Offered but Declined:_____ (employee to initial)
If on-site treatment or first aid, by whom?_____
Person providing treatment or first aid exposed to blood or body fluids? Yes____ No____
If off-site, name facility (hospital, clinic, etc)_____
Length of stay at facility:_____ Treatment Rendered:_____
Nature of Injury or Chemical Exposure (specify chemical) _____
Contusion ___ Cut ___ Infection ___ Irritation ___ Puncture ___ Strain ____ Fracture ____
Other_____
Body Part(s) Involved: (Be as specific as possible):_____

Incident Description:_____
_____
_____
What was the employee doing when the incident occurred:_____
_____
Act(s) that caused or contributed to incident:_____
_____
Emergency Services Required? Yes ___ No ___ If yes, provide affiliation:_____
Ambulance _____ Paramedics _____ Fire Dept _____ Law Enforcement _____
WITNESS(ES) NAME(S): (Provide address if witness is not an employee)
1. _____ 2. _____
_____ _____
Motor Vehicle Involved? Yes __ No __ If yes, complete Motor Vehicle Accident Report
Exposure to Toxic Materials or Radiation? Yes __ No __ If yes, specify:_____

Reported by:_____ Date: _____

For Corporate Health and Safety Use Only:

☐ OSHA Recordable ☐ First Aid ☐ Report Only ☐ Workers' Compensation

Comments:

Fax to Russ Palchak, Corporate Health and Safety Director, Pittsburgh, PA (412) 787-8210 within 24 hours.
Copy to Office Health and Safety Manager within 24 hours.

(J:FORMS\HS8-1\5/93)

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INCIDENT INVESTIGATION REPORT
(Supervisor/Project Manager to complete)

ıployee/Subcontractor Name:_____

List the names of other persons affected during the incident:_____

What was employee doing when the incident occurred?_____

What act(s) caused or contributed to the incident?_____

What condition(s) caused or contributed to the incident?_____

Was the employee using protective clothing and equipment? (Describe)_____

What action has been taken to prevent recurrence?_____

Employee new to the job? Yes ___ No ___

What on-the-job training has the employee received?_____

Was the employee informed of all the hazards associated with the job? Yes ___ No ___

Are there site or job safety rules which are applicable to the employee's job? Yes ___ No ___

Does the employee know these safety rules? Yes ___ No ___

If No, Why?_____

Is there a written procedure for the employee's job? Yes ___ No ___

Does the written procedure mention hazards? Yes ___ No ___

Have these procedures been reviewed with the employee? Yes ___ No ___

If Yes, when? _____-_____-_____

Did accident occur on overtime? Yes ___ No ___

List any other contributing factors:_____

INVESTIGATED BY:	REVIEWED BY:
_____	_____
Supervisor (Signature)	Health and Safety Manager (Signature)
_____	_____
Supervisor (Print or Type)	Health and Safety Manager (Print or Type)
_____	_____
ıte	Date

McLaren Hart

MOTOR VEHICLE ACCIDENT REPORT

(Attach to Accident/Injury Report (HS 8-1) if Motor Vehicle is Involved in Accident)

'ICLE(S) INVOLVED:

Vehicle ID #	Vehicle Make and Model	Vehicle License Plate #	Registered Owner	Insurance Co./ Policy #
1				
2				
3				
4				

DAMAGE SUSTAINED:

Vehicle ID #	Damage Description	Estimated Cost to Repair
1		
2		
3		
4		

DRIVER INFORMATION:

Vehicle ID #	Driver Name	Driver License #	Date of Birth	Citations Issued?	Passengers Involved
1				Y or N	Y or N
2				Y or N	Y or N
3				Y or N	Y or N
4				Y or N	Y or N

PASSENGERS INVOLVED:

Vehicle ID #	Name and Address	Phone Number
1		
2		
3		
4		

NOTE: Diagram the accident on reverse side of this report using the vehicle numbers above in your diagram. Include approximate speed, weather conditions, and any other pertinent details. include this diagram in any correspondence.

HEALTH AND SAFETY PLAN

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HEALTH AND SAFETY PLAN
PHASE I STUDY AREA INVESTIGATION
PULVERIZING SERVICES SITE
MOORESTOWN, NEW JERSEY

1.0 INTRODUCTION

Rizzo Associates maintains a corporate health and safety program in accordance with OSHA regulations. This Health and Safety Plan (HSP) presents the adaptation of the corporate program to the investigation of the Phase I Study Area of the Pulverizing Services Site. The project specific HSP will be implemented and followed by Rizzo Associates' personnel and subcontractors during the field investigation. Rizzo Associates' policy is to conduct all activities so that the health and safety of the project personnel and public are protected. This document provides health and safety procedures that have been or will be followed in conjunction with the following activities:

- Surveying;
- Geophysical Investigation;
- Drilling;
- Sampling; and
- Monitoring well installation.

The plan does not include health and safety procedures for site activities other than those listed above.

1.1 SITE DESCRIPTION

Industrial activities involving pesticide grinding, micronizing, and blending first occurred at the Pulverizing Services Site in 1935. Initially, inorganic pesticides such as lead arsenate, calcium arsenate, and sulfur predominated. In later years, organic pesticides such as DDT, eldrin, malathion, dieldrin, lindane, and several other common pesticides were handled on the site. It is important to note that these chemical pesticides were not actually manufactured at the site. Site operations included grinding, blending, and packaging for distribution

under various company labels. PPG owned and operated the site from approximately 1948 until the fall of 1963. During the late 1950s and early 1960s, waste may have been disposed behind the main production buildings in several trenches. Pulverizing Services, Inc. purchased the site in 1963 and operated it until 1977.

The New Jersey Department of Environmental Protection (NJDEP) initiated enforcement action in April 1985 against Pulverizing Services, Inc. Samples were taken in 1986 confirming the presence of pesticides in soils. In June of 1987, the NJDEP issued an Administrative Order against PPG. Late in 1987, the USEPA took over the lead agency role of the site. During the fall of 1987, the USEPA collected a substantial number of soil samples and performed a ground-penetrating radar survey. An Administrative Order on Consent was entered into by the USEPA and PPG for the installation of security fencing at the site in May of 1988. This investigation of the Phase I Study Area is being performed under an Administrative Order on Consent between PPG and the USEPA dated March 31, 1989 (II-CERCLA-80109).

1.2 PROJECT DESCRIPTION

The investigation of Phase I Study Area will focus on suspected land disposal locations. The Scope of Work includes a geophysical investigation and land survey (already completed) and characterization of vertical and horizontal extent of soil and groundwater contamination through the sampling of borings and monitoring wells. The investigation of the site buildings and underground tanks are not part of this project.

1.3 TERMINOLOGY AND DEFINITIONS

The following terms and abbreviations are used throughout this document:

- ACGIH - American Conference of Governmental Industrial Hygienists.
- ANSI - American National Standard Institute.

- APR - Air Purifying Respirator; these are available in two types--full-face and half-face.
- Area Monitoring - Monitoring of airborne contamination in a work area.
- Contamination Reduction Zone - Area external to exclusion zone and established as a buffer between exclusion zone and support zone.
- Exclusion Zone - Area where admittance is prohibited without protective clothing and equipment.
- HSC - Health and Safety Coordinator.
- NIOSH - National Institute for Occupational Safety and Health.
- OSHA - Occupational Safety and Health Administration
- SHSO - Site Health and Safety Officer.
- HSP - Health and Safety Plan.
- Support Zone - Area outside the exclusion zone which is reached by passing through the contamination reduction zone.
- USCG - United States Coast Guard.
- USEPA - United States Environmental Protection Agency.

1.4 REFERENCES

The following references were used in preparing this plan:

- RFP and Addendum No. 1 from PPG Industries, Inc. for performance of the Phase I Study Area investigation dated March 31, 1989 and April 13, 1989.
- REAC Trip Report (USEPA) dated March 18, 1988.

- Sampling Report - USEPA Technical Assistance Team and Environmental Response Team October 1987 Sampling Report.
- Standard First Aid Manual, American Red Cross.
- OSHA Safety and Health Standards, 29 CFR 1910 and 1926 (specifically 1910.120).
- Superfund Amendments and Reauthorization Act of 1986, (Public Law 99-499).
- American National Standard Practices for Respiratory Protection, ANSI, Z 88.2-1980.
- Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/ OSHA/USCG/USEPA, 1985.
- A Guide to Industrial Respiratory Protection, NIOSH, 1976.
- Standard Operating Safety Guides, USEPA, 1988.
- NIOSH Manual of Analytical Methods, 3rd Edition, 1984.
- Registry of Toxic Effects of Chemical Substances, NIOSH, 1981-1982.
- Occupational Health Guidelines for Chemical Hazards, NIOSH/OSHA, 1981.
- Threshold Limit Values and Biological Exposure Indices for 1988-89, ACGIH.
- Dangerous Properties of Industrial Materials, Sax, 6th Edition, 1984.
- Personal Protective Equipment for Hazardous Material Incidents: A Selection Guide, NIOSH, 1984.
- Proceedings of a NIOSH Workshop on Recommended Heat Stress Standards, NIOSH, 1979.

2.0 PROGRAM ORGANIZATION AND ADMINISTRATION

The Rizzo Associates' project manager and project staff will be responsible for continuous adherence to the safety procedures prescribed herein during the conduct of the work.

The HSP is accomplished through an integrated team effort of the following key project staff:

- J. Timothy Onstott - Project Manager
  Overall responsibility for all project activities.

- Kenneth J. Bird - Health and Safety Coordinator (HSC)
  Provides corporate health and safety support to project staff, evaluates the effectiveness of the HSP, and revises the HSP to reflect any changes in site conditions.

- On-Site Health and Safety Officer (SHSO) - (Senior Individual On Site)
  Responsible for executing HSP requirements on site.

An SHSO will be designated for each field task if Mr. Bird is not present on site. The SHSO will have the required training (Section 5.0) and experience to implement the HSP. The SHSO will be designated by Mr. Bird based on the needs of each task.

3.0 MEDICAL SURVEILLANCE

Rizzo Associates' employees participate in a medical surveillance program which is administered through the Occupational Medicine Department of the University of Pittsburgh. The physicians in the Occupational Medicine Department are aware of Rizzo Associates' projects and OSHA regulations.

Each potentially exposed employee has an individually designed medical surveillance program based on possible exposure. Employees are given a baseline physical prior to being exposed to a hazardous situation. Employees are given examinations either annually in accordance with OSHA (employee having 30 days of exposure per year) or whenever they accumulate 30 days of exposure. At a minimum, all field personnel receive a medical examination every two years. Medical examinations are also mandatory for employees who may have been exposed to hazardous substances at concentrations above the permissible limits or if the employee has developed symptoms indicating possible overexposure. The medical consultant (Occupational Medicine Department) can increase the frequency of examinations if medically necessary.

The baseline medical examination includes:

- Occupational, environmental, and family history;
- Comprehensive physical examination;
- Biochemical profile, including CBC with differential;
- Urinalysis;
- Pulmonary function test;
- Audiometric testing;
- Chest x-ray; and
- Electrocardiogram.

Chest x-ray and electrocardiograms are not part of the periodic examinations unless the physician determines it is necessary. The other

tests are performed in the periodic examination. Medical records are maintained by the University of Pittsburgh in accordance with federal regulations.

Rizzo Associates' health and safety department receives correspondence on the following three issues:

- If employee is medically able to wear a respirator;
- If employee is medically able to work on a hazardous waste site; and
- If employee has occupational-related medical limitations of which Rizzo Associates should be aware (bee sting allergy, bad back).

Non-work related medical problems are only discussed between the individual and physician.

All subcontractor personnel on site will be required to demonstrate that they participate in a medical surveillance program in accordance with OSHA.

4.0 HAZARD ASSESSMENT

An evaluation of conditions at the site has been made to determine the potential effects upon site personnel during the field activities. This evaluation is based on the following:

- Nature of potential contaminants.
- Locations of potential contaminants at specific work areas.
- Estimated levels of contamination.
- Potential for personnel and public exposure during various site activities.
- Effects of potential contaminants on human health.

Exposure potential is defined as the probability of a worker or public receiving harmful exposures.

Based upon existing data, air does not appear to be a major exposure route for toxic compounds. Volatile organics were not detected in the NJDEP or USEPA investigations. Contaminants of concern are listed in Table HSP-1. The principal concern is for dermal contact with contaminated soil or dust and possible aspiration of contaminated dust. Concentrations of pesticide compounds in the Area A surface soils ranged up to 2,400 ug/g. Inadvertent ingestion is also a potential concern. Personal protection requirements (Section 7.0) and site management controls have been explicitly designed to control this exposure potential.

Public exposure potential will not be significantly changed by the activities undertaken during this investigation.

5.0 TRAINING

Rizzo Associates' project personnel have received training in accordance with OSHA 29 CFR 1910.120. In addition, the HSC will design a site-specific training session. This training session will be held at the site prior to the initiation of work. It will be conducted by the HSC or SHSO and will include the following topics:

- Site history;
- Compounds identified on site;
- Explanation of acute and chronic effects of toxic chemicals identified at the site;
- Site requirements for personnel protection (respirators, etc.), effectiveness, and limitations;
- Prohibited actions or procedures in designated work zones;
- Safety precautions and buddy system explained;
- Accident preventive procedures;
- Decontamination procedures;
- Work zones and site control procedures;
- Health and safety personnel and organization;
- Air monitoring program; and
- Symptoms and treatment of heat-related illness.

Rizzo Associates' site personnel performing the geophysical survey and associated land surveying and performing sampling and observing drilling and well installation will receive appropriate training as described above.

The drilling subcontractor is required to have training in accordance with OSHA requirements. Documentation of the training will be retained by Rizzo Associates for the project files. Subcontractor personnel will receive site-specific training as described above.

Weekly safety meetings and other refresher training will be held by the SHSO or the HSC. Safety problems, changes in site conditions, monitoring results, and other safety-related topics, as appropriate,

will be discussed. Site visitors will be informed of the site hazards and instructed by the HSC or SHSO in the use of protective gear. Records of personnel attendance at training sessions and safety meetings will be maintained.

6.0 WORK AREAS

The unpaved areas of Area A (Figure 2 of the Site Operations Plan) are designated as the exclusion zones for this investigation. The required protective equipment varies by the activities proposed for this area as described in Section 7.0.

Soils surrounding the asphalt areas will be considered potentially contaminated; vehicles and equipment used within these areas will be steam cleaned prior to leaving the site. The contamination reduction zone will be located on the asphalt next to Building No. 29, through which entrance and egress from Area A will be controlled. During the geophysical survey and active drilling operations, entrance to the work area will be limited to those individuals having proper protective equipment.

Eating/drinking, chewing, smoking, and non-use of required protective clothing are forbidden in Area A.

7.0 PERSONNEL PROTECTIVE EQUIPMENT

7.1 GENERAL

Equipment for personnel protection is based on USEPA levels of protection as specified in the Standard Operating Safety Guides (USEPA, 1988). Protective equipment will be compatible with and provide protection from the class of compounds known to be on site and from any additional compounds identified during the investigation. The protective equipment will provide respiratory protection and skin and eye protection for personnel operating in the designated exclusion and contamination reduction zones. Protective equipment selection is also based on task-specific conditions.

The following sections describe the minimum protective equipment for initial use based on task and location. The results of the air monitoring program (Section 8.0) or observed site conditions may induce changes (upgrade/downgrade) to the protection required. Potential changes could be more or less personnel protective equipment, depending upon the type and concentration of contaminants detected.

Initially, all personnel performing sampling and analyses for the project will be required to wear the following basic clothing:

- Work clothes;
- Steel Toe/shank, chemical-resistant (Neoprene, PVC) boots;
- Eye protection; and

Additional protective equipment will be required in certain areas and at certain times, as described in the following subsections.

7.2 SURVEYING AND GEOPHYSICAL INVESTIGATION

The nature of the activities involved in surveying and geophysical investigation will not disturb the ground surface or produce significant quantities of dust. Personnel involved in these activities will wear the basic clothing and the following:

- Latex gloves
- Regular tyvek

Respiratory protection will be based on air monitoring measurements and site observations by the SHSO and as approved by the HSC.

7.3 DRILLING, SAMPLING, AND MONITORING WELL INSTALLATION ACTIVITIES

In addition to the basic work clothing requirements, the following personal protective gear will be worn during drilling, sampling, and monitoring well installation activities within Area A:

- Hooded PVC or Saran Tyvek coverall;
- Nitrile outer gloves;
- Latex inner gloves;
- Full-face air purifying respirator with GMP cartridges, high efficiency; and
- Hard hat.

Air purifying respirators will be worn at all time personnel are potentially exposed, unless more protection is required as determined by air monitoring (Section 8.0). Hoods, boots, and gloves will be properly taped to reduce potential for skin exposure. If breathing zone air measurements for organic vapors are above background, additional respiratory protection will be worn. Depending upon the measurement, supplied air systems will be worn in accordance with the action levels listed in Section 8.0.

8.0 AIR MONITORING

Although as described in Section 4.0, airborne contaminants do not appear to present a significant threat to worker or public health and safety, air monitoring will play an important part in the HSP. Area air monitoring for total volatile organic compounds will be performed with an HNu (10.2 eV) or flame ionization organic vapor analyzer. Continuous monitoring will occur during drilling.

The action levels and required responses are as follows:

- Level I
  Organic Vapor (OV) not above background ($\leq$5 ppm); no response required.

- Level II
  OV measurements of background up to 10 ppm. All workers are required to use air purifying respirators with pesticide cartridges with dust filter (or equivalent).

- Level III
  OV concentrations 10-500 ppm for unknown compounds. All workers are required to use supplied air respiratory protection equipment. This level is not anticipated to occur at this site.

- Level IV
  OV measurements in excess of 500 ppm. Remove personnel and treat as an emergency situation. Reduce exposure by suspending operations and covering the potential source with soil to reduce airborne concentrations. The USEPA shall be notified. Level IV is not anticipated to occur during this project.

OV concentration will be measured in worker breathing zone. Background will be determined daily upwind of the site.

9.0 PERSONNEL DECONTAMINATION

Decontamination will occur when the potential for the spread of contamination exists. Decontamination stations will be established at the edge of the asphalt road near Building No. 29. The stations will consist of the following:

- Boot and glove wash/rinse;
- Removal of disposable clothing;
- Hard hat and respirator wash/rinse; and
- Hands and face wash.

Disposable clothing will be stored at the facility.

10.0 RESPIRATORY PROTECTION PROGRAM

A respiratory protection program that meets OSHA Standard 29 CFR 1910.134 will be implemented. The training required by OSHA covers maintenance and cleaning of respirators. Each individual will be responsible for their own respirator. Rizzo Associates will use respiratory equipment manufactured by MSA.

Respirators will be stored in a clean, sanitary area where they are protected against mechanical damage, dust, heat, extreme cold, excessive moisture, or damage by chemical contact. A qualitative fit test will be employed for each individual required to wear a respirator.

11.0 HEAT/COLD STRESS

11.1 HEAT-RELATED ILLNESS

Adverse weather conditions are important considerations in planning and conducting site operations. Hot or cold weather can cause physical discomfort, loss of efficiency, and personnel injury. Of particular importance during this project is heat stress.

The following control measures will be employed as necessary to help control heat stress:

- Provision for adequate liquids to replace lost body fluids. Employees must replace water and salt lost through perspiration. Employees will be encouraged to drink more than the amount required to satisfy thirst, since thirst satisfaction is not an accurate indicator of adequate salt and fluid replacement.
- Replacement fluids can be a 0.1 percent salt solution, commercial mixes such as Gatorade or Quick Kick, or a combination of these with fresh water.
- Establishment of a work-rest regimen that will provide adequate rest periods for cooling down.
- Use of cooling devices such as vortex tubes or cooling vests which can be worn beneath protective garments.
- Breaks taken in a cool rest area.
- Employees not being assigned other tasks during rest periods.
- Employees being informed of the importance of adequate rest, acclimatization, and proper diet in the prevention of heat stress.

12.0 REPORTS AND RECORD KEEPING

Records of health and safety activities will be maintained in accordance with OSHA requirements. The records will document air monitoring levels, protective equipment worn, incidents, and training.

12.1 LOGS AND REPORTS

Rizzo Associates will maintain logs and reports covering the implementation of the HSP. The format will include training logs, daily logs, incident reports, and medical certificates:

- Training Logs (shall be completed for both initial training and refresher training):

  - employee signature;
  - topics covered;
  - date;
  - time; and
  - signature of trainer(s).

- Daily Logs:

  - date;
  - area (site specific) checked;
  - employees in a particular area;
  - equipment utilized by employees and job function;
  - protective clothing and devices worn by employees;
  - area air monitoring results;
  - perimeter air monitoring results; and
  - SHSO signature and date.

- Incident Report - Describing injuries, off-site release or accident.

- Medical Certificates - Documenting that a site employee is in a medical surveillance program.

- Training Certificates - Documenting that employees have received training in accordance with OSHA.

12.2 RECORD KEEPING

Rizzo Associates will maintain health and safety records for 30 years after completion of the project. Access to records by employees is permitted as required under state and federal regulations.

13.0 EMERGENCY RESPONSE

Emergency response procedures have been developed to cover extraordinary conditions that may occur. If emergencies do arise, they are anticipated to most likely be typical construction site emergencies such as equipment fire and minor worker injury (cuts, bruises). Complex situations dealing with uncontrolled hazards are very unlikely.

13.1 GENERAL RESPONSE CONSIDERATIONS

Emergencies must be dealt with in a manner that minimizes the health and safety risk to site personnel and the public. For the following sections, it is assumed the HSC is not present on site. If HSC is present, he will assume the SHSO responsibilities described below.

The following procedures shall be implemented in the event of an emergency:

- First aid or other appropriate initial action will be administered by those closest to the accident/event. This assistance will be coordinated by the ranking individual on site and will be conducted in a manner so that those rendering assistance are not placed in a situation of unacceptable risk. The primary concern is to avoid placing a greater number of workers in jeopardy.

- Employees shall report all accidents and unusual events to:

  - SHSO;
  - Project Manager; and
  - HSC.

- The SHSO is responsible for conducting the emergency response in an efficient, rapid, and safe manner. The SHSO will decide if off-site assistance and/or medical treatment is required and shall be responsible for alerting off-site authorities and arranging for their assistance.

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- The SHSO will provide to the above personnel an Incident Report which includes the following:

  - A description of the emergency (including date, time, and duration).

  - Date, time, and name of all persons/agencies notified and their response.

  - A description of corrective actions implemented or other resolution of the incident.

- All workers on site are responsible for conducting themselves in a mature, calm manner in the event of an accident/unusual event. All personnel must conduct themselves in a manner to avoid spreading the danger to themselves and to surrounding workers.

13.2 RESPONSIBILITIES

The SHSO or a designated substitute shall have responsibility for directing response activities in the event of an emergency. He will:

- Assess the situation.
- Determine required response measures.
- Notify appropriate response teams.
- Determine and direct on-site personnel during the emergency.
- Contact and coordinate with government agencies.

He shall coordinate his response activities with those of public agencies.

The SHSO shall be responsible for implementing the HSP emergency response provisions for all personnel and visitors on site. The SHSO shall know what personnel are on site and where they are located.

13.3 PUBLIC RESPONSE AGENCIES

The following is a list of public response agencies to be contacted and who may, depending on the nature of the situation, assume authority for emergency response. In the event of an emergency situation, site personnel shall lend all necessary assistance to the agency in charge.

Immediate Emergencies	Phone Numbers
Police	235-0130
Fire Department	234-1111
Ambulance	234-1111
Memorial Hospital	267-0700
Emergency Support	
New Jersey Department of Environmental Protection (NJDEP)	609-292-7172
USEPA Region II	212-264-8674
USEPA (24-hour Hotline)	201-584-8730
USEPA National Response Center	800-424-8802
Rizzo Associates Medical Consultant	412-648-3240

The nearest public telephone is located at the Citgo service station at the intersection of Camden Avenue and New Albany Road.

13.4 EMERGENCY RESPONSE EQUIPMENT

Before site operations are initiated, the following emergency equipment will be provided at the site:

- First aid kit. Contents of the kit meet OSHA Standard 1910.151 requirements.
- Portable eyewash bottles.
- Chemical fire extinguishers will be provided on site on each piece of heavy equipment; type ABC, 20 pounds.
- List of persons and phone numbers for emergency notification.

Other equipment used for the routine implementation of the worker health and safety protection and monitoring programs will be made available as needed to support any emergency response activity.

13.5 ACCIDENTS AND NON-ROUTINE EVENTS

Several types of emergencies are outlined in the following subsections. These are not intended to cover all potential situations, and the corresponding response procedures should not be followed blindly. Every accident is a unique event that must be dealt with by

trained personnel working in a calm, controlled manner. In the event of an accident/unusual event, the prime consideration is to provide the appropriate initial response to assist those in jeopardy without placing additional personnel at an unnecessary risk.

The vast majority of worker injuries on hazardous waste sites are non-chemical in nature. The injuries tend to be sprains, rashes, and lacerations which, because of protective equipment and site conditions, must be treated promptly. Follow-up care is extremely important to assure that a minor injury or illness does not become aggravated by site conditions. Employees shall be instructed to report all injuries and illnesses to the HSO.

13.5.1 Worker Injury

If a person working in a contaminated area is physically injured, Red Cross first aid procedures shall be followed. Depending on the severity of the injury, emergency medical response may be sought. If the employee can be moved, he will be taken to the edge of the work area where contaminated clothing will be removed, emergency first aid administered, and transported to the hospital.

If the injury to the worker is chemical in nature (e.g., overexposure), the following first aid procedures are generally instituted as soon as possible:

- Eye Exposure - If contaminated solid or liquid gets into the eyes, wash eyes immediately at the emergency eyewash station using large amounts of water and lifting the lower and upper lids occasionally. Obtain medical attention immediately. Use of contact lenses is not permitted in exclusion or contamination reduction zones.

- Skin Exposure - If contaminated solid or liquid gets on the skin, take appropriate actions (i.e., do not use water if substance is water reactive). Obtain medical attention immediately if symptoms warrant.

- Inhalation - If a person inhales large amounts of organic vapor, move him to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Obtain medical attention as soon as possible.

- Ingestion - If contaminated solid or liquid is swallowed, medical attention shall be obtained immediately.

13.5.2 Fires

Personnel in each work group shall be knowledgeable in fire-suppression techniques. They shall be instructed in:

- Proper use of the fire extinguishers supplied on site.

- Techniques in smothering fires using available non-combustible materials.

- Emergency evacuation of procedures in the event fires reach an out-of-control situation.

All personnel will be instructed to summon the fire department for assistance should there be even a remote chance that the fire will spread.

13.5.2.1 Small Fires: In the event of a small fire at the site, the SHSO shall, at a minimum, take the following actions:

- Evacuate all unnecessary personnel from the area to an upwind location, if possible.

- Attempt to extinguish fire using portable fire extinguishers or by smothering.

- Request emergency response assistance (ambulance, fire, hospital, poison control center) as needed for any injuries or exposures to hazardous chemicals.

- Notify the Contracting Officer of the incident.

13.5.2.2 Large Fires: In the event of a large or small fire which cannot be extinguished, the SHSO shall undertake the following actions:

- Evacuate all personnel from the area of the fire, preferably to an upwind location.
- Notify the fire department and other emergency response agencies.
- Notify the Contracting Officer of the incident.

13.6 CONTINGENCY PLAN

In the unlikely event that site evacuation is necessary, the SHSO will notify on-site personnel verbally. The SHSO is also responsible for notifying the appropriate agencies.

The evacuation route will be to a rendezvous point. The route to the rendezvous point will be reviewed in the initial training session.

A daily site log will not be maintained because the small number of personnel on site at one time will not create a problem of accounting for personnel in an emergency. If someone is missing, the SHSO will alert emergency personnel.

TABLES

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TABLE HSP-1

PESTICIDE CONSTITUENTS DETECTED IN SURFACE SOILS[1] PULVERIZING SERVICES SITE

BHC (alpha)

BHC (gamma), also known as lindane

Dichlorodiphenylethane (DDE)

DDD (tetrachlorodiphenylethane)

Dichlorodiphenyl Trichloroethane (DDT)

Dieldrin

Endosulfan Sulfate

Endrin ketone

Sulfur

Arsenic

Botran

Quintozene

1. Based upon USEPA sampling in October 1987 reported in TAT Sampling Report and USEPA sampling of December 1987 reported in the REAC Trip Report.

APPENDIX C

FIELD SCREENING TEST INFORMATION

L2000™ PCB/CHLORIDE ANALYZER

DEXSIL®

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The L2000 PCB/Chloride Analyzer

Dexsil's L2000 PCB/Chloride Analyzer is a field portable instrument that determines PCB concentration in oil and soil over a range of 5 to 2000 ppm. The L2000 tests for the total amount of chloride present in the sample, converts the amount of chloride sensed to an equivalent amount of PCB and provides an LCD readout in parts per million.

The instrument is easy to use and does not require a laboratory or an experienced technician to get accurate results. Being an on-site instrument, the L2000 eliminates the days or even weeks of waiting for the laboratory. Sample testing time is cut to 5 minutes for oil and 10 minutes for soil. The L2000 also eliminates the high cost of laboratory analysis. An average oil analysis costs $4.00 per sample and soil $8.00 per sample.

How The L2000 Works

The L2000 utilizes a chloride specific electrode to quantitatively measure the amount of PCB in a sample. A sodium reagent is reacted with the sample to convert the PCB to chloride. The instrument senses the amount of chloride in the sample and translates it to the equivalent amount of PCB.

Because different types of PCB contain different percentages of chlorine, the L2000 has an analysis range switch which allows the user to adjust for either Aroclor 1242, Aroclor 1260 or Askarel A (60% Aroclor 1260 and 40% trichlorobenzene). If the Aroclor of the sample being tested is unknown, the analysis range switch is set for 1242 for the most conservative reading.

How To Use The L2000

One L2000 can be used to test both oil and soil samples. For soil analysis, any PCB must first be extracted from the soil using the extraction solvent supplied with the instrument. After the PCB has been extracted from the soil, the procedure for oil and soil is the same. The sample is reacted with a sodium reagent to transform the PCB into chloride which the instrument then quantifies. The L2000 shows the result in parts per million PCB.

Several samples can be prepared concurrently to speed the sample testing time. In a single 8 hour day, one operator can run as many as 150 oil samples or 100 soil samples.

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Typical Application Of The L2000 PCB/Chloride Analyzer

A site suspected of formerly holding PCB transformers is being considered for the location of a new office complex. Before the property is purchased, the prospective buyer wants the entire lot checked for PCB levels. By using the L2000 Analyzer, the entire site can be quickly screened for PCB, and, if necessary, cleaned up in a fraction of the time required by conventional methods.

1. The entire site is mapped out, and using a grid system, samples are taken from different areas that will give a representative view of the entire site. Each sample can be analyzed the same day with the L2000 to determine just how much PCB is in each sample. If the instrument shows that PCBs are present, several samples are then sent to a laboratory for GC analysis to confirm that the source of the organic chloride is indeed PCB and to identify what Aroclor or Aroclors may be present.

2. As clean-up of the site starts, additional samples are analyzed with the L2000 to determine just where the points of greatest contamination are located. In less than a day, all hot-spots are located and isolated.

3. As excavation takes place, additional samples are analyzed to determine the vertical and horizontal spread of the contamination. Since results on any sample can be obtained in less than ten minutes, only the soil that is truly contaminated is actually removed. Excavation continues until the L2000 shows that the soil now being removed from the site contains PCBs below the critical level. As soon as all test sites in the area register less than the critical level (eg, 10 ppm) with the L2000, digging can stop and several final samples are sent to a laboratory for GC analysis to confirm that any remaining soil contains less than the critical concentration of PCB.

During the entire process of detecting the presence of PCBs, isolating local hot-spots, and removing contaminated soil from the site, the L2000 saves both time and expense by providing quick, accurate results on a large number of samples. Waiting for analytical results is reduced to a minimum and excavating can proceed at a continuous pace because in less than ten minutes, the L2000 lets you know right where you stand. Just the waiting time that is saved can pay for the instrument in less than a week and the decreased analytical costs can help bring you in under budget.

Whether you test oil or soil samples, the L2000 PCB/Chloride Analyzer can help you work faster and more economically, and provide you with a competitive edge in PCB analysis that will increase your productivity and improve your bottom line.

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The Complete PCB Analysis System

Each L2000 PCB/Chloride Analyzer comes complete with all the necessary equipment and reagents.

Each instrument includes:

- Carrying Case
- Step-Down Transformer
- Portable Balance
- 5 ml Pipettor
- Vial Rack
- Timer
- Enough Reagents To Perform 200 Tests.

Additional reageants are available from Dexsil. For additional information on the affordable L2000 PCB/Chloride Analyzer contact Dexsil at (203) 288-3509.

PRODUCT SPECIFICATIONS
L2000 PCB/Chloride Analyzer

Testing Range	5 ppm to 2000 ppm
Limit Of Detection	5 ppm (µg/g)
Error	< 5% measured as chloride
Temperature Range	10° C to 35° C (50° F to 95° F)
Detection Method	Chloride Specific Electrode
Voltage Requirement	120/240 V/AC
Readout	3.5 Digit LCD
Weight	1.6 Kg 3.5 lbs.
Size	20 cm x 20 cm x 11.5 cm 8" x 8" x 5"

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DEXSIL®

January 1993

L2000 PCB/CHLORIDE ANALYZER

Specifications:

Range: 5ppm-2000ppm — Limit of Detection: 5ppm

% Error: <5% (chloride) — Temperature Range: 10°C-35°C

Voltage: 120v/230v AC — Readout: 3.5 digit LCD

Wt: 1.6 kg — Size: 8" x 8" x 4.5"

Detection Method: Chloride Specific Electrode

Price Breakdown

1.	L2000 Instrument	$2,450.00
2.	Ion Specific Electrode (Chloride)	$ 460.00
3.	Power Cube (step down transformer: specific voltage + outlet configuration)	$ 45.00
4.	Portable Electronic Balance 0-80 grams	$ 125.00
5.	5 ml Pipettor	$ 160.00
6.	Carrying Case	$ 50.00
7.	Vial Rack	$ —
8.	Timer	$ 10.00
9.	*(Option 1: 40 Oil Test Reagents)	$ 200.00
	a. 40 sodium reaction tubes	
	b. Extract solution	
	c. Rinse solution	
	d. Calibration solution	
	e. Electrode filling solution	
	f. 40 disposable pipettes	
	g. 40 filters	
	h. 40 20 cc vials	
	i. Marking pen	
	j. 1 box of tissue wipes	
	k. Reaction tube rack	
	*(Option 2: 20 Soil Test Reagents)	
10.	Instructions	$ —
	Total Cost L2000 PCB Analyzer	US$3,500.00

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DEXSIL®

January 1993

L2000 REAGENT PACK SCHEDULE

L2000 OIL REAGENT PACK

QUANTITY	BULK PACK	
40 Tests/pk	COST/TEST	COST/PK
40	$5.00	$ 200.00
1000 (25 x 40 Test Packs)	$4.50	$4500.00
200 Tests/pk	COST/TEST	COST/PK
200	$4.50	$ 900.00
1000 (5 X 200 Test Packs)	$4.00	$4000.00
2000 (10 X 200 Test Packs)	$3.75	$7500.00

L2000 SOIL REAGENT PACK

QUANTITY	BULK PACK	
40 Tests/pk	COST/TEST	COST/PK
40	$10.00	$ 400.00
200 Tests/pk	COST/TEST	COST/PK
200	$ 8.00	$1600.00

1000+ Please call or write for quotation.

L2000$LIST

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MILLIPORE

EnviroGard™ DDT Soil Test Kit

Intended Use

The EnviroGard DDT Soil Test Kit is a qualitative or semi-quantitative field test for the detection of DDT and its metabolites DDD and DDE in soil. With the EnviroGard DDT Soil Test Kit, you can read test results visually or perform a semi-quantitative analysis using a photometer.

Test Principles

The EnviroGard DDT Soil Test Kit is based on the use of polyclonal antibodies that bind either DDT or DDT-Enzyme Conjugate. These antibodies are immobilized to the walls of the test tubes. When DDT is present in the sample, it competes with the DDT-Enzyme Conjugate for a limited number of antibody binding sites.

- A sample containing DDT is added to a test tube containing Assay Diluent. DDT-Enzyme Conjugate is then added to the test tube. The DDT-Enzyme Conjugate competes with the DDT for the antibody binding sites.
- After the incubation, the unbound molecules are washed away.
- A clear solution of chromogenic Substrate is then added to the test tube. In the presence of bound DDT-Enzyme Conjugate, the clear Substrate is converted to a blue color. One enzyme molecule can convert many Substrate molecules.

Since there are the same number of antibody binding sites on every test tube and each test tube receives the same number of DDT-Enzyme Conjugate molecules, a sample that contains a low concentration of DDT allows the antibody to bind many DDT-Enzyme Conjugate molecules.

Therefore, a low concentration of DDT produces a dark blue solution. Conversely, a high concentration of DDT allows fewer DDT-Enzyme Conjugate molecules to be bound by the antibodies, resulting in a lighter blue solution.

Note: Color is inversely proportional to DDT concentration.

Darker color = Lower concentration
Lighter color = Higher concentration

Performance Characteristics

The EnviroGard DDT in Soil Test Kit will not differentiate between DDT, its metabolites, and other structurally similar compounds, but will detect their presence to differing degrees. The following table shows a number of compounds and the approximate concentration of each required to yield a positive result (Lower Limit of Detection or LLD) and the concentration required to inhibit one-half of the color developed by the Negative Control (IC50). Concentration is in parts per million (ppm) in soil.

Compound	LLD	IC50
p,p'-DDT (kit calibrator)	0.04	1.25
p,p'-DDD	0.01	0.3
p,p'-DDE	0.18	3.6
o,p'-DDT	4	93
o,p'-DDD	0.4	11
o,p'-DDE	3	93
DDA	0.002	0.04
Chloropropylate	0.007	0.08
Chlorobenzilate	0.03	0.35
Dicofol	0.14	2
Tetradifon	1.2	14
Thiobencarb	5	52
Tebuconazole	7	95
Neburon	17	284
Chloroxuron	24	216
Monolinuron	25	714
Diclofop	70	>1000

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following compounds have lower limits of detection > 100 ppm:

2,4-D	4-chlorophenoxyacetic acid
Chlorbromuron	Chlordane
Chlortoluron	Dicamba
Diflubenzuron	Diuron
Lindane	Linuron
MCPA acid	MCPB
Mecoprop	

Precautions

- Treat DDT, solutions that contain DDT and potentially contaminated soil samples as hazardous materials.
- Where appropriate, use gloves, proper protective clothing, and methods to contain and handle hazardous material.
- Store all test kit components at 4°C to 8°C (39°F to 46°F) when not in use.

  Do not freeze test kit components or expose them to temperatures greater than 37°C (99°F).
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test.
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Use approved methodologies to confirm any positive results.
- Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.
- Tightly recap the DDT calibrator vials to prevent evaporative loss.
- Distribution of DDT in soils may be highly variable. The use of a composite sampling technique may be appropriate. Development of a sampling plan that assures adequate sample number and distribution is the responsibility of the analyst.
- DDT is light sensitive. Store soil extracts at 2°C to 7°C, shielded from direct light.

Materials Provided

EnviroGard DDT Soil Test Kit
This test kit contains the following items:

20 Antibody-Coated Test Tubes
1 vial of Assay Diluent
1 vial of Negative Control (methanol)
1 vial of 0.1 ppm DDT Calibrator in methanol
1 vial of 1 ppm DDT Calibrator in methanol
1 vial of 10 ppm DDT Calibrator in methanol
1 vial of DDT-Enzyme Conjugate
1 vial of Substrate
1 vial of Stop Solution
1 20-place Test Tube Rack
22 Pipette Tips (for the Gilson M-25 Microman® Positive Displacement Pipettor)

Materials Required and Ordered Separately

See "Ordering Information" for the appropriate catalogue numbers.

EnviroGard Soil Extraction Kit

Use this kit for the extraction of DDT in soil samples. This kit contains enough devices to process 12 samples:

- 12 30 milliliter (mL) Polypropylene Bottles with screw caps (each bottle contains five stainless steel mixing beads)
- 12 Filtration Devices, comprised of 12 upper (filter unit) and lower (sample tube) units
- 15 Wooden Spatulas
- 12 Screw Top Glass Vials, 4.0 mL
- 15 Weigh Boats

Methanol

Methanol, ACS reagent grade (60 mL for 12 samples), is required for soil extraction, but is not included in the EnviroGard Soil Extraction Kit. You must order it separately. (See "Ordering Information.")

Materials Required but Not Provided

You will also need several other items, some of which are included in the EnviroGard Soil Field Lab. (See "Ordering Information" for the appropriate catalogue number).

- Gilson M-25 Microman Positive Displacement Pipettor
- Eppendorf™ Repeater® Pipetter and five Combitips® (3 x 12.5 mL, 1 x 5.0 mL, and 1 x 50 mL)
- Balance capable of accurately weighing 5 grams
- Millipore Differential Photometer or Enviro-Quant Photometer
- Indelible marker for labeling test tubes
- Watch or timer
- Clean running water or a wash bottle containing tap or deionized water (500 mL)
- Calculator (optional)

Suggestions for Pipetter Use

- Practice using both pipetters (positive displacement and Repeater pipetter) with water and extra tips before you analyze your samples.
- Use a new tip each time you use the Repeater pipetter to avoid reagent cross-contamination. Label three 12.5 mL tips "Diluent", "Substrate" and "Stop," and one 5.0 mL tip "Conjugate".
- Draw the desired reagent volume into the Repeater pipetter and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
- To add reagents using the Repeater pipetter, pipette down the side of the test tube just below the rim.
- To add samples and calibrators using the positive displacement pipetter, pipette down the side of the test tube just above the liquid level.
- The carryover volume of the positive displacement tips is minimal, but may affect results if you are going from a high to low DDT concentration. Use a new pipetter tip each time you pipette a new unknown.

Assay Procedure

Collect/Store the Sample

1. Collect soil in appropriately-sized and labeled containers.
2. Take care to remove excess twigs, organic matter and rocks or pebbles from the sample. For best results, wet soils should be air-dried overnight and thoroughly mixed before testing.
3. Store soil samples at 4°C (39°F).

Prepare the Sample/Extract the Soil

1. Use the portable balance, wooden spatula, and a weigh boat to measure out 5.0 g of soil.
   - Place the balance on a level surface and press ON/MEMORY.
   - Place the weigh boat on the balance and press TARE.
   - Weigh the soil.
2. Transfer the 5.0 g of soil into an appropriately-labeled, 30 mL polypropylene extraction bottle. If you are testing more than one soil sample, cap the bottle loosely and repeat steps 1 and 2 until all soil samples are weighed out. Use a new wooden spatula and weigh boat for each sample.
3. Attach a 50 mL Combitip to the Repeater pipetter and set the dial to 5. Add 5.0 mL of methanol to the extraction vial and cap tightly.
4. Repeat steps 1-3 for each sample you test.
5. Vigorously shake the extraction bottles for at least 2 minutes and then allow the contents to settle briefly.
6. Pour the liquid contents of each vial into the appropriately-labeled lower (sample tube) piece of the filter unit. To obtain optimal filtering efficiency, do not let more than one or two mixing beads slip into the filter device.
7. Insert the plunger into the filter base unit.
8. Push down on the plunger to filter the extract. After 30 to 60 seconds, push down on the plunger again.
9. For longer term or spill-safe storage, remove the cap from the plunger and carefully pour the sample extract into an appropriately-labeled, 4.0 mL glass vial. Cap the vial. Repeat this step for each of the sample extracts.
10. If sample extracts are not going to be tested right away, store them at 2°C to 7 °C in the dark.

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erform the Test

NOTE: Allow all reagents and sample extracts to reach room temperature before you begin the test. Do not analyze more than 20 test tubes at a time.

1. The choice of calibrators to use in the test will depend on the the selection of the analysis. The use of two calibrators may be appropriate if screening for a single level of DDT.

Remove the test tubes from the plastic bag and label them as follows*:

Tube Label	Tube Contents
NC	Negative Control
C1	0.1 ppm Calibrator
C2	1 ppm Calibrator
C3	10 ppm Calibrator
S1	sample 1
S2	sample 2
etc.	

* You are not required to perform the assay in duplicate; however, doing so will increase the precision.

Place the test tubes in the test tube rack. Push down on each tube so that it is held firmly and does not fall out of the rack when shaken.

CAUTION: Do not "snap" the test tubes into the rack as this may result in a cracked tube.

2. Attach the 12.5 mL Combitip labeled "Diluent" to the Repeater pipetter and adjust the dial to 2. Add 500 microliters (µL) of Assay Diluent to each test tube.
3. Attach a clean pipette tip to the Microman pipetter and adjust the dial to "250". Add 25 µL of each calibrator to the corresponding "C" test tube by placing the end of the pipette tip against the side of the tube (just above the level of the Assay Diluent) and dispensing the volume.

CAUTION: Replace the caps on the calibrator vials immediately after use to minimize evaporation.

4. Using a clean tip for each sample, add 25 µL of each sample extract to the appropriately-labeled test tube.
5. Attach the 5.0 mL Combitip labeled "Conjugate" to the Repeater pipetter and adjust the dial to 1. Add 100 µL of DDT-Enzyme Conjugate to each test tube.
6. Shake the test tube rack to mix for 10 to 15 seconds. Leave the test tubes undisturbed for 15 minutes.
7. Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to overflowing with cool tap or distilled water, then decant and vigorously shake out the remaining water.

   Repeat this wash step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on absorbant paper.
8. Attach the 12.5 mL Combitip labeled "Substrate" to the Repeater pipetter and set the dial to 2. Add 500 µL of Substrate to each test tube. Leave the test tubes undisturbed for 10 minutes.

   NOTE: If a blue color does not develop in the Negative Control test tube within 10 minutes after adding the Substrate, the test is invalid and you must repeat it.

Interpret the Results

You can either interpret the results visually within 10 minutes after adding the Substrate to each test tube, or you can perform a more precise analysis with a photometer after you add the Stop Solution.

Visual Interpretation

After you add the Substrate, wait 10 minutes then mix the test tubes by shaking them for a few seconds until they are a uniform blue color. Compare the sample test tube to the calibrator test tubes against a white background. The test tube rack in the kit is well-suited for this purpose.

NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of *p,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE.

- If a sample test tube contains *more* color than the calibrator test tube, the sample contains DDT at a concentration *lower* than the calibrator.
- If a sample test tube contains *less* color than the calibrator test tube, the sample may contain DDT at a concentration *greater* than the calibrator.
- If the sample test tube contains color that is between the calibrator test tubes, the sample contains DDT at

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a concentration between the calibrator concentrations.

- If a sample test tube contains *approximately the same* amount of color as the calibrator test tube, the sample contains DDT at a concentration *approximately equal* to the calibrator.

- If the sample test tube contains less color than the 10 ppm Calibrator test tube, you may dilute a fraction of the soil extract in methanol (for example, 1:100) and perform the assay again. To determine the concentration of the diluted extract multiply the result by the dilution factor. (Go to "Semi-Quantitative Interpretation" for further details.)

Photometric Interpretation

After you add the Substrate, wait 10 minutes then add the Stop Solution to each test tube.

WARNING: Stop solution is 1N Hydrochloric acid. Handle carefully.

Attach the 12.5 mL Combitip labeled "Stop" to the Repeater pipetter and set the dial to 2. Add 500 µL of Stop Solution to each test tube. This converts the blue color in the test tubes to yellow.

NOTE: After you add Stop Solution to the test tubes, results should be read within 30 minutes.

Millipore Differential Photometer

1. Place a water blank test tube containing 1.5 mL of Milli-RO® or Milli-Q® water, or equivalent in the left (reference) well.

2. Place the Negative Control test tube into the right (sample) well. Record the optical density (OD) of the Negative Control.

3. Remove the Negative Control test tube and replace it with the 0.1 ppm Calibrator test tube to reactivate the photometer. Record the result. Repeat this step to determine the OD for each of the remaining calibrators and for each sample.

Semi-quantitative Interpretation

Compare the OD of each sample to the OD of each calibrator:

NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of *p,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE.

- If a sample OD is *equal* to the OD of a calibrator, the sample contains DDT at a concentration *approximately equal* to the calibrator.

- If a sample OD is *greater* than a calibrator OD, the sample contains *less* DDT than the calibrator.

- If a sample OD is *lower* than a calibrator OD, the sample may contain *more* DDT than that calibrator.

- If an assay result indicates that a soil sample contains greater than 10 ppm total DDT, but you need more specific information, the soil extract may be diluted 1:100 in neat methanol, and assayed again. You must then multiply the results of the re-assay by 100 to determine the approximate sample concentration.

NOTE: If you know in advance that the "action level" of interest is greater than 10 ppm total DDT in soil, the assay may be modified to pinpoint that particular concentration. For example:

If you wish to categorize samples as less than or greater than 250 ppm, you should dilute all sample extracts 1:250 in neat methanol (e.g. 20 µL extract plus 4.98 mL methanol) and compare the diluted extracts to the 1 ppm DDT kit calibrator. Due to the 250-fold dilution, the 1 ppm calibrator represents 250 ppm in the assay.

NOTE: If you are interested in action levels greater than 1000 ppm, please contact Millipore Technical Services for assistance.

Limitations of the Procedure

The EnviroGard DDT Soil Test Kit is a screening test only. Actual quantitation of DDT by EnviroGard immunoassay is not possible due to the Test kit's cross-reactivity with DDT breakdown products and other similar compounds and to the variations in extraction efficiency inherent in the fast extraction protocol described in this product insert.

Soil sampling error may significantly affect testing reliability. The distribution of pesticides in different soils can be extremely heterogeneous. Soils should be dried and homogenized before analysis by any method. Split samples (i.e. for GC and immunoassay) should always derive from the same homogenate.

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Actual OD values will vary. This data is for demonstration purposes only.

Tube	OD	Interpretation
NC	0.90	
C1 (0.1 ppm)	0.75	
C2 (1 ppm)	0.49	
C3 (10 ppm)	0.35	
S1	0.68	>0.1 ppm < 1 ppm
S2	0.16	> 10 ppm

Note:

The EnviroQuant Photometer is also available from Millipore. This dual wavelength instrument measures the OD at 450 nm minus 600 nm of all samples and calibrators, and provides a printout of results. See "Ordering Information" for the appropriate catalogue number.

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Ordering Information

The following table lists descriptions and catalogue numbers for the EnviroGard DDT and Soil Extraction Test Kits and related products.

Description	Catalogue Number
EnviroGard DDT in Soil Test Kit, includes the following items: • 20 Antibody-Coated Test Tubes • 1 vial of Assay Diluent • 1 vial of Negative Control • 1 vial of 0.1 ppm DDT Calibrator • 1 vial of 1 ppm DDT Calibrator • 1 vial of 10 ppm DDT Calibrator • 1 vial of DDT-Enzyme Conjugate • 1 vial of Substrate • 1 vial of Stop Solution (1N hydrochloric acid) • 1 20 - place Test Tube Rack • 22 Pipette Tips, 1 to 25 µL range (for the Gilson M-25 Microman positive displacement pipettor)	
EnviroGard Field Soil Extraction Kit, includes the following items test 12 samples: • 12 30 mL Polypropylene Bottles with screw caps, each containing 5 stainless steel mixing beads • 12 Filtration Devices, comprised of 12 upper (filter unit) and lower (sample tube) units • 15 Wooden Spatulas • 12 Screw-Top Glass Vials, 4.0 mL • 15 Weigh Boats	ENSP 000 20
Methanol for soil extraction, 100 mL bottle	ELCR 000 07
Millipore Differential Photometer: • 115 volt (V) • 230 V	 ENVR 000 00 ENVR [illegible] 30
EnviroQuant Photometer, 110V EnviroQuant Photometer, 220V EnviroQuant Replacement Paper, 12 rolls	ENV[illegible] 00 ENV[illegible] 00 EN[illegible] 11 02
EnviroGard Replacement Pipetter Tips (available separately): • Positive displacement pipetter tips, 1-25 µL range 200/pk (not preassembled) • Repeater pipetter tips, 5.0 mL, 100/pk • Repeater pipetter tips, 12.5 mL, 100/pk Repeater pipetter tips, 50 mL, 10/pk	 ENVR L04 09 ENVR L01 09 ENVR L02 09 ENVR L03 09

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Description	Catalogue Number
EnviroGard Soil Test Kit includes: • 1 Positive displacement precision pipetter • 1 Eppendorf Repeater pipetter • 1 Electronic timer • 13 Polystyrene test tubes, 12 mm X 75 mm (for blanking the spectrophotometer and sample dilutions) • 1 Portable balance with 100 gram calibrator weight • 1 Wash bottle, 500 mL • 4 Test tube racks, six-position • 8, 5.0 mL Pipette tips for the Repeater pipetter, for 0.1 mL through 0.5 mL dispensing volumes • 4, 12.5 mLPipette tips for the Repeater pipetter, for 0.25 mL through 1.250 mL dispensing volumes • 1, 50 mL Pipette tip for the Repeater pipetter, for 1.0 mL through 5.0 mL dispensing volumes • 100 pipette tips, 2.0-25 µL (not preassembled) • 100 pipette tips, 50-250 µL (not preassembled) Contact Millipore Technical Service for kit component replacement or reordering information. (See the "Technical Assistance" section for the number of the Millipore office nearest you.)	ENVR L00 09

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Technical Assistance

For additional information about Millipore products, call the Millipore office nearest you.

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COMPARISON OF DDT IMMUNOASSAY AND GAS-CHROMATOGRAPHY
"REAL WORLD" SOIL SAMPLES

Sample ID	GC Total DDT (ppm)	Immunoassay Call (ppm ranges)	Agree/ Disagree (A/D)
61701	18	> 10 < 50	A
61702	12	> 10 < 50	A
61703	11	> 10 < 100	A
61704	30	> 10 < 50	A
61706	0.44	> 0.1 < 1	A
61707	0.47	> 0.1< 1	A
61708	0.40	> 0.1 < 1	A
61709	0.33	> 0.1 < 1	A
60801	41	> 10 < 100	A
60802	52	> 10 < 100	A
60803	40	> 10 < 100	A
60804	79	> 10 < 100	A
60807	0.18	> 0.1 < 1	A
60808	0.23	> 0.1 < 1	A
60809	0.18	> 0.1 < 1	A
01	1140	> 500 < 5000	A
02	2755	> 500 < 5000	A
03	245	> 50 < 500	A
04	850	> 100 < 1000	A
05	1832	> 500 < 5000	A
06	1185	> 1000 < 5000	A
07	14,375	> 5000 < 50,000	A
08	5025	> 5000 < 50,000	A
09	460	> 100 < 1000	A
10	217	> 50 < 500	A
11	536	> 200 < 2000	A
12	308	> 50 < 500	A
13	1316	> 200 < 2000	A
14	1415	> 1000 < 10,000	A
15	1204	> 200 < 2000	A
16	1370	> 1000 < 10,000	A
17	435	> 50 < 500	A
18	4800	> 5000 < 50,000	D
19	755	> 100 < 1000	A
20	1760	> 1000 < 10,000	A
21	115	> 20 < 200	A
22	579	> 100 < 1000	A
23	129	> 30 < 300	A
24	115	> 100 < 1000	A
25	2904	> 300 < 3000	A
26	1512	> 200 < 2000	A
27	< 0.1	< 0.1	A
28	< 0.1	> 0.1 < 1	D
29	< 0.1	< 0.1	A
30	< 0.1	> 0.1 < 1	D
31	0.14	< 0.1	D
32	0.15	> 0.1 < 1	A

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SPECTRACE 6000 TRANSPORTABLE X-RAY FLUORESCENCE ANALYZER STANDARD OPERATING PROCEDURE

1.0 Scope and Application

This document addresses the application of energy dispersive X-ray fluorescence spectrometry (EDXRF) to the determination of contaminant metals in soil and sediments. The EDXRF technique is capable of both qualitative and quantitative analysis of elements with atomic numbers 11 (sodium) through 92 (uranium). This operating procedure discusses the determination of the elements listed in Table 1.

Table 1. Elements and their atomic symbols analyzed by the EDXRF method.

Arsenic (As)	Manganese (Mn)
Barium (Ba)	Mercury (Hg)
Cadmium (Cd)	Nickel (Ni)
Calcium (Ca)	Potassium (K)
Chromium (Cr)	Silver (Ag)
Copper (Cu)	Titanium (Ti)
Iron (Fe)	Zinc (Zn)
Lead (Pb)	

1.1 Principles of Operation

The Spectrace 6000 is a transportable laboratory grade EDXRF instrument. It is equipped with a single sample position.

1.1.1 Fundamentals of the X-ray spectrometric measurements

The basis of X-ray fluorescence spectrometry is the interaction of X-rays produced by the Spectrace 6000 X-ray tube with the constituents of the soil or sediment sample.

When atoms in a soil sample absorb the tube x-radiation, the energy causes the ejection of an electron from the atomic cloud. Next, electronic transitions occur to return the excited atom to a ground energy state. These transitions result in emission of a *characteristic* X-ray. The energy of the characteristic X-ray indicates the atomic number of the emitting atom and the specific electronic transition that occurred. (Reference 1,2)

The lithium drifted silicon (Si(Li))detector converts the characteristic X-ray energy into an electronic signal. The electronics in the Spectrace 6000 further processes the signal and displays the X-ray spectrum on a personal computer.

1.1.2 Generation of spectral background

When the X-ray tube shines on the soil sample, some of the X-rays are scattered toward the detector rather than absorbed by the soil. The X-ray spectrum then shows the scattered radiation as background under the elemental emission lines. The magnitude of the background limits the achievable minimum detectable limit for the target contaminant elements.

The peak fitting and deconvolution software of the Spectrace 6000 filters background signal prior to quantitative analysis. Each soil spectrum then can be compared with another without regard to background variations.

2.0 Overview of the method

Three spectra are acquired for each EDXRF measurement. Each spectrum optimizes one or more target elements or matrix constituents of the sample. Standardization of the EDXRF method relies on National Institutes of Standards and Technology (NIST) certified soil standard materials.

Elemental emission peak deconvolution is followed by quantitative analysis with a fundamental parameters method. The fundamental parameters method can accurately determine the composition of a sample even though the standards used do not cover the range of concentrations expected. Additionally, fundamental parameters can address matrix interactions from widely varying soil types.

Calibration by fundamental parameters proceeds by completely defining for the program each standard sample's composition, including the major components (ex. Fe, Ca). Many instances arise when a portion of the matrix may be better estimated by a difference component. For soil, the difference component is defined as SiO_X where X can be 2 - 5. When X = 5, a composition of 24% Si and 45% O results that approximates the concentrations found in most soils types.

The tube output distribution is calculated by the program followed by determination of the matrix independent pure element count rate (PECR) for each analyte. The routine then develops a set of theoretical standards. Next, the routine computes the theoretical X-ray intensity expected for each analyte using the Sherman formula

$$I_L = \omega_A g_L \frac{r_A - 1}{r_A} \frac{d\Omega}{4\pi} \int_{E_{abs}}^{E_{max}} \frac{I_E C_A \mu_A(E) \csc\phi}{\mu_M(E)\csc\phi + \mu_M(E_A)\csc\psi} \qquad \text{Eq. 2.1}$$

where:

I_L - intensity of the analyte emission line;
ω_A - fluorescent yield of analyte A;
g_L - fractional value of the measured analyte line (L) in its series;
r_A - absorption edge jump ratio of analyte A;
$\frac{d\Omega}{4\pi}$ - fractional value of the fluorescent X-ray that is directed toward a detector;
E_{max} - maximum tube output X-ray energy;
E_{abs} - absorption edge energy of analyte A;
I_E - intensity of the primary X-ray beam at energy (E);
C_A - concentration of analyte A;
$\mu_A(E)$ - mass absorption coefficient on analyte A for X-ray energy (E);
ϕ - incident angle of primary beam;
$\mu_M(E)$ - mass absorption coefficient of the matrix for X-ray energy (E);
$\mu_M(E_A)$ - mass absorption coefficient of the matrix for analyte X-ray;
ψ - take off angle of the fluorescent emission.

Combining the theoretical standard and intensity information, alpha influence coefficients are determined by solving simultaneous equations of the form developed by Lachance and Traill shown in equation 2.2 below. Alpha coefficients describe the quantitative effect of each matrix element on an analyte's measured X-ray intensity.

$$C_i = B_i + K_i I_i \left(1 + \sum_{j=1}^{n} \alpha_{ij} C_j\right) \qquad \text{Eq. 2.2}$$

where:

I_i = X-ray intensity of element i;
α_{ij} = alpha coefficient describing the matrix interaction of element j on analyte i;
C_i = concentration of analyte element i;
K_i = slope term;
B_i = intercept term;
C_j = concentration of matrix element j.

Unknowns are quantified by estimating analyte concentration from the measured intensity and the alpha coefficients. Next, theoretical intensities are calculated from equation 2.1 and compared to the measured intensities. If the intensities are more than 1% different, a new estimate of concentration is made and the theoretical intensity computation is repeated. The iteration continues until the theoretical and measured intensities converge

3.0 Sample conatiners and handling

After sample collection by EPA approved protocol, samples should be stored in either metal free glass jars or polyethylene bags. In the lab, only disposible wooden or plastic spatulas and clean glass drying containers should contact the soil samples.

4.0 Interferences

The EDXRF method is a total elemental analysis technique. Errors can arise for the following sources:

4.1 Sample preparation error

The nature of soil samples require an approach to such variables as moisture, particle size, and heterogeneity and representitiveness of the sample to the soil at the site. Sample preparation as outlined in this SOP minimizes or eliminates the effects of many of these variables on the EDXRF result.

4.2 Spectral interference

Emission line overlap can be severe depending on the spectral peak position and relative concentrations of the overlapping elements. The Spectrace software utilizes a multiple linear least squares deconvolution method produces accurate analyte peak intensities for subsequent quantification.

4.2.1 Lead [Pb] Lα line and arsenic [As] Kα line overlap

Accurate peak deconvolution is obtained at lead concentration to arsenic concentration ratios of 17:1 (Reference 1).

4.3 Chemical matrix interferences

Quantitative X-ray fluorescence analysis is a comparison analytical method; EDXRF spectrometer response for an elemental emission line is evaluated with respect to the known concentration of the element in a standard material. Contaminated soils are complex samples in regard to their overall elemental composition. Matrix effects are defined as the impact that the concentration variations of major soil components (ex. Ca, Fe, Si) has on measured analyte X-ray emission intensity.

The fundamental parameters method mathematically corrects analyte intensities with respect to concentration variations of sample matrix components

5.0 Equipment and apparatus

5.1 Description of the Spectrace 6000

The Spectrace 6000 is a transportable, laboratory grade, energy dispersive X-ray fluorescence (EDXRF) spectrometer. It has found wide application in the analysis of soils, sediments, hazardous waste, and waste oil materials.

The instrument utilizes a high flux rhodium [Rh] anode X-ray tube which is positioned to direct X-rays onto the sample. Standard equipment includes an electronically cooled lithium drifted silicon [Si(Li)] solid state X-ray detector.

Pulse processing electronics in the Spectrace 6000 card cage module communicate spectral data to a personal computer. The personal computer displays and processes spectral information, and outputs elemental concentration data.

5.2 Equipment and apparatus list

5.2.1 Spectrace 6000 Analyzer System

The complete Spectrace 6000 Analyzer system includes:

- Spectrace 6000 spectrometer and card cage modules
- Spectrace 6000 operators manual
- Personal computer (a 486 is recommended) with SSXRF.EXE software installed and a printer.
- Spectrace 6000 technical manual

5.2.2 Sample preparation and presentation materials

- Sample communition device - mechanical grinder (tungsten carbide) or manual grinding device - mortar and pestle (tungsten carbide)
- Drying oven - conventional (115 degrees Celsius) or microwave
- 10 mesh brass sieve
- Drying containers (glass or polyethylene)
- Disposable X-ray cups (32 mm diameter closed end)
- Polypropylene window film (0.25 mil thickness)

5.2.3 Limits and Precautions

Spectrace 6000 operators are protected from accidental exposure to X-rays by a lid lock and side panel interlocks. If any problems arise with the X-ray on indicator light on the front panel or with the interlock system contact your Spectrace Instruments service engineer.

Beryllium windows are present on the X-ray tube and detector. Do not allow any sample or debris to fall on the window avoid puncturing it. If a window should break note the beryllium metal is poisonous. Use extreme caution when collecting broken beryllium pieces. Consult your Spectrace Instruments service engineer for advice on cleanup of broken windows.

5.3 Instrument maintenance

5.3.1 Detector cooling

Ensure the free flow of air around the Spectrace 6000 spectrometer cabinet (especially fan vents). Additionally, a supply of uninterruptable 110V AC power is recommended so that the electronically cooled detector is always at operational temperatures.

5.3.2 Sample chamber cleaning

Keep debris and dust wiped from the chamber. Do not use forced air to dust the chamber because the detector beryllium window is fragile.

6.0 Reagents

A first step in the determination are used to acquire a library of reference peak shapes for emission peak deconvolution.

6.1 The following reagents for the acquisition of peak shape spectra:

Potassium carbonate
Calcium carbonate
Titanium dioxide
Vanadium oxide
Chromium oxide
Manganese oxide
Iron oxide
Copper metal
Cobalt oxide
Nickel metal or chloride
Zinc metal or oxide
Tungsten oxide
Mercury oxide
Lead oxide or metal
Arsenic oxide
Rubidium carbonate
Strontium carbonate
Zirconium oxide
Silver metal
Cadmium metal
Tin metal
Antimony oxide
Barium carbonate

All compounds or metals should be Analyzed Reagent grade or better.

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6.2 Interference sample

When lead [Pb] metal or oxide is used to produce the reference lead emission lines the Lα and Lβ ratio does not accurately reflect found the ration found in soil spectra. For this reason, an interference sample is used to generate the lead reference spectrum.

6.2.1 Composition of interference sample

Boric acid [H_2BO_3] - 15.8%
Silicon dioxide [SiO_2] - 50%
Aluminum trioxide [Al_2O_3] - 20%
Calcium oxide [CaO] - 11%
Iron oxide [Fe_2O_3] - 1%
Iron metal [Fe] - 2%
Lead nitrate [$Pb(NO_3)_2$] - 0.2%

6.2.2 Formulation of the interference sample

The materials listed in section 6.2.1, weighed to proper proportions, are introduced into a mechanical grinding vessel and ground for 3 minutes. This material is available from the Spectrace Instruments Applications Department upon request.

7.0 Procedures

7.1 Prerequisites

7.1.1 Site location

The instrument must be placed so that free air flow around the instrument is supplied. Operate at temperatures of 50 -90 degrees Fahrenheit and relative humidity between 20 - 80% only. Power requirements are 115 V AC, 14 amps and a power conditioner/filter is recommended. Power both the Spectrace 6000 instrument and the person computer from the same AC line.

7.1.2 Detector cooling

Electronically cooled Si(Li) detectors require AC power during operation, however, Spectrace Instruments recomends continuous power to the system so that the detector remains cool and electronics stablized.

7.1.3 Energy calibration

Once every twenty four hours, the gain setting of the instrument must be checked using the copper calibration disk supplied by the manufacturer. Execute the automated "ENRGY CALIB" function from the procedure menu. The computer automatically adjusts electronics gain to ensure proper calibration. This function normally takes between 8 and 10 minutes. Figure 1 shows the copper emission spectrum displayed during energy calibration.

7.1.4 Acquisition of reference peak shape spectra

The acquisition of reference spectra is only required once. As long as no pulse processing modules have changed, these peak shape references remain valid. Often, this procedure can be done at the manufacturers site.

Place the materials listed in section 6.1 and the interference sample described in section 6.2 in individual X-ray cups with polymer window film seals. Then acquire the following spectra in a subdirectory labeled \REFS with the conditions listed below. Reference is made to DOS file naming structure below. Consult Chapter 2 of the Operators Manual or a DOS manual.

- 12 kV, aluminum filter, - for the elements potassium, calcium, titanium, vanadium, chromium, and iron. Save the spectra individually in the \REFS subdirectory with the spectrum number equal to the atomic number (i.e. potassium [K] - SPECTRUM.19). Place the cursor on the Kα peak of each element emission and allow at least 30,000 counts to acquire in the channel.

- 35 kV, thick palladium filter - for the elements cobalt [Co], nickel [Ni], zinc [Zn], bromine [Br], arsenic [As], rubidium [Rb], strontium [Sr], and zirconium [Zr]. Save the spectra individually in the \REFS subdirectory with the spectrum number equal to the atomic number (i.e. cobalt [Co] - SPECTRUM.27). Place the cursor on the Kα peak of each element emission and allow at least 30,000 counts to acquire in the channel. For the L line emission of mercury [Hg], lead [Pb], and tungsten [W] allow at least 10,000 counts to acquire in the Lα channel. Save the spectrum as 100 + the atomic number (mercury [Hg] - SPECTRUM.180). For the interference sample, use the same conditions as above but allow the spectrum to acquire for 1000 seconds livetime and save the spectrum as SPECTRUM.199.

- 50 kV, copper filter - for the elements silver [Ag], cadmium [Cd], tin [Sn], antimony [Sb], and barium [Ba]. Save the spectra individually in the \REFS subdirectory with the spectrum number equal to the atomic number (i.e. silver [Ag] - SPECTRUM.47). Place the cursor on the Kα peak of each element's emission line and allow at least 30,000 counts to acquire in the channel.

7.2 Standardization of the Method

Standardization of the Spectrace 6000 uses a set of NIST certified reference materials and/or site specific calibration standards. See the discussion in Appendix A on the prerequisites of site specific standards. By using NIST certified materials, the EDXRF standardization then can be referenced to NIST certificates or an independent analysis referee method. Standardization adjustment can be affected by including or omitting a particular analyte element in a standard or addition or removal of a standard sample altogether.

7.2.1 Setting up the standards file

For the standards NIST 2709, 2710, 2711, 2704, 8407, 8408 type in the standards file as shown in Figure 1. Parentheses indicate that the concentration is to be used to define the matrix of the standard but <u>not</u> to be used for estimation of analyte sensitivity. Generally, those concentrations in the standard less than 50 ppm are defined in parentheses. A blank indicates that the component is either not present or is at a concentration near the minimum detectable limit of the method. Consult the Spectrace Instruments Fundamental Parameters software manual for further details.

7.2.2 Setting up the reference and spectrum processing file.

Enter the reference file as listed in the Figures 2, 3, and 4 following the instructions in Chapter 3 of the Spectrace Instruments users manual.

7.2.3 Set up the analysis technique menu in the combination procedure. see setup of mulitple procedures in Chapter 5 of the Spectrace 6000 operators manual.

7.2.4 Set up the acquisition conditions as follows:

Low Z procedure: 13 kV tube voltage, 0.30 mA tube current, aluminum (0.13 mm) filter, and 100 seconds analysis livetime.

Mid Z procedure: 35 kV tube voltage, 0.10 mA tube current, thick palladium [Pd] filter (0.13 mm), 200 seconds analysis livetime.

High Z procedure: 50 kV tube voltage, 0.35 mA tube current, copper filter (0.63 mm), 200 seconds analysis livetime.

7.2.4 Run the analysis of the standards.

7.2.5 Evaluation of the standardization:

Table 2 lists the pure element count rates for the standardization. The pure element count rates should be within 20% relative of these values.

Table 2. Pure element count rates for the elements analyzed.

Element	Pure Element Count Rate	Element	Pure Element Count Rate
Arsenic	40325	Manganese	36326
Barium	9560	Mercury	5949
Cadmium	25667	Nickel	9086
Calcium	9734	Potassium	5960
Chromium	31464	Silver	25341
Copper	20830	Titanium	18577
Iron	40761	Zinc	23541
Lead	10242		

7.2.6 Set up three procedures for the analysis of unknowns, identical to steps 7.2.2 to 7.2.4 except indicate a combination procedure in the mode "Run unknowns" in the analysis technique menu.

7.3 General keys and menu software

The Spectrace Instruments Operators manual and Fundamental Parameters software manual should be consulted for operation and flow of analysis of the Spectrace software.

7.4 Pre operational checks

7.4.1 Control sample check

One of the six NIST soil standards or a well characterized sample from the site is analyzed as an unknown. Plot the concentration response for at least one element (preferably a target analyte) from each of the three acquisition conditions on a control chart. For those analytes with greater than 100 ppm concentration, the result should be within 20% of the known elemental concentration. If a result exceeds the 20% limit, restandardize the method as described in section 7.2.

If more than one measurement of an element in a set of five exceeds the Upper or Lower control limit (± 3 x standard deviation of the charted data) perform a resolution and stability check.

7.4.2 Resolution and stability check

A resolution and stability check should be performed if the test outlined in section 7.4.1 fails. See the sample output in Figure 6 or consult the Spectrace Instruments Model 6000 Technical manual for details. Contact a Spectrace service engineer if the instrument fails this test.

7.5 Analysis of unknown soil samples

7.5.1 Prepare samples as indicated in Section 8.0

7.5.2 On the procedure menu, select the run unknowns combination procedure setup in Section 7.2.6.

7.5.3 Hit the F1 "RUN" key and enter the number of samples to analyze and the indentifying label for the first soil sample. Repeat for all individual samples in the set to be analyzed.

7.6 Minimum Detectable Limits (MDL's) of the method for the Spectrace 6000

7.6.1 Non target elements method MDL's

Determination of the non-target elements' MDL's proceeds by averaging the counting statistic error from the "CONCENTRATION ERROR" printout header for the element in each of the six standards. This average, multiplied by 3 is the MDL reported for that element.

7.6.2 Target elements MDL's

For the target elements, select the NIST standard which comes closest to the action level concentration for the target. Then using the "PEAK BACKGROUND" printout format use the equation found in section 8.1 to determine MDL's.

7.6.3 Typical MDL's for the Spectrace 6000 for soils are listed in Table 3.

Table 3. Minimum detectable levels of EDXRF analyzed elements.

Element	MDL (mg/kg)	Element	MDL (mg/kg)
Arsenic	8	Manganese	12
Barium	35	Mercury	8
Cadmium	2	Nickel	7
Calcium	90	Potassium	120
Chromium	16	Silver	2
Copper	4	Titanium	18
Iron	11	Zinc	4
Lead	4		

8.0 Sample preparation

8.1 First agitate the sample in its glass or bag container to mix the soil material.

8.2 Using a wooden spatula, transfer approximately 15 grams of sample material onto the glass drying container.

8.3 Place in the drying oven and drive off moisture.

8.4 Seive dried material through 10 mesh screen and discard the fraction held in the screen.

8.5 Transfer the material passing through the screen to the grinding vessel and grind for a preset period of time to assure -250 mesh material.

8.6 Transfer approximately 8 grams of the dried material into a labeled disposable X-ray cup and seal with one sheet of polypropylene window film.

9.0 Calculations

9.1 MDL calulation for non-target elements

Calculation of the MDL for the non-target elements is three times the average counting statistics error. Use the "CONCENTRATION ERROR" report format to obtain the data.

9.2 MDL calulation for target elements

Calculation of the MDL for the target elements uses Equation 9.1 below and requires peak and background counting data from NIST certified standard reference material containing a concentration of the target element closest to the predetermined action level. Use the report format "PEAK BACKGROUND" to obtain the data for this calculation.

$$MDL = \frac{3 \times \sqrt{B}}{P} \times conc. \quad \text{Eq. 9.1.}$$

Where the variables are defined as:

MDL - minimum detection level;
B - background counts under fitted analyte peak;
P - fitted peak counts of analyte peak;
conc. - concentration of analyte from standard reference material certificate provided by NIST.

10.0 Quality assurance/quality control

10.1 Precision

Precision data is accumulated in the pre-operational check listed section 7.4. Keep in mind that precision is a function of counting time and the number of peak counts and background counts collected for the analyte. Precision in EDXRF measurements is predicted by counting statistics as shown in the following equation:

$$Rel.\ \sigma_{theo} = \frac{\sqrt{P + 2 \times B}}{P} \times 100\% \qquad \text{Eq. 10.1}$$

The Spectrace Instruments model 6000 typically provides measurement precision (indicated by the control chart plotted in section 7.4) 1.2 - 1.8 times the σ theoretical. Several acquisitions of the control sample are required prior to making this determination.

10.2 Accuracy

The accuracy of an EDXRF measurement can be verified in two ways. First, certified soil reference materials (procured from the NIST or other standard generating entity) not utilized in the Spectrace 6000 standardization are analyzed as soil unknowns as outlined in Section 7.5. Verify that results for target elements near the predetermined action concentration level are within 20% of the certified concentration for the standard.

Second, a subset of samples (10% at the minimum), preferably near the action concentration level, are submitted to a laboratory for acid digestion and ion-coupled plasma emission or atomic absorption spectrometric (ICP/AA) analysis. The split submitted should be taken of the prepared soil material subsequent to Section 8.5. Target elements exhibiting a regression analysis (EDXRF data versus ICP/AA data) r^2 value greater than 0.95 has been considered QA3 level data. Regression analysis producing r^2 values of 0.7 or above can be considered QA1 level (field screening) data. To improve the r^2 value, ICP/AA analyzed soil materials maybe added to the standards file setup in 7.2.1. The utmost discretion should be used to alter the standardization method of section 7.2. Laboratory data should be provided for all the elements listed in the standards file (Figure 2). Short of that potassium, calcium and iron along with the target elements must be determined in the potential site specific standard soil samples.

10.2.1 Matrix considerations

Complimentary QA/QC verification include analysis of soils with varying matrix composition. Of interest is the effect on EDXRF results when analyzing samples with high siliceous or calcareous soils or soils with high iron composition.

Additionally, acid digestion and ICP/AA analyzed site specific standards can introduce error due to variable recoveries, caused by soil matrix differences, of target elements (see Reference 7). When site specific standards are used, errors are propagated into the EDXRF standardization.

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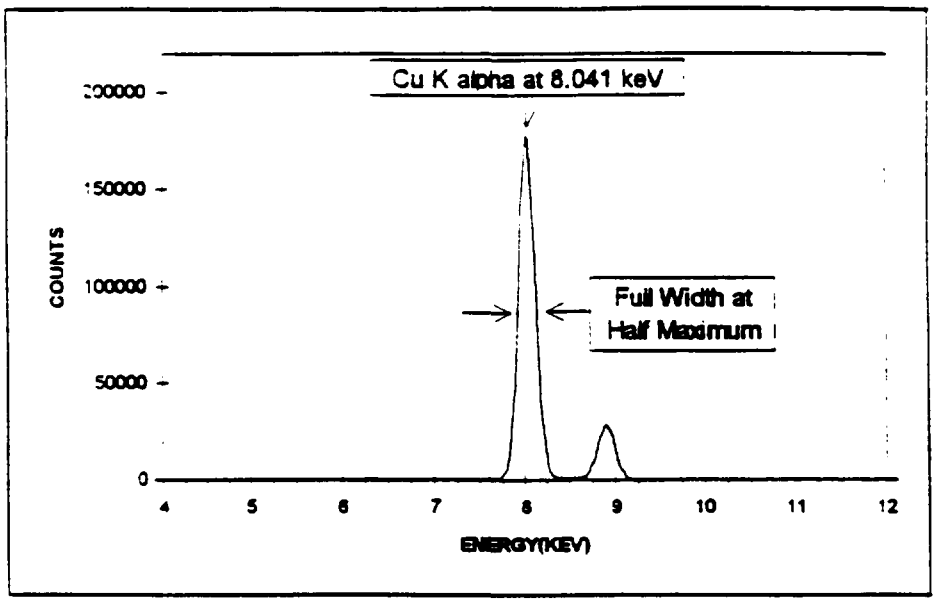

Figure 1. Spectrum of copper (Cu) calibration sample.

Sample ID	K %	CA %	TI %	CR PPM	MN PPM	FE %	NI PPM	CU PPM	ZN PPM	AS PPM	PB PPM	HG PPM	AG PPM	CD PPM	BA PPM	SIO5 DIFF
2710	2.11	1.25	0.283	(39)	10110	3.38		2950	6952	626	5532	(32)	35	(22)	707	
2711	2.45	2.88	0.306	(47)	638	2.89		114	350	105	1162			42	726	
2704	2	2.6	0.457	135	555	4.11		99	438	(23)	161				414	
2709	2.03	1.89	0.342	130	538	3.5	88	35	106						968	
8408	(1.5)		(0.56)	(88)	(1400)	(2.6)			(160)		(80)	107			(360)	
8407	(1.7)		(0.44)	(98)	(970)	(2.42)			(230)		(96)	50			(400)	

Figure 2. Standards file data.

SPECTRUM PROCESSING- PROCEDURE 2- LOW Z STANDARDS

1.	*AR K	- XML-FITTING	< 2280 - 3660 EV >
2.	K KA	- XML-FITTING	< 2580 - 4120 EV >
3.	CA KA	- XML-FITTING	< 2960 - 4540 EV >
4.	TI KA	- XML-FITTING	< 3760 - 5460 EV >
5.	*V KA	- XML-FITTING	< 4360 - 5940 EV >
6.	CR KA	- XML-FITTING	< 4660 - 6500 EV >
7.	MN KA	- XML-FITTING	< 5140 - 7040 EV >
8.	FE KA	- XML-FITTING	< 5640 - 7620 EV >
9.	*BA L	- XML-FITTING	< 3420 - 6400 EV >

Figure 3. Reference file setup for Low Z standards.

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SPECTRUM PROCESSING - PROCEDURE 3 MID Z STANDARDS

1.	*MN	- XML-FITTING	< 5240 - 7020 EV >
2.	*FE	- XML-FITTING	< 5740 - 7640 EV >
3.	*CO	- XML-FITTING	< 6220 - 8220 EV >
4.	NI KA	- XML-FITTING	< 6700 - 8780 EV >
5.	CU KA	- XML-FITTING	< 7260 - 9480 EV >
6.	ZN KA	- XML-FITTING	< 7840 - 10140 EV >
7.	AS KA	- XML-FITTING	< 9720 - 12460 EV >
①8.	PB LB	- XML-FITTING	< 9860 - 13560 EV >
②9.	*PBLL	- XML-FITTING	< 8500 - 9680 EV >
②10.	*PBLG	- XML-FITTING	< 13840 - 16180 EV >
11.	*RB	- XML-FITTING	< 12660 - 15740 EV >
12.	*SR	- XML-FITTING	< 13340 - 16540 EV >
13.	*ZR	- XML-FITTING	< 14880 - 18500 EV >
14.	*BR	- XML-FITTING	< 10960 - 13940 EV >
15.	HG LB	- XML-FITTING	< 9240 - 12980 EV >
16.	*W LA	- XML-FITTING	< 7720 - 10700 EV >

①- Region No. 8 is defined using the interference sample spectrum C:\REFS\SPECTRUM.199.

②- Regions Nos. 9 and 10 are defined using a pure Pb sample spectrum C:\REFS\SPECTRUM.182.

Figure 4. Mid Z reference file setup.

SPECTRUM PROCESSING- PROCEDURE 4 HIGH Z STANDARDS

1.	AG KA	- XML-FITTING	< 21100 - 26140 EV >
2.	CD KA	- XML-FITTING	< 22080 - 27340 EV >
3.	SN KA	- XML-FITTING	< 24120 - 29860 EV >
4.	SB KA	- XML-FITTING	< 25180 - 31120 EV >
5.	BA KA	- XML-FITTING	< 30840 - 38020 EV >

Figure 5. High Z reference file setup.

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SPECTRACE INSTRUMENTS

SPECTRUM : 2711

TUBE VOLTAGE : 13 KV
TUBE CURRENT : 0.30 mA
ATMOSPHERE : AIR

FILTER USED : ALUMINUM
LIVETIME : 100 SEC

F.S. = 8K

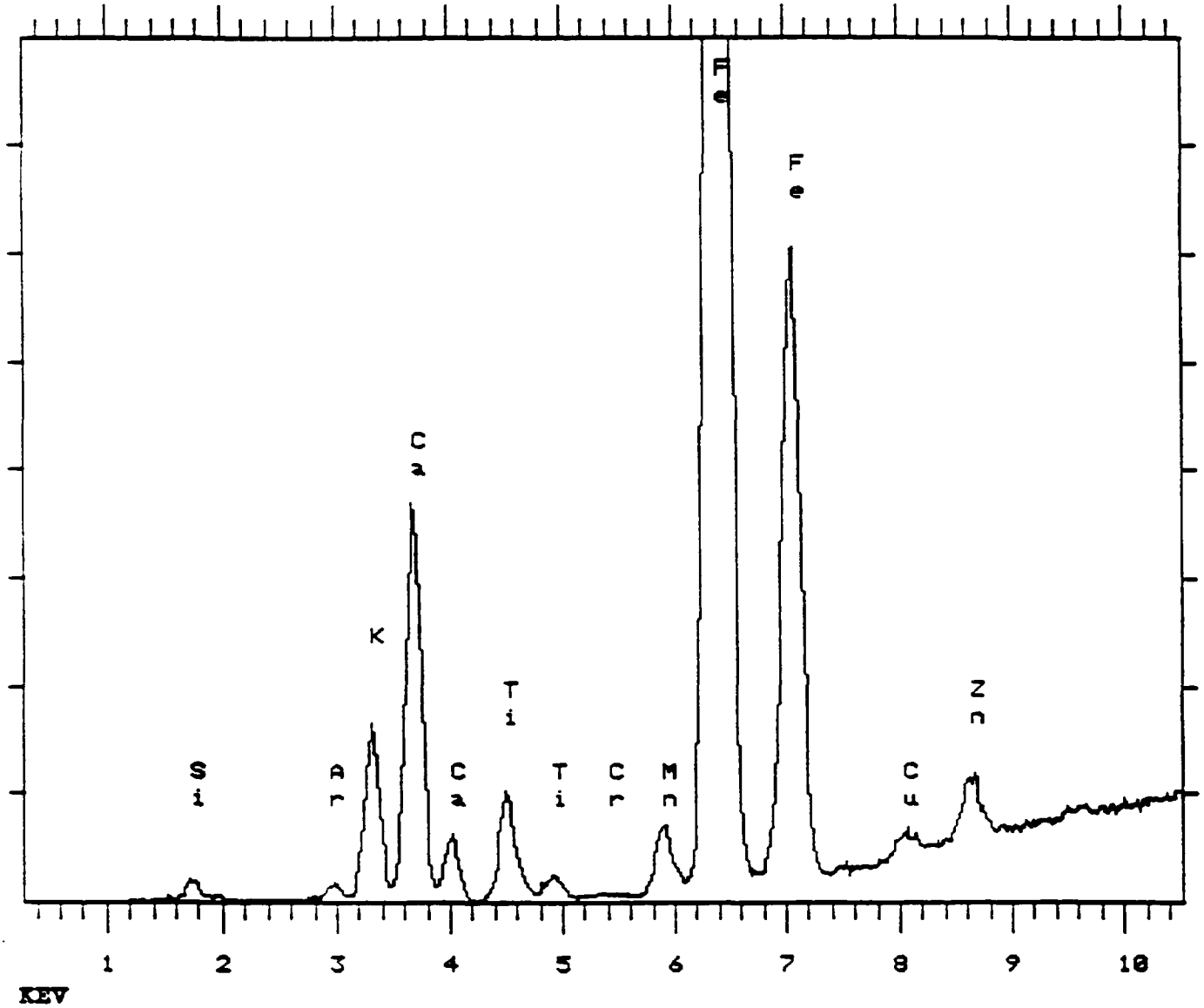

Figure 6. Example of a Low Z acquired spectrum.

SPECTRACE INSTRUMENTS

SPECTRUM : 2711

TUBE VOLTAGE : 35 KV
TUBE CURRENT : 0.10 mA
ATMOSPHERE : AIR

FILTER USED : PD THICK
LIVETIME : 200 SEC

F.S. = 2K

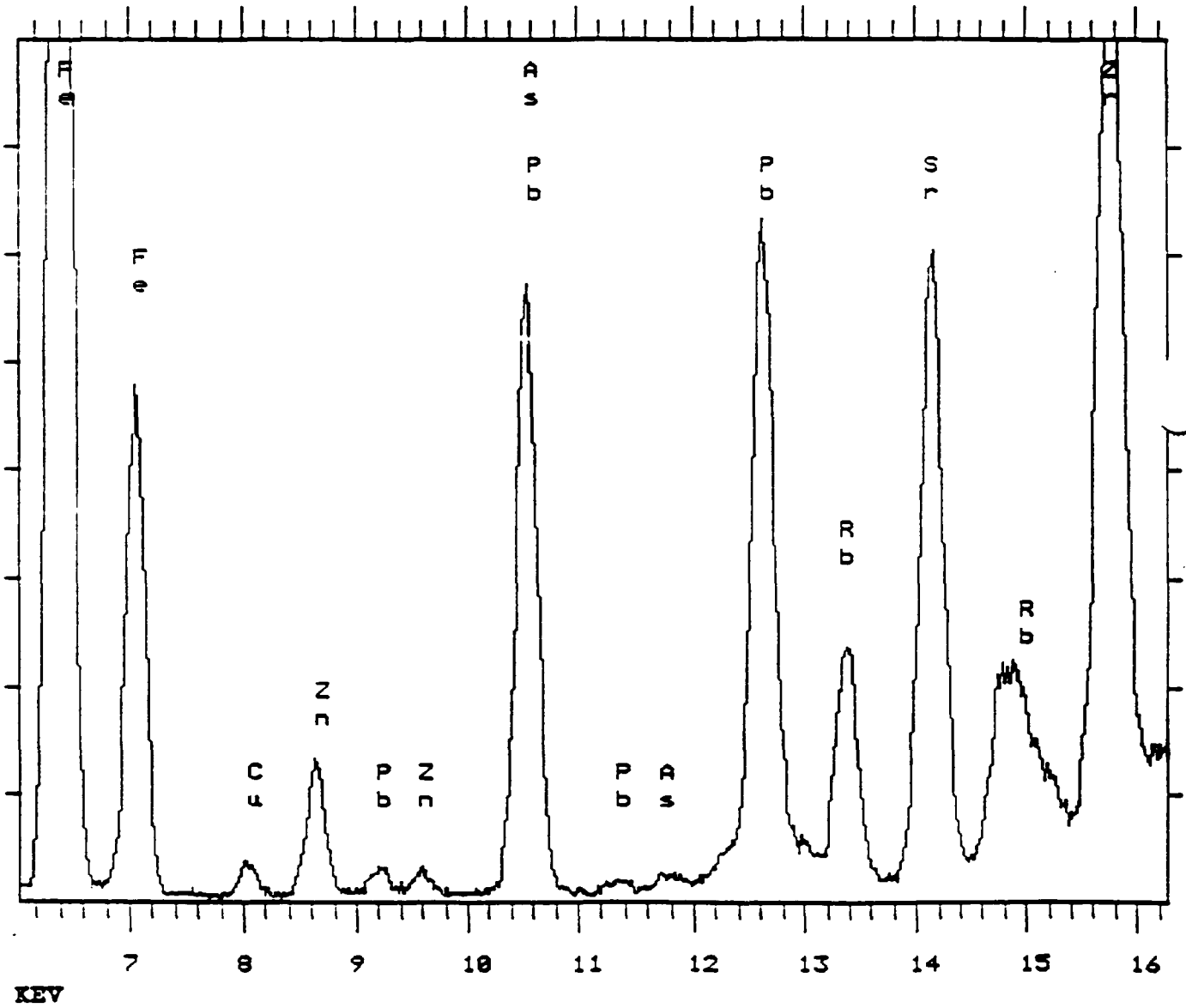

Figure 7. Example of a Mid Z acquired spectrum.

SPECTRACE INSTRUMENTS

SPECTRUM : 2711

TUBE VOLTAGE : 50 KV
TUBE CURRENT : 0.35 mA
ATMOSPHERE : AIR

FILTER USED : COPPER
LIVETIME : 200 SEC

F.S. = 2K

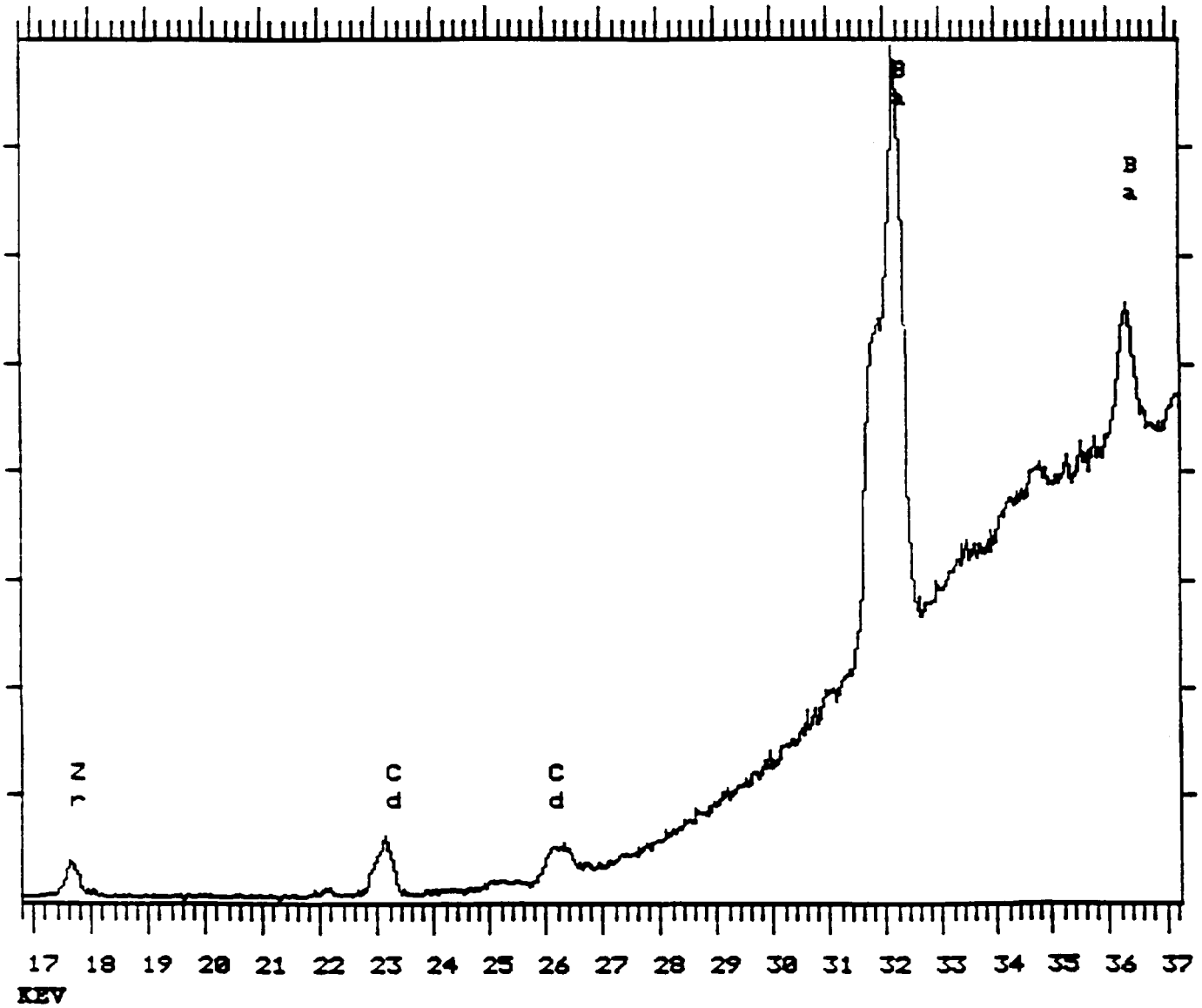

Figure 8. Example of a High Z acquired spectrum.

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EDXRF ANALYSIS
SPECTRACE INSTRUMENTS

PROCEDURE : LOW Z UNKNOWNS
FILTER USED : ALUMINUM — TUBE VOLTAGE : 13 KV
ATMOSPHERE : AIR — TUBE CURRENT : 0.30 MA
COUNT RATE RANGE : MED — LIVETIME : 100 SEC
ANALYSIS METHOD : NO ANALYSIS — PRESET COUNT : 0 K

TIME : 1:58 pm — DATE : 5/20/94

EDXRF ANALYSIS
SPECTRACE INSTRUMENTS

PROCEDURE : MID Z UNKNOWNS
FILTER USED : PD THICK — TUBE VOLTAGE : 35 KV
ATMOSPHERE : AIR — TUBE CURRENT : 0.10 MA
COUNT RATE RANGE : MED — LIVETIME : 200 SEC
ANALYSIS METHOD : NO ANALYSIS — PRESET COUNT : 0 K

TIME : 1:58 pm — DATE : 5/20/94

EDXRF ANALYSIS
SPECTRACE INSTRUMENTS

PROCEDURE : HIGH Z UNKNOWNS
FILTER USED : COPPER — TUBE VOLTAGE : 50 KV
ATMOSPHERE : AIR — TUBE CURRENT : 0.35 MA
COUNT RATE RANGE : MED — LIVETIME : 200 SEC
ANALYSIS METHOD : NO ANALYSIS — PRESET COUNT : 0 K

TIME : 1:58 pm — DATE : 5/20/94

EDXRF ANALYSIS
SPECTRACE INSTRUMENTS

PROCEDURE : 6+7+8
FILTER USED : PD THIN — TUBE VOLTAGE : 30 KV
ATMOSPHERE : AIR — TUBE CURRENT : 0.02 MA
COUNT RATE RANGE : MED — LIVETIME : 100 SEC
ANALYSIS METHOD : FUN. PARAMS. — PRESET COUNT : 0 K

TIME : 1:58 pm — DATE : 5/20/94

SAMPLE	ELEMENT	CONCENTRATION			ERROR
ID 41267					
	K	0.946	%	+/-	0.0160
	CA	0.396	%	+/-	0.0071
	TI	0.375	%	+/-	0.0050
	CR	16.816	PPM	+/-	8.7725
	MN	905.854	PPM	+/-	11.0601
	FE	2.425	%	+/-	0.0044
	NI	22.736	PPM	+/-	7.0572
	CU	93.608	PPM	+/-	7.5829
	ZN	936.812	PPM	+/-	10.7608
	AS	9.505	PPM	+/-	3.1224
	PB	181.592	PPM	+/-	3.7554
	HG	N D			
	AG	N D			
	CD	0.866	PPM	+/-	0.7174
	BA	3580.220	PPM	+/-	12.1648
	SIO5	95.283	DIFF		

Figure 9. Example of a Spectrace 6000 soil sample printout.

RUN #	FWHM*	PEAK CENTROID	TOTAL COUNTS	PEAK COUNTS	TIME
1	209.6	8041	2187969 ± 0.00%	1345906 ± 0.00%	12:30:28
2	210.2	8042	2189712 ± 0.04%	1346270 ± 0.01%	12:38:01
3	211.2	8042	2188745 ± 0.03%	1344938 ± 0.04%	12:45:28
4	210.3	8042	2187722 ± 0.04%	1344136 ± 0.06%	12:52:55
5	209.6	8042	2189506 ± 0.04%	1347528 ± 0.09%	13:00:22
6	211.1	8042	2189264 ± 0.03%	1345296 ± 0.08%	13:07:50
7	210.9	8042	2186424 ± 0.05%	1343684 ± 0.09%	13:15:16
8	210.0	8042	2194782 ± 0.11%	1350504 ± 0.15%	13:22:44
9	210.4	8042	2191210 ± 0.10%	1347079 ± 0.14%	13:30:12
10	210.7	8042	2191683 ± 0.10%	1347479 ± 0.14%	13:37:39

*- FWHM - full width at half maximum of the Cu K alpha peak. See Figure 1.

Figure 10. Example of resolution and stability test procedure printout.

Appendix A: INTRODUCTION

Government agencies as well as private industry are actively investigating and remediating soils at sites contaminated by heavy metals. Energy dispersive X-ray fluorescence analysis (EDXRF) is well suited to the determination of heavy metals in contaminated soils because it is a non-destructive and accurate elemental analysis technique. Energy dispersive X-ray fluorescence spectrometers are field portable or transportable in a van or trailer. Transportable EDXRF systems can provide minimum detectable levels (MDL) below 10 ppm for most metals. Field portable EDXRF instruments provide MDLs below 50 ppm for most metals. EDXRF results correlate well with other metal analysis methods (AA or ICP).

Quantitative X-ray fluorescence analysis is a comparison analytical method; EDXRF spectrometer response for an elemental emission line is evaluated with respect to the known concentration of the element in a standard material. Simple sample matrix types such as polymers or hydrocarbons, exhibit linear X-ray intensity response over limited metal concentration ranges. Contaminated soils, by comparison, are complex samples in regard to their overall elemental composition. Matrix effects are defined as the impact that the concentration variations of major soil components (ex. Ca, Fe, Si) has on measured analyte X-ray emission intensity.

Matrix effects produce a non-linear measured X-ray intensity versus metal concentration response when analyzing soils. A classical method that addresses matrix effects, called site specific standardization, relies on a suite of standards and regression mathematics. When this aspect of EDXRF is applied to contaminated soils analysis several concerns arise as listed in Table 1. The standard materials selected for EDXRF calibration must contain metal analyte concentrations that bracket the range found at the contaminated site. In addition, the concentrations of iron, calcium, and silicon in the standard materials must bracket those found in the soil types at the site.

Table 1. Comparison of two standardization methods.

FP Standardization	Site Specific Standardization
❒ No site sampling required prior to project initiation;	O Soil samples to be used as standards must be collected well in advance of project initiation date;
❒ Pure elements and/or readily available certified reference materials are used as standards (see Appendix B);	O High costs associated with lab analysis of soils and significant time required to receive data back;
❒ Concentration levels of matrix and metals in the standards need not bracket the levels found at the site.	O A large number of standards are required so that the matrix and metal concentrations bracket the entire range found at the site.

Figure 1 is an example of a site specific standardization for lead in soil. The samples earmarked to be used as standards were collected during an expeditionary sampling trip to the site followed by independent analysis of the collected samples. Soil samples selected as standards (usually significantly fewer than were collected) are designated "site specific" and hopefully bracket the concentration ranges required. Some reasons for omitting a soil sample from the suite of standards may include: (1) Having several other samples possessing the same general concentrations of metals and matrix, or (2) questionable independent analysis data for a metal or matrix component.

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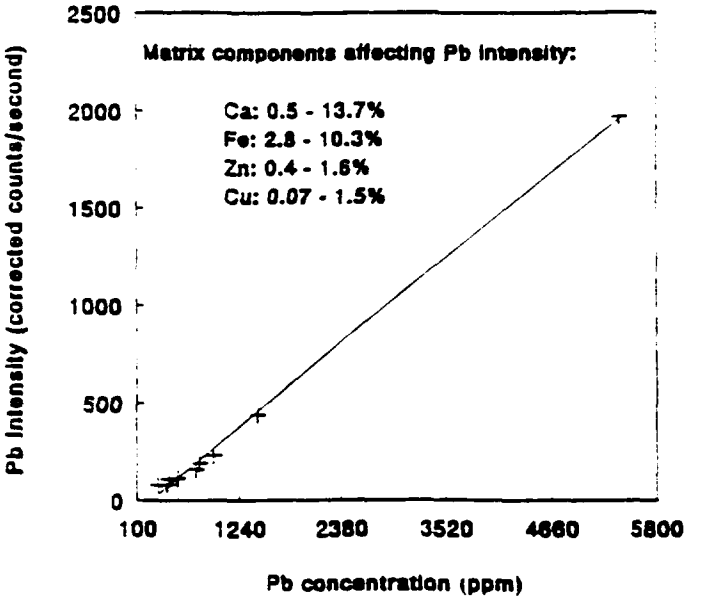

Figure 1. Lead calibration obtained using site specific standards.

Generating site specific standards may be prohibitive for two reasons: (1) Cost considerations concerning expeditionary sampling and sufficient independent analysis to have confidence in the soil metal and matrix compositions, or (2) time constraints involving sampling and waiting for independent analysis results.

The Pb calibration curve shown in Figure 1 is only valid if unknown soil compositions fall between 400 and 5200 ppm Pb and possess a matrix makeup in the ranges listed on the figure. While at some sites this requirement may be met, other sites may provide samples that routinely fall outside the required concentration ranges.

As a result of the constraints imposed by site specific standardization, a mathematical treatment of matrix effects designated "Fundamental Parameters" (FP) has been developed (see Table 1). The FP routines discussed in this report provide accurate metal concentration determinations in contaminated soil samples that have wide ranges of metal and matrix concentrations. Calibration of an FP method requires only a limited set of certified generic soil standards or pure elements.

Spectrace data analysis software uses one of two types of FP calibration methods. The Spectrace 9000, a HgI_2 detector based field portable EDXRF spectrometer uses radioisotope excitation that classifies the FP method as an effective energy computation. The Spectrace models 6000 and 5000 use X-ray tube excitation and the FP routine is classified as a tube modeling routine.

TUBE MODELING FP ROUTINES

Fundamental parameter routines based on tube modeling use equations describing X-ray tube intensity output distributions. Mathematical derivation of these algorithms occurred sometime ago; they have only recently been executed on personal computers.

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Tube modeling routines can use pure elements or oxides of the pure elements as standards. Unfortunately, however, X-ray excitation conditions appropriate for analysis of a pure metal sample do not result in optimum sensitivities and minimum detectable metal levels in a soil matrix.

To achieve optimum X-ray analysis conditions for metals in soils, a limited set (typically four) of soil standard materials is selected. The National Institute of Standards and Technology (NIST) provides several certified reference materials (Appendix B) satisfactory for standardization of the tube modeling FP routines.

Prior to FP standardization X-ray spectra of the standards are collected and analyzed for analyte and matrix element emission intensities. Calibration proceeds by completely defining for the program each standard sample's composition, including the major components (ex. Fe, Ca). Many instances arise when a portion of the matrix may be better estimated by a difference component. For soil, the difference component is defined as SiO_X where X can be 2 - 5. When X = 5, a composition of 24% Si and 45% O results that approximates the concentrations found in most soils types.

The tube output distribution is calculated by the program followed by determination of the matrix independent pure element count rate (PECR) for each analyte. The routine then develops a set of theoretical standards. Next, the routine computes the theoretical X-ray intensity expected for each analyte using equation A-1 shown in Appendix A. Combining the theoretical standard and intensity information, alpha influence coefficients are determined by solving simultaneous equations of the form in equation A-2 (Appendix A). Alpha coefficients describe the quantitative effect of each matrix element on an analyte's measured X-ray intensity.

Unknowns are quantitated by estimating analyte concentration from the measured intensity and the alpha coefficients. Next, theoretical intensities are calculated from equation A-1 and then the computed intensities are compared with the measured intensities. If the intensities are more than 1% different, a new estimate of concentration is made and the theoretical intensity computation is repeated. The iteration continues until the theoretical and measured intensities converge. Figure 2 illustrates correlation between a tube modeling FP routine for 150 soil samples analyzed by EDXRF for Pb and Cd and inductively coupled plasma analysis (ICP) data.

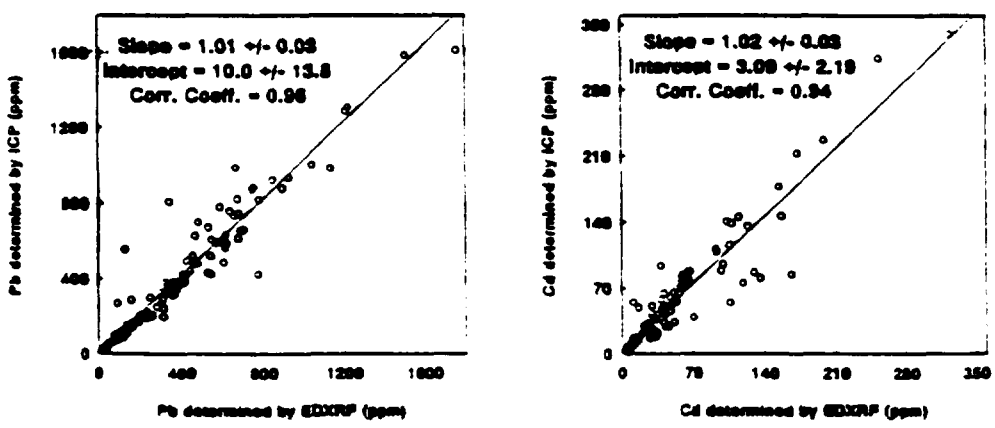

Figure 2. Correlation plots for Pb and Cd analyzed in 150 soil samples by both EDXRF and ICP.

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Field portable spectrometers incorporate, almost exclusively, radioisotope excitation rather than the X-ray tubes found in transportable EDXRF spectrometers. Advantages of radioisotopes are that the spectrometer can be small and lightweight. Compared to X-ray tube excitation, however, a radioisotopes X-ray flux is much smaller and higher minimum detectable metal concentrations result.

Effective energy fundamental parameter methods are just as accurate as their X-ray tube modeling counterparts, but are mathematically simpler. This is due to the nature of the radioisotope output, which is monochromatic, and therefore equation A-1 is not integrated over a broad range of energies.

The effective energy routine offered in the Spectrace 9000 relies on spectrometer response measured from pure elements and fundamental parameters to ascertain matrix interactions in soil. Determination of alpha coefficients during standardization proceeds by determining a set of theoretical standards. Next, using a variation of equation A-1, without the integral, theoretical intensities are calculated. Equation A-2 is then solved as a set of simultaneous equations using the theoretical information. Calibration of the Spectrace 9000 occurs at the factory using the effective energy FP routine then alpha coefficients are downloaded into the permanent memory of the Spectrace 9000 electronics module.

Computation of unknown concentrations proceeds in an iterative fashion using equations of the form A-3 shown in Appendix A. For these calculations, concentrations are compared and adjusted between each iteration for all the analyte elements. Figure 3 illustrates Pb and Cu results obtained for soil samples analyzed using the Spectrace 9000. The EDXRF results are correlated in the plots with atomic absorption data.

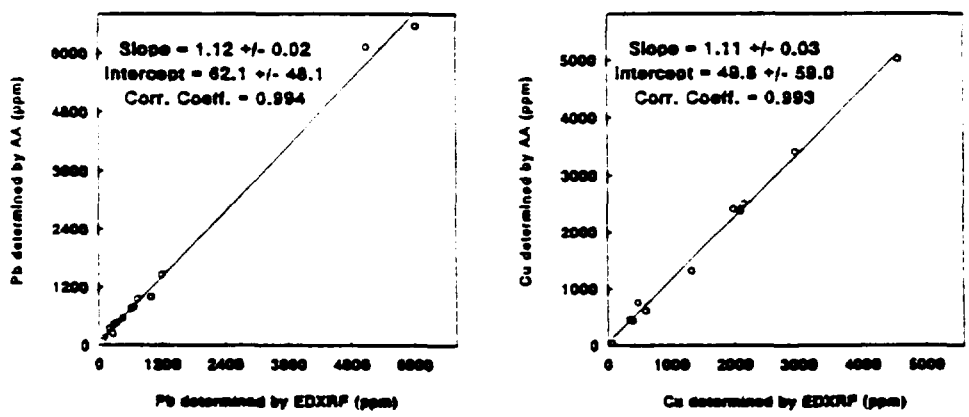

Figure 3. Correlation plots for Pb and Cu analyzed in soil samples by both EDXRF and AA.

CONCLUSION

The incorporation of fundamental parameter routines for EDXRF analysis of soils provides accurate estimates of metal contaminant concentration with a minimum number of standard materials. FP standardization offers several advantages when compared with site

specific standardization. Fundamental parameters standardization relies on pure element standards or certified standard reference soil materials. These materials are readily available, unlike site specific standards. Universal application to all soil matrix types is another merit of FP standardization. When analyzing soil samples collected from a site EDXRF results correlate well with AA and ICP methods. Fundamental parameters analysis methods provide rapid and cost effective EDXRF measurement data in contrast to site specific standardization that requires an initial sampling trip and the time interim waiting for laboratory results.

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302211

Principles of Energy Dispersive X-ray Fluorescence Spectrometry

Spectrace Instruments, Inc.

Energy dispersive X-ray fluorescence spectrometry (EDXRF), developed in the early 1970's, is a flexible elemental analysis technique used for qualitative and quantitative composition determinations of solid, liquid, thin film, and powder samples. EDXRF is capable of analyzing elements with atomic number 11 (sodium) and higher in the periodic table in concentration ranges of a few ppm to 100% by weight.

The versatility of EDXRF spectrometry is indicated by the myriad of sample types effectively analyzed by the technique. Table 1 is a breakdown of three analytical areas where EDXRF has found extensive use: quality assurance and quality control of finished products, environmental samples, and process streams. Advantages of the EDXRF method compared to other elemental analysis techniques include: EDXRF is non-destructive, requires little or no sample preparation, analysis turnaround time is fast (usually three to twenty minutes) and the interpretation of X-ray emission spectra acquired from a sample is straightforward.

Quality Control/Quality Assurance	
	Magnetic media..... Cr and Co film thickness
	Silicon wafers..... P_2O_5 in a SiO_2 layer
	Dry copier toner..... Si, S, Zn, and Fe
	Finished polymers..... V in HDPE; Ca in butyl rubber
	Oil blending..... additive components
Environmental	
	Contaminated soil..... Pb, As, Cr and Cd
	Air filters..... Heavy metals
	Hazardous waste..... Cr, Pb, As, and Zn
	Wastewater effluent..... Heavy metals
Process Control	
	Catalyst depletion..... Mn, Co, and Br in acetic acid
	Plating baths..... Fe, Cu, Zn, Pt, and S in acids
	Hydrocarbon fuel..... S in diesel and gasoline

Table 1. Applications of EDXRF spectrometry.

This general guide describes the fundamental aspects of EDXRF analysis to assist in the understanding of the method applied for the determination of material composition. The discussion of hardware will focus on Spectrace Instruments' EDXRF spectrometers.

I. PRODUCTION OF X-RAY EMISSION AND ACQUISITION OF EDXRF SPECTRA

Figure 1 is a schematic of the interacting modules used in combination to produce the X-ray emission spectrum of a sample. The EDXRF spectrometer components diagrammed in Figure 1 will be discussed in the following three subsections:

A) X-ray excitation: X-rays tubes with primary radiation filters and radioisotope sources;

B) Interaction of X-rays with matter: X-ray absorption followed by X-ray emission;

C) Solid state X-ray detectors and supporting electronics: Generation of the X-ray emission spectrum.

A. X-RAY EXCITATION

The Spectrace EDXRF spectrometers use one of two types of X-ray excitation source. They are X-ray tubes coupled with primary radiation filters or sealed radioisotope sources.

1. X-ray tube/primary radiation filters- The X-ray tube, shown in Figure 2, is an evacuated glass envelope that is comprised of a:

a. Filament with adjustable current control;

b. Pure metal anode which may be Rh, W, or Ag;

c. Beryllium window (2 to 5 mils thick) through which the X-rays radiate toward the sample.

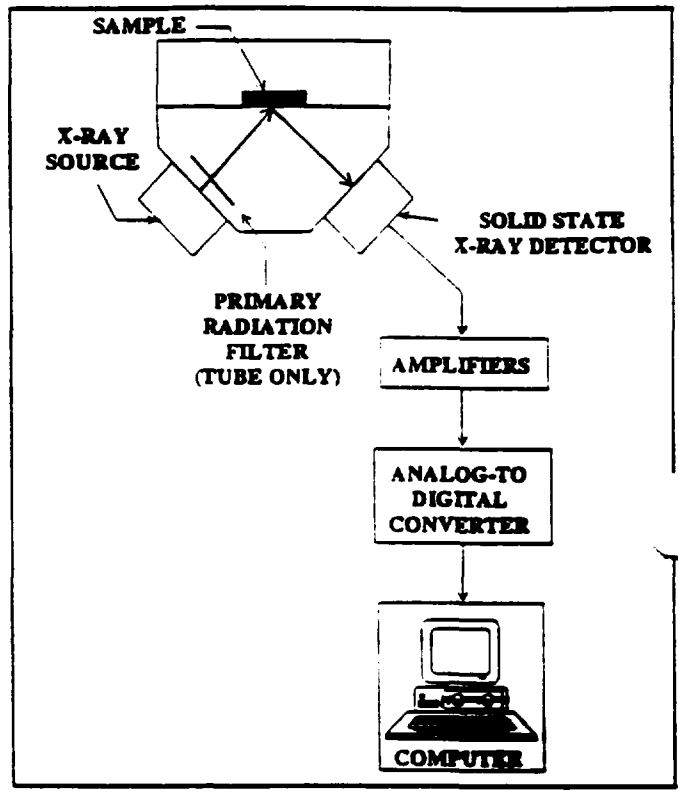

Figure 1. Schematic of an EDXRF spectrometer.

The X-ray tube and high voltage power supply combination provides a stable, high intensity x-radiation source. The X-ray tube is electronically interlocked to cabinet doors and the chamber lid to prevent accidental exposure to operators. The high voltage differential between the filament and anode (Figure 2) is adjustable (4 to 50 kV) by computer control of the X-ray tube power supply. The highest energy X-ray output from the tube is equal to the maximum energy imparted to an electron by the accelerating voltage.

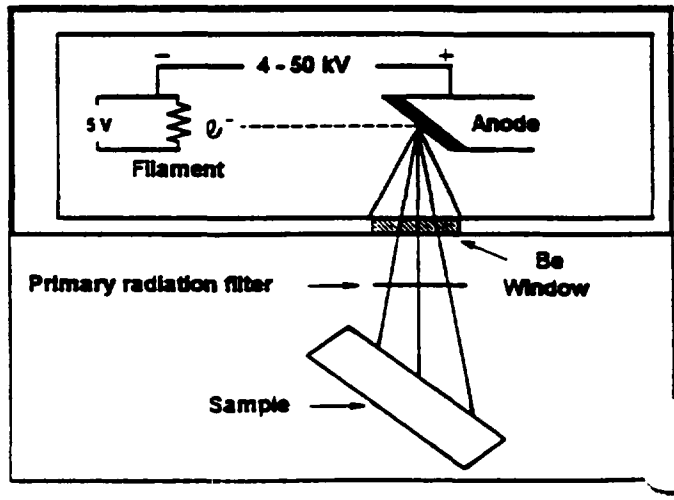

Figure 2. X-ray tube and primary radiation filter position.

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Equation 1 describes this relationship:

$$E_{max}\ \text{output (keV)} = \text{tube voltage (kV)} \qquad \text{Eq. 1}$$

Two separate processes generate X-rays in the tube. First, the electrons strike the anode and gradually decelerate producing a continuum of X-rays. Furthermore, the electron energy imparted to anode atoms may be sufficient to initiate production of characteristic X-ray emission (see the discussion of characteristic X-ray emission later in section B). As a result of these processes, the output distribution from the X-ray tube is a superposition of intense characteristic anode X-rays and continuum, sometimes called bremsstrahlung, x-radiation. Direct X-ray tube excitation provides high spectral flux output with

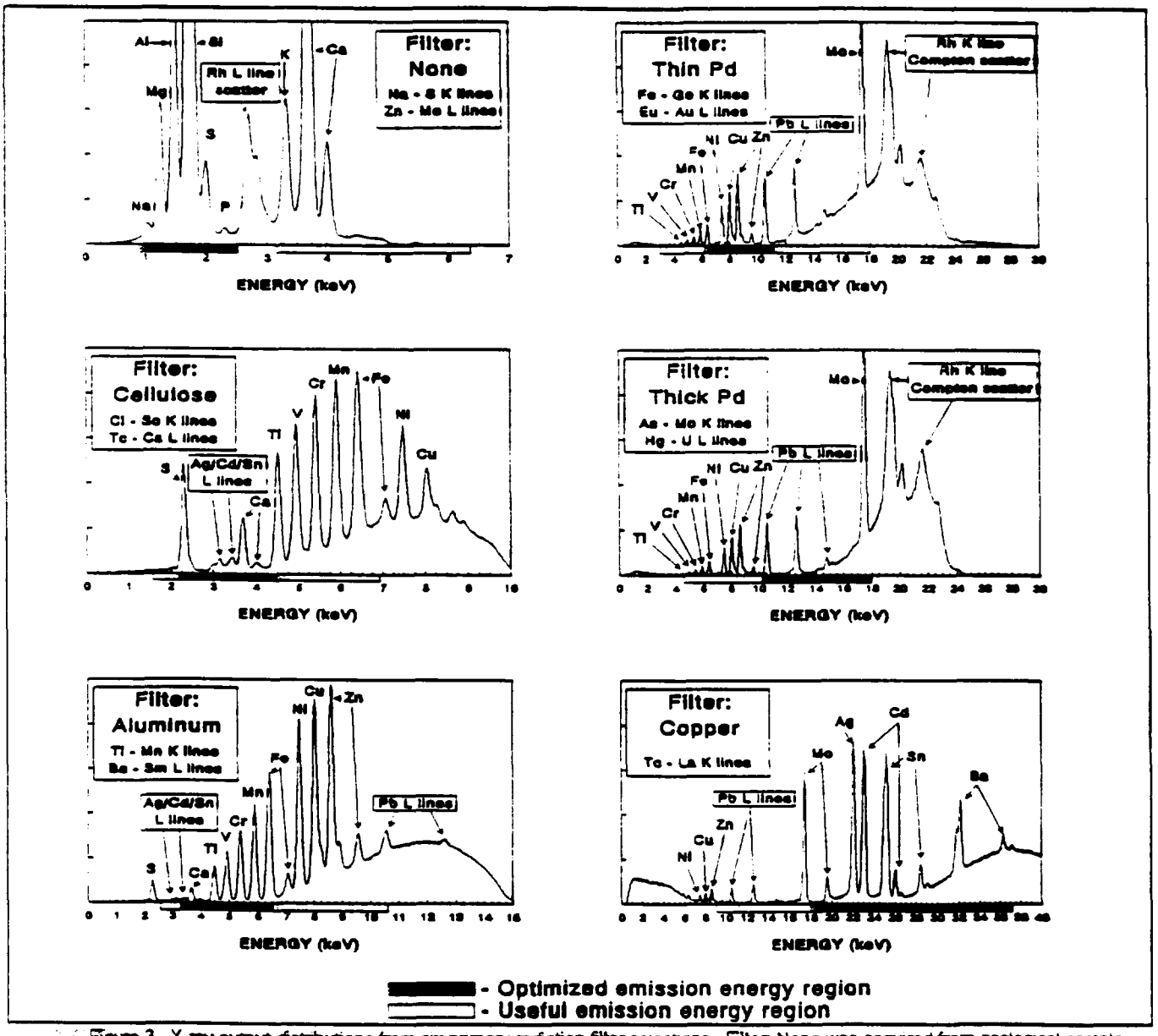

Figure 3. X-ray output distributions from six primary radiation filter selections. Filter: None was acquired from geological sample BR (basalt). All other spectra were acquired from a Conostan S-21 standard containing 900 ppm of each element.

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small power supply requirements (50 watts maximum). In practice, X-ray tube voltage is selected in combination with a primary radiation filter to optimize the excitation conditions for the elements of interest in a sample.

Primary radiation filters (see Figure 1) modify the X-ray tube output distribution before striking the sample. Five primary filters are available and each one is used to optimize the X-ray spectrum acquisition conditions in one or more ways: (1) reduce background beneath element emission lines in a spectral region; (2) eliminate X-ray tube anode, usually Rh, characteristic line radiation; (3) transmit X-rays of sufficient energy to excite the elements of interest. Computer automation allows selection of multiple sets of X-ray tube voltage and primary radiation filters to achieve optimum excitation conditions for complex samples.

Primary X-rays from the tube and primary radiation filter combination can be scattered either elastically (Rayleigh) or inelastically (Compton) by the sample. Scatter results in background in the X-ray spectrum. Figure 3 illustrates the effect of each of six filter positions on Rh anode X-ray tube output shown after scattering from a sample into the detector. Listed on each spectrum are the K and L emission lines and energy regions optimized using a particular filter.

2. Sealed radioisotopes- Some unstable isotopes emit X-rays upon radioactive decay. Radioisotope X-ray sources consist of a radioactive salt sealed in a capsule that includes a window for X-rays to escape. The Spectrace portable spectrometer is equipped with three radioisotope sources mounted on a computer automated motor. The sources are rotated so that X-rays are directed toward the sample. No primary radiation filter is used between the source and the sample, and the source, because of its small size, can be positioned very close to the sample. The X-rays produced by a radioisotope source can be one or more discrete emission lines. This is in contrast to the broad distribution of energies output from the X-ray tube. Table 2 lists the half-lives, emission line energies and useful elemental analysis regions for each source. Radioisotope sources are smaller and lightweight but provide less output flux than X-ray tubes that require a high voltage power supply. Radioisotope equipped spectrometers can be portable for field use but sensitivity is reduced compared to X-ray tube excited EDXRF spectrometers. Figure 4 is a plot of lower limits of detection (LLD) provided by the tube excited EDXRF spectrometers compared to their radioisotope excited counterparts. The LLD's were determined using a geological sample and 200 seconds analysis livetime.

Nuclide	Half-life ($T_{1/2}$)	Emission	Elemental Analysis Rgn.
Fe 55	2.7 yr.	5.9 keV; Mn K X-rays	K - Cr K lines In - La L lines
Cd 109	453 days	22.1 keV: Ag K X-rays 87.7 keV gamma	Cr - Pd K lines Ce - U L lines
Am 241	458 yr.	26.4 keV gamma 59.6 keV gamma	Y - Tm K lines

Table 2. Radioisotopes used in EDXRF spectrometers.

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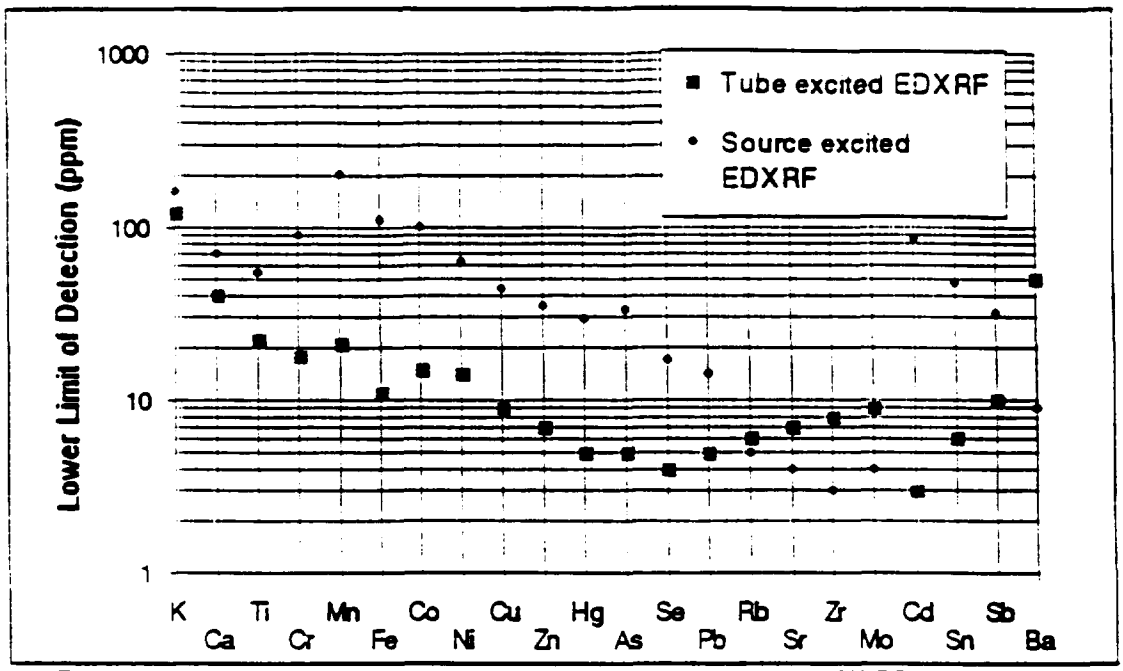

Figure 4. Lower limit of detection comparison of source excited and tube excited EDXRF spectrometers.

B. INTERACTION OF X-RAYS WITH MATTER

1. X-ray absorption- EDXRF relies on interactions between x-radiation from the primary X-ray source and the atoms in the sample. A prerequisite to X-ray emission is X-ray absorption. Source X-rays striking a pure material (shown in Figure 5) with intensity I_0 can be absorbed by the material resulting in an attenuated intensity, I, that passes through. Equation 2 describes this relationship,

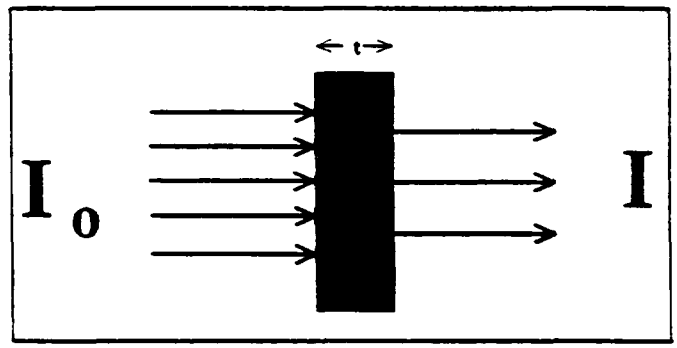

Figure 5. X-ray absorption by a material.

$$I = I_0 e^{-(\mu/\rho)\rho t} \quad \text{Eq. 2}$$

where:

- μ/ρ - mass absorption coefficient;
- ρ - density (g/cm^3) of the material;
- t - thickness (cm) of the pure material;
- e - base of the natural log.

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The mass absorption coefficient, in units of cm^2/g, describes the X-ray absorption characteristics of any material. Figure 6 is a plot of μ/ρ versus X-ray energy impinging on pure lead (Pb, atomic number 82). Note two features: (1) the gradual decrease in μ/ρ with increasing X-ray energy, and (2) the sharp discontinuities corresponding to X-ray absorption.

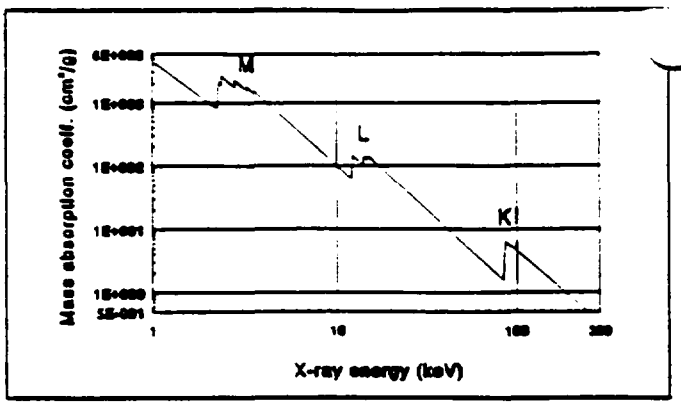

Figure 6. μ/ρ versus energy for Pb, atomic number 82.

Discontinuities or "jumps" in the plot represent photoelectric absorption edges in the Pb atom. The absorption edge represents the amount of energy required to eject an electron from its orbital. For an atom to undergo photoelectric absorption some fraction of the source X-rays incident on the sample must exceed an absorption edge energy. Photoelectric absorption is illustrated on the atomic scale in Figure 7. The photoelectron (e^- in Figure 7) is ejected from the electron cloud with some kinetic energy.

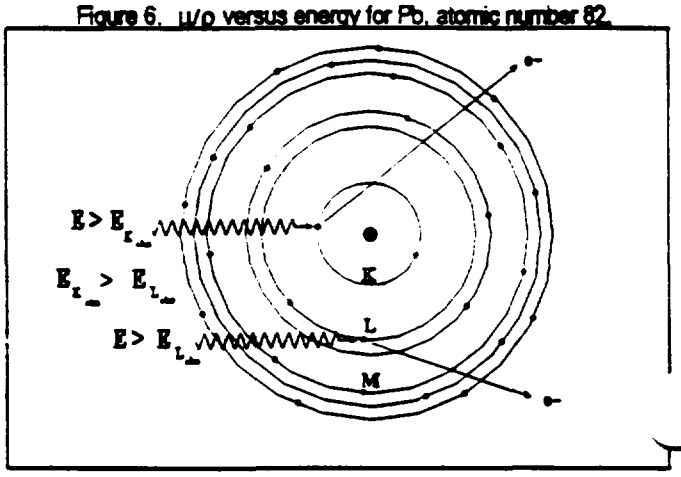

Figure 7. X-ray absorption and photoelectron ejection.

As shown in Figure 7, the innermost electron orbitals are designated the K and L shell. The graph in Figure 6 indicates that, for a particular atom, the energy required to eject a K shell photoelectron ($E_{K\ abs}$) is much larger than the $E_{L\ abs}$ for the L shell because the inner K shell electron is held more tightly to the atom. Each atom in the periodic table has unique values of $E_{L\ abs}$ and $E_{K\ abs}$. Photoelectric absorption by the M shell is rarely of interest.

After photoelectric absorption, a vacancy is created in the electron orbital and the atom is in an excited, unstable energy state. To return to the ground state, the excited atom undergoes electronic rearrangement to fill the electron vacancy. The relaxation processes of excited atoms are discussed in the following section.

2. <u>X-ray emission</u>- An electronic rearrangement that produces an X-ray photon is called a fluorescence transition. An X-ray photon emitted by such a transition is designated a "characteristic" X-ray and its energy indicates: (1) the atomic number of the emitting atom and, (2) the specific fluorescence transition giving rise to the X-ray emission. Subsequent analysis of emitted characteristic X-rays is the basis for energy dispersive X-ray fluorescence spectrometry. To date there are two accepted notation conventions for X-ray transitions: Siegbahn and IUPAC. The IUPAC notation is the most recent and is included in

this text in brackets []. Extensive tabulations of known elemental emission line energies are accessed and displayed by the data analysis computer.

Consider the electronic transitions shown in Figure 8. If an electron vacancy occurs in the K shell, either the Kα [K-L_3] (from the L shell) transition or the Kβ [K-$M_{2,3,5}$] (from the M shell) transition may follow. The Kα [K-L_3] transition is approximately ten times more probable than the Kβ [K-$M_{2,3,5}$] transition and the K emission of an element in the EDXRF spectrum can be identified by a pair of emission lines at an intensity ratio of 10:1. Note the Cu Kα [K-L_3] at 8.041 keV and Cu Kβ [K-$M_{2,3,5}$] at 8.903 keV peaks in Figure 9, which is the X-ray fluorescence spectrum of a brass sample.

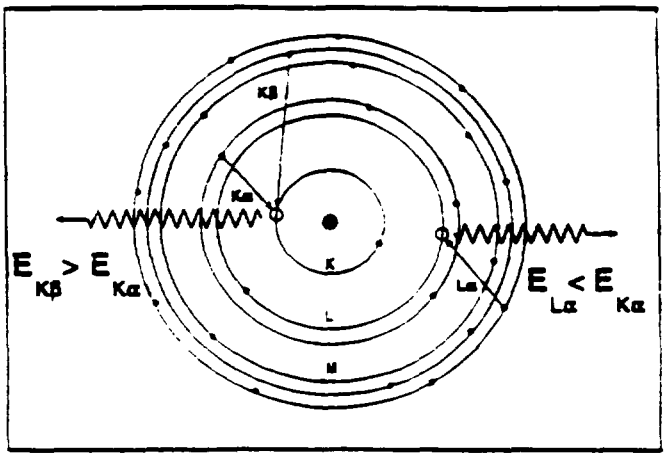

Figure 8. X-ray fluorescence transitions.

When an L shell vacancy occurs, electrons from the M or N shell can fill the vacancy as illustrated in Figure 8. For an individual atom, photons emitted from L transitions are always less energetic than those produced from K transitions. The pattern of the L emission lines from an element is an intense pair of peaks (Pb Lα[L_3-$M_{4,5}$] at 10.550 keV, Pb Lβ[L_3-N_5] at 12.612 keV) accompanied by other minor emission lines (Figure 9). Multiple L emission lines arise because there are three separate L shell electron energy levels that can be excited as indicated by the L(I), L(II) and L(III) absorption edges shown in Figure 6.

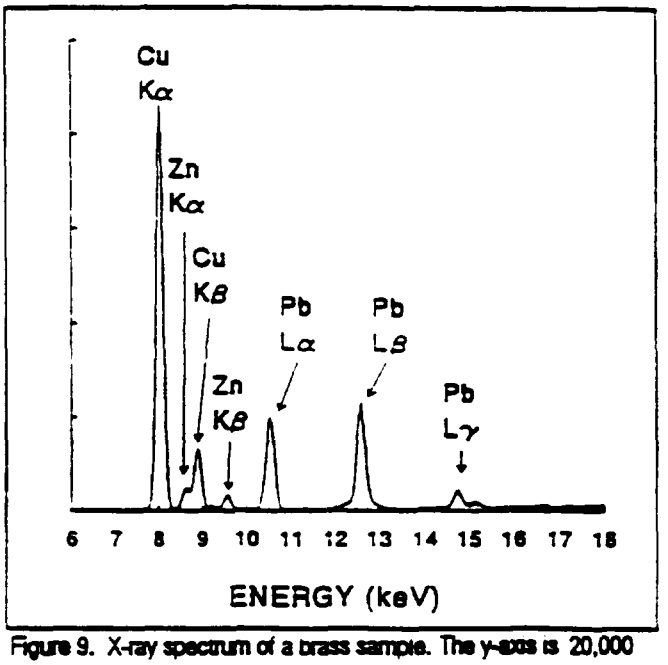

Figure 9. X-ray spectrum of a brass sample. The y-axis is 20,000 counts full scale.

The L emission lines are useful for determination of those elements with atomic number 47 (Ag) and above in the periodic table. Since the X-ray tube maximum output energy is limited to 50 keV and the extent of the useful spectral

range is 0–40 keV it follows that L line emission must be used for determination of those elements above atomic number 60 (Nd).

The solid state X-ray detector (shown schematically in Figure 1) in the EDXRF spectrometer analyzes characteristic X-ray photons emitted by fluorescence transitions to determine the X-ray spectrum of a sample. Quantitative analysis using EDXRF relies on the intensity of either L or K emission lines to represent elemental concentrations.

C. SOLID STATE X-RAY DETECTORS AND SUPPORTING ELECTRONICS

The solid state X-ray detector, shown schematically in Figure 10, can be either a lithium drifted silicon (Si(Li) crystal or HgI_2 crystal. The HgI_2 crystal is operated near room temperature, in contrast to the Si(Li) crystal must be cooled to at least -90 °C for operation. There are two mechanisms of Si(Li) detector cooling:

1) Thermal contact with liquid nitrogen(LN);

2) Thermoelectric cooling with the Peltier effect (ECD).

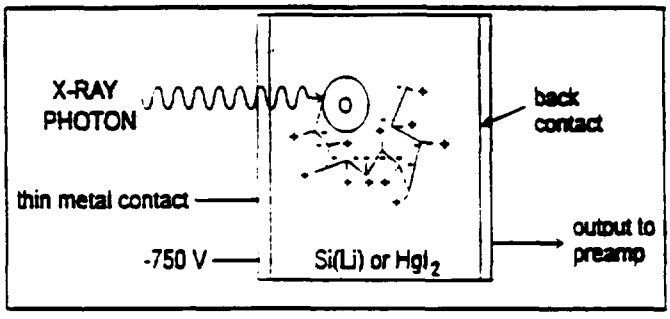

Figure 10. A solid state X-ray detector.

The term resolution defines the ability of an X-ray detector to separate X-ray photons very near one another in energy. A smaller full width at half maximum (FWHM) represents improved detector resolution. Table 3 lists the detector operational temperatures attained by the two cooling methods and the resolution provided by Spectrace systems configured with either the Si(Li) or the HgI_2 detector.

Solid state X-ray Detector	Cooling Mechanism	Operational Temperature	Resolution (FWHM at Mn Kα X-ray)
Si(Li)	liquid N_2	-170 °C	155 eV
Si(Li)	Peltier	-90 °C	185 eV
HgI_2	Peltier	-5 °C	294 eV

Table 3. Solid state EDXRF detectors

The solid state detector converts X-ray photon energy into a measurable electronic signal. Its operation is as follows: When an X-ray photon enters the crystal, as shown in Figure 10, it ionizes atoms in the active area and produces electron (-) and hole (+) pairs. The number of electron/hole pairs formed is directly proportional to the energy of the X-ray photon that entered the detector. The basis for energy discrimination of X-ray photons emitted from a sample is the proportionality of the detector output signal to the incident X-ray photon energy.

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Detector output signals must first be amplified and then sorted with respect to their magnitude by a pulse height analyzer (PHA). Components of the PHA, shown in Figure 1, include a preamplifier and main amplifier, as well as an analog-to-digital converter. The output from the PHA is the EDXRF emission spectrum and is a plot of the number of X-ray events detected, or counts, on the y-axis versus pulse height, or X-ray energy, on the x-axis. The EDXRF spectrum shown in Figure 9 is transmitted to the personal computer for display and further processing.

The acquisition time selected for collection of the EDXRF emission spectrum depends largely on the goals of a particular analysis method. Precision of an analysis can be improved by acquiring the spectrum for a longer duration thereby reducing the effect of random counting errors on the overall analytical error. Typical EDXRF spectrum acquisition times can range from a few seconds to 30 minutes depending on the application.

II. EDXRF SPECTRUM PROCESSING AND PEAK FITTING METHODS

Quantitative determinations performed by EDXRF require that elemental emission lines observed in the X-ray spectrum of a sample be analyzed for subsequent correlation to concentration or thin film thickness. Computer processing of X-ray spectra (Figure 9) integrates peaks to accurately represent the intensity of emission peak (or peaks) of an individual element. Accurate peak intensities from each spectrum are required because any errors are mathematically correlated to elemental concentration variation in the quantitative analysis portion of the method.

When no peak overlaps are present, analyte intensity can be represented by a simple gross (peak plus background) or net (peak above background) integrated area incorporating the emission line. A major concern in EDXRF analysis methods is analyte emission peak overlaps in complex spectra acquired from multi-component samples. Features in complex spectra that can overlap analyte peaks are: a) emission lines from other elements in the sample; b) detector artifacts, or; c) spectral feature such as scatter peaks arising from the X-ray tube anode.

To address peak overlaps, a multiple linear least squares fitting routine, also called XML, is available in the EDXRF data analysis software. Before XML fitting, a zero weight digital filter is applied to the X-ray spectrum to remove backgroun variations. The XML fitting routine uses a library of previously acquired reference spectra that represent unobstructed elemental emission peak shapes. Fitting continues by mathematically minimizing spectral differences between the reference spectra and the sample spectrum of interest. After XML processing, the resultant peak above background determined for eac reference spectrum is saved as the analyte intensity. The XML method of fitting successfully extracts overlapped peaks and tabulates accurate analyte peak intensities for quantitative analysis.

III. QUALITATIVE AND QUANTITATIVE ANALYSIS USING EDXRF

The nature of the characteristic X-ray photon, by definition, makes EDXRF a highly selective and rapid qualitative analysis technique. Qualitative analysis is a simple method by which each peak in the X-ray spectrum is matched to known, tabulated elemental emission lines. Automatic peak identification programs are available to identify the elemental emission peaks observed in an EDXRF spectrum. The informational content provided by the X-ray spectrum permits unequivocal confirmation of the presence of an element in a sample.

After analyte peak integration, the next step in the sequence of quantitative EDXRF analysis is correlation of measured analyte peak intensities to element concentration or thin film loading. A crucial component to the success of quantification is the selection of the mathematical model (analysis method) that best describes the analyte emission line intensity versus concentration relationship. The method selected depends on the nature of the samples to be analyzed, availability of standard materials, and objectives of the analysis.

A simple method for correlating analyte peak intensity to component concentration is a linear mathematical model as represented by Equation 3:

$$I_i = B_i + K_i C_i \qquad \text{Eq. 3}$$

where:

C_i = analyte element (i) concentration;

I_i = analyte element (i) intensity (counts/second).

B_i = intercept (intensity at $C_i = 0$);

K_i = slope of the linear least squares regression line;

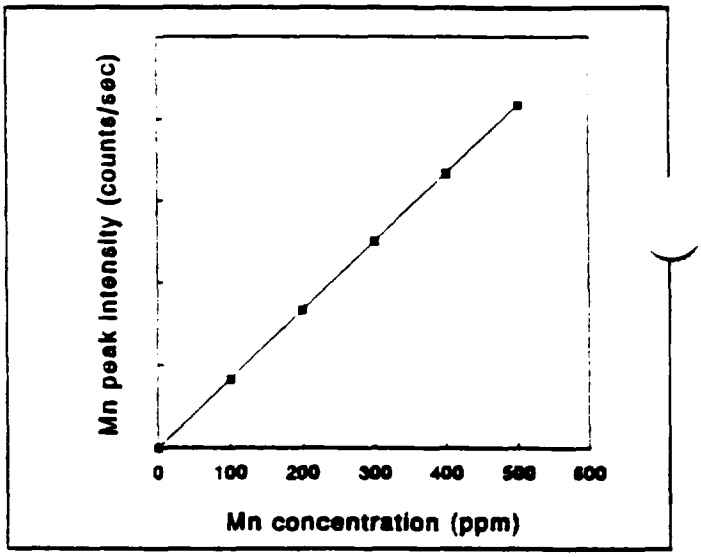

Figure 11. Calibration curve for Mn in an acetic acid solution.

Figure 11 illustrates a linear calibration for Mn (0 to 500 ppm) in a catalyst solution composed of acetic acid, water, and xylene. Other elements present in the process stream are Co and Br in the 300 to 800 ppm concentration range. Sample types that are likely to exhibit a linear analyte intensity versus concentration response are those in which the analyte concentration varies over a limited range and the sample matrix is relatively invariant sample to sample. In this example application, the acetic acid mixture makes up the bulk of the sample matrix and analyte concentration variations are insignificant in comparison.

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In some cases the analyte element itself, because its concentration varies over a large range, can effect the overall mass absorption coefficient of a set of samples. The term used for this phenomenon is self-absorption and can be addressed mathematically by fitting a second order polynomial or quadratic function of the type in Equation 4 to the data.

$$I_i = B_i + K_i C_i + \alpha_{ii} C_i^2 \qquad \text{Eq. 4}$$

where α_{ii} represents the exponential coefficient. However, if a sample is complex and composed of several matrix elements it is advisable to evaluate one of the more explicit matrix correction methods described below.

When an analyte peak intensity versus concentration plot produces a non-linear response, the underlying cause is some type of matrix interaction. To account for matrix interactions, empirical matrix correction algorithms are sometimes used. The function of these routines is to solve a set of simultaneous equations of the type listed in Equation 5.

$$C_i = B_i + K_i I_i \left(1 + \sum_{j=1}^{j=n} \alpha_{ij} I_j\right) \qquad \text{Eq. 5}$$

where:

α_{ij} = alpha coefficient describing the matrix interaction of element j on analyte i;

I_j = intensity measured for matrix element j.

Alpha coefficients are the quantitative representation of the matrix elements' (j's in Eq. 5) effect on the measured intensity of analyte element i. To mathematically determine the coefficients in equation 5, enough type standards with concentrations of both the analyte and matrix elements bracketing the unknown compositions must be available. A "type standard" has a known composition and a matrix composition identical to the unknown samples to be analyzed.

Stainless steel is an example of a sample type that exhibits severe matrix interactions but which are routinely analyzed with mathematical corrections. Chromium's absorption edge energy (Cr K_{abs} = 6.00 keV) is just below the Fe Kα (6.40 keV) emission line energy. An increased Cr measured peak intensity is observed arising from Fe emission, to a greater or lesser degree, depending on Fe concentration. Graphing measured Cr intensity versus Cr concentration results in the non-linear plot shown in Figure 12. Concurrently, iron emission X-rays are selectively absorbed by chromium, resulting in the non-linear plot of Fe intensity versus Fe concentration also shown in Figure 12.

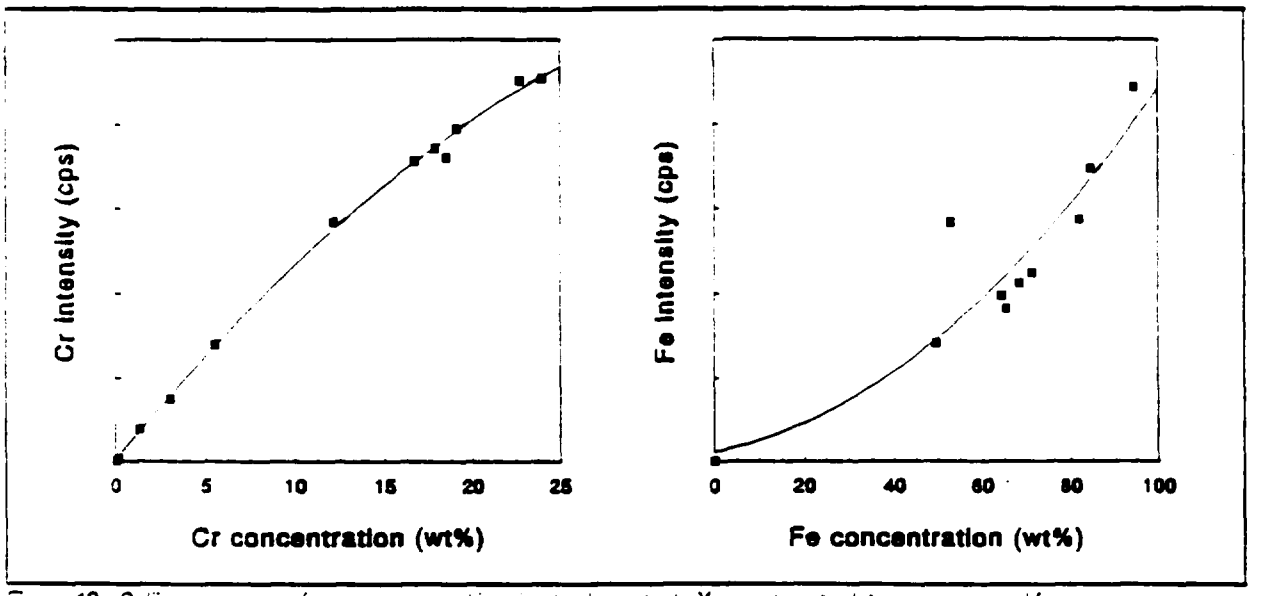

Figure 12. Calibration curves for chromium and iron in stainless steel. X-ray intensity data is uncorrected for matrix interactions.

After solving equation 5 and calculating alpha coefficients, corrected calibration curves are plotted. Figure 13 illustrates the calibration curves resulting from analyte intensities corrected for matrix effects arising from Cr and Fe interaction in stainless steels.

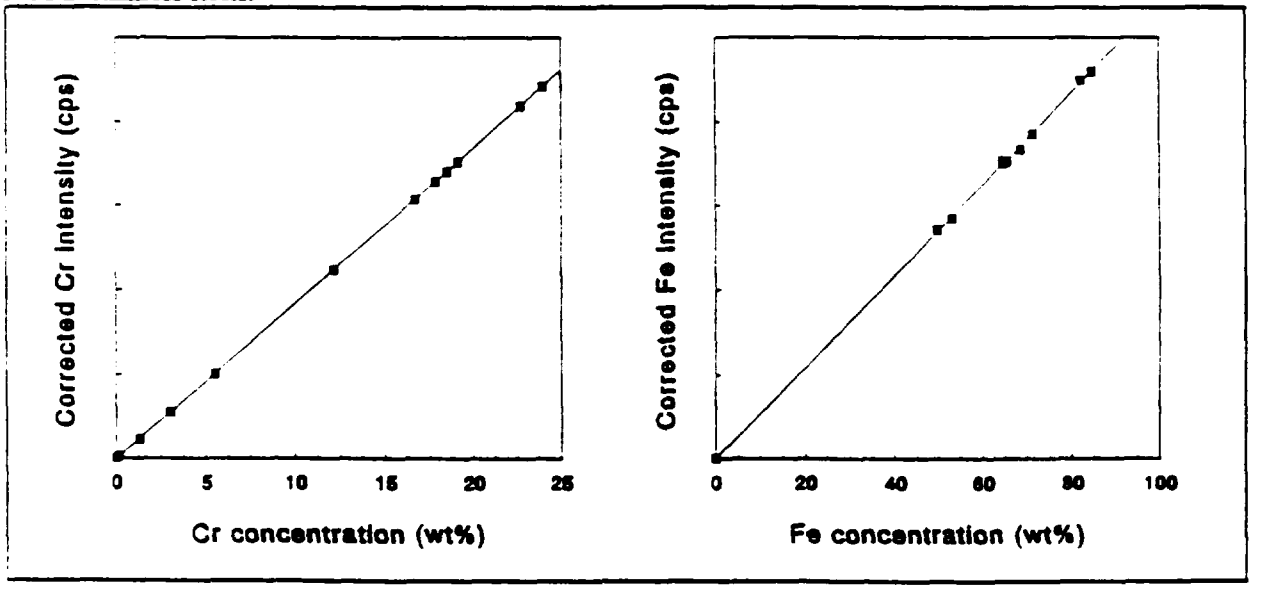

Figure 13. Calibration curves for chromium and iron in stainless steels. X-ray intensity data is corrected for matrix interactions.

The linearity of the plots in Figure 13 indicates that over the range of Cr and Fe concentrations in the standards, the empirical correction model accurately accounts for the effect of significant Cr and Fe matrix interactions.

Many situations arise where complex samples are addressed by EDXRF and a suite of type standards is unavailable to solve equation 5 for all significant matrix interactions. In these cases a fundamental parameter analysis method can be employed. The fundamental parameters (FP) method establishes EDXRF spectrometer response measured from a limited

standards set and uses first principles of X-ray absorption and emission to correct for matrix interactions. The FP method makes use of the X-ray spectrometer response from one or more comparison methods listed in the following table:

Comparison materials for FP standardization:

1. Multiple type standards
2. Single type standard
3. Dissimilar standard(s)
4. Pure element foils, salts or oxides
5. Standardless

Any combination of the five categories listed can be accommodated by FP software to ascertain the composition and/or thin film thickness of an unknown material.

Regardless of the number or type of standards used there are several advantages of the FP method compared to the empirical matrix correction algorithm discussed previously. By making use of the standard materials listed in the table above, all interactions arising from matrix components can be addressed, even if a component is not measurable by EDXRF. In contrast to empirical correction methods, the composition of the standard materials need not bracket the entire concentration ranges expected for the analyte or the matrix components. Accuracy of the FP method with a limited standards set can be comparable to or better than the accuracy achieved by empirical methods. The FP technique also permits the analyst greater flexibility regarding the types of unknowns that can be addressed for quantitative determination of composition.

IV. CONCLUSION

The principles of EDXRF measurements are straightforward and, when combined with computer automation, provide a powerful analytical technique for determination of elemental composition in solid, powder, thin film and liquid samples. Energy dispersive X-ray fluorescence spectrometry has found wide application for quality control and quality assurance of both intermediate and finished products. Many process streams and environmental samples are ideally addressed by non-destructive EDXRF methods that are versatile regarding the sample types that can be analyzed, often with little or no sample preparation before analysis.

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On-site x-ray fluorescence spectrometry mapping of metal contaminants in soils at Superfund sites

By W. Watson, J.P. Walsh, and B. Glynn

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On-site x-ray fluorescence spectrometry mapping of metal contaminants in soils at Superfund sites

ANALYSIS AND remediation of contaminated solid waste sites requires determination of the nature, extent, and magnitude of priority pollutants in the soil. This information must be made immediately available to on-site personnel for rapid and effective cleanup action. Energy dispersive x-ray fluorescence analysis (EDXRF) is a versatile and reliable method of identifying and quantifying those elements with atomic numbers greater than 11.[1] This article describes an EDXRF method developed for soil screening that requires minimal sample preparation and is readily implemented in the field, using the Spectrace 6000 (Tracor Xray, Mountain View, California).

Background

Metal contamination of soils is an important environmental concern. Lead, for example, was identified as a major contaminant on about 30% of 546 Superfund sites evaluated. Arsenic, cadmium, chromium, and zinc were each of concern on about 15% of the sites. Western metal mining and smelter sites, particularly where soil metal contamination is the major concern, often cover square miles; East Helena, Montana; Leadville, Colorado; Telluride, Colorado; and Kellog, Idaho, are all within Superfund sites.

Mr. Watson and Ms. Glynn are with Tracor Xray, Mountain View, California. Mr. Walsh is President, James P. Walsh and Associates, Boulder, Colorado.

The reauthorization of Superfund mandated that cleanup activities be completed on no fewer than 175 facilities by 1989 and 200 more by 1991.[2]

At a typical contamination site, the investigative team needs to collect, analyze, and map soil samples for the EPA's (Environmental Protection Agency) hazardous substance list shown in *Table 1*. To date, the Contract Laboratory Program (CLP) has been the primary means for analyzing Superfund samples. However, the delay of several weeks between submission of sample and receipt of results frequently requires remobilizing crews to the site for additional samples and mapping. The cost of CLP analysis ($200 or more per sample) and the necessity to remobilize based on interpretation of the initial results, makes mapping of a site a formidable expense.

In order to streamline mapping and cleanup operations, the EPA has instituted a program (FASP—Field Analytical Screening Program) to facilitate on-site screening.[3] The availability of a field mobile, analytical quality EDXRF system makes it possible for a crew to collect, analyze, and map data for samples while on location. Statistical analysis can also be applied to determine what, or where, additional samples should be collected and analyzed in order to define the extent of contamination. This program minimizes the number of samples sent to CLPs and eliminates the need for remobilizing crews.

EDXRF is accepted by the EPA as a level II analytical method according to the guidelines for Data Quality Objectives for Remedial Response Activities.[4] EDXRF is a good method for soil screening because it: 1) Requires minimal sample preparation; 2) provides rapid turnaround times; 3) is capable of analyzing a wide range of elements; 4) has a dynamic range that corresponds to typical soil contamination (ppm to 100%); and 5) furnishes an accurate quantitative analysis with results comparable to those obtained using CLP methodology.

Types of EDXRF systems

Two general types of EDXRF systems have been used for soil analysis: hand-held portable units that typically use radioisotopic source excitation and proportional counter detectors, and laboratory systems using x-ray tube excitation and high resolution lithium drifted silicon [Si(Li)] detectors.

Proportional counter detectors are rugged and lightweight, important

Table 1
EDXRF detectable EPA pollutant metals on the hazardous substance list

Aluminum	Copper	Potassium
Antimony	Iron	Selenium
Arsenic	Lead	Silver
Barium	Magnesium	Sodium
Cadmium	Manganese	Thallium
Calcium	Mercury	Vanadium
Chromium	Nickel	Zinc
Cobalt		

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features for components of a portable analyzer. However, the resolution of a proportional counter is inadequate for separation of the primary characteristic lines of elements close in atomic number.

Radioisotopic sources have the advantage of being light and inexpensive, making them useful for a portable analyzer; however, they lack the flexibility and intensity of an x-ray tube, resulting in higher limits of detection. Also, analysis of a wide range of elements requires the use of two or three different isotope/proportional counter combinations, increasing the cost and complexity of this type of unit.

By comparison, the Si(Li) detector resolves primary characteristic lines, thereby allowing qualitative identification of the elements present in a sample by simple inspection of the spectrum (see *Figure 1*), as well as improving the accuracy of quantitative analysis. These differences mean that x-ray tube/Si(Li) detector type EDXRF systems provide significantly better precision, accuracy, and sensitivity.

A typical laboratory EDXRF unit utilizes a liquid nitrogen (LN) cooled solid-state Si(Li) detector; thus the unit tends to be bulky and not easily mobilized. However, a recently developed Peltier thermoelectrically cooled Si(Li) detector, which does not require LN, establishes a new category of instruments called field mobile analytical quality EDXRF. This detector has been installed and used for various applications over the past two years,[5] and has shown a high degree of reliability, stability, and durability. Use of a 12.5 μm beryllium window enables detection of emission lines from elements as low in atomic number as sodium. The typical resolution of the detector is 185 eV compared with 155 eV for LN cooled Si(Li) detectors. This 19% degradation has little effect on the applicability of the system to analysis of the priority pollutants listed in Table 1.

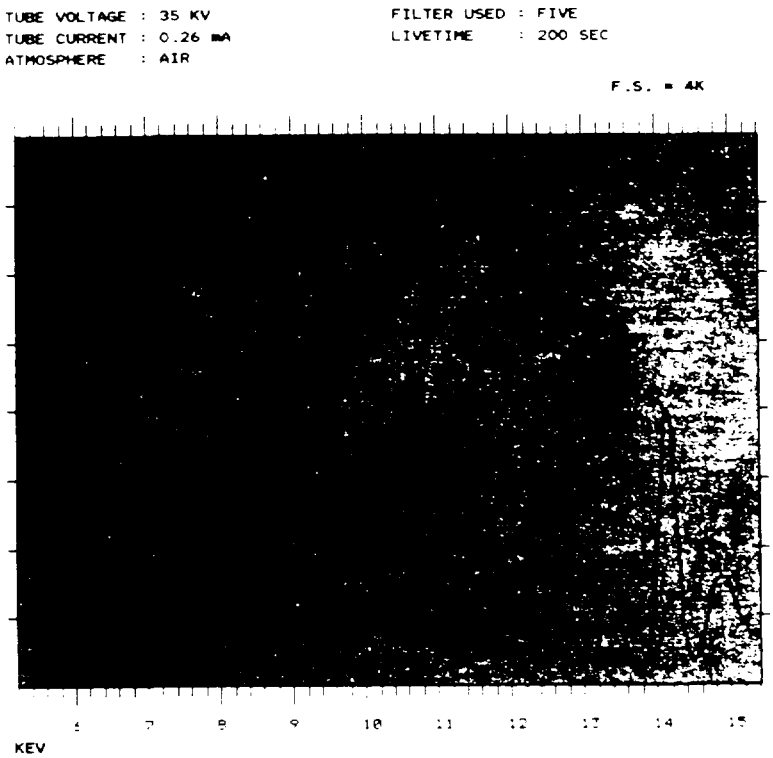

Figure 1 *Spectrum of contaminated soil showing the clear separation of the primary emission lines of the elements of interest.*

Instrumentation

The Spectrace 6000 is an analytical quality EDXRF system which uses the high resolution Peltier-cooled detector. It is composed of three basic modules: the spectrometer, containing the x-ray tube, detector, and sample chamber; a card cage housing the electronics; and an IBM PC, PS/2, or compatible computer. This computer can also be used to run geostatistical analysis software. Using an automated 50 kV x-ray source with a selection of automated primary radiation filters, this EDXRF system provides the low ppm detection limits required for on-site field screening. Lower limits of detection (LLDs) obtained for analysis of pollutant metals in soil are listed in *Table 2*.

The Spectrace TX-6000 is easily installed in an on-site laboratory van (see *Figure 2*) for movement to, or within, a contamination site and requires 700 W of 110 V ac power for operation. It is rugged enough so that vibration from transportation does not affect calibration.

Sample collection and preparation

Sample preparation for the EDXRF analysis requires considerably less time than a CLP digestion. Although sample preparation for EDXRF can be as simple as collecting soil in a sample cup, good accuracy and precision do require that the sample be dried, sieved, and pulverized before analysis.

Soil samples are collected by hand with trowels and/or augers or

Table 2

EDXRF detection limits (LLDs) for pollutant metals on the hazardous substance list

Antimony	5 ppm	Copper	16 ppm	Nickel	14 ppm
Arsenic	8 ppm	Iron	19 ppm	Potassium	0.03%
Cadmium	4 ppm	Lead	6 ppm	Silver	8 ppm
Calcium	0.02%	Manganese	21 ppm	Zinc	11 ppm
Chromium	16 ppm				

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with drill rigs for subsurface sampling. The larger the sample the better; 1 kg samples can be mixed and subsampled, but a minimum of 100-200 g of sample should be delivered to the laboratory. Recommended procedures for sample collection are described by the EPA.[6]

Samples must be dried, either in a conventional or microwave oven. Foreign objects such as twigs, leaves, grass, and pebbles are removed, agglomerates and lumps are broken up but not ground, and the material is sieved with a 10 mesh (2 mm) sieve and the coarse factor discarded. The sample is mixed and split, and about 10 g are ground with a mortar and pestle or a grinder. If the soils are ground mechanically, all samples should be ground under the same conditions. Our research recommends grinding to 100 mesh. The resulting powder is placed in a sample cup fit with a mylar window, and the soil is ready for analysis in the EDXRF spectrometer. *Figure 3* shows the sequence of steps involved in this procedure. Since the sample should be brought back to an on-site laboratory for preparation, a hand-carried EDXRF instrument is not necessary.

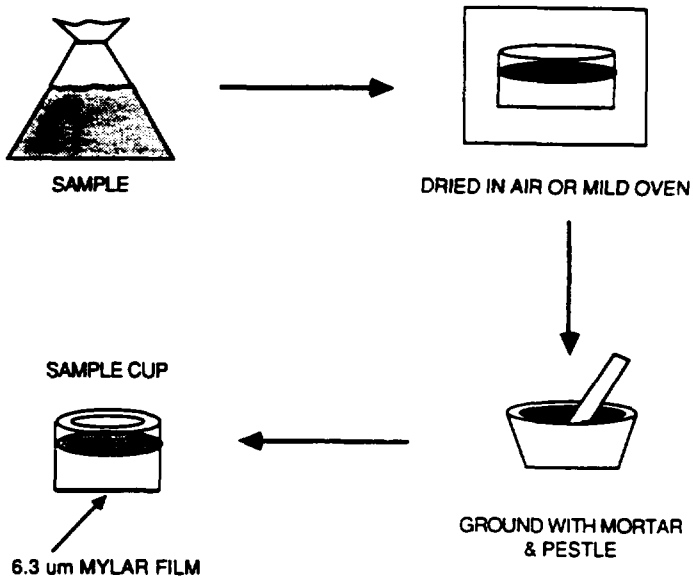

Figure 3 *Sample preparation for XRF soil screening.*

Data acquisition

In this application, 14 elements were analyzed. Three excitation conditions are necessary to optimize analysis of the elements of interest. The elements analyzed and conditions used are shown in *Table 3*. Sample throughput, using the sample preparation and analysis described here, was 25 per day. Figure 1 is the Mid Z spectrum of a contaminated waste sample. Clearly visible are peaks for Mn, Fe, Ni, Cu, Zn, and Pb. Also visible is the K-beta emission line for arsenic (an emission line not easily detected with a proportional counter when lead is present in the sample). After acquisition, the spectral data are automatically processed to correct for overlapping peaks and background.

Figure 2 *Installation of the Spectrace 6000 in an instrument van.*

Fundamental parameters analysis

Quantitative analysis was performed using a Fundamental Parameters (FP) program based on the method of Criss.[7] FP eliminates the need for a large number of standards (as many as 30 for some programs), or for site specific standards (sam-

Table 3
Excitation conditions and elements analyzed

Low Z	13 kV, 0.10 mA 0.13 mm Al filter 200 sec live time	K-Fe
Mid Z	30 kV, 0.17 mA 0.13 mm Rh filter 200 sec live time	Co-Se (K-lines) Hg-Pb (L-lines)
High Z	50 kV, 0.35 mA 0.63 mm Cu filter 200 sec live time	Ag-Ba

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ples previously collected and analyzed by CLP methods). The FP program automatically corrects for any matrix enhancement or absorption effects based on stored physical constants. *Table 4* shows the standards used for the analysis. The soils are of different matrices—two soil samples, a copper concentrate, nonferrous dust, lead concentrate, and two ores. The total time required for analysis of unknown samples is approximately 30 min, including sample preparation.

Quality control

The quality control procedure should follow QA/QC requirements for CLP. Following is a three-part quality control strategy:

1. Duplicate samples are run at 10-20% frequency. This measures both the precision of the analysis and the homogeneity of the sample. The control limit is 35% relative deviation. If the results for a particular element fall out of this limit, the data are flagged for that element.
2. One of the standard materials is analyzed at a frequency of 10-20% to verify the continued calibration of the instrument. If the standard concentration falls out of the 30% relative deviation limit, the procedure is restandardized. Typically, the x-ray system remains standardized over a period of months because it has no moving parts.
3. Sample splits are sent to laboratories for atomic absorption (AA) or inductively coupled plasma (ICP) comparison analysis at a 10-20% frequency.

Table 4

Standards used for EDXRF analysis with Fundamental Parameters analysis

Standard Reference Material	Source	Description
NBS 1648	NIST*	Urban particulate
SO-3	CANMET**	Soil sample
CCU-1A	CANMET	Copper concentrate
PD-1	CANMET	Nonferrous dust
CPB-1	CANMET	Lead concentrate
SO-1	CANMET	Soil sample
KC-1A	CANMET	Zn, Pb, Sb, Ag ore
UM-1	CANMET	Ni, Cu, Co ore

* NIST: National Institute for Standards and Technology (Gaithersburg, Maryland).

** CANMET: Canada Centre for Mineral and Energy Technology (Ottowa, Ontario, Canada).

Table 5

CLP vs EDXRF (ppm)*

Sample	CLP Element		EDXRF Run 1	Run 2	Run 3
A2-6bx	Cd	9.4	9	9	9
	As	23	31	37	48
	Pb	138	143	155	127
	Zn	167	128	131	125
B4-16cx	Cd	88	60	54	61
	As	27	28	28	31
	Pb	168	155	159	161
	Zn	625	548	477	563
A1-11cx	Cd	22	33	28	31
	As	71	88	78	79
	Pb	323	303	315	318
	Zn	186	140	123	139

* CLP: ICP for Cd, Pb, and Zn; GFAA for As.

Results

Three contaminated soil samples were split and submitted independently for both CLP and EDXRF analysis. The CLP laboratory, following EPA protocols SOW-785, used ICP for the determination of three of the four contaminants of interest: Cd, Pb, and Zn; graphite furnace atomic absorption (GFAA) was used to determine arsenic. *Table 5* lists the results of the CLP analysis for the metals, Cd, As, Pb, and Zn. Three replicates of each sample were analyzed by EDXRF, and those data are also presented in Table 5. In general, the CLP and EDXRF results agree within 30% relative. The EDXRF sample-to-sample precision is within 10% relative standard deviation (RSD) in all cases, except arsenic in sample A2-6bx.

Accuracy of the EDXRF and ICP methods is demonstrated by the analysis of standard reference materials; these results are presented in *Table 6*. Standard SO-1, used in the EDXRF method standardization, when resubmitted as an unknown for EDXRF analysis, shows good agreement with the known concentrations of Pb and Zn. The ICP analysis for lead in standard SO-1 is in error by almost 100% at the 21 ppm Pb level. The ICP result for Pb in standard SO-2, shown in Table 6, is within 10% of the known lead concentration; however, the Zn result is in error by 100%. Sample SO-2, not used in the EDXRF standardization, when submitted for EDXRF analysis, shows accuracy better than 20% relative at the 20 ppm Pb concentration and excellent accuracy for zinc at the 124 ppm Zn level.

Mapping and interpretation

The next step in the evaluation process is interpretation of results. It is important that the site personnel have a means of rapidly identifying the areas of contamination. A contour map such as the one shown in *Figure 4*, or an isometric plot, shown in *Figure 5*, allows visualization of priority pollutant locations with respect to the site layout.

The great advantage of a field mobile EDXRF analyzer is the capability to provide sampling with real time data on the nature and extent of contamination. The metal or metals of greatest concern can be identified using the multielement capability EDXRF. The depth and horizontal extent of contamination are determined from analyzed bore hole

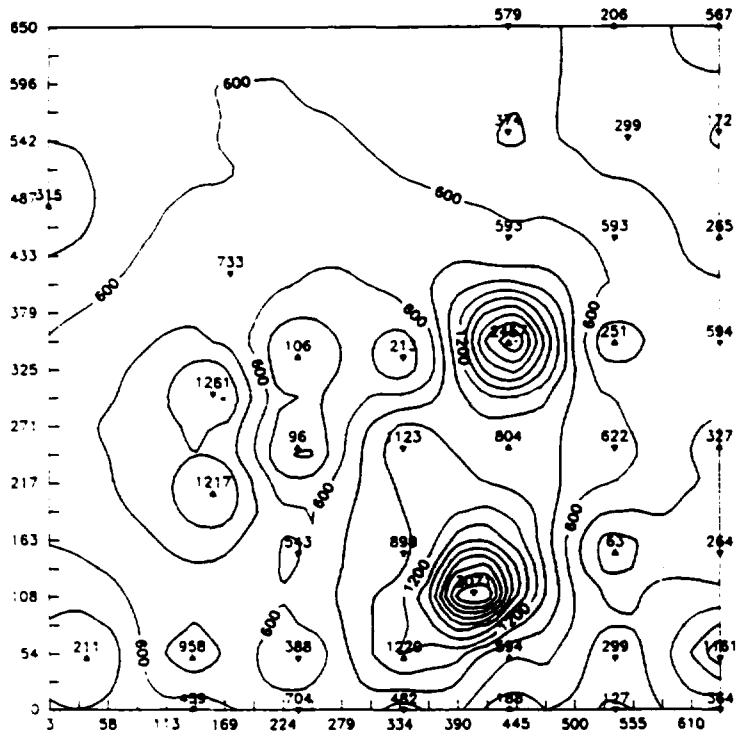

Figure 4 *Contour map showing lines of equal concentration of lead within the site.*

samples. If samples are delivered to a mobile laboratory and run daily, each day's sampling effort can focus on the areas near the "action level"; i.e., the areas that are neither the "hottest" nor cleanest, but those areas that are on the borderline between clean enough to leave in place and contaminated enough to require cleanup. With information in hand, immediate decisions can be made as to where additional samples should be collected, and which samples should be sent for CLP analysis.

The conventional approach is to collect a grid of samples, submit the samples to a fixed base laboratory, wait several weeks for results, and then return to the site for more sampling on another grid. With the mobile EDXRF approach, the number of uncontaminated samples submitted for AA-ICP analysis is reduced, and the most expensive part of the site investigation—sampling—can be optimized.

Table 6
Analysis of Standard Reference Materials (ppm)

	ICP	EDXRF	Known
Sample SO-1 (used in EDXRF standardization)			
Pb	41	14	21
Zn	129	147	146
Sample SO-2 (not used in EDXRF standardization)			
Pb	19	17	21
Zn	55	123	124

Conclusions

An instrument such as the Spectrace 6000, which combines the features of low LLDs, field mobility, and ease of use, represents a category of instruments suitable for field mobile analytical quality EDXRF and is ideal for on-site screening and mapping of contaminated soils.

References

1. LEYDEN, D.E., *Spectroscopy 2* (6), 28-36 (1987).
2. PORTER, J.W., Communication to regional EPA administrators, August 19, 1987.
3. FISK, J. et al., paper presented at *EPA Conference on Field Screening Methods for Hazardous Waste Site Investigations*, "Field monitoring methods in use for Superfund analyses" (Las Vegas, NV, October 1988).
4. USEPA, *Data Quality Objectives for Remedial Response Activities Development Process*, EPA/540/G-7/003 (1987).
5. HARDING, A.R., *Am. Lab. 19* (12), 24-31 (1987).
6. BARTH, D.S. and MASON, B., *Soil Sampling Quality Assurance Users Guide*, USEPA Environmental Monitoring Systems Lab, EPA-600/4-84-043 (1984).
7. CRISS, J.W., BIRKS, L.S. and GILFRICH, J.V., *Anal. Chem. 50*(1), 33-37 (1978).

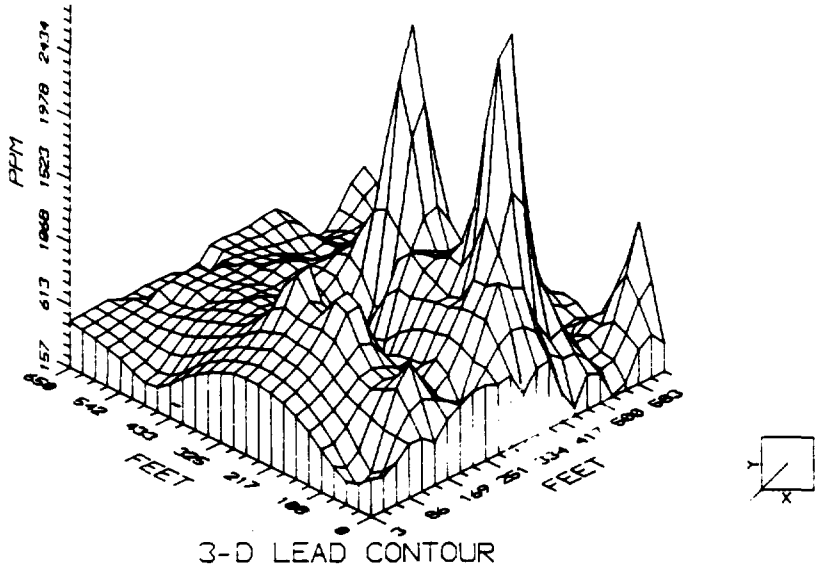

Figure 5 *Isometric plot of concentration vs position within an area of the site.*

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APPENDIX D

SUPPORTING INFORMATION

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(\PPG\0801461\R002.NJM)

CALCULATION OF USEPA SOIL SCREENING LEVEL FOR DDT AND ITS DEGRADATION PRODUCTS BASED ON THE PROTECTION OF GROUNDWATER

Objective

USEPA Draft Soil Screening Levels (SSLs) for the protection of groundwater (USEPA, 1993) were calculated for DDT, DDD, and DDE as discussed below. These values will be used to assist field personnel in the delineation of subsurface soils at the PPG facility in Moorestown, New Jersey. These SSLs will be used by field personnel to determine if a subsurface soil sample will be sent to a fixed laboratory for GC/MS analysis. If subsurface sample is determined to have concentrations of DDT, DDD, and DDE in excess of the SSLs, the soil sample will be sent to the laboratory for more detailed chemical analysis.

Method

The USEPA SSLs for the protection of groundwater were calculated using the algorithms presented in the *Draft Soil Screening Level Guidance* (USEPA, 1993). The SSLs were calculated using the Tier I default soil parameters and the USEPA Region III Risk-Based Concentrations for exposure to tap water (USEPA, 1994). The Region III Risk-Based Concentrations are based on exposure to chemicals in drinking water and are calculated in the same manner as the USEPA Maximum Contaminant Level Goals (MCLGs) for drinking water (USEPA, 1994). The equation used to derive the SSL for protection of groundwater is:

$$SSL = C_w * [(K_{oc} * f_{oc}) + (\theta * S/BD)] * DAF$$

where:

SSL = Soil Screening Level for the protection of groundwater (mg/kg);

C_w = Target concentration in groundwater (mg/L); Set equal to Region III Risk-Based Concentration for DDT, DDD, and DDE presented in Table 1 (USEPA, 1994);

K_{oc} = Organic carbon partitioning coefficient (ml/g); Set equal to published K_{oc} values for DDT, DDD, and DDE presented in Table 1 (USEPA, 1986);

f_{oc} = Fraction of organic carbon in soil (unitless); The default value of 0.01 was used;

θ = Soil porosity (L/L); The USEPA default value of 0.5 L/L was used;

S = Fraction water content (L/L); The USEPA default value of 0.3 L/L was used;

BD = Soil bulk density (kg/L); The USEPA default value of 1.5 kg/L was used;

DAF = Dilution/attenuation factor (unitless); The USEPA recommended value of 10 for sites greater than one acre was used.

Results

The calculated USEPA SSLs based on the protection of groundwater for DDT, DDD, and DDE are 5 mg/kg, 23 mg/kg, and 88 mg/kg, respectively. Since the field screening device only measures the total concentration of DDT and its degradation products and the possibility exists that all of the DDT related compounds in a sample could be DDT, the SSL for DDT of 5 mg/kg should be used by field personnel.

References

U.S. Environmental Protection Agency (USEPA). (1986). *Superfund Public Health Evaluation Manual*. EPA/540/1-86/060. October.

U.S. Environmental Protection Agency. (USEPA). 1993. Distribution of DRAFT Soil Screening Level Guidance. Memorandum from Henry L. Longest II dated September 30, 1993. Office of Emergency and Remedial Response, Washington, DC. PB93-963508.

U.S. Environmental Protection Agency (USEPA). (1994). *Risk-Based Concentration Table, Third Quarter 1994*. Memorandum from R. L. Smith, USEPA Region III Senior Toxicologist.

TABLE 1
CHEMICAL-SPECIFIC PARAMETER VALUES AND CALCULATED USEPA SSLs FOR DDT, DDD, AND DDE

Chemical	USEPA Risk-Based Concentration (mg/L)[a]	Organic Carbon Partitioning Coefficient, K_{oc} (L/kg)[b]	Calculated USEPA Soil Screening Level (mg/kg)[c]
DDT	0.0002	243,000	5.0
DDD	0.0002	770,000	23
DDE	0.0003	440,000	88

[a] From USEPA, 1994
[b] From USEPA, 1986
[c] SSL based on protection of groundwater; calculated using methods in USEPA, 1993

GWSSL.XLS

USEPA SOIL SCREENING LEVEL FOR PROTECTION OF GROUNDWATER

Date:	07/08/94		
Fraction of organic carbon:	foc =	0.01	
Total porosity:	theta =	0.5	L/L
Moisture content:	S =	0.3	L/L
Soil bulk density:	BD =	1.5	kg/L
Dilution Factor:	DAF =	10	
Chemical	MCLG	Koc	SSL
	mg/L	L/kg	mg/kg
DDD	0.0003	770000	23.1
DDE	0.0002	4400000	35.0
DDT	0.0002	243000	1.9

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Frequently the decision regarding the number of confirming laboratory analyses to make per group of sample field analyses is chosen empirically. It is determined more by tradition and intuition than solid statistical reasoning. The problem is similar to determining the calibration frequency of a laboratory instrument such as a gas chromatograph/mass spectrometer (GC/MS). Calibration monitoring is done by including a known standard every fixed number of samples. A procedure for determining the optimal calibration monitoring frequency has been provided by Yfantis, et al (1987). This procedure was presented at the Chemometrics Workshop sponsored by the U. S. Environmental Protection Agency (USEPA) and held in August 1986.

Yfantis, et al consider the situation in which a number of samples are to be analyzed using an analytical procedure such as GC/MS where periodic calibration checks are required. The probability of achieving an "accurate" analysis is assumed to be known. An accurate analysis is defined as one in which the measured value is within the desired tolerance of the true value. In addition, the probability of successfully calibrating the instrument is also assumed to be known as well as the cost of calibration and cost of an analysis. Given this information, Yfantis provides a procedure for determining the optimal frequency for the analysis of standard samples to monitor instrument calibration.

The use of field screening analytical techniques for remedial decision making is an exact parallel to the problem considered by Yfantis. A site specific "calibration" of the field analytical method is conducted by comparing field screening results with paired laboratory analyses for an initial set of "calibration" samples. A successful calibration is achieved if the field analytical technique "correctly" classifies the samples with respect to a specified action level. A correct classification is defined as being the same as the classification by laboratory analysis.

Periodically during the remedial investigation paired field and laboratory analyses will be conducted on the same sample to monitor the "calibration" of the field screening technique. If continued calibration is demonstrated all results obtained since the previous calibration check are accepted as valid. If there is a significant deviation from calibration, all the intervening results are suspect and, sampling and analysis may have to be repeated.

Based upon past experience with field screening analyses for total DDT, the probability of "correctly" classifying a sample is estimated to be 0.93 (93 percent). Considering the variation among duplicate laboratory analyses, this is approximately the same probability as can be achieved using only laboratory analyses. This reflects the inherent variation in total DDT concentration in the site soil. Thus, the probability of a successful "calibration" check is assumed to be 0.93. This probability will also be assumed as the minimum probability of a "successful" field analysis. The nominal cost of a laboratory analysis is taken to be $1,300. The cost of a field screening analysis is approximately $40. Applying the Yfantis statistical estimation technique the optimal number of field analysis samples per "calibration" sample can be estimated. These are presented in the following table.

302236

Probability of a successful field analysis	Number of samples per calibration check	Relative frequency of laboratory analysis (Percent)
0.93	4	25
0.95	5	20
0.99	11	9

Reference

Yfantis, E. A., G. T. Flatman and F. C. Garner, (1987) "Optimum Frequency of Calibration Monitoring" , Environmental Monitoring Systems Laboratory, Office of Research and Development, USEPA, Las Vegas, Published in Chemometrics and Intelligent Laboratory Systems.

302237

STATE OF NEW JERSEY

DEPARTMENT OF ENVIRONMENTAL PROTECTION AND ENERGY

Let's protect Earth

Certifies That

Wadsworth/Alert, Inc.
4101 Shuffel Drive NW
North Canton, OH. 44720

having duly met the requirements of the

Regulations Governing Laboratory Certification
And Standards Of Performance N.J.A.C. 7:18 et. seq.

is hereby approved as a

State Certified Environmental Laboratory

To perform the analyses as indicated on the Annual Certified Parameter List
which must accompany this certificate to be valid

#74008
PERMANENT CERTIFICATION NUMBER

August 14, 1992
DATE

Scott A. Weiner
COMMISSIONER, DEPARTMENT OF
ENVIRONMENTAL PROTECTION AND ENERGY

302238

This certification is subject to unannounced laboratory inspections as specified by N.J.A.C. 7:18-2.11(d) and agreed to by the Laboratory Manager on filing the application

TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST.

STATE OF NEW JERSEY
DEPARTMENT OF ENVIRONMENTAL PROTECTION
OFFICE OF QUALITY ASSURANCE
ANNUAL CERTIFIED PARAMETER LIST FOR 1993-1994

ENSECO-WADSWORTH/ALERT LABS. (74008) IS CERTIFIED TO PERFORM THE ANALYSES BELOW UNTIL JUNE 30 1994.

WATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

00076 TURBIDITY

00080 COLOR

00095 SPECIFIC CONDUCTANCE

00299 DISS OXYGEN-ELECTRODE

00310 BOD(5/20 DAY)

00340 COD

00400 HYDROGEN ION-PH

00410 ALKALINITY

00436 ACIDITY

00500 TOT SOLIDS

00505 TOT VOLATILE SOLIDS

00530 SUSP SOLIDS

00545 SETT SOLIDS-VOLUMETRIC

00556 OIL AND GREASE

00605 ORGANIC NITROGEN

00610 AMMONIA NITROGEN

00615 NITRITE

302239

ATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

00625 TOT KJELDAHL NITROGEN

00630 NITRATE

00680 ORGANIC CARBON, TOTAL

00720 CYANIDE, TOTAL

00722 CYANIDE, AMEN TO CHLOR

00740 SULFITE

00745 SULFIDE

00900 HARDNESS

00940 CHLORIDE

00945 SULFATE

00951 FLUORIDE, TOTAL

00955 SILICA

01032 CR HEX

32730 PHENOLS

38260 SURFACTANTS

50060 CHLORINE RESIDUAL

70300 TOT DISS SOLIDS

METALS

00915 CALCIUM (ICAP)

00925 MAGNESIUM (ICAP)

00929 SODIUM (ICAP)

302240 LAB 74009
10/29/93

\TER POLLUTION LABORATORY CERTIFICATION

METALS

00935 POTASSIUM (ICAP)

01002 ARSENIC (AA)

01005 BARIUM (ICAP)

01010 BERYLLIUM (ICAP)

01020 BORON (ICAP)

01025 CADMIUM (ICAP)

01030 CHROMIUM (ICAP)

01035 COBALT (ICAP)

01040 COPPER (ICAP)

01045 IRON (ICAP)

01049 LEAD (ICAP)

01055 MANGANESE (ICAP)

01065 NICKEL (ICAP)

01085 VANADIUM (ICAP)

01090 ZINC (ICAP)

01105 ALUMINUM (ICAP)

01145 SELENIUM (ICAP)

01147 SELENIUM (AA)

71900 MERCURY (COLD VAPOR)

ORGANICS

624 PURGEABLES (GC/MS)

302241

ATER POLLUTION LABORATORY CERTIFICATION

ORGANICS

625 B/N, ACIDS & PEST (GC/MS)

99007 PESTICIDES
39330 ALDRIN
39380 DIELDRIN
39360 DDD
39365 DDE
39370DDT
39410 HEPTACHLOR
39350 CHLORDANE

THIS LIST MUST BE CONSPICUOUSLY DISPLAYED WITH THE PERMANENT CERTIFICATE AT THE LABORATORY

LAB 74008
10/29/93

302242

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: OH048

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
ALUMINUM	1	671	681	550- 784	579- 755	ACCEPTABLE
	2	< 200	140	107- 182	117- 173	UNUSABLE DATA
ARSENIC	1	427	492	408- 587	430- 565	CHECK FOR ERRO
	2	77.1	74.3	56.7- 92.9	61.2- 88.4	ACCEPTABLE
BERYLLIUM	1	423	461	382- 533	401- 514	ACCEPTABLE
	2	222	240	198- 279	208- 268	ACCEPTABLE
CADMIUM	1	172	165	138- 194	145- 187	ACCEPTABLE
	2	63.9	61.0	50.8- 72.0	53.5- 69.4	ACCEPTABLE
COBALT	1	905	880	775- 980	801- 954	ACCEPTABLE
	2	56.2	53.8	45.4- 61.9	47.5- 59.8	ACCEPTABLE
[illegible]MIUM	1	746	730	604- 843	634- 813	ACCEPTABLE
	2	23.8	23.4	17.4- 29.0	18.9- 27.6	ACCEPTABLE
COPPER	1	622	601	524- 657	541- 640	ACCEPTABLE
	2	18.7	18.7	13.6- 24.1	14.9- 22.8	ACCEPTABLE
IRON	1	50.0	58.0	43.5- 72.5	47.2- 68.8	ACCEPTABLE
	2	1140	1100	950- 1270	991- 1230	ACCEPTABLE
MERCURY	1	8.69	9.38	7.31- 11.9	7.89- 11.3	ACCEPTABLE
	2	6.21	6.67	5.00- 8.48	5.44- 8.04	ACCEPTABLE
MANGANESE	1	621	600	536- 660	551- 644	ACCEPTABLE
	2	76.0	73.5	64.0- 82.7	66.4- 80.4	ACCEPTABLE
NICKEL	1	873	860	766- 952	789- 928	ACCEPTABLE
	2	341	340	296- 383	307- 372	ACCEPTABLE
LEAD	1	1260	1200	1060- 1350	1100- 1310	ACCEPTABLE
	2	776	738	636- 833	661- 809	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302243

PERFORMANCE EVALUATION REPORT

DATE: 12/27/9

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: OH048

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
SELENIUM	1	190	229	156- 277	171- 262	ACCEPTABL
	2	341	387	268- 486	296- 459	ACCEPTABLE
VANADIUM	1	954	940	832- 1040	860- 1020	ACCEPTABLE
	2	175	170	146- 198	151- 183	ACCEPTABLE
ZINC	1	854	842	737- 947	763- 921	ACCEPTABL
	2	50.0	46.3	37.5- 56.1	39.3- 53.7	ACCEPTABLE
ANTIMONY	3	49.9	94.5	56.4- 119	64.3- 111	NOT ACCEPTABLE
	4	101	189	108- 244	125- 227	NOT ACCEPTABLE
SILVER	3	81.1	73.9	60.6- 86.8	63.9- 83.5	ACCEPTABLE
	4	23.5	25.8	21.1- 30.4	22.2- 29.2	ACCEPTABLE
THALLIUM	3	62.4	62.8	47.0- 77.3	50.3- 73.4	ACCEPTABLE
	4	577	539	421- 643	450- 615	ACCEPTABLE
MOLYBDENUM	3	< 500	24.5	18.6- 30.6	20.2- 29.0	UNUSABLE DATA
	4	< 500	81.5	64.4- 95.0	68.3- 91.1	UNUSABLE DATA
STRONTIUM	3	19.8	19.1	14.4- 23.3	15.5- 22.1	ACCEPTABLE
	4	73.8	73.4	62.0- 84.5	64.9- 81.5	ACCEPTABLE
TITANIUM	3	122	130	109- 151	114- 145	ACCEPTABLE
	4	42.7	43.0	34.0- 53.4	36.5- 50.9	ACCEPTABLE
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
PH-UNITS	3	9.64	9.50	9.19- 9.75	9.26- 9.68	ACCEPTABLE
	4	9.61	4.70	4.62- 4.77	4.64- 4.75	NOT ACCEPTABLE
SPEC. COND. (UMHOS/CM AT 25 C)	1	936	899	840- 980	857- 963	ACCEPTABLE
	2	4.29	398	368- 435	377- 427	NOT ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302244

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: OH048

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
TDS AT 180 C	1	589	577	396- 759	441- 713	ACCEPTABLE
	2	229	223	168- 284	183- 269	ACCEPTABLE
TOTAL HARDNESS (AS CACO3)	1	285	279	236- 307	245- 298	ACCEPTABLE
	2	53.2	57.4	51.2- 63.8	52.8- 62.2	ACCEPTABLE
CALCIUM	1	108	110	87.4- 126	92.2- 121	ACCEPTABLE
	2	7.81	7.00	5.95- 8.19	6.23- 7.91	ACCEPTABLE
MAGNESIUM	1	< 5.00	0.960	0.771- 1.18	0.822- 1.13	UNUSABLE DATA
	2	10.3	9.70	8.38- 11.0	8.71- 10.6	ACCEPTABLE
SODIUM	1	63.7	61.7	56.2- 67.8	57.6- 66.3	ACCEPTABLE
	2	27.5	26.3	23.6- 29.2	24.3- 28.5	ACCEPTABLE
POTASSIUM	1	7.95	7.50	6.39- 8.81	6.70- 8.50	ACCEPTABLE
	2	42.4	40.0	34.5- 45.6	35.9- 44.2	ACCEPTABLE
TOTAL ALKALINITY (AS CACO3)	1	124	120	106- 133	109- 130	ACCEPTABLE
	2	< 20.0	12.0	7.85- 15.3	8.77- 14.3	UNUSABLE DATA
CHLORIDE	1	188	199	183- 213	187- 210	ACCEPTABLE
	2	39.8	41.2	36.5- 45.4	37.6- 44.3	ACCEPTABLE
FLUORIDE	1	4.03	3.30	2.83- 3.77	2.95- 3.65	NOT ACCEPTABLE
	2	0.453	0.380	0.319-0.457	0.336-0.439	CHECK FOR ERRO
SULFATE	1	11.8	14.0	10.8- 16.8	11.5- 16.0	ACCEPTABLE
	2	76.7	92.0	78.1- 105	81.5- 102	NOT ACCEPTABLE
NUTRIENTS IN MILLIGRAMS PER LITER:						
AMMONIA-NITROGEN	1	7.50	7.70	6.10- 9.16	6.47- 8.79	ACCEPTABLE
	2	0.666	0.730	0.491-0.982	0.550-0.923	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302245

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: OH048

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
NUTRIENTS IN MILLIGRAMS PER LITER:						
NITRATE-NITROGEN	1	.546	0.520	0.378-0.658	0.412-0.625	ACCEPTABLE
	2	11.4	11.0	8.84- 13.0	9.34- 12.5	ACCEPTABLE
ORTHOPHOSPHATE	1	0.169	0.150	0.114-0.187	0.122-0.178	ACCEPTABLE
	2	4.30	4.10	3.48- 4.68	3.62- 4.54	ACCEPTABLE
KJELDAHL-NITROGEN	3	10.1	14.0	10.3- 17.1	11.1- 16.3	NOT ACCEPTABLE
	4	0.840	0.710	0.226- 1.32	0.357- 1.19	ACCEPTABLE
TOTAL PHOSPHORUS	3	6.51	7.40	5.57- 8.05	5.86- 7.75	ACCEPTABLE
	4	0.468	0.490	0.341-0.569	0.369-0.542	ACCEPTABLE
DEMANDS IN MILLIGRAMS PER LITER:						
COD	1	66.4	70.8	52.4- 84.6	56.5- 80.5	ACCEPTABLE
	2	200	207	163- 230	172- 221	ACCEPTABLE
	1	32.8	28.0	23.9- 32.8	25.0- 31.6	CHECK FOR ERROR
	2	92.9	82.0	69.5- 95.3	72.8- 91.9	CHECK FOR ERROR
5-DAY BOD	1	27.1	44.9	26.1- 60.1	30.3- 55.8	CHECK FOR ERROR
	2	92.4	131	75.4- 180	88.5- 167	ACCEPTABLE
CARBONACEOUS BOD	1	22.5	38.5	16.5- 60.4	22.5- 54.4	ACCEPTABLE
	2	58.5	112	52.8- 171	69.0- 155	CHECK FOR ERROR
PCB'S IN MICROGRAMS PER LITER:						
PCB-AROCLOR 1254	1	2.42	1.87	0.938- 2.71	1.21- 2.49	ACCEPTABLE
PCB-AROCLOR 1260	2	4.57	4.63	2.79- 5.96	3.19- 5.56	ACCEPTABLE

*BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302246

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

ABORATORY: OH048

NALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
PCB'S IN OIL IN MILLIGRAMS PER KILOGRAM:						
CB IN OIL- 1016/1242	2	22.5	35.3	8.02- 46.6	13.0- 41.6	ACCEPTABLE
CB IN OIL- 1254	1	36.0	43.9	13.5- 61.8	19.8- 55.5	ACCEPTABLE
PESTICIDES IN MICROGRAMS PER LITER:						
HLORDANE	3	8.46	8.21	4.91- 9.72	5.52- 9.11	ACCEPTABLE
	4	1.60	2.21	1.07- 2.77	1.29- 2.56	ACCEPTABLE
LDRIN	1	0.559	0.539	0.122-0.754	0.202-0.674	ACCEPTABLE
	2	0.111	0.086	.0171-0.121	.0303-0.108	CHECK FOR ERROR
IELDRIN	1	0.506	0.475	0.203-0.710	0.267-0.645	ACCEPTABLE
	2	0.203	0.173	.0861-0.239	0.106-0.219	ACCEPTABLE
DD	1	1.11	0.866	0.433- 1.15	0.524- 1.06	CHECK FOR ERROR
	2	0.318	0.202	.0956-0.268	0.117-0.246	NOT ACCEPTABLE
DE	1	0.636	0.539	0.235-0.756	0.301-0.690	ACCEPTABLE
	2	0.195	0.173	.0788-0.236	.0989-0.216	ACCEPTABLE
DT	1	0.795	0.796	0.362- 1.06	0.450-0.972	ACCEPTABLE
	2	0.184	0.142	.0570-0.216	.0772-0.196	ACCEPTABLE
EPTACHLOR	1	0.781	0.669	0.187-0.918	0.279-0.825	ACCEPTABLE
	2	0.317	0.216	.0756-0.269	0.100-0.245	NOT ACCEPTABLE
EPTACHLOR EPOXIDE	1	0.523	0.478	0.260-0.640	0.308-0.592	ACCEPTABLE
	2	0.218	0.174	.0858-0.235	0.105-0.216	CHECK FOR ERROR

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302247

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: OH048

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE HALOCARBONS IN MICROGRAMS PER LITER:						
1,2 DICHLOROETHANE	1	13.6	15.1	10.2- 20.4	11.5- 19.1	ACCEPTABLE
	2	51.6	55.8	36.2- 73.4	40.9- 68.7	ACCEPTABLE
CHLOROFORM	1	17.1	11.8	7.85- 15.9	8.86- 14.8	NOT ACCEPTABLE
	2	106	64.4	38.3- 86.7	44.4- 80.6	NOT ACCEPTABLE
1,1,1 TRICHLOROETHANE	1	11.6	13.4	8.68- 18.2	9.89- 17.0	ACCEPTABLE
	2	54.0	37.8	22.7- 50.0	26.2- 46.6	NOT ACCEPTABLE
TRICHLOROETHENE	1	5.89	7.57	5.10- 10.3	5.75- 9.62	ACCEPTABLE
	2	99.5	62.7	38.6- 80.5	43.9- 75.2	NOT ACCEPTABLE
CARBONTETRACHLORIDE	1	13.1	16.4	10.9- 22.2	12.3- 20.8	ACCEPTABLE
	2	45.8	37.1	23.0- 50.1	26.4- 46.6	ACCEPTABLE
TETRACHLOROETHENE	1	6.50	9.24	6.08- 12.5	6.89- 11.7	CHECK FOR ERROR
	2	65.5	51.3	31.2- 66.7	35.7- 62.2	CHECK FOR ERROR
BROMODICHLOROMETHANE	1	12.8	10.8	7.33- 14.2	8.21- 13.3	ACCEPTABLE
	2	96.5	36.1	24.9- 53.6	28.5- 50.0	NOT ACCEPTABLE
DIBROMOCHLOROMETHANE	1	14.9	13.1	8.48- 17.0	9.55- 15.9	ACCEPTABLE
	2	69.3	58.1	34.1- 80.2	39.9- 74.4	ACCEPTABLE
BROMOFORM	1	16.9	14.5	7.45- 20.0	9.04- 18.4	ACCEPTABLE
	2	43.0	42.3	25.5- 59.9	29.9- 55.6	ACCEPTABLE
METHYLENE CHLORIDE	1	9.62	10.6	6.59- 16.1	7.79- 14.9	ACCEPTABLE
	2	93.5	54.1	30.7- 76.5	36.5- 70.7	NOT ACCEPTABLE
CHLOROBENZENE	1	16.3	16.0	11.4- 20.1	12.5- 19.0	ACCEPTABLE
	2	67.5	63.7	41.3- 79.8	46.2- 74.9	ACCEPTABLE
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
BENZENE	1	46.0	40.1	25.7- 56.0	29.5- 52.2	ACCEPTABLE
	2	10.1	8.25	5.24- 11.6	6.04- 10.8	ACCEPTABLE

BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302248

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

ABORATORY: OH048

NALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
THYLBENZENE	1	74.7	66.9	36.0- 94.3	43.4- 86.9	ACCEPTABLE
	2	17.4	14.0	9.26- 19.0	10.5- 17.8	ACCEPTABLE
OLUENE	1	53.4	49.2	30.2- 65.2	34.6- 60.8	ACCEPTABLE
	2	11.4	9.51	6.09- 12.8	6.93- 11.9	ACCEPTABLE
,2-DICHLOROBENZENE	1	59.8	65.5	32.7- 93.9	40.5- 86.2	ACCEPTABLE
	2	8.75	8.88	5.85- 11.7	6.59- 11.0	ACCEPTABLE
,3-DICHLOROBENZENE	1	48.3	47.9	29.4- 61.7	33.5- 57.6	ACCEPTABLE
	2	16.7	16.6	11.6- 21.1	12.9- 19.9	ACCEPTABLE
,4-DICHLOROBENZENE	1	59.4	62.5	34.4- 85.8	41.0- 79.3	ACCEPTABLE
	2	13.2	12.4	7.49- 17.8	8.79- 16.5	ACCEPTABLE
MISCELLANEOUS PARAMETERS:						
OTAL CYANIDE	1	.619	0.860	0.598- 1.15	0.668- 1.08	CHECK FOR ERROR
IN MG/L)	2	.131	0.160	0.102-0.208	0.116-0.194	ACCEPTABLE
ON-FILTERABLE RESIDUE	1	52.0	61.0	47.7- 64.6	49.8- 62.5	ACCEPTABLE
IN MG/L)	2	76.7	83.0	62.3- 91.3	66.4- 87.8	ACCEPTABLE
IL AND GREASE	1	4.55	8.30	3.07- 12.2	4.22- 11.0	ACCEPTABLE
IN MG/L)	2	41.6	48.4	33.7- 56.0	36.5- 53.2	ACCEPTABLE
OTAL PHENOLICS	1	0.662	0.595	0.312-0.878	0.384-0.805	ACCEPTABLE
IN MG/L)	2	3.41	3.13	1.66- 4.60	2.04- 4.23	ACCEPTABLE
OTAL RESIDUAL CHLORINE	1	3.60	3.70	3.06- 4.52	3.25- 4.32	ACCEPTABLE
IN MG/L)	2	1.40	1.50	1.09- 1.77	1.18- 1.68	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302249

Quanterra

Environmental Services

1 of 2

(formerly IT Analytical Services)
5103 Old William Penn Highway
Export, PA 15632
412/327-7231 fax
412/731-8806

To: Neale Misguitta / Brian Smith

Location: North Canton

Telephone: 412-934-5944 / 216-497-9396

From: Maureen DeRubeis Date: 8-2-94

No. of Pages (including cover sheet): 15

Remarks: New Jersey Certificate
WP31, WS033 Results

ITAS PT/07-94/0126/FaxCov.Sht

302250

State of New Jersey

DEPARTMENT OF ENVIRONMENTAL PROTECTION AND ENERGY

CHRISTINE TODD WHITMAN
Governor

ROBERT C. SHINN, JR.
Commissioner

July 13, 1994

International Technology Corp.
5103 Old William Penn Highway
Export, PA 15632

Manager: Tom Daniels

Lab ID# 77564

Dear Mr. Daniels:

Enclosed is your 1994-95 Annual Certified Parameter List. This list replaces the 1993-94 form and must be conspicuously displayed at the laboratory, along with your permanent Laboratory Certificate.

Your cooperation in this matter is appreciated.

Sincerely,

Dottie Correnti 4y

Dottie Correnti
Administrative Analyst I
Bureau of Revenue

DCP:ch-208
Enclosure
cc: Jerry Bundy

302251

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STATE OF NEW JERSEY
DEPARTMENT OF ENVIRONMENTAL PROTECTION
OFFICE OF QUALITY ASSURANCE
ANNUAL CERTIFIED PARAMETER LIST FOR 1994-1995

INTERNATIONAL TECHNOLOGY CORP. (77564) IS CERTIFIED TO PERFORM THE ANALYSES BELOW UNTIL JUNE 30 1995.

DRINKING WATER LABORATORY CERTIFICATION

LIMITED CHEMISTRY

002 ALKALINITY, ELECT TITRIM

934 NITRATE, AUTO CD REDUC

935 FLUORIDE-ELECTRODE

944 TURBIDITY

947 CHLORIDE, HG OR AG NITRAT

948 COLOR, PLATINUM COBALT

951 PH, GLASS ELECTRODE

952 TOT DISS SOLIDS, TOT RES

953 HARDNESS, EDTA

956 SULFATE, GRAVIM OR TURBID

METALS

912 HG,MANUAL COLD VAPOR

914 AS, GRAPHITE FURNACE

916 CD, GRAPHITE FURNACE

917 CR, GRAPHITE FURNACE

918 PB, GRAPHITE FURNACE

LAB 77564
07/05/94

302252

DRINKING WATER LABORATORY CERTIFICATION

METALS

920 SE, GRAPHITE FURNACE

921 AG, GRAPHITE FURNACE

954 NA, ATOMIC ABSORPTION

961 BARIUM, ICAP

962 CADMIUM, ICAP

963 CHROMIUM, ICAP

964 LEAD, ICAP

965 SILVER, ICAP

966 COPPER, ICAP

967 IRON, ICAP

968 MANGANESE, ICAP

969 ZINC, ICAP

ORGANICS

941 ORGANOCHLORINE PESTICIDES
ENDRIN
LINDANE
METHOXYCHLOR
TOXAPHENE

524.2 VOC (PT/GC-MS)

WATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

00010 TEMPERATURE

00076 TURBIDITY

WATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

00095 SPECIFIC CONDUCTANCE

00310 BOD(5/20 DAY)

00340 COD

00400 HYDROGEN ION-PH

00410 ALKALINITY

00436 ACIDITY

00500 TOT SOLIDS

00530 SUSP SOLIDS

00556 OIL AND GREASE

00610 AMMONIA NITROGEN

00615 NITRITE

00625 TOT KJELDAHL NITROGEN

00630 NITRATE

00665 PHOSPHORUS, TOT AS P

00671 ORTHOPHOSPHATE AS P

00680 ORGANIC CARBON, TOTAL

00681 ORGANIC CARBON, DISSOLVED

00720 CYANIDE, TOTAL

00745 SULFIDE

00940 CHLORIDE

00945 SULFATE

00951 FLUORIDE, TOTAL

LAB 77564
07/05/94

302254

WATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

01032 CR HEX

32730 PHENOLS

38260 SURFACTANTS

70300 TOT DISS SOLIDS

METALS

00915 CALCIUM (ICAP)

00925 MAGNESIUM (ICAP)

00929 SODIUM (ICAP)

00935 POTASSIUM (ICAP)

01002 ARSENIC (AA)

01005 BARIUM (ICAP)

01010 BERYLLIUM (ICAP)

01020 BORON (ICAP)

01025 CADMIUM (ICAP)

01027 CADMIUM (AA)

01030 CHROMIUM (ICAP)

01034 CHROMIUM (AA)

01035 COBALT (ICAP)

01040 COPPER (ICAP)

01045 IRON (ICAP)

01049 LEAD (ICAP)

WATER POLLUTION LABORATORY CERTIFICATION

METALS

01051 LEAD (AA)

01055 MANGANESE (ICAP)

01057 THALLIUM (ICAP)

01059 THALLIUM (AA)

01060 MOLYBDENUM (ICAP)

01065 NICKEL (ICAP)

01075 SILVER (ICAP)

01077 SILVER (AA)

01085 VANADIUM (ICAP)

01090 ZINC (ICAP)

01095 ANTIMONY (ICAP)

01097 ANTIMONY (AA)

01105 ALUMINUM (ICAP)

01145 SELENIUM (ICAP)

01147 SELENIUM (AA)

71900 MERCURY (COLD VAPOR)

ORGANICS

608 PESTICIDES & PCBS (GC)

624 PURGEABLES (GC/MS)

625 B/N, ACIDS & PEST (GC/MS)

09032 PENTACHLOROPHENOL

WATER POLLUTION LABORATORY CERTIFICATION

ORGANICS

99007 PESTICIDES
39330 ALDRIN
39380 DIELDRIN
39360 DDD
39365 DDE
39370DDT
39410 HEPTACHLOR
39350 CHLORDANE

THIS LIST MUST BE CONSPICUOUSLY DISPLAYED WITH THE PERMANENT CERTIFICATE AT THE LABORATORY

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PLUK2 77564

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
ALUMINUM	1	676	681	550- 784	579- 755	ACCEPTABLE
	2	128	140	107- 182	117- 173	ACCEPTABLE
ARSENIC	1	488	492	408- 587	430- 565	ACCEPTABLE
	2	73.6	70.3	56.7- 92.9	61.2- 88.4	ACCEPTABLE
BERYLLIUM	1	433	461	382- 533	401- 514	ACCEPTABLE
	2	221	240	188- 278	208- 268	ACCEPTABLE
CADMIUM	1	179	165	138- 194	145- 187	ACCEPTABLE
	2	67.6	61.0	50.8- 72.8	53.5- 69.4	ACCEPTABLE
COBALT	1	956	880	775- 980	801- 954	CHECK FOR ERROR
	2	57.8	53.8	45.4- 62.9	47.5- 59.8	ACCEPTABLE
CHROMIUM	1	762	730	604- 843	634- 813	ACCEPTABLE
	2	22.5	23.6	17.6- 29.0	18.9- 27.6	ACCEPTABLE
COPPER	1	626	601	524- 657	541- 640	ACCEPTABLE
	2	19.8	18.7	13.6- 20.1	14.9- 22.8	ACCEPTABLE
IRON	1	53.4	58.0	43.5- 72.5	47.2- 68.8	ACCEPTABLE
	2	1100	1100	950- 1270	991- 1230	ACCEPTABLE
MERCURY	1	7.52	9.38	7.31- 11.9	7.89- 11.3	CHECK FOR ERROR
	2	5.13	6.67	5.08- 8.88	5.44- 8.08	CHECK FOR ERROR
MANGANESE	1	636	600	536- 660	551- 648	ACCEPTABLE
	2	76.2	73.5	64.0- 82.7	66.4- 80.4	ACCEPTABLE
NICKEL	1	896	860	766- 952	789- 928	ACCEPTABLE
	2	362	340	296- 353	307- 372	ACCEPTABLE
LEAD	1	1240	1200	1060- 1350	1100- 1310	ACCEPTABLE
	2	734	738	636- 833	661- 809	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

Score = 97%

302258

PERFORMANCE EVALUATION REPORT DATE: 12/31/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PLUMZ

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
SELENIUM	1	215	228	156- 277	171- 262	ACCEPTABLE
	2	383	387	268- 486	296- 459	ACCEPTABLE
VANADIUM	1	938	940	832- 1040	860- 1020	ACCEPTABLE
	2	197	170	146- 185	151- 183	NOT ACCEPTABLE
ZINC	1	527	542	337- 947	763- 911	CHECK FOR ERROR
	2	48.3	46.3	37.5- 56.1	39.9- 53.7	ACCEPTABLE
ANTIMONY	3	68.5	94.5	56.4- 119	64.3- 111	ACCEPTABLE
	4	153	169	108- 244	125- 227	ACCEPTABLE
SILVER	3	71.7	73.9	60.6- 86.8	63.9- 93.5	ACCEPTABLE
	4	24.3	25.8	21.1- 30.4	22.2- 29.2	ACCEPTABLE
THALLIUM	3	60.1	62.8	47.0- 77.3	50.8- 73.4	ACCEPTABLE
	4	555	539	421- 643	450- 615	ACCEPTABLE
MOLYBDENUM	3	22.3	24.5	16.6- 30.6	20.2- 29.0	ACCEPTABLE
	4	66.6	81.6	68.4- 95.0	68.3- 91.1	CHECK FOR ERROR
STRONTIUM	3	19.6	19.1	14.4- 23.3	15.5- 22.1	ACCEPTABLE
	4	78.5	73.4	62.0- 84.5	64.9- 81.5	ACCEPTABLE
TITANIUM	3	127	130	109- 151	110- 145	ACCEPTABLE
	4	43.0	43.0	34.0- 53.4	36.5- 50.9	ACCEPTABLE
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
PH-UNITS	3	9.52	9.50	9.19- 9.75	9.25- 9.68	ACCEPTABLE
	4	4.67	4.70	4.62- 4.77	4.64- 4.75	ACCEPTABLE
SPEC. COND.	1	857	899	840- 980	857- 963	ACCEPTABLE
(UMHOS/CM AT 25 C)	2	394	398	368- 435	377- 427	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302259

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PL042

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
TDS AT 180 C	1	523	577	396- 759	481- 713	ACCEPTABLE
	2	223	233	168- 284	193- 269	ACCEPTABLE
TOTAL HARDNESS (AS CACO3)	1	260	279	236- 307	245- 298	ACCEPTABLE
	2	60	57.4	51.2- 63.8	52.8- 62.2	ACCEPTABLE
CALCIUM	1	108	110	87.4- 126	92.2- 121	ACCEPTABLE
	2	7.63	7.00	5.95- 8.13	6.23- 7.91	ACCEPTABLE
MAGNESIUM	1	1.03	0.960	0.771- 1.18	0.822- 1.13	ACCEPTABLE
	2	10.8	9.70	8.38- 11.0	8.71- 10.6	ACCEPTABLE
SODIUM	1	64.4	61.7	56.2- 67.8	57.6- 66.3	ACCEPTABLE
	2	27.1	26.3	23.6- 29.2	24.3- 28.5	ACCEPTABLE
POTASSIUM	1	7.84	7.50	6.39- 8.81	6.70- 8.50	ACCEPTABLE
	2	41.7	40.0	34.5- 45.6	35.9- 44.2	ACCEPTABLE
TOTAL ALKALINITY (AS CACO3)	1	122	120	106- 133	109- 130	ACCEPTABLE
	2	12	11.0	7.85- 15.3	8.77- 14.3	ACCEPTABLE
CHLORIDE	1	215	199	183- 213	187- 210	NOT ACCEPTABLE
	2	43.6	41.2	36.5- 45.8	37.6- 44.3	ACCEPTABLE
FLUORIDE	1	3.82	3.30	2.83- 3.77	2.95- 3.65	NOT ACCEPTABLE
	2	0.508	0.380	0.319-0.457	0.335-0.439	NOT ACCEPTABLE
SULFATE	1	10.9	14.0	10.8- 16.8	11.5- 15.0	CHECK FOR ERROR
	2	92.2	92.0	78.1- 105	81.5- 102	ACCEPTABLE
NUTRIENTS IN MILLIGRAMS PER LITER:						
AMMONIA-NITROGEN	1	7.78	7.70	6.10- 9.16	6.47- 8.79	ACCEPTABLE
	2	0.437	0.330	0.691-3.982	1.550-0.723	NOT ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302260

PERFORMANCE EVALUATION REPORT DATE: 12/17/91

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PE042

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
NUTRIENTS IN MILLIGRAMS PER LITER:						
NITRATE-NITROGEN	1	0.569	0.520	0.370-0.658	0.412-0.625	ACCEPTABLE
	2	10.1	11.0	8.86- 13.0	9.34- 12.5	ACCEPTABLE
ORTHOPHOSPHATE	1	0.146	0.150	0.114-0.187	0.122-0.178	ACCEPTABLE
	2	4.10	4.10	3.48- 4.68	3.62- 4.58	ACCEPTABLE
KJELDAHL-NITROGEN	3	13.5	14.0	10.3- 17.1	11.1- 16.3	ACCEPTABLE
	4	0.920	0.710	0.226- 1.32	0.357- 1.19	ACCEPTABLE
TOTAL PHOSPHORUS	3	7.08	7.40	5.57- 8.05	5.96- 7.75	ACCEPTABLE
	4	0.463	0.490	0.381-0.569	0.369-0.582	ACCEPTABLE
DEMANDS IN MILLIGRAMS PER LITER:						
COD	1	75.0	70.8	52.4- 84.6	56.5- 80.5	ACCEPTABLE
	2	200	207	163- 230	172- 221	ACCEPTABLE
TOC	1	38	28.0	23.9- 32.8	25.0- 31.6	NOT ACCEPTABLE
	2	79	82.0	69.5- 95.3	72.8- 91.9	ACCEPTABLE
5-DAY BOD	1	40	49.9	26.1- 60.1	30.3- 55.9	ACCEPTABLE
	2	130	131	75.8- 190	88.5- 167	ACCEPTABLE
PCB'S IN MICROGRAMS PER LITER:						
PCB-AROCLOR 1254	1	1.09	1.87	0.988- 2.71	1.21- 3.49	CHECK FOR ERROR
PCB-AROCLOR 1260	2	4.77	4.63	2.79- 5.96	3.19- 5.56	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302261

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PA042

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
PCB'S IN OIL IN MILLIGRAMS PER KILOGRAM:						
PCB IN OIL- 1016/1242	2	26.2	35.3	8.02- 46.5	13.0- 41.4	ACCEPTABLE
PCB IN OIL- 1254	1	31.7	43.9	13.5- 61.8	19.9- 55.5	ACCEPTABLE
PESTICIDES IN MICROGRAMS PER LITER:						
CHLORDANE	3	8.07	8.21	4.91- 9.73	5.52- 9.11	ACCEPTABLE
	4	2.28	2.21	1.07- 2.77	1.29- 2.56	ACCEPTABLE
ALDRIN	1	0.459	0.539	0.122-0.754	0.202-0.578	ACCEPTABLE
	2	0.064	0.086	.0171-0.121	.0303-0.138	ACCEPTABLE
DIELDRIN	1	0.416	0.475	0.203-0.710	0.267-0.645	ACCEPTABLE
	2	0.130	0.173	.0861-0.239	0.106-0.210	ACCEPTABLE
DDD	1	0.453	0.566	0.433- 1.15	0.524- 1.06	ACCEPTABLE
	2	0.171	0.202	.0956-0.268	0.117-0.246	ACCEPTABLE
DDE	1	0.498	0.539	0.235-0.756	0.301-0.690	ACCEPTABLE
	2	0.136	0.173	.0788-0.236	.0989-0.216	ACCEPTABLE
DDT	1	0.722	0.796	0.362- 1.06	0.450-0.972	ACCEPTABLE
	2	0.108	0.142	.0570-0.216	.0772-0.196	ACCEPTABLE
HEPTACHLOR	1	0.500	0.669	0.187-0.918	0.279-0.825	ACCEPTABLE
	2	0.177	0.216	.0756-0.269	0.140-0.245	ACCEPTABLE
HEPTACHLOR EPOXIDE	1	0.415	0.474	0.260-0.680	0.305-0.592	ACCEPTABLE
	2	0.140	0.174	.0858-0.235	0.105-0.216	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

R=95%

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302262

PERFORMANCE EVALUATION REPORT

DATE 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: P1042

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
VOLATILE HALOCARBONS IN MICROGRAMS PER LITER:						
1,2 DICHLOROETHANE	1	18.0	15.1	10.2- 20.8	11.5- 19.1	ACCEPTABLE
	2	44.2	55.8	36.2- 73.4	40.9- 68.7	ACCEPTABLE
CHLOROFORM	1	11.9	11.8	7.85- 15.9	8.86- 14.9	ACCEPTABLE
	2	55.9	64.4	38.3- 86.7	48.4- 80.6	ACCEPTABLE
1,1,1 TRICHLOROETHANE	1	12.9	13.8	8.68- 18.2	9.89- 17.0	ACCEPTABLE
	2	30.2	37.8	22.7- 50.3	26.2- 46.6	ACCEPTABLE
TRICHLOROETHENE	1	7.12	7.57	5.10- 10.3	5.75- 9.63	ACCEPTABLE
	2	51.4	62.7	38.6- 80.5	43.9- 75.2	ACCEPTABLE
CARBONTETRACHLORIDE	1	15.9	16.4	10.9- 22.7	12.3- 21.8	ACCEPTABLE
	2	31.9	37.1	23.0- 50.1	26.8- 46.6	ACCEPTABLE
TETRACHLOROETHENE	1	8.73	9.24	6.08- 12.5	6.99- 11.7	ACCEPTABLE
	2	41.2	51.3	31.2- 66.7	35.7- 62.2	ACCEPTABLE
BROMODICHLOROMETHANE	1	10.2	10.8	7.33- 14.2	8.21- 13.3	ACCEPTABLE
	2	32.7	38.1	24.9- 53.5	28.5- 51.0	ACCEPTABLE
DIBROMOCHLOROMETHANE	1	11.4	13.1	6.46- 17.0	9.55- 15.9	ACCEPTABLE
	2	46.1	58.1	34.1- 80.2	39.3- 74.4	ACCEPTABLE
BROMOFORM	1	12.3	14.5	7.85- 20.0	9.04- 18.4	ACCEPTABLE
	2	32.2	42.3	25.5- 59.9	29.9- 55.6	ACCEPTABLE
METHYLENE CHLORIDE	1	11.6	10.6	6.59- 16.1	7.79- 14.9	ACCEPTABLE
	2	48.9	54.1	30.7- 76.5	36.5- 70.7	ACCEPTABLE
CHLOROBENZENE	1	16.1	16.0	11.4- 20.1	12.5- 19.0	ACCEPTABLE
	2	54.0	63.7	41.3- 79.8	46.2- 74.9	ACCEPTABLE
VOLATILE AROMATICS IN MICROGRAMS PER LITER:						
BENZENE	1	33.4	40.1	25.7- 56.0	29.5- 52.2	ACCEPTABLE
	2	6.82	8.25	5.24- 11.6	6.04- 10.9	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

R-95%

PERFORMANCE EVALUATION REPORT DATE: 12/27/93

WATER POLLUTION STUDY NUMBER WP031

LABORATORY: PA062

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TOLATILE AROMATICS IN MICROGRAMS PER LITER:						
ETHYLBENZENE	1	56.2	66.9	36.0- 94.3	43.4- 86.9	ACCEPTABLE
	2	11.3	14.0	9.26- 19.0	10.5- 17.9	ACCEPTABLE
TOLUENE	1	45.9	49.2	30.2- 65.2	34.6- 60.8	ACCEPTABLE
	2	7.85	8.51	6.09- 12.6	6.93- 11.9	ACCEPTABLE
1,2-DICHLOROBENZENE	1	55.4	65.5	32.7- 93.9	40.5- 86.2	ACCEPTABLE
	2	8.99	8.98	5.85- 11.7	6.59- 11.0	ACCEPTABLE
1,3-DICHLOROBENZENE	1	39.8	47.9	29.4- 51.7	33.5- 57.6	ACCEPTABLE
	2	14.9	16.6	11.6- 21.1	12.9- 19.9	ACCEPTABLE
1,4-DICHLOROBENZENE	1	53.4	62.5	34.4- 85.8	41.6- 79.3	ACCEPTABLE
	2	11.7	12.8	7.89- 17.8	8.79- 16.5	ACCEPTABLE
MISCELLANEOUS PARAMETERS:						
TOTAL CYANIDE (IN MG/L)	1	1.25	0.860	0.598- 1.15	0.669- 1.08	NOT ACCEPTABLE
	2	0.183	0.160	0.102-0.208	0.116-0.194	ACCEPTABLE
NON-FILTERABLE RESIDUE (IN MG/L)	1	53	61.0	47.7- 64.6	49.6- 62.5	ACCEPTABLE
	2	72	83.0	62.8- 91.3	66.4- 87.9	ACCEPTABLE
OIL AND GREASE (IN MG/L)	1	10	8.30	3.07- 12.2	4.22- 11.0	ACCEPTABLE
	2	47.8	48.4	33.7- 56.0	36.5- 53.2	ACCEPTABLE
TOTAL PHENOLICS (IN MG/L)	1	0.668	0.595	0.312-0.878	0.384-0.805	ACCEPTABLE
	2	3.55	3.13	1.66- 4.60	2.04- 4.23	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302264

Quanterra

Environmental Services

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2 of 2

To: Neale Misquitta / Brian Smith

Location: North Canton

Telephone: 412-934-5944 / 216-497-9396

From: Maureen Dehulieu Date: 8-2-94

No. of Pages (including cover sheet): 8

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WP031, WS033 Results

ITAS PT/07-94/0126/FaxCov.Sht

302265

PERFORMANCE EVALUATION REPORT DATE: 2/10/94

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
TRACE METALS IN MICROGRAMS PER LITER:					
ALUMINUM	1	1430	1400	1260- 1510	ACCEPTABLE
ANTIMONY	2	45.7	48.9	34.2- 63.6	ACCEPTABLE
ARSENIC	1	40.3	39.5	33.1- 45.1	ACCEPTABLE
BARIUM	2	1750	1800	1530- 2070	ACCEPTABLE
BERYLLIUM	1	8.4	9.07	7.71- 10.4	ACCEPTABLE
IRON	2	1680	1700	1500- 1990	ACCEPTABLE
[illegible]IUM	1	52.8	49.0	39.2- 58.8	ACCEPTABLE
CHROMIUM	1	165	159	135- 183	ACCEPTABLE
COPPER	1	1940	1900	1710- 2090	ACCEPTABLE
LEAD	1	75.3	78.7	55.1- 102	ACCEPTABLE
MANGANESE	1	67.0	63.3	56.7- 67.7	ACCEPTABLE
MERCURY	1	1.4	1.77	1.24- 2.30	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

Score = 91%

302266

PERFORMANCE EVALUATION REPORT DATE: 2/10/94

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
TRACE METALS IN MICROGRAMS PER LITER:					
MOLYBDENUM	2	116	130	89.2- 166	ACCEPTABLE
NICKEL	1	524	490	417- 563	ACCEPTABLE
SELENIUM	1	100	98.3	78.6- 118	ACCEPTABLE
SILVER	2	65.4	66.9	57.1- 75.6	ACCEPTABLE
THALLIUM	2	9.5	9.56	6.69- 12.4	ACCEPTABLE
TOC	1	2210	2090	1920- 2240	ACCEPTABLE
NITRATE/NITRITE/FLUORIDE IN MILLIGRAMS PER LITER:					
NITRATE AS N	1	8.70	9.80	8.82- 10.8	NOT ACCEPTABLE
NITRITE AS N	1	1.47	1.40	1.19- 1.61	ACCEPTABLE
FLUORIDE	1	6.83	6.60	5.94- 7.26	ACCEPTABLE
INSECTICIDES IN MICROGRAMS PER LITER:					
ALACHLOR	5	4.01	4.27	2.35- 6.19	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302267

PERFORMANCE EVALUATION REPORT DATE: 2/10/94

WATER SUPPLY STUDY NUMBER W5033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
INSECTICIDES IN MICROGRAMS PER LITER:					
ATRAZINE	5	6.68	3.42	1.88- 4.96	NOT ACCEPTABLE
CHLORDANE (TOTAL)	3	3.30	2.76	1.52- 4.00	ACCEPTABLE
ENDRIN	1	2.60	2.63	1.84- 3.42	ACCEPTABLE
HEPTACHLOR	4	1.51	1.73	0.951- 2.51	ACCEPTABLE
HEPTACHLOR EPOXIDE	4	0.662	0.679	0.373-0.985	ACCEPTABLE
HEXACHLOROBENZENE	4	1.37	1.32	0.505- 1.84	ACCEPTABLE
HEXACHLOROCYCLOPENTADIENE	4	2.40 **	2.92	0.116- 3.44	ACCEPTABLE
LINDANE	1	0.137	0.136	.0748-0.197	ACCEPTABLE
METHOXYCHLOR	1	40.8	42.3	23.3- 61.3	ACCEPTABLE
SIMAZINE	5	18.8	8.73	2.07- 14.5	NOT ACCEPTABLE
TOXAPHENE	2	11.1	9.23	5.08- 13.4	ACCEPTABLE
HERBICIDES IN MICROGRAMS PER LITER:					
2,4-D	1	5.57	7.57	3.78- 11.4	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.
** SIGNIFICANT BIAS IS ANTICIPATED FOR THIS RESULT.

302268

PERFORMANCE EVALUATION REPORT DATE: 2/17/94

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
HERBICIDES IN MICROGRAMS PER LITER:					
2,4,5-TP (SILVEX)	1	4.39	5.60	2.80- 8.40	ACCEPTABLE
PENTACHLOROPHENOL	1	1.40	2.72	1.36- 4.08	ACCEPTABLE
TRIHALOMETHANES IN MICROGRAMS PER LITER:					
BROMODICHLOROMETHANE	1	17.1	15.8	12.6- 19.0	ACCEPTABLE
BROMOFORM	1	43.9	34.4	27.5- 41.3	NOT ACCEPTABLE
CHLORODIBROMOMETHANE	1	32.7	28.7	23.0- 34.4	ACCEPTABLE
[illegible]IOFORM	1	35.1	36.7	29.4- 44.0	ACCEPTABLE
TOTAL TRIHALOMETHANE	1	128.8	115.6	92.5- 139	ACCEPTABLE
VOLATILE ORGANIC COMPOUNDS IN MICROGRAMS PER LITER:					
BENZENE	1	12.1	12.0	9.60- 14.4	ACCEPTABLE
CARBON TETRACHLORIDE	1	12.9	13.4	10.7- 16.1	ACCEPTABLE
CHLOROBENZENE	2	20.5	18.4	14.7- 22.1	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302269

PERFORMANCE EVALUATION REPORT DATE: 2/10/94

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
VOLATILE ORGANIC COMPOUNDS IN MICROGRAMS PER LITER:					
1,2 DICHLOROBENZENE	2	15.3	15.5	12.4- 18.6	ACCEPTABLE
1,4-DICHLOROBENZENE	1	15.3	15.1	12.1- 18.1	ACCEPTABLE
1,2-DICHLOROETHANE	1	17.7	16.9	13.5- 20.3	ACCEPTABLE
1,1-DICHLOROETHYLENE	1	12.3	12.9	10.3- 15.5	ACCEPTABLE
C 1,2 DICHLOROETHYLENE	2	18.5	16.5	13.2- 19.8	ACCEPTABLE
T 1,2 DICHLOROETHYLENE	2	15.7	13.9	11.1- 16.7	ACCEPTABLE
DICHLOROPROPANE	2	14.9	14.3	11.4- 17.2	ACCEPTABLE
ETHYLBENZENE	2	23.4	17.0	13.6- 20.4	NOT ACCEPTABLE
STYRENE	2	14.5	13.0	10.4- 15.6	ACCEPTABLE
TETRACHLOROETHYLENE	2	13.9	12.9	10.3- 15.5	ACCEPTABLE
TOLUENE	2	13.3	12.3	9.84- 14.8	ACCEPTABLE
1,1,1-TRICHLOROETHANE	1	13.7	14.6	11.7- 17.5	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302270

PERFORMANCE EVALUATION REPORT DATE: 2/10/90

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE		TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
VOLATILE ORGANIC COMPOUNDS IN MICROGRAMS PER LITER:						
TRICHLOROETHYLENE	1	15.1		14.9	11.9- 17.9	ACCEPTABLE
VINYL CHLORIDE	1	8.44		7.35	4.41- 10.3	ACCEPTABLE
TOTAL XYLENES	2	12.8		11.6	9.28- 13.9	ACCEPTABLE
CHLOROETHANE	3	16.3		16.8	10.3- 24.6	ACCEPTABLE
DIBROMOMETHANE	3	13.3		15.2	10.5- 18.7	ACCEPTABLE
DICHLOROMETHANE	3	11.8		12.8	9.25- 16.3	ACCEPTABLE
1,1,2,2-TETRACHLOROETHANE	3	8.33	**	8.63	6.37- 9.98	ACCEPTABLE
1,2,4-TRICHLOROBENZENE	3	16.0		16.8	12.0- 20.1	ACCEPTABLE
1,1,2-TRICHLOROETHANE	3	14.9		15.7	9.66- 19.7	ACCEPTABLE
MISCELLANEOUS ANALYTES:						
TURBIDITY (NTU'S)	1	1.35	**	1.10	0.836- 1.50	ACCEPTABLE
TOTAL FILTERABLE RESIDUE (MILLIGRAMS PER LITER)	1	478		384	255- 567	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.
** SIGNIFICANT BIAS IS ANTICIPATED FOR THIS RESULT.

302271

PERFORMANCE EVALUATION REPORT DATE: 2/10/94

WATER SUPPLY STUDY NUMBER WS033

LABORATORY PA042

ANALYTES	SAMPLE NUMBER	REPORTED VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	PERFORMANCE EVALUATIONS
MISCELLANEOUS ANALYTES:					
CALCIUM (MG. CACO3/L)	1	204	200	187- 212	ACCEPTABLE
PH-UNITS	1	9.18	9.10	8.86- 9.31	ACCEPTABLE
ALKALINITY (MG. CACO3/L)	1	42	41.0	37.3- 46.4	ACCEPTABLE
CORROSIVITY (LANGELIER IND. AT 20C)	1	0.340	1.057	0.679- 1.39	NOT ACCEPTABLE
SODIUM (MILLIGRAMS PER LITER)	1	20.8	18.8	17.1- 20.5	NOT ACCEPTABLE
SULFATE (MILLIGRAMS PER LITER)	1	155	170	152- 185	ACCEPTABLE
CYANIDE (MILLIGRAMS PER LITER)	1	0.387	0.340	0.255-0.425	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

302272