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REMEDIAL DESIGN WORK PLAN

SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

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OCT 30 1991

QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: Remedial Design Work Plan
Summit National Superfund Site
Deerfield Township of Portage County, Ohio

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Deerfield Township of Portage County, Ohio

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REMEDIAL DESIGN WORK PLAN

SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

OCTOBER 1991

REF. NO. 2372 (13)

CONESTOGA-ROVERS & ASSOCIATES

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1.0 INTRODUCTION

1.1 PURPOSE AND ORGANIZATION OF REPORT

Potentially Responsible Parties (PRPs) entered into negotiations with the United States Environmental Protection Agency (USEPA) and the Ohio Environmental Protection Agency (OEPA) to complete a Remedial Design/Remedial Action (RD/RA) for the Summit National Superfund Site (Site) in Deerfield Township of Portage County, Ohio. The negotiations culminated in a agreement between a group of Settling Defendants and USEPA which agreement has been incorporated into a Consent Decree. The United States District Court for the Northern District of Ohio, Eastern Division by Judge David D. Dowd entered the Consent Decree on June 11, 1991, in Civil Action No. C81-1961. Pursuant to the terms of the Consent Decree, the Consent Decree was recorded with the Portage County Recorder in the Portage County Record of Deeds, Volume IIII, page 780-1140.

The RD Work Plan, presented herein, is submitted on behalf of the Settling Defendants by the Trustees of the Summit National Facility Trust (SNFT), pursuant to their authority under the trust agreement to provide for performance of work as required by the Consent Decree. This RD Work Plan is submitted to USEPA and OEPA as the first task associated with the RD/RA for the Site, as required by Paragraph VI Sections E.1 and E.2 of the Consent Decree.

The RD Work Plan is organized as follows:

i) Section 1 states the purpose of the RD Work Plan, presents the organization of the RD Work Plan, presents a general introduction and history of the Site, presents a summary of the proposed remedial action determined for the Site, presents the project organization and presents the basis for the RD;

ii) Section 2 presents the Health and Safety Plan for the pre-design and design phases;

iii) Section 3 presents the Sampling and Analysis Plan for the pre-design and design phases;

iv) Section 4 presents the work plan for the pre-design activities;

v) Section 5 presents the work plan for the design activities;

vi) Section 6 identifies the reports and documentation required during the RD activities;

vii) Section 7 presents the community relations requirements during the RD activities; and

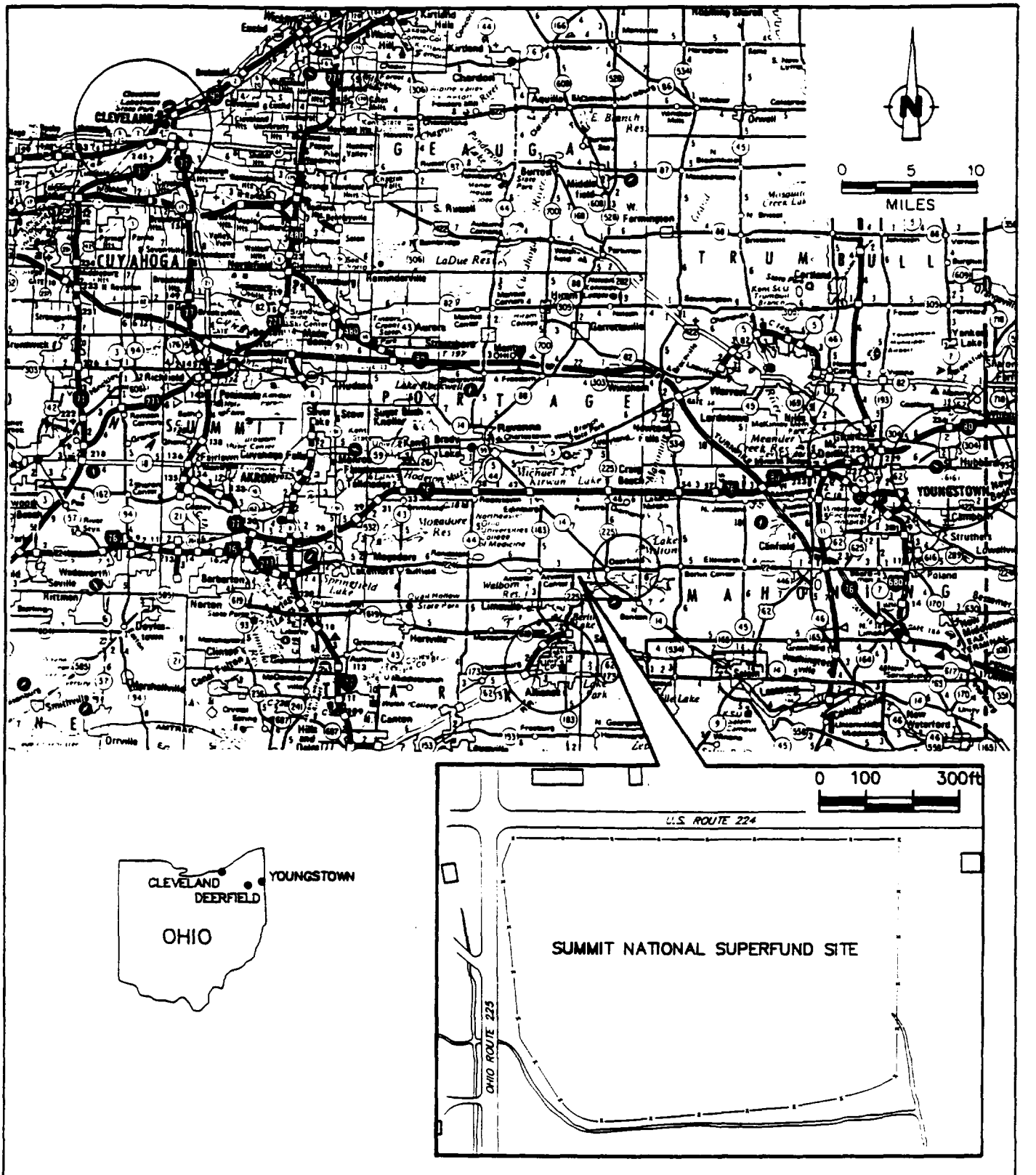
viii) Section 8 presents the schedule of activities for the RD.

1.2 SITE DESCRIPTION

The Site is located in Deerfield Township of Portage County, Ohio, at the intersection of Ohio Route 225 and U.S. Route 224, approximately 45 miles southeast of Cleveland, Ohio, and 20 miles west of Youngstown, Ohio (see Figure 1.1). The Site occupies approximately 11.5 acres and is approximately rectangular in shape.

Prominent features remaining at the Site from previous operations include a 15-foot high coal tipple, loading dock, scale house and concrete debris in the northwest corner of the Site, two ponds in the midsection of the Site, an abandoned incinerator and two buildings in the southeast corner of the Site and two abandoned buildings in the northeast corner of the Site. Little vegetation remains at the Site since most of the Site was graded following previous surface cleanup activities performed at the Site.

A 6-foot high fence encompasses the Site, with two locked gates from U.S. Route 224 and one locked gate from Ohio Route 225 for entrance.



SOURCE: OHIO OFFICIAL TRANSPORTATION MAP, 1987

figure 1.1

SITE LOCATION
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

1.3 SITE HISTORY

1.3.1 History of Operations

The Site was originally used as a coal strip mine and contained a coal wash pond and coal stockpile, prior to its use by Donald Georgehoff, Summit National Services and/or Summit National Liquid Disposal Services for the operation of a hazardous waste treatment, recycling, storage and disposal facility.

In 1973, OEPA issued a "Permit to Install" for a liquid waste incinerator at the Site. Commencing in 1974, Summit National Liquid Services received a variety of liquid wastes for incineration by authority of an OEPA "Permit to Operate". In June 1975, the OEPA Northeast District Office investigated a complaint of an unauthorized wastewater discharge at the Site. Subsequently, the OEPA requested USEPA to investigate the Site. USEPA investigated the Site on October 29, 1976 and found evidence of chemical leaks and spills. As a result, the Site operator was officially notified in December 1976 of the need for a Spill Prevention Control and Countermeasure Plan. On June 12, 1978, OEPA ordered Summit National to cease operations.

1.3.2 History of Remedial Actions

In 1980, OEPA fenced and graded the Site to control access and drainage. Liquid wastes stored on the Site were characterized and staged. During the fall of 1980, USEPA removed three bulk tanks and their contents and some contaminated soil, and treated contaminated water under a 311 action (Section 311 of the Clean Water Act).

During 1980 and 1981, some companies that had previously used Summit's services voluntarily removed waste from the Site. In 1981 and 1982, a surface waste cleanup was conducted at the Site by a number of generators and the State of Ohio.

An emergency response action was conducted by USEPA Region V Emergency Response Section , in the Spring of 1987, to control surface water drainage and remove a buried tank.

1.3.3 History of Investigative Programs

A Remedial Investigation (RI) was initiated at the Site in 1984. Field investigations were conducted in November and December 1984, December 1985 and January 1986, and June through September 1986.

The Remedial Investigation/Feasibility Study (RI/FS) for the Site was prepared by CH_2M Hill under contract to USEPA, Region V. The final RI/FS report was completed on February 12, 1988.

1.3.4 Regulatory History

The OEPA Director issued Final Findings and Orders on June 12, 1978, requiring Summit National to cease receiving waste materials and to remove all liquid waste from the Site.

On October 1, 1981, the State of Ohio filed in the United States District Court for the Northern District of Ohio, Eastern Division, a Complaint against 42 Defendants for the recovery of response costs incurred or to be incurred to prevent, minimize or eliminate the threat posed by the hazardous substances at the Summit National property, for the recovery of damages to the State's natural resources, for nuisance abatement, for injunctive relief, for declaratory relief and for civil penalties. Prior to filing of the Complaint, the State had entered into agreements with 22 of the Defendants and resolved all State claims against those parties. Subsequent to filing of the Complaint, the State entered into settlements with 20 of the Defendants, resolving all or part of their liability to the State.

USEPA, pursuant to § 105 of CERCLA 42 U.S.C. § 9605, placed the Summit National Facility in Portage County, Ohio on the National

Priorities List (NPL), which is set forth in 40 C.F.R. Part 300, and published notice of the placement in the Federal Register on September 15, 1983.

In the fall of 1984, USEPA commenced an RI/FS, pursuant to 40 C.F.R. 300.68, for the Site in response to a release or a substantial threat of release of a hazardous substance at or from the Site. The field investigations were conducted in November and December 1984, December 1985 and January 1986 and June through September 1986.

In December 1986, USEPA observed that the eastern pond on the Site was flooding, the embankment about the pond was eroding and an underground tank was leaking. Consequently, in March 1987, the USEPA issued a Section 106(a) CERCLA Unilateral Administrative Order (AO) to the PRPs for the Site. The AO was issued to contain and terminate the actual or threatened release into the environment of hazardous substances due to the deteriorating Site conditions. To satisfy their obligations under the AO the PRPs agreed to reimburse USEPA for response costs related to this emergency action.

USEPA completed the RI/FS for the Site on February 12, 1988. Pursuant to § 117 of CERCLA, as amended by the Superfund Amendments and Reauthorization Act (SARA), 42 U.S.C. § 9617, USEPA published notice of completion of the RI/FS and of the proposed plan for remedial action identified in the FS Report and provided opportunity for public comment. Considering the information in the Administrative Record, including that developed in the RI, the evaluation of remedial alternatives in

the FS and the proposed plan for remedial action identified therein and the public comments received, USEPA selected a remedial action plan alterative which is embodied in a document called a Final Record of Decision, dated June 30, 1988.

On April 1, 1988, USEPA, pursuant to § 122 of CERCLA, 42 U.S.C. § 9622, notified certain entities that USEPA determined each entity to be a PRP regarding the proposed remedial action at the Site. In accordance with § 121(f)(1)(F) of CERCLA, 42 U.S.C. § 9621(f)(1)(F), USEPA notified the State of Ohio on November 2, 1987 of negotiations with PRPs regarding the scope of the remedial action for the Site, and USEPA provided the State with an opportunity to participate in such negotiations and be a party to any settlement. Pursuant to § 122(j) of CERCLA, 42 U.S.C. § 9622(j), USEPA notified the Federal and State natural resource trustees of negotiations with PRPs on the subject of addressing the release or threatened release of hazardous substances at the Site.

Pursuant to § 122 of CERCLA, 42 U.S.C. § 9622, USEPA, OEPA and PRPs participated in negotiations leading to the Settling Defendants' agreement in 1988 to perform remedial design and remedial action work at the Summit National Facility, which work is embodied in the Consent Decree and the Appendices thereto. The work constituted an amended remedial action, within the meaning of § 117 of CERCLA, 42 U.S.C. § 9617, for the Summit National Facility and therefore required provisions for further public comment and issuance of a revised Record of Decision. The Consent Decree was entered by the Court following a public comment and

evaluation period, and after further evaluation by the Court became effective on June 11, 1991.

1.4 PROPOSED REMEDIAL ACTION

The RA to be implemented at the Site is detailed in Appendix B of the Consent Decree, in the document entitled "Statement of Work and Appendices to Statement of Work, Appendix A - Soil Removal and Treatment, Appendix B - Groundwater Extraction System, Appendix C - Site Cover, Appendix D - Groundwater Treatment System, Appendix E - Schedule of Activities, Summit National Superfund Site, Deerfield Township of Portage County, Ohio" printed on December 14, 1989 (Statement of Work). The Statement of Work (SOW) delineates and describes in detail the RA to be implemented at the Site. The major components of the RA include the following:

i) excavation and on-Site incineration of on-Site contaminated soils, pond sediments, contaminated soils associated with buried containerized materials, buried containerized materials and existing stockpiled soils, and of off-Site east-perimeter contaminated soils;

ii) excavation and placement on Site of off-Site contaminated pond and drainage ditch sediments;

iii) demolish on-Site buildings and structures to grade and place resulting debris under on-Site cap;

iv) installation and operation of a groundwater extraction system for hydraulic containment, collection and extraction of Site-related contaminated groundwater, consisting of an overburden pipe and media drain installed along the southern boundary of the Site and extending along the east and west boundaries of the Site, a bedrock well system of six new extraction wells along the alignment of the overburden pipe and media drain, and conversion of two existing on-Site monitoring wells to temporary extraction wells;

v) design, installation and operation of a water treatment system to treat extracted groundwater and water from pond dewatering, perimeter sediment dewatering and construction dewatering operations;

vi) installation and maintenance of a 2-foot thick permeable vegetated soil cap over the Site;

vii) removal of buried drums, containers and underground tanks;

viii) installation of an 8-foot high chain link fence to secure the Site;

ix) implementation of access/deed restrictions;

x) implementation of an effectiveness groundwater monitoring program, including installation of additional monitoring wells and piezometers, hydraulic monitoring and groundwater quality monitoring; and

xi) monitor Site surface runoff.

1.5 PROJECT ORGANIZATION

Pursuant to the Consent Decree the Settling Defendants have formed the SNFT and have empowered the Trustees to provide for the performance of the RD/RA. CRA will be the primary consultant to the SNFT for the pre-design investigation and the RD portion of the RD/RA. Additional subcontractors will be selected to perform specific tasks during the pre-design and RD phases, such as boundary and topographic surveys, analytical testing, soil borings, geophysical testing, geotechnical testing and exploratory excavations.

1.6 BASIS FOR REMEDIAL DESIGN

The RD for the RA selected for the Site will be based on the provisions of the Consent Decree and the SOW attached thereto, USEPA Superfund Remedial Design and Remedial Action Guidance, and other appropriate guidances that may be provided by USEPA.

A design criteria document establishing the basis for the RD of the RA will be prepared and submitted to USEPA and OEPA within 75 calendar days of the effective date of the Consent Decree, in accordance with Paragraph VI Section E.3.a of the Consent Decree.

2.0 HEALTH AND SAFETY PLAN

A Site-specific Health and Safety Plan (HSP) is required to ensure that all pre-design and design activities are performed safely and in accordance with applicable regulatory requirements, and that all persons on Site, the general public and the environment are protected from exposure to Site-related contaminated material. A Site-specific HSP has been developed for the pre-design and design activities associated with the RD for the Site and is presented in Appendix A.

The HSP will be adhered to for all activities conducted at or in the vicinity of the Site during the pre-design and design phases of the RD for the Site.

3.0 SAMPLING AND ANALYSIS PLAN

A Site-specific Sampling and Analysis Plan (SAP) is required to ensure that all pre-design and design activities requiring sampling and analyses are performed to established and accepted protocols. All sampling and analyses will be part of a quality assurance program to ensure meaningful results are obtained and to ensure that all activities are comparable.

A Site-specific SAP has been developed for the pre-design sampling and analysis activities for the Site and is presented in Appendix B. The associated Site-specific Quality Assurance Project Plan (QAPP) is presented in Appendix C. These plans will be adhered to for all sampling and analysis activities required during the pre-design and design phases of the RD for the Site.

4.0 PRE-DESIGN ACTIVITIES

4.1 GENERAL

In order to perform the RD of the RA selected for the Site, additional investigations are required to supplement the data presented in the RI Report. The pre-design activities required to provide the additional data consist of the following:

i) securement of access agreements;
ii) boundary and topographical surveys;
iii) investigation of magnetic anomalies;
iv) investigation of underground tanks;
v) characterization of the Tipple well;
vi) bedrock profiling;
vii) groundwater characterization;
viii) groundwater treatability studies;
ix) Upper Sharon Aquifer characterization; and
x) investigation of sediments.

All pre-design activities will be performed in accordance with the HSP, as stated in Section 2.0. The pre-design sampling and analysis activities will be performed in accordance with the SAP, as stated in Section 3.0, whereas pre-design investigative activities will be conducted in accordance with the specific guidelines and procedures detailed in the following subsections.

4.2 ACCESS AGREEMENTS

Prior to conducting the pre-design activities it will be necessary to obtain on- and off-Site access. All on- and off-Sites access agreements required to implement the pre-design and design activities will be obtained by the SNFT, with assistance from USEPA as required, in accordance with the provisions of Paragraph X of the Consent Decree. Site access agreements will extend for the duration and implementation of the RD/RA and will include allowances for all operation and maintenance considerations and State and Federal oversight activities.

4.3 BOUNDARY AND TOPOGRAPHICAL SURVEY

Prior to commencing design activities at the Site, it is necessary to establish the legal boundaries for the Site. Also, a topographical survey of the Site is required to complete design activities and provide cost estimates.

The topographical survey will include, the following:

i) cross-sections coincident with the 100-foot grid established at the Site by USEPA;

ii) establishment of the alignment of the proposed pipe and media drain to facilitate the bedrock profiling along the drain; and

iii) location of all existing groundwater monitoring wells, including elevation of ground surface and top of well casing at each monitoring well site.

The surveys will be performed to a vertical tolerance of plus or minus one-hundredth of a foot (±0.01 feet) and a horizontal tolerance of ± 0.1 feet.

The topographical survey and establishment of the Site legal boundaries will be performed by a surveyor licensed to practice in the State of Ohio. All surveying activities will be performed according to the requirements of the HSP developed for the Site.

The results of the boundary and topographical survey will be utilized in the design of the following RA components:

i) pipe and media drain;
ii) soil removal and replacement;
iii) groundwater extraction system;
iv) perimeter fence relocation;
v) groundwater treatment system;
vi) incinerator location; and
vii) Site cap and final grading plan.

4.4 INVESTIGATION OF MAGNETIC ANOMALIES

The SOW requires that exploratory trenches be excavated in areas of magnetic anomalies which were identified during the RI to contain buried containerized materials or for which no test pits were excavated. The purpose of the exploratory trenching is to visually confirm the presence of buried containerized materials in order to delineate the areas of buried material to be removed during the RA.

The exploratory trenches will be excavated to the standard width of a backhoe bucket (42 to 60 inches) at 15-foot centers and at the approximate locations detailed on Figure 4.1. The excavations will proceed from the perimeters of the magnetic anomaly areas toward the centers of the areas, until buried containerized material is encountered at each test trench location. The exploratory trenches will be excavated to a minimum depth of eight feet below surface grade unless buried containerized material is encountered, or to the depth of the water table if greater than eight feet below surface grade. Excavated soils will be stockpiled adjacent to the trench on 6-mil polyethylene sheeting.

A qualified field engineer or geologist will observe all excavation activity and record in the Site log record for each excavated trench, the following:

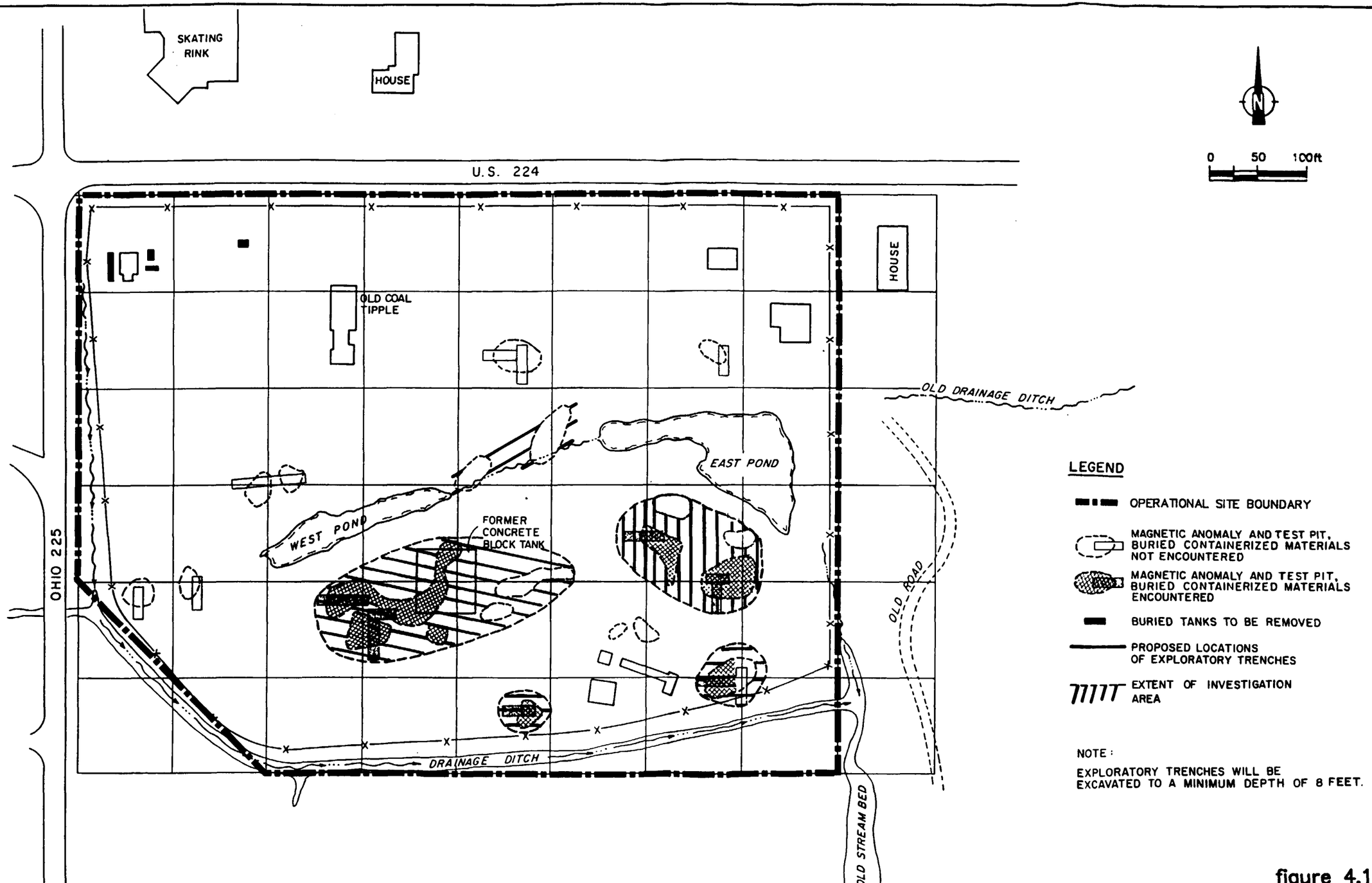


figure 4.1

PROPOSED LOCATIONS OF EXPLORATORY TRENCHES
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

i) time of excavation;

ii) weather conditions;

iii) stratigraphic description;

iv) location, width and depth of excavation;

v) location and description of visual soil contamination, and buried containerized materials;

vi) depth to groundwater; and

vii) ambient organic vapor concentrations as measured by field measurement equipment such as a total organic vapor analyzer (OVA).

In addition, a photographic log of each trench excavation will be maintained.

The performance of this work will be contingent upon being able to excavate with a reasonable amount of effort, consistent with health and safety considerations for on-Site personnel.

Buried containers encountered during the exploratory excavations will be left in place. The soil excavated from the trenches, with the polyethylene sheeting, will be used to backfill the trenches on completion of the excavations in a manner to ensure standing water would not collect on the trench alignment. The location of the ends of each trench will be staked and surveyed consistent with the topographic survey protocols discussed in Section 4.3.

In the event a buried drum is ruptured during the trench excavation, the ruptured drum will be placed in an overpack drum and staged

on-Site for sampling and disposal during the RA. Adjacent soils visually contaminated by the ruptured drum would be collected and contained in Department of Transport (DOT) approved 55-gallon drums, and staged on-Site for sampling and disposal during the RA.

All excavation and backfilling activities will be performed in accordance with the Site-specific HSP. Following completion of excavation and backfilling activities, the backhoe will be decontaminated in accordance with the protocols for equipment decontamination presented in the Site-specific SAP.

The extent of buried containerized materials as determined by the exploratory trenches will be utilized in the RD to specify the extent of buried containerized materials to be removed during the RA. Compatibility testing and characterization of the buried containerized materials will be performed during the RA, in accordance with a work plan to be developed for the RA activities.

4.5 INVESTIGATION OF UNDERGROUND TANKS

Four underground tanks in the northwest corner of the Site were identified during the RI. The SOW requires that the contents of these tanks, if any, be sampled, analyzed, characterized and disposed of in accordance with the appropriate requirements for the disposition of the materials. In addition, visually contaminated soils above the watertable, if

any, associated with the underground tanks are to be removed and treated with the on-Site soils.

It is anticipated that the contents of the underground tanks, if any, will be disposed of by on-Site incineration during the RA. To quantify the materials to be removed during the RA, the tank access hatches or fill ports will be uncovered by hand tools or a backhoe, as necessary, and the tank will be opened. A qualified field engineer or geologist will observe all excavation activity and the opening of each tank, and record in the Site log record the following:

i) time of excavation;

ii) weather conditions;

iii) location and description of visual soil contamination, if any;

iv) location, description and estimated size, if possible, of each underground tank;

v) depth to groundwater, if encountered;

vi) visual characterization of the contents of each underground tank, if any, as solid/sludge or liquid, and the estimated thickness of each such layer. An oil-phase interphase probe, steel tape covered with "water paste" sensing material, glass or stainless-steel sampling thief, or similar investigative sampling devices may be used to assist in the visual characterization of the contents of each underground tank; and

vii) ambient organic vapor concentrations as measured by field measurement equipment such as an OVA.

In addition, a photographic log of each uncovered tank will be maintained.

The performance of this work will be contingent upon the tanks being opened with a reasonable amount of effort, consistent with health and safety considerations for on-Site personnel.

Underground tanks opened during the investigative activities will be sealed water-tight and the soil excavated to expose the tanks will be used to backfill the excavations on completion of the investigation. The location of each tank will be staked and surveyed, consistent with the topographic survey protocols discussed in Section 4.3.

All excavation, investigation and backfilling activities will be performed in accordance with the Site-specific HSP. Following completion of excavation and backfilling activities, the backhoe will be decontaminated in accordance with the protocols for equipment decontamination presented in the Site-specific SAP.

The visual characterization of the underground tanks, the contents of the underground tanks and the soils adjacent to the underground tanks will be utilized in the RD to specify the quantity and nature of tank contents and associated contaminated soils to be removed during the RA. Compatibility testing and characterization of the underground tank contents will be performed during the RA, in accordance with a work plan to be developed for the RA activities.

4.6 CHARACTERIZATION OF THE TIPPLE WELL

The SOW requires that the Tipple well be characterized prior to closure. Characterization of the Tipple well with respect to groundwater quality and physical conditions will be performed during the pre-design activities, whereas closure, if required, will be performed during the RA.

Geologic records from the RI will be reviewed to determine the probable lithology in the vicinity of the Tipple well. An approximate geologic cross-section will then be prepared for use in the field.

A drill rig will be set up over the Tipple well to attempt to open the well for subsequent investigations. It is understood that the well was abandoned by the installation of a wooden plug (i.e. a telephone pole). This wooden plug will be drilled out using wet-rotary drilling methods. Once the plug has been removed, the remainder of the well bore will be cleaned by lowering the drill rods to the bottom of the well and removing any siltation and rock debris.

Following the opening and cleaning of the Tipple well, it's stratigraphy and completion details will be determined by means of geophysical logging. The suite of logs to be run will include spontaneous potential (SP), short and long normal resistivity, natural gamma and caliper. The traces of the various logs will be interpreted to determine the depth and

diameter of casing, the borehole diameter, geologic units penetrated and location of fracture zones.

Once the details of the well have been determined, the water bearing zones will be investigated by means of a borehole flow meter and/or water injection testing using industry accepted standards Areas of major contribution will be selected for zone specific groundwater sampling and analyses, such that groundwater samples are collected as follows:

i) one sample from the Lower Sharon Aquifer (if the well is completed to this unit);
ii) one sample from the Upper Sharon Unit;
iii) two samples from the intermediate unit (IU); and
iv) one sample from the water table unit (WTU) (if the well is open to this unit).

The zone specific groundwater samples from the Tipple well will be collected and analyzed for the Target Compound List (TCL) parameters, as detailed in the Site-specific SAP.

Characterization of the Tipple well as described above will be performed under the supervision of a qualified field engineer or geologist. Observations and measurement data will be recorded in the Site log record.

The performance of this work will be contingent upon the Tipple well being opened with a reasonable amount of effort, consistent with health and safety considerations for on-Site personnel.

Following characterization of the Tipple well, the well will be secured with a lockable cover. The location of the Tipple well will be surveyed, consistent with the topographic survey protocols discussed in Section 4.3.

The characterization activities will be performed in accordance with the Site-specific HSP. Following completion of the characterization activities, the drill rig and sampling equipment will be decontaminated in accordance with the protocols for equipment decontamination presented in the Site-specific SAP. Liquids and solids generated as a result of the characterization activities will be handled consistent with the waste material handling protocols presented in the Site-specific SAP.

Characterization of the Tipple well will provide the data required to determine whether the Upper Sharon Aquifer has adversely been impacted by past operations at the Facility. In the event that extraction of groundwater from the Upper Sharon Aquifer is determined to be necessary, separate work plans and remedial designs will be prepared and submitted to USEPA and OEPA for review, modification and approval. Otherwise, the Tipple well will be properly abandoned during the RA.

4.7 BEDROCK PROFILING

The SOW requires that the profile of the bedrock along the centerline of the proposed pipe and media drain be determined during the pre-design activities. To determine the bedrock profile, boreholes will be installed, as detailed in Section 8.4.2 of the SAP, to the depth of bedrock along the alignment of the drain, at approximately 100-foot centers, as shown on Figure 4.2. A qualified engineer or geologist will observe all drilling activities and record in the Site log record for each soil boring the following:

i) time of drilling;

ii) weather conditions;

iii) location and stratigraphic description;

iv) depth to groundwater;

v) depth to bedrock, as determined by auger refusal; and

vi) organic vapor concentrations of soil samples collected at the proposed elevation of the pipe bedding and at three equally spaced intervals between the pipe bedding elevation and surface grade, as measured by field measurement equipment such as an OVA. The measurements will be taken directly from the auger cuttings at each borehole location, as the cuttings are brought to surface grade.

All on-Site boreholes will be backfilled with the native soil cuttings and all off-Site boreholes will be backfilled with native soil except for the last foot of backfill which will be backfilled with a cement-bentonite

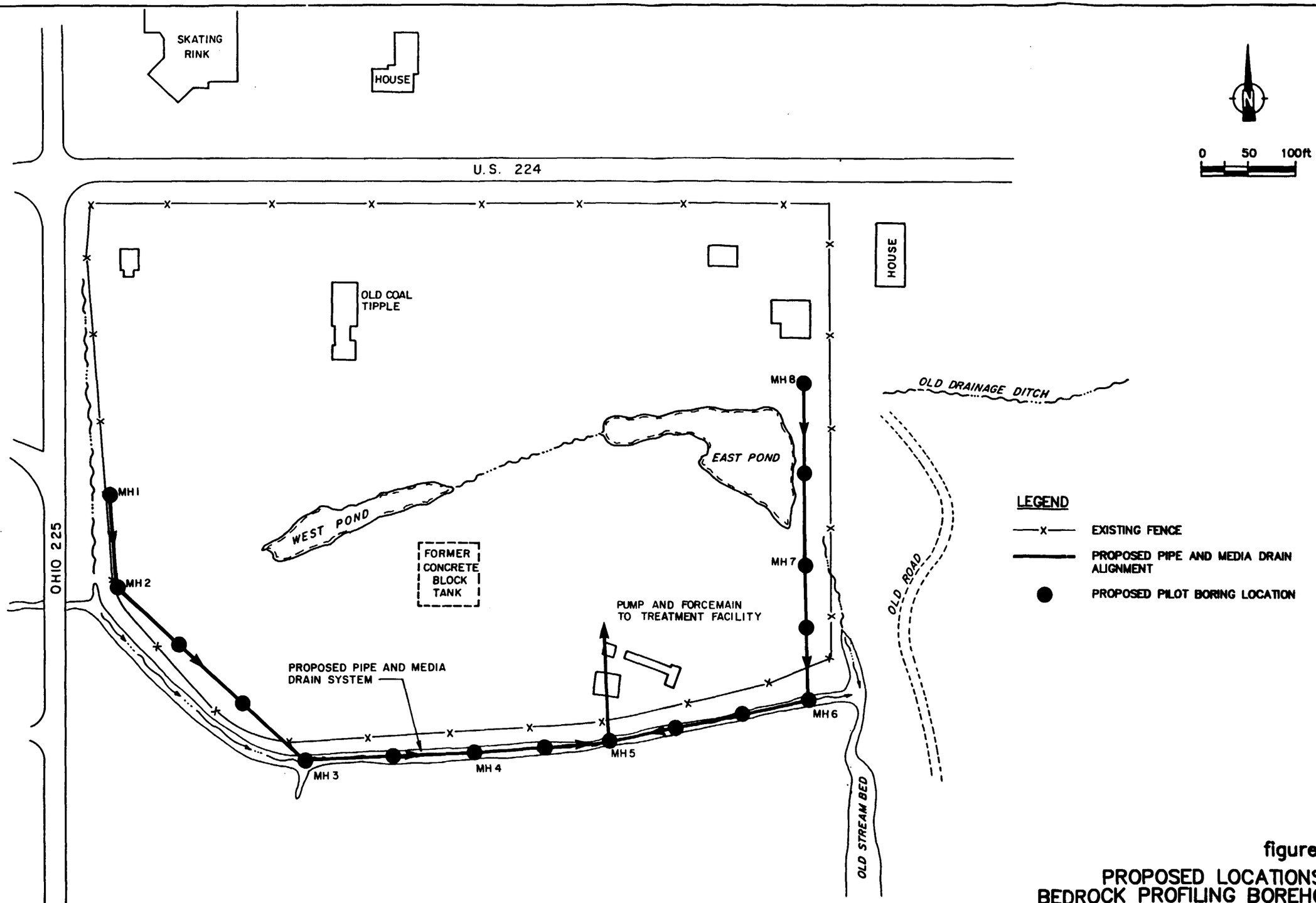


figure 4.2
PROPOSED LOCATIONS OF
BEDROCK PROFILING BOREHOLES
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

mixture. Surplus visually clean soil cuttings will be collected and disposed of consistent with the waste material handling protocols presented in Section B.7.0 of the SAP. Following completion of the bedrock profiling activities, the drill rig and sampling equipment will be decontaminated in accordance with the protocols for equipment decontamination presented in the SAP. The location of each soil boring will be staked and surveyed, consistent with the topographic survey protocols discussed in Section 4.3.

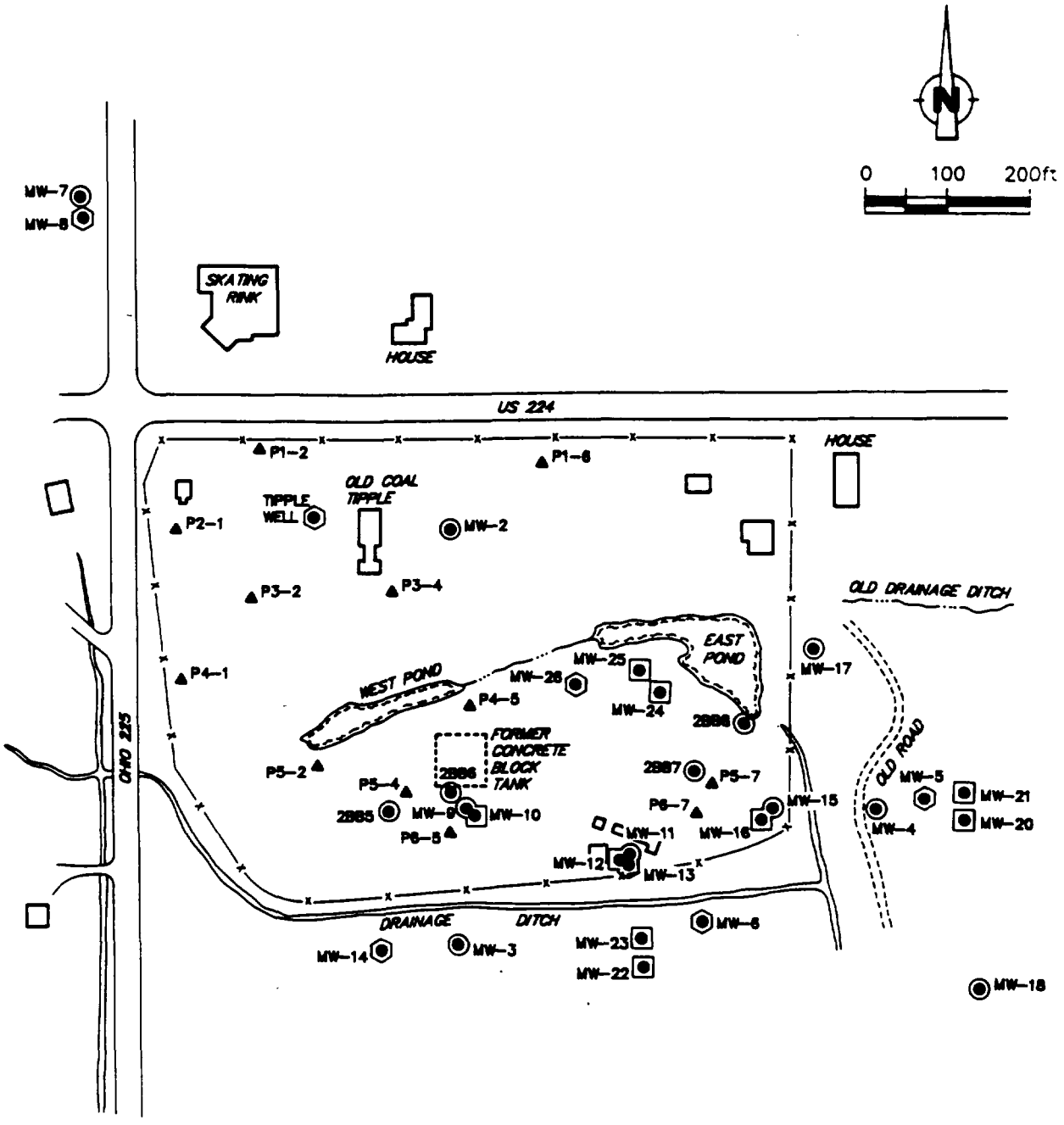
The bedrock profile will be utilized to complete the final design, elevation and slope of the pipe and media drain overburden groundwater collection system. In addition, soil samples will be collected from each borehole at the proposed elevation of the pipe bedding and at three equally spaced intervals above the pipe bedding elevation. These soil samples will be analyzed for grain size distribution to determine the gradation requirements of the bedding and granular backfill materials of the pipe and media drain, such that the potential for blinding of these materials by infiltration of fines from the adjacent native materials will be minimized.

All soil boring and sampling activities required to complete the bedrock profiling and grain size analyses will be conducted in accordance with the Site-specific HSP and SAP. Work zones will be established at each off-Site soil boring location as detailed in Section A.7.0 of the HSP.

4.8 GROUNDWATER CHARACTERIZATION

As stated in the SOW, the groundwater collection systems for the Site must effectively contain, collect and extract Site-related contaminated groundwater in the WTU and IU beneath and off the Site. The proposed locations of the pipe and media drain system for the WTU and the extraction well system for the IU, as presented in the SOW, are based on the groundwater quality data presented in the RI Report, which are based on groundwater samples collected during the RI investigative period of 1984 to 1986.

During the pre-design activities, it is proposed that one complete round of groundwater sampling and analyses and two complete rounds of water level measurements be performed, for all existing on- and off-Site groundwater monitoring wells, as shown on Figure 4.3. The groundwater samples will be analyzed for the TCL parameters, to provide a current definition of the nature and extent of Site-related contaminated groundwater. In addition, in conjunction with the groundwater treatability studies discussed in Section 4.10, it is proposed that pumping tests be conducted to better define the hydraulic characteristics of the aquifers. If conditions have changed, from those presented in the RI Report, that will necessitate revisions to the proposed groundwater extraction systems as presented in the SOW in order to achieve the containment, collection and extraction criteria for Site-related contaminated groundwater as required by the Consent Decree, separate work plans and designs will be prepared and submitted to USEPA and OEPA for review, modification and/or approval.



LEGEND

- ▲ WATER TABLE UNIT PIEZOMETER LOCATION
- ◉ WATER TABLE UNIT MONITORING WELL LOCATION
- ▣ INTERMEDIATE UNIT MONITORING WELL LOCATION
- ⬢ UPPER SHARON AQUIFER MONITORING WELL LOCATION

figure 4.3

EXISTING MONITORING WELL LOCATIONS
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

All groundwater sampling and analyses will be performed in accordance with the protocols described in the Site-specific HSP and SAP.

4.9 SURFACE WATER CHARACTERIZATION

As stated in the SOW, the west and east ponds on Site must be dewatered, and the drainage ditch along the south and east boundaries of the Site may require dewatering, to facilitate sediment removal. Such surface waters are to be treated by an on-Site water treatment system.

The most recent surface water quality data are presented in the RI Report, which are based on surface water samples collected during the RI investigative period of 1984 to 1986. During the pre-design activities, it is proposed that one composite surface water sample be collected from each pond, drainage ditch and impoundment, and analyzed for the TCL parameters to provide current surface water quality data. The current data then would be used to design the surface water treatment requirements. In addition, the volume of surface water in each area will be estimated, either by topographic survey or visual estimates, to provide an indication of the volume of surface water that may require treatment prior to the sediment removal activities to be conducted during the RA.

All surface water sampling and analyses will be performed in accordance with the protocols described in the Site-specific HSP and SAP.

4.10 GROUNDWATER TREATABILITY STUDIES

As stated in the SOW, extracted groundwater will be treated on-Site prior to discharge to the drainage ditch at the northeast corner of the Site. The groundwater treatment process, based on the groundwater quality data presented in the RI, as specified in the SOW, is to consist of clarification, sludge dewatering, filtration, air stripping and carbon adsorption.

The groundwater is expected to contain dissolved and suspended compounds which typically deposit or precipitate on groundwater treatment components, including air strippers and carbon filters. This deposition and precipitation typically adversely affects the performance of the treatment systems. Therefore, the SOW provides for groundwater treatability studies to be conducted during the pre-design phase.

The groundwater treatability studies will consist of both bench-scale treatability evaluations and pilot scale treatability evaluations. The treatability studies will be conducted on Site with the following components:

i) groundwater collection/storage;

ii) a trailer equipped with bench-scale test equipment including chemicals, glassware, drying equipment and weigh-scale equipment;

iii) a pilot air stripper unit; and

iv) a pilot carbon unit.

Prior to the conductance of the treatability studies, a complete round of groundwater samples will be collected from the existing monitoring wells as discussed in Section 4.8. All groundwater samples will be analyzed for TCL parameters and select samples will be analyzed for general water quality parameters. The results of these analyses will be used to develop a Site-specific parameter list (SSPL) for the analyses of water samples collected during the conductance of the treatability studies.

Groundwater required for the treatability studies will be pumped to an on-Site equilization tank from a combination of existing wells at the Site, at various flow rates, to provide an influent water quality consistent with the design basis for the treatment system. The specific wells and flow rates to be used will be based on the results of the groundwater characterization discussed in Section 4.8.

The pilot study unit will be tested at a flow rate of 5 to 10 gallons per minutes (gpm). During the pilot study program, the treated groundwater (7,200 to 14,400 gallons per day (gpd)) will be discharged through an activated carbon adsorption system and disposed of in accordance with all applicable or relevant and appropriate regulations.

During the treatment study program, bench-scale and pilot-scale tests will be conducted as follows:

i) Test 1: bench-scale jar test for treatment by clarification for solids/metals removal;

ii) Test 2: bench-scale test for treatment by filtration for solids removal;

iii) Test 3: bench-scale test for treatment by aeration for VOC removal solids precipitation;

iv) Test 4: bench-scale test for treatment by carbon adsorption; and

v) Test 5: pilot-scale test for treatment by a combination of air stripping and carbon filtration.

The procedures for bench-scale tests, consisting of pH adjustment, aeration, coagulation and filtration, are detailed in the Site-specific SAP.

After completion of the bench-scale tests, the pilot process will be operated for a period of three weeks (Test 6) at a liquid loading rate of 5 to 10 gpm with an optimal air to water ratio determined from the initial experimental tests. This longer duration test will be used to determine the impact potential of scale formation and biological growth conditions, if any.

Prior to commencement of the pilot process (Test 6), a step-drawdown pumping test consisting of three 30-minute steps at varying flow rates will be conducted on monitoring wells selected to be used during the pilot process treatability study. The tests will be performed to confirm that the selected monitoring wells are capable of producing the required flow rates for the treatability study as well as to better define the hydraulic characteristics

of the aquifers. The resulting groundwater will be pumped to an on-Site storage tank, pending treatment in the pilot groundwater treatment system prior to discharge off Site.

During the conductance of the pilot-scale treatability studies, the extracted groundwater will be treated by a combination of the pilot air stripper/carbon filter unit. The unit will be sized to treat a hydraulic capacity of up to 10 gpm. The final size and configuration of the pilot air stripper/carbon filter will be selected based on the results of the groundwater characterization discussed in Section 4.8.

During the pilot-scale treatability study, the effect of the liquid loading rate, air:water ratios and packing height on the removal process will be determined. Water loading rates will be tested at 20 and 30 gpm/ft^2 while air:water ratios will be tested at 30:1 and 100:1 for each respective flow rate. Samples will be collected at 2-foot intervals of packing and analyzed for the SSPL, along with influent and effluent samples.

During the extended test period, samples of the influent and effluent will be collected daily, for the first week, and then twice weekly for the remaining two weeks. At the end of the test run, the pilot unit will be shut down and inspected for signs of deposition, precipitation and biological growth.

The samples collected will be analyzed for the SSPL to be developed for the groundwater treatability studies. The rates of deposition,

precipitation and biological growth will be determined by visual inspection. As necessary, samples of deposits will be collected with follow-up analysis for compounds which appear (i.e. calcium, iron, sulphate). Sampling and analyses will be conducted according to the Site-specific SAP.

The volume of sludge and the volume of solids to be expected in the final groundwater treatment system will be determined from the results of the treatability studies. The final design of the sludge dewatering system (i.e. type, size, auxiliary equipment), will be based on the results of these treatability studies and in consultation with USEPA and OEPA. Dewatered materials may or may not be hazardous waste, and a final determination will be made on the basis of chemical analyses. Provisions for disposal of dewatered materials will be consistent with their chemical characterization and State and Federal requirements. During the conductance of the pilot testing, dewatered materials, if any, will be characterized and secured on Site in DOT approved hazardous waste drums, pending treatment during the RA.

The results of the pilot test will define the parameters to be used for the final design of the groundwater treatment system components. The standards of performance against which the pilot process system will be evaluated will be appropriate Best Available Treatment Technology (BATT) or water quality criteria, whichever are more stringent.

The groundwater treatability will be conducted in compliance with the Site-specific HSP.

In addition, during pumping from the selected wells for conductance of the pilot study, water levels will be measured in various monitoring wells in the vicinity of the pumped wells, to better define hydraulic parameters of the aquifers.

4.11 UPPER SHARON AQUIFER CHARACTERIZATION

As required by the SOW, all Upper Sharon well installation and development records produced during the RI will thoroughly be reviewed and, as stated in Section 4.8, two rounds of water level measurements will be collected from all existing RI wells completed in the Upper Sharon Unit. As required by the SOW, in the event that the groundwater flow direction in the Upper Sharon Unit is determined to be to the northeast, or cannot be conclusively determined, or if the Upper Sharon Aquifer is found to contain Site-related contamination through characterization of the Tipple well (see Section 4.6), one additional monitoring well will be installed in the Upper Sharon Unit in the northeast sector of the Site.

Should it be determined that an additional monitoring well is required in the Upper Sharon Unit, separate work plans and remedial designs will be prepared and submitted to USEPA and OEPA for review, modification and/or approval.

The water level measurements will be conducted in accordance with the Site-specific HSP and SAP.

4.12 INVESTIGATION OF SEDIMENTS

Sediment removal from the drainage ditch along the south and east perimeters of the Site and from the first impoundment southeast of the Site will be performed during the RA. As stated in the SOW, the depth of sediment removal will be based on the actual depth of sediment as determined visually.

To estimate the quantity of sediments to be removed during the RA, split-spoon samples of the sediments will be collected during the pre-design activities. The samples will be collected during dry weather conditions at 100-foot intervals along the center line or edges of the drainage ditch, and at a 100-foot grid spacing within the impoundment area. All locations will be staked and surveyed in accordance with the topographic survey protocols discussed in Section 4.3.

Sediment samples will be collected and classified as discussed for soil samples in the Site-specific SAP.

5.0 REMEDIAL DESIGN ACTIVITIES

5.1 GENERAL

RD activities generally consist of preparing engineering drawings, specifications, bid documents and supporting calculations for the various components of the RA. The design of each component of the RA also reflects the requirements imposed by all applicable local, State and Federal laws, regulations and permits. The design process is sometimes an iterative process where a preliminary design is developed and reviewed by appropriate regulatory Agencies before permit approval requirements, which affect the final design, can be incorporated into the final design.

The SOW requires documents in support of the design of the RA for the Site as follows:

i) design criteria document;
ii) preliminary design (30 percent complete);
iii) pre-final design (95 percent complete);
iv) final design (100 percent complete); and
v) remedial construction work plan.

These documents will be submitted as specified in the SOW and as presented in Section 6.0

Review of submittals by USEPA and OEPA and dispute resolution will be governed by the relevant sections of the Consent Decree.

5.2 PERMITTING REQUIREMENTS

As stated in the Consent Decree, USEPA and OEPA have determined that no Federal, State or local permits are required for work conducted entirely on-Site. Also, pursuant to § 121(e)(1) of CERCLA, 42 U.S.C. § 9621(e)(1), all permits or approvals necessary under Federal, State and local laws for off-Site work, including transport and/or disposal of waste materials removed from the Site, will be obtained by submitting timely applications and requests for any such permits and approvals.

During the initial design phases, each component of the remedy will be evaluated to determine which permit programs are potentially applicable. This will be accomplished through discussion with local, State and Federal Agencies. At this time, it is envisioned that the major permit programs to be considered are compliance with the substantive requirements for on-Site soil incineration (40 CFR Part 264, Standards for Owners and Operators of Hazardous Waste Treatment, Storage and Disposal Facilities), and off-Site inorganic liquids disposal (RCRA 40 CFR Part 264/265). As stated in Section 5.0 of Appendix D to the SOW, discharge of treated groundwater to surface water shall meet appropriate National Pollution Discharge Elimination System (NPDES) standards. Other permit programs

requiring consideration would be related to Site construction/demolition activities.

The design requirements imposed by the applicable permit programs will be incorporated in the pre-final and final design submissions.

5.3 DESIGN CRITERIA DOCUMENT

As required by the Consent Decree, a design criteria document will be prepared and presented to USEPA and OEPA for review and approval. The design criteria document will establish the basis for the design of the RA and will be based on the Consent Decree with specific reference to the SOW for the Site as presented in Appendix B to the Consent Decree, USEPA Superfund Remedial Design and Remedial Action Guidance, and any additional guidance documents provided to the SNFT by USEPA and OEPA.

5.4 PRELIMINARY DESIGN (30 PERCENT)

The preliminary or 30 percent design report consisting of construction plans and technical specifications for all aspects of the selected remedy will be submitted to USEPA and OEPA for review and approval. The preliminary design report will include verification of existing conditions and

the results of pre-design investigations, and will reflect the design effort at 30 percent completion with calculations that reflect the same percentage of completion as the designs they support. Supporting data and documentation will be provided with the design documents defining the functional aspects of the remedy. As well, the preliminary design report will address all technical requirements of the project so that these aspects may be reviewed to confirm that the final design will be consistent with the SOW and Applicable or Relevant and Appropriate Requirements (ARARs) and will provide an operable and usable RA. At the 30 percent design stage, an assessment of permitting requirements will be made. This will include the identification of:

i) construction/operating permits potentially required;
ii) the permitting authority;
iii) regulations governing applications, exemption and variances;
iv) information required by each permit application; and
v) time required by the permitting agency to process the application.

This information will be used to develop a program to assess the impact of the various permit programs on the design and to develop and submit the required applications.

At a minimum, the preliminary design submittal will include:

i) documentation of Site boundaries and topography (including Site cross-sections), and locations of utilities;

ii) discussion of utility and Site access requirements necessary to implement the RA;

iii) results from exploratory trenches in the vicinity of on-Site magnetic anomalies and delineation of the areal extent of excavation required for removal of buried containerized material;

iv) results of initial investigations of underground storage tanks in the northwest corner of the Site;

v) results of sampling and analysis of monitoring wells;

vi) results of the groundwater treatability study and an evaluation of the effectiveness of the proposed treatment process, including a review of permitting requirements to determine the necessity for conducting an air quality impact assessment;

vii) results of the groundwater pumping tests conducted concurrent with the groundwater treatability study;

viii) results of bedrock profiling and grain size distribution along the alignment of the proposed pipe and media drain;

ix) results of the review of the Upper Sharon Aquifer characterization;

x) results of the sampling and analysis of surface waters with an initial assessment of surface water treatment requirements;

xi) results of the off-Site sediment depth investigations;

xii) preliminary design of the soil incineration system directed toward developing specifications for the full scale rotary kiln treatment system, including evaluating the logistics of operating the equipment on the Site, the destruction removal efficiency to be achieved for individual constituents and the air quality limits to be imposed;

xiii) preliminary requirements and procedures for the on-Site incineration trial burn during the RA;

xiv) determination of the alignment and profile of the proposed pipe and media drain, and gradation requirements for the pipe and media drain backfill;

xv) preliminary design of the extracted groundwater transfer system, including pumphouses, electrical, mechanical and control requirements, forcemains and pump systems;

xvi) preliminary groundwater treatment system design based on the results of the groundwater treatability study, including component sizing and operation principles, and an assessment of the proposed treatment systems compliance with ARARs;

xvii) proposed design contours for the final Site cover including typical cross-sections reflecting the results of the topographical survey of the Site, material and vegetation specification for the Site cover, erosion control and gas venting measures;

xviii) preliminary plans and specifications, conceptual aspects of the design and preliminary construction drawings reflecting the design effort at 30 percent completion, as well as an assessment of conformance with ARARs and performance standards.

5.5 PRE-FINAL DESIGN (95 PERCENT)

A pre-final design report reflecting the 95 percent completion stage will be submitted for review and approval by the USEPA and OEPA. The pre-final design will have addressed all comments generated from the review of the preliminary design and will clearly show any modifications of the design resulting from incorporation of these comments.

The pre-final design will include, as a minimum, the following:

i) possible sources of error and references to potential problems and how potential problems may be resolved;

ii) details and extent of soil and sediment removal, including dewatering requirements for on- and off-Site ponds and ditches;

iii) designated spoil areas for excavated sediments, well boring cuttings and material from the pipe and media drain excavation;

iv) procedure for drum excavation, compatibility testing and characterization, and on and/or off-Site disposal requirements, including on-Site incineration;

v) technical specifications for the selected incinerator and disposal criteria for fly ash;

vi) details and operation procedures of all components of the groundwater extraction and collection systems;

vii) details and operation of all components of the extracted groundwater treatment system and confirmation of the treatment systems compliance with air emission and effluent discharge criteria, including the air quality impact assessment reflecting any air quality modelling, if required (as determined in the preliminary design submission);

viii) construction drawings and technical specifications suitable for bid purposes;

ix) construction schedule for implementation of the RA; and

x) assessment of conformance with ARARs, performance standards and permitting requirements.

5.6 FINAL DESIGN (100 PERCENT)

The final design report containing design plans and specifications at 100 percent completion will be submitted to USEPA and OEPA for review and approval. This submittal will clearly address all comments from these Agencies' review of the pre-final design. The final design will include, as a minimum, the following:

i) complete plans and specifications for both construction and bid purposes;

ii) complete design analyses, including design calculations;

iii) final construction schedule;

iv) construction cost estimate; and

v) assessment of conformance with ARARs, performance standards and permitting requirements.

The final design will reflect a level of effort such that the technical requirements of the RA selected for the Site have been addressed, and outlined such that it may be reviewed to determine substantial compliance with applicable requirements of the Consent Decree.

5.7 DRAFT RC WORK PLAN

A Draft RC Work Plan will be submitted to the USEPA and OEPA with the pre-final design submittal. The Draft RC Work Plan will detail the implementation of the RC tasks associated with the RA for the Site and will include the following:

i) draft RC HSP;
ii) draft RC SAP;
iii) draft RC QAPP;
iv) draft RC operation and maintenance plan for the remedial action;
v) schedule for implementation of the RC tasks; and
vi) schedule for submittal of RC reports.

The Draft RC Work Plan will be developed in accordance with the Consent Decree, USEPA Superfund Remedial Design and Remedial Action Guidance, and any additional guidance document provided to the SNFT by USEPA, as well as the design criteria document and the RD plans and specifications.

5.8 FINAL RC WORK PLAN

A Final RC Work Plan will be submitted to the USEPA and OEPA following USEPA's and OEPA's review of the Draft RC Work Plan. The Final RC Work Plan will reflect USEPA's and OEPA's comments on the Draft RC Work Plan and will include the following:

i) final plans, construction specifications and schedule for performance of the RA at the Site;

ii) final RC HSP;

iii) final RC SAP;

iv) final RC QAPP; and

v) final RC operation and maintenance plan for the remedial action.

The final RC Work Plan will be the guidance document used to govern the conduct of construction, sampling, monitoring, operation and maintenance of the RA for the Site.

6.0 REPORTS AND DOCUMENTATION

6.1 QUARTERLY PROGRESS REPORTS

Quarterly progress reports will be provided to USEPA and OEPA as required by the Consent Decree and will include the following major items:

i) description of the actions, including any corrective actions directed toward problem areas, which have been taken toward achieving compliance with the Consent Decree and RD Work Plan during the previous quarter;

ii) results of sampling and tests and all other data received by the SNFT during the course of the design activities during the previous quarter;

iii) all plans and procedures completed according to the RD Work Plan during the previous quarter;

iv) descriptions of all actions, data collection, and plans which are scheduled for completion during the next quarter;

v) information regarding percentage of completion, unresolved delays encountered or anticipated that may affect the future schedule for implementation of the RD Work Plan, a description of efforts made to

mitigate those delays or anticipated delays and any other plans required to be submitted pursuant to the Consent Decree; and

vi) identification of each failure during the previous quarter to attain any deadline of milestone identified in Paragraph XVIII.A of the Consent Decree.

The progress reports will be submitted by the tenth day of the first month of each calendar quarter, commencing with the second calendar quarter following the effective date of the Consent Decree, until the completion of all construction required under the RC Work Plan.

If the date for submission of any item or notification required by the Consent Decree falls upon a weekend or State or Federal holiday, the submission date will be extended until the next business day following the weekend or holiday.

6.2 EVENT OCCURRENCE REPORTING

Upon the occurrence of any event during performance of the RD Work Plan which, pursuant to § 103 of CERCLA, requires reporting to the National Response Center, the SNFT will promptly orally notify the USEPA Project Manager (RPM) and OEPA Project Coordinator, or in the event of the unavailability of the USEPA RPM, the Emergency Response

Section, Region V, USEPA and OEPA Office of Emergency Response, in addition to the reporting required by § 103.

Within 20 calendar days of the onset of such an event, the SNFT will furnish to USEPA and OEPA a written report setting forth the events which occurred and the measures taken, and to be taken, in response thereto.

Within 30 calendar days of the conclusion of such an event, the SNFT will submit a report setting forth all actions taken to respond thereto.

6.3 DESIGN CRITERIA DOCUMENT

A design criteria document, as presented in Section 5.0, establishing the basis for the design of the RA, will be submitted within 75 calendar days of the effective date of the Consent Decree.

6.4 PRE-DESIGN SUPPORTING DOCUMENTS

The supporting documents for the pre-design investigations include:

i) the HSP;

ii) the SAP and QAPP; and

iii) the RD Work Plan,

all as presented herein.

6.5 PRE-DESIGN TECHNICAL MEMORANDA

The preliminary results of the pre-design activities will be provided in a series of technical memoranda, to USEPA and OEPA, as the pre-design activities are completed. The final results and conclusions of the pre-design activities will be included as part of the preliminary design (30 percent) submission.

6.6 PRELIMINARY DESIGN REPORTS (30 PERCENT)

A preliminary design report reflecting 30 percent design completion will be submitted to USEPA and OEPA within 60 calendar days of final approval of the Design Criteria Document by USEPA and OEPA. The requirements of the preliminary design report are presented in Section 5.4.

6.7 PRE-FINAL DESIGN REPORT (95 PERCENT)

The pre-final design report reflecting 95 percent design completion will be submitted to USEPA and OEPA within 90 calendar days of

final approval of the preliminary design report by USEPA and OEPA. The requirements of the pre-final design report are presented in Section 5.5.

6.8 FINAL DESIGN REPORT (100 PERCENT)

The final design report reflecting 100 percent design completion will be submitted to USEPA and OEPA within 30 calendar days of final approval of the pre-final design report by USEPA and OEPA. The requirements of the final design report are presented in Section 5.6.

6.9 DRAFT RC WORK PLAN

A Draft RC Work Plan as required by the Consent Decree and as detailed in Section 5.7, will be submitted to USEPA and OEPA with the pre-final (95% complete) design submittal within 90 calendar days following approval of the pre-design report (30% complete) by USEPA and OEPA.

6.10 FINAL RC WORK PLAN

The Final RC Work Plan, as required by the Consent Decree and as detailed in Section 5.7, will be submitted to USEPA and OEPA with the final design report (100% complete) within 30 days following receipt of USEPA's and OEPA's comment on the Draft RC Work Plan.

7.0 COMMUNITY RELATIONS

The SNFT will cooperate with USEPA and OEPA in providing information to the public. As requested by USEPA and OEPA, the SNFT will participate in the preparation of and provide technical information to be disseminated to the public or to be used in public meetings which may be held or sponsored by USEPA or OEPA to explain activities at or concerning the Site. The SNFT may take part in such meetings.

8.0 SCHEDULE

Figure 8.1 presents the preliminary schedule for the RD.

Scheduling of field activities such as the pre-design investigation are subject to weather considerations and may be modified as the project develops.

The schedule assumes a review and approval period for submittals as presented in the SOW. Longer review and approval periods will require similar schedule extensions.

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The item(s) listed below are not available in SDMS. In order to view original document or document pages, contact the Superfund Records Center.

SITE NAME	Summit National		
DOC ID #	128553		
DESCRIPTION OF ITEM(S)	Remedial Design Work Plan (Proposed Rd Schedule)		
REASON WHY UNSCANNABLE	_X_OVERSIZED	OR	___ FORMAT
DATE OF ITEM(S)	October 1991		
NO. OF ITEMS	1		
PHASE	Remediation		
PRP	Summit Nation Liquid Services		
PHASE (AR DOCUMENTS ONLY)	___ Remedial ___ Removal ___ Deletion Docket ___ AR ____ Original ____ Update # ____ Volume ____ of ___		
O.U.			
LOCATION	Box # _2_ Folder # _6_ Subsection _K,N_		
COMMENT(S)			

A

APPENDIX A
HEALTH AND SAFETY PLAN

SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

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A.1.0 GENERAL

The pre-design investigation program to be implemented to satisfy the pre-design requirements, as presented in the Statement of Work (SOW) of the Consent Decree for the Summit National Superfund Site (Site), will involve investigations on the Site and in the immediate vicinity of the Site. During these operations personnel may come in contact with soils, groundwater, sludge and/or sediments which potentially contain hazardous wastes or hazardous waste constituents. To ensure that any direct contact with potentially contaminated material by Site-personnel is minimized, a Site-specific Health and Safety Plan (HSP) has been developed, as presented herein. This HSP has been developed to ensure the following:

i) that Site-personnel are not adversely exposed to the compounds of concern;

ii) that public welfare or the environment are not adversely impacted by off-Site migration of contaminated materials due to work activities at the Site;

iii) compliance with applicable governmental and non-governmental [American Conference of Governmental Industrial Hygienist (ACGIH)] regulations and guidelines. In particular, the amended rules of the Occupational Safety and Health Act for Subpart H of Part 1910 (Title 29 Code of Federal Regulations (CFR) Part 1910.120) will be implemented for all Site work; and

iv) initiation of proper emergency response procedures to minimize the potential for any adverse impact to Site workers, the general public or the environment.

For the purpose of this HSP all sampling and investigative activities performed on Site involving contact with potentially contaminated materials will be considered contaminated operations requiring personal protective equipment (PPE). Similar activities occurring off Site are considered non-contaminated operations requiring a modified level of PPE from that for on-Site work. A detailed description of the PPE required is presented in Section A.8.

All sampling and investigative activities at the Site will be conducted in accordance with the provisions of this Site-specific HSP. Cost and/or scheduling considerations will not be considered as justification for modifying this plan. A copy of this HSP and applicable Material Safety Data Sheets (MSDS) will be maintained on Site whenever Site activities associated with the pre-design or design activities are in progress.

A.2.0 SITE CHARACTERIZATION AND POTENTIALLY HAZARDOUS COMPOUNDS

A Site-specific data base has been compiled for the Site, as a result of the Remedial Investigation (RI) performed by CH_2M Hill on behalf of the United States Environmental Protection Agency (USEPA). The chemicals identified during the RI on or in the vicinity of the Site are summarized in Table A.1.

TABLE A.1

CHEMICALS IDENTIFIED DURING THE RI ON OR IN THE VICINITY OF THE SITE (1) SUMMIT NATIONAL SUPERFUND SITE DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Volatile Organics	*Base Neutral and Acids*		*Pesticides and PCBs*	*Inorganics*
Vinyl Chloride	N-Nitrosodimethylamine	Diethylphthlate	Alpha-BHC	Aluminum
Methylene Chloride	Phenol	Fluorene*	Beta-BHC	Antimony
Acetone	Aniline	N-Nitrosodiphenylamine*	Delta-BHC	Arsenic
Carbondisulfide	1,3-Dichlorobenzene	Hexachlorobenzene	Gamma-BHC	Barium
Benzene	1,4-Dichlorobenzene	Pentachlorophenol	Heptachlor	Beryllium
2-Hexanone*	Benzyl Alcohol	Phenanthrene*	Aldrin	Cadmium
4-Methyl-2-Pentanone*	1,2-Dichlorobenzene	Anthracene*	Heptachlor Epoxide	Calcium
Tetrachloroethene	2-Methylphenol	Di-n-Butylphthlate	Endosulfan	Chromium
Toluene	4-Methylphenol*	Fluoranthene**	Dieldrin	Cobalt
Chlorobenzene	Hexachloroethane	Pyrene*	Endrin	Copper
Ethylbenzene	Isophorone	Butylbenzylphthalate*	4,4'-DDT	Iron
Styrene	2-Nitrophenol	Benzo (a) Anthracene	Toxaphene	Lead
Total Xylenes	2,4-Dimethylphenol*	Bis (2-ethylhexyl) Phthalate	Aroclor 1232***	Cyanide
1,1-Dichloroethene	Benzoic Acid	Chrysene	Aroclor 1242***	Magnesium
1,1-Dichloroethane	1,2,4-Trichlorobenzene	Di-n-Octyl Phthalate*	Aroclor 1248***	Manganese
1,2-Dichloroethane	Naphthalene*	Benzo (b) Fluoranthene**	Aroclor 1254***	Mercury
Trans-1,2-Dichloroethene	4-Chloro-3-Methylphenol	Benzo (k) Fluoranthene**	Mirex	Nickel
Chloroform	Hexachlorocyclopentadiene	Benzo (a) Pyrene**		Potassium
2-Butanone*	Dimethylphthalate	Indeno (1,2,3-cd) Pyrene**		Selenium
1,1,1-Trichloroethane	Acenaphthylene	Dibenzo (a,h) Anthracene**		Silver
1,1,2,2-Trichloroethane	Acenaphthene	Benzo (g,h,i) Perylene*		Sodium
Trichloroethene	4-Nitrophenol			Thallium
1,1,2-Trichloroethane	Dibenzofuran*			Tin
				Vanadium
				Zinc

Notes:

(1) Reproduced from Table 6-2 of the "Final Remedial Investigation Report", Volume 1, Summit National Site, Deerfield, Ohio, dated February 10, 1988.

* Lacks chronic toxicity data (USEPA, 1986a).

** Carcinogenic polynuclear aromatic hydrocarbons (PAHs) for which the potency factor for Benzo (a) Pyrene applies.

*** Potency factor for polychlorinated biphenyls (PCBs) applies to sum of Aroclors.

A.3.0 BASIS

The Occupational Safety and Health Administration (OSHA) Standards and Regulations contained in Title 29, CFR, Parts 1910 and 1926 (29 CFR 1910 and 1926), including the amended sections in 29 CFR 1910.120 and current Threshold Limit Values (TLVs) as provided by the ACGIH, provide the basis for this HSP. Some of the specifications within this section are in addition to OSHA regulations and reflect the positions of USEPA, the National Institute for Occupational Safety and Health (NIOSH) and the United States Coast Guard (USCG) regarding procedures required to ensure safe operations at potential hazardous waste sites. In addition, the following references have been used as guidance in developing the HSP:

i) CERCLA Sections 104(f) and 111(c) (6);

ii) EPA Order 1440.2 - Health and Safety Requirements for Employees Engaged in Field Activities;

iii) EPA Order 1440.1 - Respiratory Protection;

iv) EPA Occupational Health & Safety Manual

v) EPA Interim Standard Operating Safety Guide (September 1982);

vi) OSHA Standards for General Industry;

vii) NIOSH, Manual of Analytical Methods, Volumes I-VII;

viii) Threshold Limit Values (TLV) for Chemical Substances and Physical Agents in the Work Environment with Intended Changes Adopted by ACGIH;

ix) ANSI Z 88.2-1980, American National Standard, Practices for Respiratory Protection;

x) Air Sampling Instruments for Evaluation of Atmospheric Contaminants, 6th edition, 1983, American Conference of Governmental Industrial Hygienists;

xi) Appropriate health and safety statutes; and

xii) Superfund Public Health Evaluation Manual, U.S. EPA (October 1986).

The safety and health of the public and on-Site personnel and the protection of the environment will take precedence over cost and schedule considerations for all project work. Each employer will designate an on-Site individual as Site Safety Officer (SSO) during on-Site pre-design and design activities. The SSO will be responsible for decisions regarding when work will be stopped or started for health or safety considerations. Each employer will develop a Site-specific HSP related to its specific activities at the Site. Each employer's Site-specific HSP will be reviewed by Conestoga-Rovers & Associates (CRA) for completeness and compliance with the minimum standards set forth in this HSP, prior to commencing work involving contact with potentially contaminated materials at the Site.

A.4.0 RESPONSIBILITIES AND ADMINISTRATION

The SSO will supervise the implementation of the HSP and will be responsible for all decisions regarding operations and work stoppages due to health and safety considerations.

The responsibilities of the SSO will be as follows:

i) be responsible for implementation of the HSP at the initiation of Site work;

ii) conduct the initial briefing sessions for all on-Site personnel with regard to the HSP and other safety requirements to be observed during field sampling, including:

   a) potential hazards, including heat/cold stress,
   b) personal hygiene principles,
   c) PPE,
   d) respiratory protection equipment usage, and
   e) emergency procedures dealing with fire and medical situations;

iii) review and modify the HSP as more information becomes available concerning the hazardous materials involved, and review all monitoring reports;

iv) supervision and enforcement of safety equipment usage;

v) supervision and inspection of equipment cleaning;

vi) personnel training in safety equipment usage and emergency procedures;

vii) monitoring of the health and safety program under the direction of an industrial hygienist;

viii) suspend work activity if unsafe working conditions develop, including unsafe working conditions generated by adverse wind, rain and lightning;

ix) inform workers of the nature of chemical exposure risk as required by the "Right-to-Know" Law;

x) recommend medical examination when worker appears to require it;

xi) coordination of emergency procedures; and

xii) maintain a sign in/out log for personnel and visitors.

A.5.0 MEDICAL SURVEILLANCE

In accordance with requirements detailed in 29 CFR 1910.120 and 29 CFR 1910.134, all Site personnel who will come in contact with potentially contaminated materials will have received, within one year prior to starting field activities, medical surveillance by a licensed physician or physician's group.

Medical records for all Site personnel who will come in contact with potentially contaminated materials will be maintained by their respective employers. The medical records will detail the tests that were taken and will include a copy of the consulting physician's statement regarding the tests and the employee's suitability for work. These medical records must be available to the employee or his designated representative upon written request, as outlined in 29 CFR 1910.120, Section (f).

Each employer will ensure and certify to the SSO that its personnel involved in activities associated with potentially contaminated materials will have had all necessary medical examinations prior to commencing work within contaminated areas. Personnel not obtaining medical certification and who do not have their records up-to-date will not perform work within contaminated areas.

Interim medical surveillance will be completed if an individual exhibits poor health or high stress responses due to on-Site activity or when accidental exposure to elevated concentrations of contaminants occurs.

A.6.0 TRAINING

The SSO will require that all Site personnel who will come in contact with potentially contaminated materials complete training sessions in accordance with 29 CFR 1910.120 prior to entering the Site. This training shall consist of a minimum of 40 hours of instruction off Site and three days of actual field experience under direct supervision. Each employer will maintain documentation stating that its on-Site personnel have complied with this regulation.

Prior to commencing Site activities, all personnel will be required to attend a Site-specific initiation session. These sessions, conducted by the respective employer's SSO, will be used to instruct the Site personnel as to what the potential Site hazards are, level of PPE required, Site-specific requirements, and the basis of the HSP. At this session it will be confirmed that all on-Site personnel have the 40 hours of training required in accordance with 29 CFR 1910.120. All personnel who attend this session will sign a Training Acknowledgement Form, an example of which is presented as Attachment A.1.

A.7.0 WORK AREAS

All Site work zones will be clearly laid out and identified in the field prior to initiation of all Site work activities. The establishment of all the Sites work zone boundaries may be modified by the SSO and/or Site Coordinator. The purpose of establishing these work zones is to limit access to potentially contaminated areas or areas which may cause physical injury to individuals and to prevent the migration of potentially hazardous materials into adjacent non-contaminated areas. These designated work zones are described as follows:

i) Exclusion Zone (EZ) - this zone will include all areas where potentially contaminated soils, sediments, waters or waste material are located, excavated, removed, transfered, stored, or disposed of and all areas where contaminated equipment or personnel travel including the decontamination area. Specifically the EZ will include all areas designated for soil and sediment removal as shown on Figures 2 to 6 inclusive, of Appendix A of the SOW. Figures 2 to 6 are reproduced in Attachment A-2 for ease of reference. Sufficient area will be provided for efficient movement of personnel and equipment as well as contaminant control. The EZ will be delineated in the field with fencing, flagging or warning barriers. Access to the EZ will be restricted to personnel who are wearing the proper PPE, have received and passed the required medical examination and have undergone the required health and safety training. A log of entry to and exit from the

EZ will be maintained for the purpose of stress monitoring and determining exposure times;

ii) Contamination Reduction Zone (CRZ) - this zone lies immediately upwind of the EZ and occurs at the interface of the EZ and Support Zone (SZ) and will provide for the transfer of construction materials, personnel and equipment, a place for donning and disposing of PPE, the storing of emergency first aid equipment and supplemental safety supplies. Access to the CRZ will be restricted to personnel who have received and passed the required medical examination and have undergone the required health and safety training; and

iii) Support Zone (SZ) - this area is comprised of the remainder of the designated work area at the Site and is defined as being an area outside the zone of significant air, soil or surface water contamination. The SZ will be clearly delineated and procedures will be implemented to prevent active or passive contamination from the other work zones. The function of the SZ includes:

a) an entry area for personnel, material and equipment to the CRZ,
b) an exit for decontaminated personnel, materials and equipment from the CRZ,
c) a storage area for, clean safety and work equipment; and
d) an area for eating, drinking and smoking.

A.8.0 PERSONAL PROTECTIVE EQUIPMENT(PPE)

The SSO will require that all personnel are equipped with PPE appropriate for the nature of work being completed. The SSO will require that all safety equipment and protective clothing are kept clean, well-maintained and that their integrity is intact.

Safety equipment and apparel as required for general work on Site will be Level D and/or Level C, or Level B protective equipment if air monitoring results indicate the need as discussed in Section A.9.0. The level of protection as well as the anticipated hazards/risks for the anticipated pre-design and design Site activities are presented in Table A.2.

Level B PPE which will be used on-Site consists of the following:

i) supplied-air respirator (MSHA/NIOSH approved). Respirators may be positive pressure-demand, self-contained breathing apparatus (SCBA) or positive pressure-demand, airline respirator (with escape bottle for Immediate Danger to Life and Health (IDLH) or potential for IDLH atmosphere);

ii) chemical-resistant clothing (overalls and long-sleeved jacket; hooded one or two-piece chemical-splash suit; disposable chemical-resistant; one-piece suits);

iii) long cotton underwear or long-sleeved shirt and pants;

TABLE A.2

LEVEL OF PERSONNEL PROTECTIVE EQUIPMENT
PRE DESIGN AND DESIGN ACTIVITIES
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Work Activity	*Anticipated Hazards/Risks*	*Level of PPE Required*
Site Preparation Activities including Land Surveying	• slip/trip/fall hazards • potential back injuries from lifting and moving • direct contact with contaminated soil and/or water	Level D
Soil/Sediment Excavation, Trenching Operations, Tipple Well Characterization	• slip/trip/fall hazards • potential personnel injuries • direct contact with contaminated soil, sediment and water • potential dust and/or vapor emissions • potential contact with unknown waste materials • hazards presented by the use of heavy equipment	Level C or Level B if air monitoring results indicate the need
Drum Removal and Handling; if required	• slip/trip/fall hazards • potential personnel injuries • direct contact with contaminated soil, sediment and water • potential dust and/or vapor emissions • potential contact with unknown waste materials • hazards presented by the use of heavy equipment and drum removal equipment	Level B
Sample Collection Activities (soil, sediment, surface water, groundwater)	• slip/trip/fall hazards • potential personnel injuries • direct contact with contaminated soil, sediment and water • potential dust and/or vapor emissions • hazards presented by the use of heavy equipment and drum removal equipment	Level C or Level B if air monitoring results indicate the need

TABLE A.2

LEVEL OF PERSONNEL PROTECTIVE EQUIPMENT
PRE DESIGN AND DESIGN ACTIVITIES
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNHSIP OF PORTAGE COUNTY, OHIO

Work Activity	*Anticipated Hazards/Risks*	*Level of PPE Required*
Groundwater Treatability Study	• slip/trip/fall hazards • potential personnel injuries • potential contact with contaminated soil, sediment and water • potential dust and/or vapor emissions • hazards presented by the use of sampling equipment • potential contact with an unknown waste	Level C or Level B if air monitoring results indicate the need
Decontamination Activities	• slip/trip/fall hazards • potential personnel injuries • potential contact with contaminated soil, sediment and water, waste material and cleaning solvents • potential vapor emissions • hazards presented by the use of specialized decontamination equipment (i.e. steam cleaning unit)	Level C or Level B if air monitoring results indicate the need

iv) gloves (outer), chemical-resistant;

v) gloves (inner), chemical-resistant;

vi) boots (outer), chemical-resistant, steel toe and shank;

vii) boot covers (outer), chemical-resistant (disposable);

viii) hard hat (face shield); and

ix) 2-way radio communications (intrinsically safe).

Level C PPE which will be used on Site consists of the following:

i) air-purifying respirator, half-face or full-face (MSHA/NIOSH approved), equipped with organic vapor/acid gas (OV/AG) cartridge with a high efficiency particulate air (HEPA) filter;

ii) chemical-resistant clothing (coveralls; hooded one-piece or two-piece chemical splash suit; chemical-resistant hood and apron; disposable chemical-resistant coveralls);

iii) long cotton underwear or long-sleeved shirt and pants;

iv) gloves (outer), chemical-resistant;

v) gloves (inner), chemical-resistant;

vi) boots (outer), chemical-resistant, steel toe and shank;

vii) boot covers (outer), chemical-resistant (disposable);

viii) hard hat (face shield);

ix) escape mask; and

x) 2-way radio communications (intrinsically safe).

Level D PPE which will be used on-Site consists of the following:

i) coveralls;
ii) gloves;
iii) boots/shoes (leather or chemical-resistant) with steel toe and shank;
iv) safety glasses; and
v) hard hat.

Additional protective equipment usage guidelines to be implemented include:

i) prescription eyeglasses in use on the Site will be safety glasses;

ii) contact lenses will not be permitted;

iii) all disposable or reusable nitrile, latex and/or cotton gloves worn on the Site will be changed, decontaminated or discarded at the end of each day;

iv) during periods of respirator usage, respirator cartridges and filters will be changed daily, or upon breakthrough, whichever occurs first;

v) on-Site personnel who have not passed a respirator fit test will not be permitted to enter or work in the EZ. Personnel will not be permitted to have beards, or long sideburns or mustaches as these interfere with a proper fit of the respirator;

vi) all PPE worn on Site will be decontaminated or discarded at the end of each work day;

vii) duct tape will be used to ensure that disposable coveralls and gloves are tightly secured when personnel are working within the EZ; and

vii) no watches, rings or other accessories will be permitted during drilling and sampling activities.

A.9.0 RESPIRATORY PROTECTION PROGRAM

Prior to arriving at the Site, all on-Site personnel will have received training in the use of, and have been fit tested for, either half- or full-facepiece respirators. All on-Site personnel will be required to comply with their employer specific written respiratory protection program developed in accordance with OSHA 29 CFR 1910.134.

The HNu Photoionization Detector (HNu) will be used in a survey mode to determine if organic vapors are present. A background reading will be established prior to commencing work activities at each monitoring well, sampling location or work area.

Action levels to determine the level of respiratory protection necessary during field activities are based on the concentration of unknown organic vapors measured within the breathing zone. The action levels and appropriate respiratory protection for Site activities are as follows:

HNu Sustained Organic Vapor Reading Above Background within Breathing Zone	*Action Taken*
0 or background	half- or full-facepiece air purifying respirator available
1 - 5 ppm	half- or full-face air purfying respirator
5 - 50 ppm	supplied air respirator

>50 ppm	shut down activities, implement additional engineering controls or remedial action

However, if the ambient concentrations of organic vapors are due to identifiable substanaces, the level of respiratory protection may be altered by the SSO.

Periodic air monitoring data may be obtained to correlate with total organic vapor readings from which the level of respiratory protection may be adjusted.

A.10.0 PERSONAL HYGIENE

The SSO will require that all personnel performing or supervising work within the EZ observe and adhere to the personal hygiene-related provisions of this section.

On-Site personnel found to be disregarding the personal hygiene-related provisions of this HSP will, at the discretion of the SSO, be barred from the Site.

Each employer will ensure that the following equipment/facilities are available for the personal hygiene of its on-Site personnel:

i) suitable disposable outerwear, gloves, respiratory protection and footwear on a daily basis for the use of its on-Site personnel;

ii) disposal containers for used disposable outerwear; and

iii) potable water and a suitable sanitation facility.

The SSO also will enforce the following regulations for personnel actively participating in the on-Site pre-design and design activities:

i) on-Site personnel will wear appropriate PPE when in the EZ;

ii) used disposable outerwear will not be reused if deemed to be unsuitable to provide the necessary protection, and when removed, will be placed inside disposal containers provided for that purpose;

iii) smoking, eating and drinking will be prohibited within the EZ and the CRZ. These activities will be permitted only within the area of the SZ; and

iv) on-Site personnel will thoroughly cleanse their hands, face, neck area and other exposed areas before smoking, eating, drinking or using toilets and before leaving the Site.

A.11.0 AIR MONITORING

During the progress of active work, air quality on Site will be monitored. Monitoring will be conducted on a daily basis and additionally as required by special or work-related conditions. Monitoring equipment will be calibrated on a daily basis in accordance with the manufacturers specifications, and such calibrations will be recorded in the Site daily log book.

The daily monitoring program will consist of monitoring with an HNu equipped with an 11.7 eV probe for volatile organic vapors.

Immediately upon identifying elevated levels of volatile vapors (greater than 5 parts per million), the air monitoring results shall be reported to the SSO, who will determine when PPE should be upgraded or operations shut down and restarted. Air monitoring will be performed within the breathing zone at each active work site. It will be re-evaluated if sustained levels greater than 5 ppm in the breathing zone exist.

The results of the air monitoring program will be recorded in a log book which will be maintained on Site.

A.12.0 COMMUNICATIONS

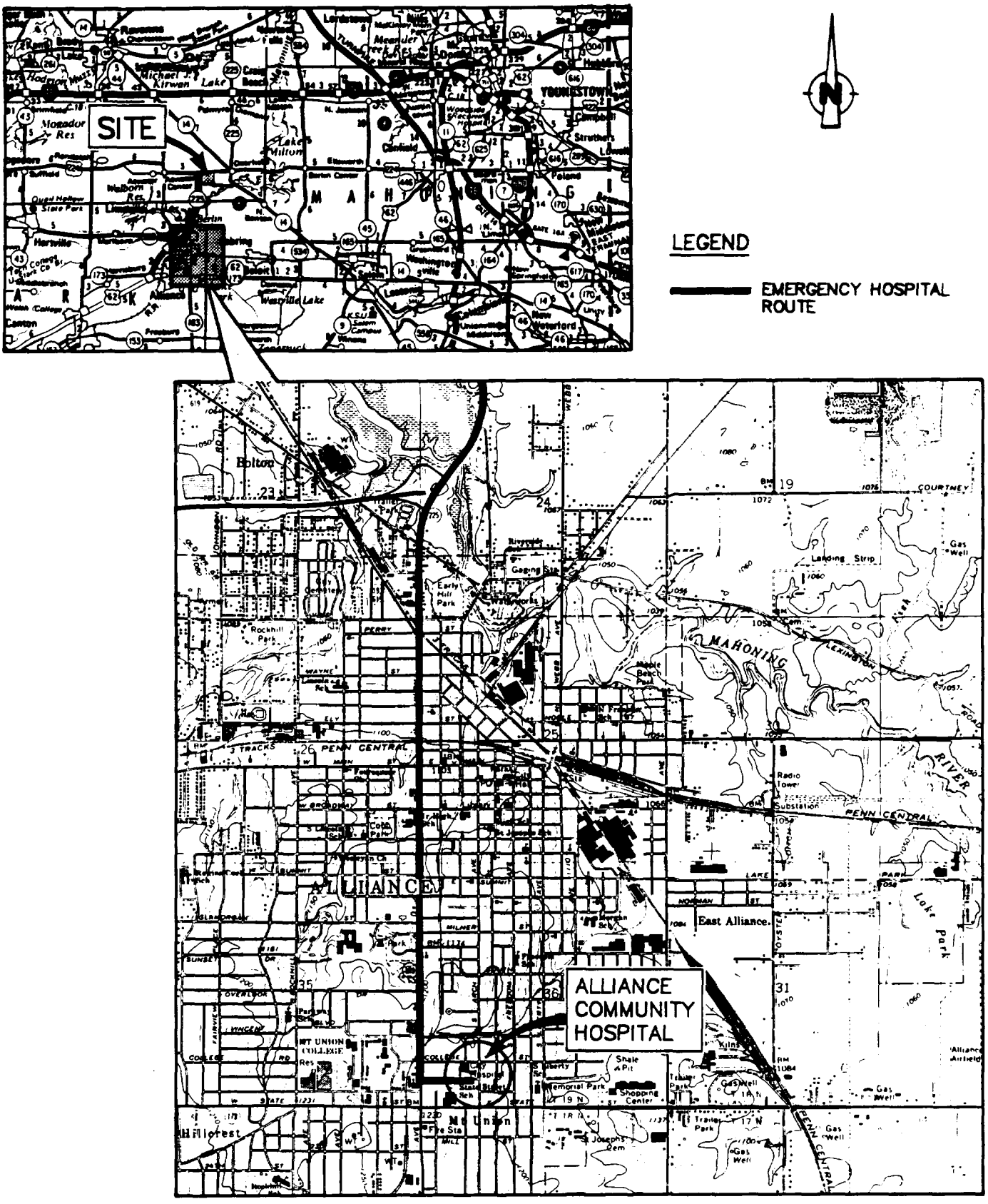
Emergency numbers including police, fire, ambulance, hospital and appropriate Regulatory Agencies are presented in Table A.3 and will be prominently posted near the telephone(s).

A route map to the nearest emergency medical facility is presented in Figure A.1 and will be posted in each vehicle. Prior to initiating Site activities, the emergency medical facility will be notified of Site activities to ensure preparedness to respond to any Site-related injuries.

TABLE A.3

EMERGENCY TELEPHONE NUMBERS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

General Emergency (Portage County Sheriff's Dept.)	911 or (216) 678-7012
Ambulance	911 or 584-2222
Hospital (Alliance Community Hospital)	(216) 821-1000
Ohio DOH (Akron)	(216) 379-1300
Ohio EPA (District)	(216) 425-9171
Ohio EPA Local Air Agency (Regional - Akron)	(216) 375-2480
Poison Control Center	(800) 962-1253
USEPA National Response Center	(800) 438-2427
State Police	(216) 297-1441
Portage County Health Department	(216) 296-9919



SOURCES: OHIO OFFICIAL TRANSPORTATION MAP, 1987
AND USGS QUADRANGLE MAP; ALLIANCE, OHIO

figure A.1

EMERGENCY HOSPITAL ROUTE
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

A.13.0 EMERGENCY AND FIRST AID EQUIPMENT

Emergency safety equipment will be available for use by Site personnel and will be located and maintained on Site. The safety equipment will include, but is not limited to, the following:

i) portable emergency eye wash;

ii) two 20-pound ABC type dry chemical fire extinguishers;

iii) approved first-aid kit for a minimum of 10 personnel;

iv) fire blanket;

v) two SCBA units;

vi) portable air horn.

A.14.0 EMERGENCY RESPONSE PLAN

Prior to commencing work, the SSO will coordinate the development of an emergency contingency plan. The plan is intended to provide immediate response to a serious Site occurrence such as injury, explosion or fire.

In the event of injury to on-Site personnel, the following protocol will be followed:

i) activate Site Alarm (three short blasts on air horn);

ii) notify the SSO;

iii) contact the designated hospital and describe the injury;

iv) decontaminate personnel if possible, and administer appropriate first aid. If personnel cannot be decontaminated, alert hospital to possible problems of contamination; and

v) transport personnel to the medical facility along a predefined route.

In the event of a significant release of toxic or hazardous vapors, the source of such vapors shall be immediately isolated, the material identified, and the SSO notified. If possible, the source of the vapors shall be controlled. All personnel shall utilize SCBA during such operations. Continuous air monitoring of the area shall commence. Appropriate

regulatory and emergency agencies will be notified of the situation. A list of the phone numbers for each agency is presented in Table A.3 which will be prominently posted near each telephone.

A.15.0 EQUIPMENT AND PERSONNEL DECONTAMINATION

During the initiation of the field investigative program, procedures will be implemented to reduce the amount of contact of both personnel and equipment with the waste constituents. These procedures include the following:

i) proper work practices that would lead to minimal direct contact with potentially contaminated material (e.g. avoid contact in areas of obvious contamination, remote sampling and handling procedures, etc.);

ii) use of disposable equipment and clothing as much as practicable; and

iii) encase source of contaminants (with plastic sheeting).

All equipment leaving the EZ which came in contact with potentially contaminated material will be decontaminated, as specified in the Site-specific SAP, within that part of the Site designated for capping.

Personnel decontamination will take place at the exit from the EZ and will, as a minimum, consist of a glove wash with detergent and removal of gloves, disposable suit and hard hat.

All personnel will remove their protective clothing and wash their hands, face, neck area and other exposed areas before entering the lunch and break areas to eat, drink, smoke or use the toilet facilities.

A.16.0 CONTAMINATION MIGRATION CONTROL

All vehicles and equipment used within the EZ will be decontaminated on Site within the area of the Site to be capped as determined necessary by the SSO prior to leaving the Site. Decontamination, when required, will consist of the thorough cleaning, using a high pressure cleaner, of those parts of the equipment which come in contact with potentially contaminated material. A temporary decontamination pad will be constructed at the double gate entrance to the Site from US Route 224, and will be utilized for final decontamination of vehicles and equipment prior to leaving the Site. The SSP will certify that each piece of equipment is clean or has been decontaminated prior to removal from Site.

Personnel engaged in vehicle decontamination will wear protective equipment including suitable disposable clothing, respiratory protection and face shields.

Sampling equipment wash water, purge water, well development water and water generated from opening and cleaning of the Tipple well will be collected and stored in an on-Site tank pending treatment at the pre-design pilot groundwater treatment system or at the groundwater treatment system to be installed during the RA, prior to discharge. Decontamination solvents will be collected and stored on Site in drums pending disposal during the RA.

ATTACHMENT A-1
TRAINING ACKNOWLEDGEMENT FORM

SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

TRAINING ACKNOWLEDGEMENT FORM

Please Print:

NAME: _____

ADDRESS: _____

SOCIAL SECURITY NUMBER: _____

EMPLOYER: _____

JOB SITE: _____

I have attended and understood the mandatory Site-specific initiation session for the above-referenced job site. This program referenced the following topics:

i) known potential hazards on Site;

ii) level of personal protection equipment required;

iii) emergency procedures for the Site; and

iv) the basics of the Site-specific Health and Safety Plan.

I further confirm that I have the required 40 hours of training to comply with 29 CFR 1910.120 and have a respirator for which I have been fit tested.

_____ Date

_____ Signature

ATTACHMENT A-2

FIGURES 2 TO 6 OF APPENDIX A OF THE STATEMENT OF WORK

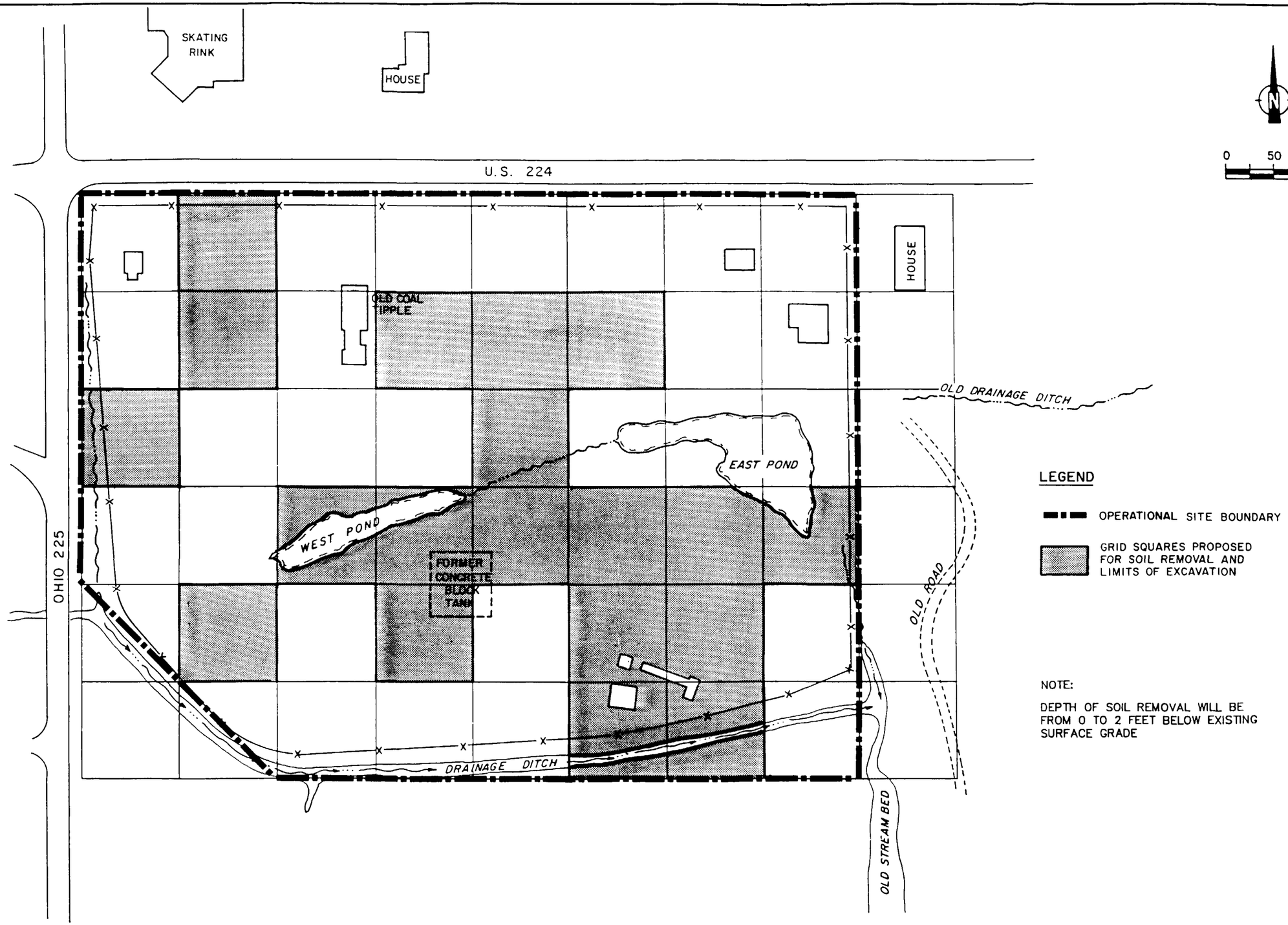
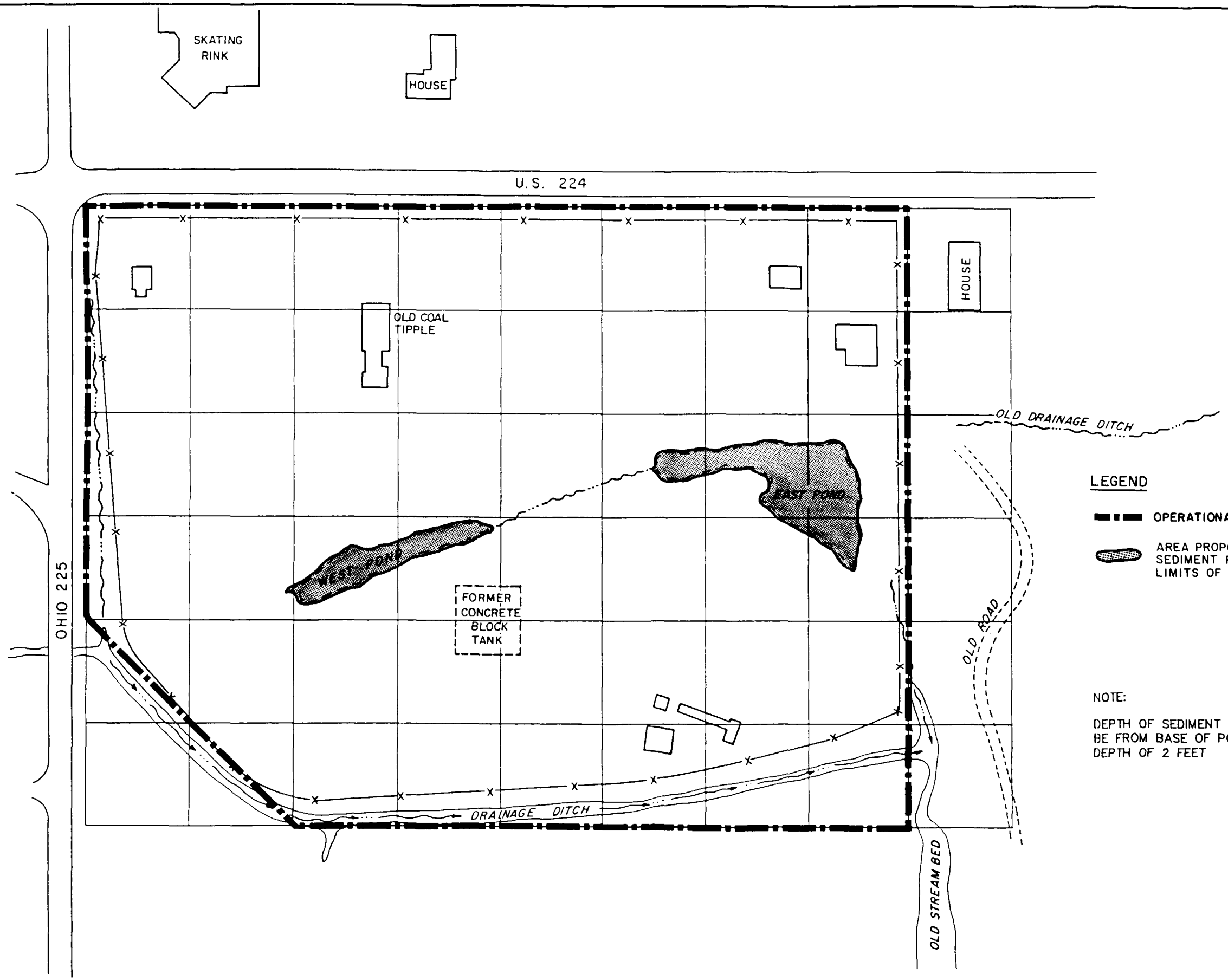


figure 2

PROPOSED ON-SITE SURFICIAL SOIL REMOVAL
Summit National Site

CRA



LEGEND

OPERATIONAL SITE BOUNDARY

AREA PROPOSED FOR SEDIMENT REMOVAL AND LIMITS OF EXCAVATION

NOTE:

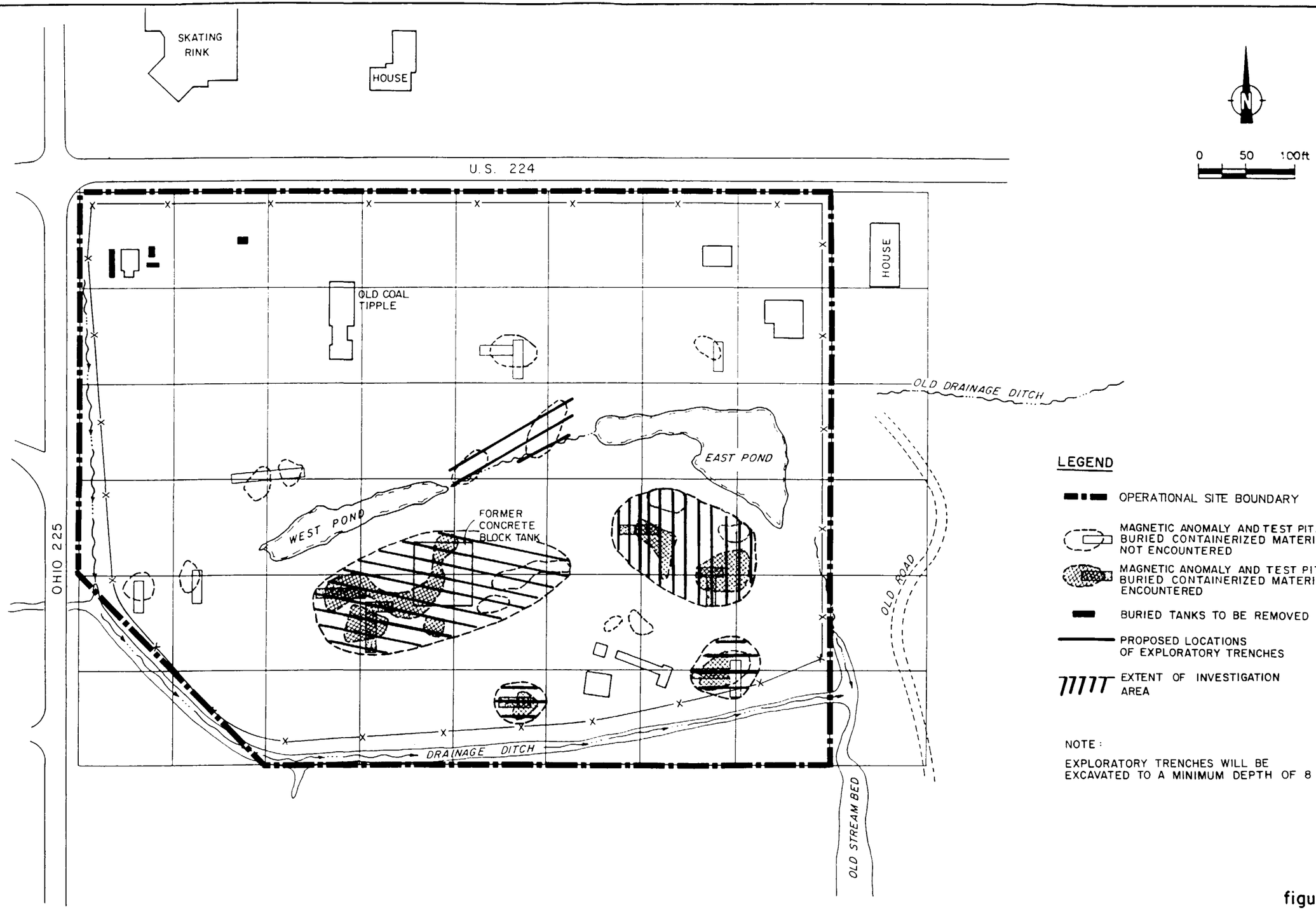
DEPTH OF SEDIMENT REMOVAL WILL BE FROM BASE OF POND TO A DEPTH OF 2 FEET

figure 3

PROPOSED ON-SITE SEDIMENT REMOVAL
Summit National Site

CRA

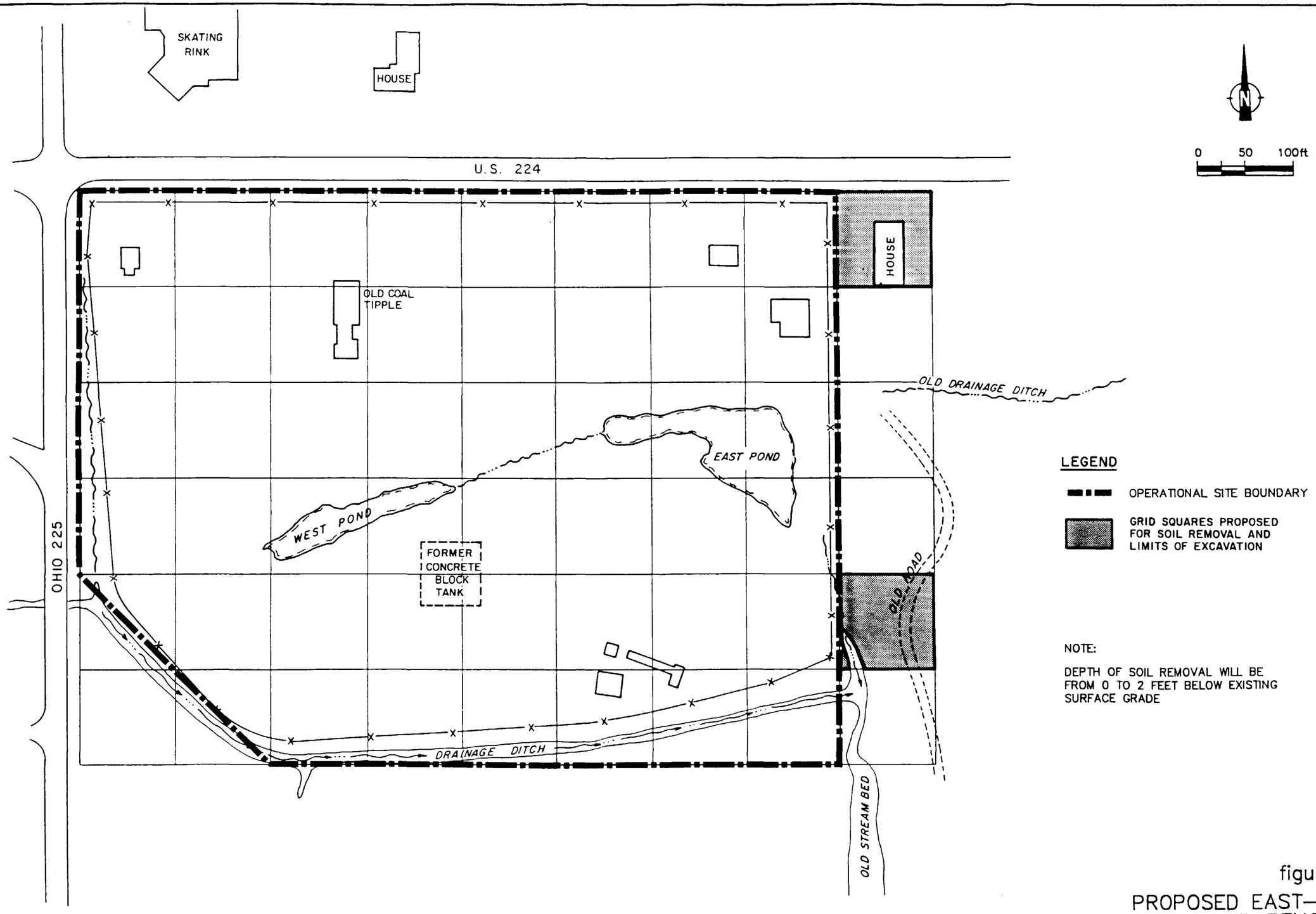
2372-24/10/88-10-D-0



CRA

figure 4

MAGNETIC ANOMALIES
Summit National Site

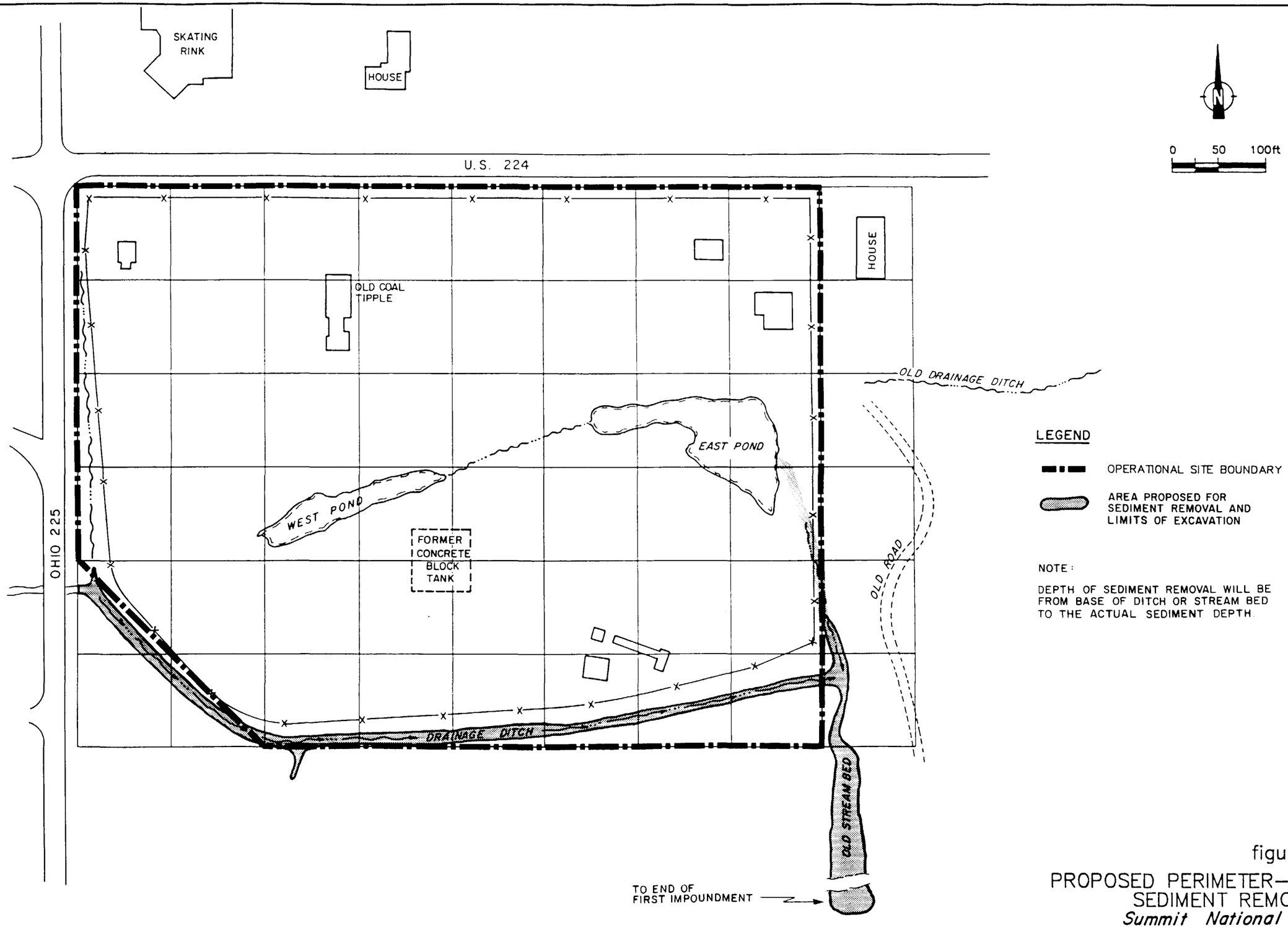


CRA

figure 5

PROPOSED EAST-SITE SURFICIAL SOIL REMOVAL

Summit National Site



CRA

figure 6
PROPOSED PERIMETER–SITE SEDIMENT REMOVAL
Summit National Site

B

APPENDIX B
SAMPLING AND ANALYSIS PLAN

SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

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B.1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared as part of the Remedial Design (RD) Work Plan for the Summit National Superfund Site (Site) located in Deerfield Township of Portage County, Ohio.

The SAP presents sampling and analytical protocols to be adhered to during the RD, to ensure that all data collection activities are consistent with the protocols contained in the United States Environmental Protection Agency (USEPA) document entitled "Guidelines for the Preparation of Standard Operating Procedures (SOPs) of Field and Laboratory Measurements", Revision 0, dated March 16, 1989, and that the analytical results are representative of field conditions. The additional data collection activities, when implemented, will supplement the existing database for the Site resulting from completion of the Remedial Investigation and Feasibility Study (RI/FS), and will permit the completion of the RD.

The Health and Safety Plan (HSP) which will be implemented during the data collecting activities and the Quality Assurance Project Plan (QAPP) which describes the specific Quality Assurance (QA) and Quality Control (QC) activities designed to achieve the specific data quality goals for the Site are presented in Appendicies A and C, respectively.

B.2.0 SCOPE OF WORK

The SAP has been designed to obtain supplemental data, which when combined with the RI database for the Site, will permit completion of the final design of the primary components of the Remedial Action (RA) selected for the Site. Additional data required for the design of each of the primary components of the RA for the Site, as identified in the RD Work Plan, are as follows:

Primary Remedial Component	*Supplemental Investigative Activities*	*Supplemental Sampling and Analyses Activities*
Soil Removal and Treatment	• boundary and topographical survey • investigation of magnetic anomalies • investigation of underground tanks	
	• surface water characterization	• collection and analyses of surface water samples
	• perimeter-Site sediments	
Groundwater Extraction System	• boundary and topographical survey	
	• characterization of the Tipple well	• collection and analyses of groundwater samples
	• bedrock profiling	• collection and grain size analysis of soil samples
	• groundwater characterization	• water level measurements • collection and analyses of groundwater samples
	• Upper Sharon Aquifer characterization	• water level measurements • collection and analyses of groundwater samples
Site Cover	• boundary and topographical survey	
Groundwater Treatment System	• boundary and topographical survey	
	• groundwater treatability studies	• bench scale testing • collection and analyses of water samples prior and post treatment

The procedures and protocols for the supplemental investigative activities are detailed in Section 4.0 of the RD Work Plan.

The procedures and protocols for the supplemental sampling and analyses activities are detailed in the following sections of this Site-specific SAP.

B.3.0 GENERAL PROTOCOLS

B.3.1 EQUIPMENT CLEANING

Upon mobilization of the drill rig and excavation equipment to the Site, the drill rig and associated equipment and the excavation equipment will be thoroughly cleaned by a high-pressure, hot-water wash to remove oil, grease, mud and other foreign matter. The drill rig and excavation equipment will be inspected by the engineer or geologist to ensure that all seals and gaskets are intact, and that no fluids are leaking. After the visual inspection has been completed, the engineer or geologist will determine if additional cleaning is required. If required, the drill rig and/or the excavation equipment will receive a thorough recleaning using a high-pressure, hot-water wash.

Prior to the collection of any samples designated for chemical analyses, all sampling equipment and tools, except for disposable bailers, will be cleaned using the cleaning protocols as follows:

i) wash thoroughly with laboratory detergent and distilled water using a brush to remove particulate matter or surface film if any;

ii) rinse with distilled/deionized water;

iii) rinse with pesticide-grade methanol;

iv) rinse with pesticide-grade isopropanol;

v) rinse with pesticide-grade methanol;

vi) rinse with distilled/deionized water;

vii) air dry;

viii) wrap in aluminum foil (sampling equipment for organics analysis) or polyethylene (sampling equipment for inorganics analysis) until required and during transport to the sampling site.

Electric water level tapes will be cleaned prior to use at each monitoring well location by rinsing with pesticide-grade methanol followed by a distilled/deionized water rinse.

Soil sampling equipment for collection of soil samples for grain size analysis will be cleaned prior to collection of each sample using a high-pressure, hot-water wash.

Prior to commencing and upon completion of drilling activities at the Tipple well and for bedrock profiling investigations, and upon completion of test trench excavations to delineate buried containerized material areas, the drill rig and excavation equipment will be thoroughly cleaned by a high-pressure, hot-water wash to remove soils and other foreign matter prior to demobilization from the Site.

Fluids used for cleaning will not be recycled. Organic decontamination fluids will be collected and containerized, pending disposal during implementation of the RA. Wash water, rinse water and development waters will be disposed of as discussed in Section B.7.0.

B.3.2 SAMPLE LABELLING

Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. The sample numbering system to be used is described in Section C.6.1 of the QAPP.

B.3.3 FIELD LOG

The field log book will be a bound document with consecutively numbered pages. The entries for each day commence on a new page which will be dated. All entries will be made only in indelible ink. Corrections will be made by marking through the error with a single line, so as to remain legible, and initialing this action followed by writing the correction. The field log books generated will be numbered consecutively and maintained by Conestoga-Rovers & Associates (CRA) in its Rosemont, Illinois office.

The following information will be recorded in the field log book for each sample collected:

i) site location identification;

ii) unique sample identification number;

iii) date and time (in 2400 hour time format) of sample collection;

iv) weather conditions;

v) designation as to the type of sample (groundwater, soil, etc.);

vi) designation as to the means of collection (grab, bailer, etc.);

vii) name of sampler;

viii) analyses to be performed on sample; and

ix) any other relevant comments such as odor, staining, texture, filtering, preservation, etc.

For the investigation of magnetic anomalies and underground tanks and the bedrock profiling, specific requirements for recording in the field log book are detailed in Sections 4.4, 4.5 and 4.7, respectively, of the RD Work Plan.

Well development details, the results of slug tests on monitoring wells, if performed, and the results of pumping tests during the groundwater treatability studies, will be recorded on CRA standard forms (see Attachment B-1), a copy of which will be kept in CRA's project files.

B.4.0 SAMPLING PROTOCOLS

B.4.1 GENERAL

A new pair of disposable latex gloves will be used for each sample handled. Disposable gloves will be collected and disposed of as discussed in Section B.7.0.

B.4.2 SOIL SAMPLING PROTOCOLS

For bedrock profiling purposes, boreholes will be installed to the bedrock surface along the proposed alignment of the pipe and media drain, as discussed in Section 4.7 of the RD Work Plan. Each borehole will be installed using 4 1/4-inch inside diameter (ID) [8-inch outside diameter (OD)] hollow stem augers until competent bedrock is encountered.

Soil samples will be collected continuously during augering to identify and classify soil materials. All soil samples will be collected using the standard penetration test method (ASTM D1586-84) or a continuous sampling system should soil conditions allow its use, and will be described and classified by visual observation according to the Unified Soil Classification System (USCS). These samples will be collected into sample jars and logged with respect to borehole depth and location.

At the completion of each borehole, at which time the depth of the borehole will be known, the soil sample representative of the

depth of the pipe bedding and the three soil samples representative of the three equally spaced intervals above the pipe bedding elevation will be retained and submitted to the project laboratory for grain size distribution analysis using ASTM Method D422.

The soil sampling equipment will be cleaned as described in Section B.3.1.

B.4.3 GROUNDWATER MONITORING WELL SAMPLING PROTOCOLS

Groundwater samples from all existing monitoring wells, as detailed in Section 4.8 of the RD Work Plan, will be collected using the sampling protocol which follows:

i) the depth of water in each well will be measured to the nearest 0.01 foot using an electric tape. The measuring device will be precleaned prior to use in each well using the cleaning sequence provided in Section B.3.1;

ii) prior to sampling, each well will be purged using a precleaned, disposable polyethylene bottom-filling bailer. The monitoring wells will be purged by removing a minimum of three standing well volumes of groundwater where the volume of standing water is calculated as follows:

$$V = 0.041\ d^2\ h$$

where:

V = volume of standing water in gallons
d = diameter of the well in inches
h = depth of water in feet

Field measurements of pH, conductivity and temperature of the evacuated water will be obtained and recorded following removal of each standing well volume and prior to sample collection. Calibration of field instruments, as specified in the QAPP, will be undertaken prior to use at each well location. Well purging will continue until three consecutive and consistent readings (± five percent) of pH, conductivity and temperature are obtained or a maximum of five standing well volumes have been removed. In the event that a well is bailed dry prior to achieving three well volumes, groundwater will be permitted to recover to a level sufficient for sample collection. The time that the well was bailed dry will be recorded and the well will be monitored for recovery. Upon recovery, the sample will be collected. All waste groundwater not collected for analysis will be disposed of as discussed in Section B.7.0;

iii) after purging the required volume of well water or immediately after well development, water samples will be collected using a precleaned, disposable polyethylene bottom-filling bailer attached to polypropylene rope. The bailer will be emptied directly into the appropriate sample containers, where containers will be filled in order of decreasing analyte volatility, using techniques which will minimize sample agitation. The groundwater samples may be collected from the bailer

used to purge the monitoring well as described above. New polypropylene rope will be used for each monitoring well;

iv) groundwater samples collected for inorganic analysis will be field filtered and noted as such on the chain-of-custody forms, such that results will be reported as dissolved compound concentrations;

v) concurrent with groundwater samples collected for inorganic analysis, some representative unfiltered untreated groundwater samples will be obtained to provide initial concentration data for contaminants associated with the solids in unfiltered groundwater;

vi) a field duplicate sample will be collected at a frequency of one per ten investigative samples collected or at a minimum of one per sampling event. All sample containers for the initial sample and the duplicate sample will be opened at the same time and each bailer of sample will be proportioned equally among the sample containers for the initial and duplicate samples. Sample containers will be filled in order of decreasing volatility;

vii) samples will be collected for matrix spike analysis at a frequency of one per 20 investigative samples collected or at a minimum of one per sampling event. The sample for matrix spike analysis will be collected from a well representative of the condition of the majority of the monitoring wells, turbid or non turbid. Samples will be collected from the representative monitoring wells using the same protocol as for the field duplicate. The chain-of-custody forms sent to the project

laboratory will indicate the samples collected for matrix spike analysis, if required; and

viii) a rinsate blank sample will be collected at a frequency of one per ten investigative samples collected or at a minimum of one per sampling event. This sample will consist of distilled/deionized water poured into, and then sampled out of, a precleaned bailer. This will provide a quality assurance check on the decontamination procedures employed for the disposable bailers and sample containers.

B.4.4 GROUNDWATER TIPPLE WELL SAMPLING PROTOCOLS

Zone-specific groundwater samples, as detailed in Section 4.6 of the RD Work Plan, will be collected from the Tipple well by means of a pump set between a set of inflatable rubber packers. The packers will be set so that the sample interval will be approximately 10 feet. The string will be installed in the well to the desired level and the packers will be inflated to isolate the sampling interval. The pump will be activated and five times the volume of water between the packers will be removed prior to collection of the sample. The groundwater sample will be collected directly into the appropriate containers in order of decreasing analyte volatility. All waste groundwater not collected for analysis will be disposed of as discussed in Section B.7.0.

Field measurements of pH, conductivity and temperature of the evacuated water, sample filtration, and collection of quality control samples (duplicates, matrix spike and rinsate blank) will be conducted as described for collection of groundwater samples from monitoring wells in Section B.4.3.

B.4.5 SURFACE WATER SAMPLING PROTOCOLS

Surface water samples, as detailed in Section 4.9 of the RD Work Plan, will be collected using the sampling protocol which follows:

i) samples representative of the depth of water at the sampling locations will be collected by the grab sample method from four locations within the surface water body, combined in a precleaned container and then distributed into laboratory prepared sample containers for shipment to the analytical laboratory;

ii) samples collected for inorganic analysis will be split in the field. One of the split samples will be field filtered and the other split sample will be unfiltered;

iii) a field duplicate sample will be collected at a frequency of one per ten investigative samples collected or at a minimum of one per sampling event. All sample containers for the initial sample and the duplicate sample will be opened at the same time and surface water will be

proportioned equally among the sample containers for the initial and duplicate samples;

iv) samples will be collected for matrix spike analysis at a frequency of one per sampling event. Samples will be collected using the sample protocol as for the field duplicate. The chain-of-custody forms sent to the project laboratory will indicate the samples collected for matrix spike analysis, if required; and

v) a rinsate blank will be collected at a frequency of one per sampling event if sampling instruments are used. This sample will consist of distilled/deionized water poured into or through the sampling instrument and collected directly in sample containers. This will provide a quality assurance check on the field decontamination procedure employed for the sampling instrument.

B.4.6 BENCH-SCALE GROUNDWATER TREATABILITY TESTS

Bench-scale tests will be conducted during the groundwater treatability studies as discussed in Section 4.10 of the RD Work Plan. Specific tests to be conducted consist of pH adjustment, aeration, coagulation, Manganese green sand and multi-media as detailed in the following subsections.

B.4.6.1 pH Titration

pH titration adjustment tests will be performed to determine the effect, if any, of altering the pH of the extracted groundwater. The procedure to be implemented for conducting the bench scale pH adjustment test will be as follows:

i) collect representative sample of the groundwater, measure and record pH and temperature;
ii) fill two nominal 1-litre clear glass test beakers with groundwater;
iii) maintain one beaker as a control sample;
iv) adjust one beaker to higher pH in one pH unit steps, using sodium hydroxide solution;
v) inspect beakers for signs of precipitates at each pH unit increase step;
vi) conduct filtration tests on each beaker; and
vii) analyze filtrate and solids for SSPL.

The addition of pH adjusting solution will be completed under kinetic mixing conditions.

B.4.6.2 Aeration

Bench-scale aeration tests will be performed to determine the effectiveness of oxidation and low level air to water air stripping, and to determine the potential for the formation of precipitates during air stripping.

The procedure to be implemented for conducting the bench-scale aeration tests is as follows:

i) collect representative sample of the groundwater;

ii) fill three nominal 2-litre clear plastic aeration test beakers with groundwater;

iii) aerate one beaker for one hour, aerate the second beaker for two hours, or longer if necessary, and maintain one beaker as a control;

iv) inspect beakers for signs of precipitates;

v) conduct filtration tests on each beaker; and

vi) analyze filtrate and solids for SSPL.

A standard laboratory air compressor pump will be used to provide the volume of air necessary for the aeration test. The air flow to each beaker will be controlled.

B.4.6.3 Coagulation

The need to conduct bench-scale coagulation tests will be determined in the field, based on visual observations of the turbidity of the extracted groundwater and whether a precipitate is generated during the performance of bench-scale aeration tests and pH adjustment tests. The purpose of the bench-scale coagulation test will be to determine the requirements for chemical aids to promote coagulation, if required.

If bench-scale coagulation tests are assessed to be necessary, a jar test program will be conducted. The bench scale coagulation tests will be performed using procedures similar to those presented in Methods 4500-OD, 4500-OE and 4500-OF of the 17th Edition of the Standard Methods for the Examination of Water and Wastewater.

B.4.6.4 Manganese Green Sand

The need to conduct a bench-scale green sand filter test will be determined based on the presence (non-presence) of iron and manganese. A sample of the filtered extracted groundwater will be filtered through a conditioned bed of green sand media at a rate of 1.5 gallons per minute per square foot (gpm/ft^2). The test green sand filter will consist of a 60-inch long by 3-inch diameter Lucite column, packed with six inches of pea gravel, 30 inches of manganese green sand and 12 inches of anthracite. Influent and effluent samples will be collected and analyzed for the following:

i) metals;
ii) iron total;
iii) iron soluble;
iv) manganese total;
v) manganese soluble;
vi) total dissolved solids; and
vii) total suspended solids.

B.4.6.5 Multi-Media

A sample of the groundwater will be filtered through a conditioned bed of multi-media at a rate of 5 gpm/ft^2. The filter will consist of a 24-inch long by 3-inch diameter Lucite column, packed with 4 inches of pea gravel, 4 inches of garnet sand, 4 inches of filter sand and 12 inches of anthracite. Influent and effluent samples will be collected and analyzed for:

i) total dissolved solids; and

ii) total suspended solids.

B.4.7 PILOT GROUNDWATER TREATABILITY TESTS

Pilot tests will be conducted during the groundwater treatability studies as discussed in Section 4.10 of the RD Work Plan. The frequency of sample collection and designated analyses also are discussed in Section 4.10 of the RD Work Plan.

Samples collected during the pilot tests will be collected from sample ports directly into the appropriate sample containers in order of decreasing analyte volatility.

B.4.8 RESPONSE TEST

Pending the results of the pumping tests to be performed during the groundwater treatability studies, as discussed in Section 4.10 of the RD Work Plan, in situ testing may be performed to determine the hydrogeologic properties of the various stratigraphic units. Single well response tests (slug tests), if performed, involve changing the water level within a well and monitoring the time required for the level to return to the static position. Two types of response tests are normally performed, namely; falling head tests, where a slug of known volume is introduced into the standing water column; and rising head tests, where a known volume of water is removed from the well and the recovery is monitored.

The displacement of the water level will be accomplished by means of a "slug" consisting of 1 1/2-inch diameter aluminum rods three feet in length. The water level will be monitored until it returns to the static level. The slug and water level measuring device will be decontaminated between wells as specified in the cleaning protocols presented in Section B.3.1.

Several methods have been developed for determination of hydraulic conductivity values from response test data. These methods consider well construction, hydrogeologic setting and time lag response as factors to calculate in situ hydraulic conductivity. The method of Hvorslev (1951) will be used, if possible, to analyze the results of response tests. If this method is found not to be applicable, other methods which may be employed include Cooper et al. (1967), Papadopulos et al. (1973), and Bouwer and Rice (1976).

B.5.0 SAMPLE SHIPMENT AND CONTAINERS

B.5.1 CHAIN-OF-CUSTODY FORMS

CRA chain-of-custody records will be used to track all samples from time of sampling to the arrival of samples at the laboratory.

Each sample container being shipped to the laboratory will contain a chain-of-custody form. The chain-of-custody form consists of four copies which are distributed to the sampler, to the shipper, to the contract laboratory and to the office file of CRA. The sampler and shipper will maintain their copies while the other two copies are enclosed in a waterproof enclosure within the sample container. The laboratory, upon receiving the samples, will complete the remaining copies. The laboratory will maintain one copy for its records. The executed original will be returned to CRA with the data deliverables package. A typical chain-of-custody form is presented in Figure B.5.1.

B.5.2 SAMPLE CONTAINERS AND HANDLING

Required sample containers, sample preservation methods, maximum holding times and filling instructions are provided in Table B.5.1.

All samples will be placed in appropriate sample containers supplied by Wadsworth/Alert Laboratories, Inc., labeled and

CRA
CONESTOGA-ROVERS AND ASSOCIATES, INC.
10400 West Higgins Road – Suite 103
Rosemont, IL 60018 708/299-9933

SHIPPED TO (Laboratory name):

CHAIN OF CUSTODY RECORD

PROJECT Nº:

PROJECT NAME:

SAMPLER'S SIGNATURE _____ (SIGN)

SEQ. Nº	SAMPLE Nº	DATE	TIME	SAMPLE LOCATOIN	SAMPLE TYPE	Nº OF CONTAINERS	REMARKS
				TOTAL NUMBER OF CONTAINERS			

ANTICIPATED CHEMICAL HAZARDS:

RELINQUISHED BY: 1 (SIGN)	DATE/TIME	RECEIVED BY: 2 (SIGN)
RELINQUISHED BY: 2 (SIGN)	DATE/TIME	RECEIVED BY: 3 (SIGN)
RELINQUISHED BY: 3 (SIGN)	DATE/TIME	RECEIVED BY: 4 (SIGN)
RELINQUISHED BY: 4 (SIGN)	DATE/TIME	RECEIVED BY: 5 (SIGN)
RELINQUISHED BY: 5 (SIGN)	DATE/TIME	RECEIVED BY: 6 (SIGN)
RELINQUISHED BY: 6 (SIGN)	DATE/TIME	RECEIVED BY: 7 (SIGN)

METHOD OF SHIPMENT:	SHIPPED BY:	RECEIVED FOR LABORATORY BY: (SIGN)	DATE/TIME
CONDITION OF SEAL UPON RECEIPT: GENERAL CONDITION OF COOLER:		COOLER OPENED BY: (SIGN)	DATE/TIME

WHITE – CRA OFFICE COPY
YELLOW – RECEIVING LABORATORY COPY
PINK – CRA LABORATORY COPY
GOLDEN ROD – SHIPPERS

figure B.5.1

SAMPLE CHAIN OF CUSTODY RECORD
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

2372-18/07/91-13-0

TABLE B.5.1

CONTAINER, PRESERVATION, SHIPPING AND PACKING REQUIREMENTS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Analysis	*Sample Containers*	*Preservation*	*Maximum Holding Time From Sample Collection*[1]	*Volume of Sample*	*Shipping*	*Normal Packaging*
Groundwater and Surface Water						
TCL[2] Volatile Organic Compounds (VOC)	Three 40-mL volatile organic (VOA) vials	HCl to pH <2, Iced to 4° C	14 days for analysis	Fill completely, no air bubbles	Federal Express Priority 1	Vermiculite or Foam Chips
TCL Base/Neutral-Acid Extractable Compounds (BNA)	Two 1-liter glass bottles[3]	Iced to 4° C	7 days for extraction 40 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
TCL Pesticide/PCB	Two 1-liter glass bottles[3]	Iced to 4° C	7 days for extraction 40 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
TAL[4] Metals	One 1-liter plastic bottle	HNO_3 to pH <2,	6 months (mercury -28 days) for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Cyanide	One 1-liter plastic bottle	NaOH to pH ≥ 12, Iced to 4° C	14 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
General Water Quality Parameters						
Chloride, Sulfate	One 1-litre plastic bottle	Iced to 4°C	28 days fore analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
TDS, TSS	One 1-litre platsic bottle	Iced to 4°C	7 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Phenols	One 1-litre glass bottle	H_2SO_4 to pH <2 Iced to 4°C	28 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips

TABLE B.5.1

CONTAINER, PRESERVATION, SHIPPING AND PACKING REQUIREMENTS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Analysis	*Sample Containers*	*Preservation*	*Maximum Holding Time From Sample Collection*	*Volume of Sample*	*Shipping*	*Normal Packaging*
Groundwater Treatability[5]						
TCL VOC	Three 40-ml VOA vials	HCl to pH <2 Iced to 4°C	14 days for analysis	Fill completely no air bubbles	Federal Express Priority 1	Vermiculite or Foam Chips
TCL BNA	Two 1-liter glass bottles[3]	Iced to 4°C	7 days for extraction 40 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Groundwater Treatability						
TCL Pesticides/PCB	Two 1-liter glass bottles[3]	Iced to 4°C	7 days for extraction 40 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
General Water Quality Parameters						
Manganese, Iron, Sodium	One 1-liter plastic bottle	HNO_3 to pH <2	6 months for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Chloride, Sulfate	One 1-liter plastic bottle	Iced to 4°C	28 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
TDS, TSS	One 1-liter plastic bottle	Iced to 4°C	7 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Phenols	One 1-liter glass bottle	H_2SO_4 to pH <2 Iced to 4°C	28 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips

TABLE B.5.1

CONTAINER, PRESERVATION, SHIPPING AND PACKING REQUIREMENTS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Analysis	*Sample Containers*	*Preservation*	*Maximum Holding Time From Sample Collection*	*Volume of Sample*	*Shipping*	*Normal Packaging*
Solids from Treatability Studies						
TCL VOC	One 4-ounce glass jar	Iced to 4°C	14 days for analysis	Fill completely	Federal Express Priority 1	Vermiculite or Foam Chips
TCL BNA	One 16-ounce glass jar	Iced to 4°C	14 days for extraction 40 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
General Water Quality Parameters						
Manganese, Iron, Sodium	One 4-ounce glass jar	Iced to 4°C	6 months for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Chloride, Sulfate, Phenols	One 4-ounce glass jar	Iced to 4°C	28 days for analysis	Fill to shoulder of bottle	Federal Express Priority 1	Vermiculite or Foam Chips
Soil Samples						
Geotechnical	One 16-ounce glass jar	--	none	--	Federal Express Priority 1	Vermiculite or Foam Chips

1 Maximum holding time periods were consistent with methods referenced in SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd edition, November 1986 and "1990 Annual Book of ASTM Standards", American Society for Testing and Materials and "Methods for the Chemical Analysis of Water and Wastes", EPA 600/4-79-020, revised March 1983.

2 TCL - Target Compound List

3 With Teflon Lined Caps

4 TAL - Target Analyte List

5 A Site-specific parameter list (SSPL) based on the pre-design groundwater characterization will be developed upon receipt of results of the groundwater characterization.

properly sealed. The sample labels will include sample number, place of collection, date and time of collection and analyses to be performed. Samples will be cushioned within the shipping coolers by the use of vermiculite and/or bubble pack. Samples will be kept cool by the use of plastic bags of ice or cooler packs, as required. A trip blank will accompany each shipment of groundwater samples submitted for VOC analyses.

Samples will be shipped by commercial courier on a daily basis to the project laboratory. The exception to this will be samples which are collected on a Sunday or holiday. For samples collected on a Sunday or holiday, additional ice will be placed in the coolers, as required, the coolers will be sealed and kept under surveillance, and picked up by the courier on the next business day.

Two seals comprised of CRA chain-of-custody tape will be placed over the lid on the front and back of each shipping cooler prior to shipment to secure the lid and provide evidence that the samples have not been tampered with en route to the laboratory. Clear tape will be placed over the seals to ensure that they are not accidentally broken during shipment.

Upon receipt of the cooler at the laboratory, the cooler will be inspected by the designated sample custodian. The condition of the cooler and seal will be noted on the chain-of-custody form by the sample custodian. The sample custodian will document the date and time of receipt of the cooler and sign the chain-of-custody form.

The sample custodian then will check the contents of the cooler with those samples listed on the chain-of-custody form. If damage or discrepancies are noticed, they will be recorded in the remarks column of the chain-of-custody form, dated and signed. They will be reported to the laboratory supervisor who will inform the laboratory manager and QA officer.

Sample disposal will be the responsibility of the laboratory. Upon disposal, the laboratory shall sign the next open "Relinquished by" box, and the word "Disposed" shall be written in the "Received by" box.

B.6.0 ANALYTICAL PROTOCOLS

B.6.1 SCOPE

Groundwater and surface water samples collected for chemical analysis, as described in Section B.4.0, will be analyzed for TCL/TAL and general water quality parameters, and soil samples will be collected for grain size analysis. Samples collected during the groundwater treatability studies will be analyzed for a Site-specific parameter list to be developed, as discussed in Section 4.10 of the RD Work Plan.

B.6.2 SAMPLE ANALYSIS

All samples collected for chemical analysis will be analyzed using approved analytical methods as outlined in Section C.8.0 of the QAPP. Practical Quantification Limits (PQLs) for water are also presented in the QAPP. PQLs are highly matrix dependent, thus, the PQLs listed are provided for guidance and may not always be achievable.

B.6.3 DATA QUALITY ASSESSMENT

B.6.3.1 Laboratory Data Assessment

The laboratory quality assurance officer will review data and identify results where additional work is required. This additional work may take the form of re-analysis or, resampling and analysis.

Internal quality control tests will be conducted by the laboratory in accordance with their standard operating procedures and the individual method requirements.

B.6.3.2 CRA Data Assessment

This step is carried out by the CRA Quality Assurance Officer to evaluate the quality and utility of the data, as a level of quality assurance beyond that provided by the laboratory. The analytical data will be evaluated based on criteria outlined in the QAPP.

The quality and acceptability or unacceptability of the data will be determined following the reporting of the laboratory analysis. The data will be examined for the acceptability of trip blanks, field blanks, duplicates, percent recoveries of matrix spikes and surrogate spikes and other laboratory quality assurance data. In the event that data is deemed unacceptable, a decision will be made by CRA regarding the appropriate

corrective action. The corrective action may include resampling, reanalysis or data-qualification.

B.7.0 WASTE MATERIAL HANDLING

All sampling generated organic decontamination fluids will be collected and contained in Department of Transport (DOT)-approved 55-gallon drums. The drums will be stored on-Site until implementation of the RA. At the completion of the construction of the on-Site groundwater treatment system, all containerized aqueous solutions will be processed in the groundwater treatment system.

All surplus sampling generated solid and aqueous wastes will be collected, secured and stored on Site for sampling and disposal during the RA. Used PPE will be collected and secured in 6-mil plastic bags and placed under the sediment stockpile cover on Site for disposal during the RA.

Soils excavated during trench excavations will be used to backfill the trenches from which they were excavated. Visually clean drill cuttings and/or locally obtained soils will be used during closure of bedrock profiling boreholes. All excess drill cuttings will be collected, secured and stored on Site for disposal during the RA.

B.8.0 ON-SITE HEALTH AND SAFETY PLAN

The SAP involves the collection of soil, surface water and groundwater samples at the Site. During the program, personnel may come in contact with materials that contain VOCs, BNAs, elevated levels of metals or other hazardous substances. Therefore, provisions for health and safety will be implemented which are designed to ensure:

i) personnel working on Site are not adversely exposed to Site contaminants;

ii) the health and safety of the general public and the environment is not compromised by off-Site migration of contaminated materials; and

iii) compliance with applicable governmental and non-governmental (American Conference of Governmental Industrial Hygienists) regulations and guidelines.

The Site-specific HSP under which this work will be completed is presented in Appendix A to the RD Work Plan.

ATTACHMENT B-1

CRA STANDARD FORMS

WELL DEVELOPMENT AND STABILIZATION FORM

PROJECT NAME _____ PROJECT NO. _____
DATE OF WELL DEVELOPMENT _____
DEVELOPMENT CREW MEMBERS _____
SUPERVISOR _____
PURGING METHOD _____

WELL INFORMATION

WELL NUMBER _____
WELL TYPE (diameter/material) _____
MEASURING POINT ELEVATION _____
STATIC WATER DEPTH _____ ELEVATION _____
BOTTOM DEPTH _____ ELEVATION _____
WATER COLUMN LENGTH _____
SCREENED INTERVAL _____
WELL VOLUME _____

Note: For 2" dia well, 1 foot= 0.14 gallons(imp) or 0.16 gallons(us).
1 meter= 2 liters.

DEVELOPMENT DATA

	1	2	3	4	5	TOT / AVG
VOLUME PURGED (# bails/tot. volume)						
FIELD pH						
FIELD TEMPERATURE ____						
FIELD CONDUCTIVITY ____						
CLARITY						
COLOR						
ODOR						
COMMENTS						

COPIES TO: _____

SINGLE WELL RESPONSE TEST - DATA

DATE (D/M/Y)	PAGE No.	FILE No. CODE
	OF	

PROJECT			PIEZOMETER NUMBER
LOCATION		WELL DIA.	BOREHOLE DIA.
CRA SUPERVISOR	SLUG DIMENSIONS (L X Ø)	SLUG DISPLACEMENT (H-Ho)	SCREEN INTERVAL TO B.D.
AQUIFER THICKNESS	DATUM POINT	DATUM POINT ELEV. AMSL	STATIC WATER LEVEL CONDITIONS (H) B.D. ☐ RISING ☐ FALLING

RUN 1

TIME			WATER LEVEL (h)	H-h	$\frac{H-h}{H-Ho}$
HR.	MIN.	SEC.			

RUN 2

TIME			WATER LEVEL (h)	H-h	$\frac{H-h}{H-Ho}$
HR.	MIN.	SEC.			

PUMPED WELL - DRAWDOWN DATA

DATE (D/M/Y)	PAGE No.	FILE No. CODE
/ /	OF	/

PROJECT	PUMPED WELL No.	TEST No.

LOCATION	TYPE OF TEST

CRA SUPERVISOR	PUMP SETTING	SCREEN INTERVAL	PUMPING RATE (Q)
	B.D.	TO B.D.	

AQUIFER THICKNESS	DATUM POINT	DATUM POINT ELEV.	STATIC WATER LEVEL CONDITIONS
		AMSL	B.D. ☐ CONFINED ☐ UNCONFINED

TIME			ELAPSED TIME (min.)	WATER LEVEL	DRAWDOWN	DISCHARGE METER READING	Q	REMARKS
DAY	HR.	MIN.						

PUMPED WELL
- RECOVERY DATA

DATE (D/M/Y)	PAGE No.	FILE No. CODE
	OF	

PROJECT	PUMPED WELL No.	TEST No.

LOCATION	TYPE OF TEST

CRA SUPERVISOR	W/L BEFORE PUMPING CEASED	SCREEN INTERVAL	PUMPING RATE (Q)
		TO	

AQUIFER THICKNESS	DATUM POINT	DATUM POINT ELEV.	STATIC WATER LEVEL CONDITIONS
		AMSL	B.D. ☐ CONFINED ☐ UNCONFINED

TIME			TIME(t) SINCE PUMPING STARTED (min.)	TIME(t') SINCE PUMPING STOPPED (min.)	RATIO t/t'	WATER LEVEL	RESIDUAL DRAWDOWN	REMARKS
DAY	HR.	MIN.						

OBSERVATION WELL - DRAWDOWN DATA

DATE (D/M/Y)	PAGE No.	FILE No. CODE
	OF	

PROJECT	JOB CAPTAIN
LOCATION	SCREEN INTERVAL TO / TEST No.

OBSERVATION WELL No.	PUMPED WELL No.	DIST. TO PUMPED WELL	STATIC WATER LEVEL AMSL	BELOW DATUM AMSL

AQUIFER THICKNESS	DATUM POINT	DATUM POINT ELEV. AMSL	PUMPING RATE	CONDITIONS ☐ CONFINED ☐ UNCONFINED

TIME			WATER LEVEL	TIME SINCE PUMPING STARTED (MIN.)	DRAWDOWN
DAY	HR.	MIN.			

TIME			WATER LEVEL	TIME SINCE PUMPING STARTED (MIN.)	DRAWDOWN
DAY	HR.	MIN.			

C

APPENDIX C
QUALITY ASSURANCE PROJECT PLAN (QAPP)

SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

LIST OF ACRONYMS AND SHORT FORMS

ACT	-	Applied Construction Technology, Inc.
ASTM	-	1990 Annual Book of ASTM Standards
BFB	-	4-Bromofluorobenzene
BNA	-	Base/Neutral-Acid Extractable Compounds
CRA	-	Conestoga-Rovers & Associates
DFTPP	-	Decafluorotriphenylphosphine
DQO	-	Data Quality Objective
GC	-	Gas Chromatography
GC/MS	-	Gas Chromatography/Mass Spectrometry
MDL	-	Method Detection Limit
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
°C	-	Degree Centigrade
PCB	-	Polychlorinated Biphenyls
PE	-	Performance Evaluation
QA	-	Quality Assurance
QA/QC	-	Quality Assurance/Quality Control
QAO	-	Quality Assurance Officer
QAPP	-	Quality Assurance Project Plan
QAS	-	Quality Assurance Section
QC	-	Quality Control
RD	-	Remedial Design
RPD	-	Relative Percent Difference
RPM	-	Remedial Project Manager
Site	-	Summit National Superfund Site
SNFT	-	Summit National Facility Trust
SOP	-	Standard Operating Procedures
SSPL	-	Site Specific Parameter List
SW-846	-	SW-846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", 3rd Edition, November 1986
TAL	-	Target Analyte List
TCL	-	Target Compound List
USEPA	-	United States Environmental Protection Agency
USEPA-WW	-	"Method for the Chemical Analysis of Water and Wastes", EPA 600/4/79-020, revised March 1983.
VOC	-	Volatile Organic Compounds
WAL	-	Wadsworth/Alert Laboratories, Inc.

QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: Remedial Design Work Plan
Summit National Superfund Site
Deerfield Township of Portage County, Ohio

PREPARED BY: CONESTOGA-ROVERS & ASSOCIATES (CRA)

Approved By: _____ Date: _____
Chairperson -
Summit National Facility Trust
Gary Gifford

Approved By: _____ Date: _____
Principal-in-Charge - CRA
Richard G. Shepherd

Approved By: _____ Date: _____
Project Manager - CRA
Jack Michels

Approved By: _____ Date: _____
Project Coordinator - CRA
Stephen Whillier

Approved By: _____ Date: _____
Quality Assurance/
Quality Control Officer
Analytical Activities - CRA
Steven Day

Approved By: _____ Date: _____
Field Quality Assurance/
Quality Control Officer - CRA
Joseph Campisi

Approved By: _____ Date: _____
Project Laboratory Manager - WAL
Thomas Himes

QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: Remedial Design Work Plan
Summit National Superfund Site
Deerfield Township of Portage County, Ohio

PREPARED BY: CONESTOGA-ROVERS & ASSOCIATES (CRA)

Approved By: _____ Date:_____
Quality Assurance/
Quality Control Officer -
Laboratory Activities - WAL
Jeff Graham

Approved By: _____ Date:_____
Sample Custodian - WAL
Alicia Danford

Approved By: _____ Date:_____
Project Laboratory Manager - ACT
LeRoy Mazer

Approved By: _____ Date:_____
Quality Assurance/Quality Control
Officer - Laboratory Activities - ACT
LeRoy Mazer

Approved By: _____ Date:_____
Sample Custodian - ACT
Emile Szocska

Approved By: _____ Date:_____
USEPA Region V
Remedial Project Manager
Anthony Rutter

Approved By: _____ Date:_____
USEPA Region V
Quality Assurance Officer/Chief of
Monitoring and Quality Assurance Branch -
Valerie J. Jones

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ATTACHMENT C-1 LABORATORY STANDARD OPERATING PROCEDURES

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents the policies, organization, objectives, functional activities and specific Quality Assurance (QA) and Quality Control (QC) activities designed to achieve the specific data quality goals associated with the Remedial Design (RD) activities for the Summit National Superfund Site (Site) located in Deerfield Township of Portage County, Ohio. The purpose and objective of the QAPP is to ensure that the analytical results are accurate and representative of the media sampled.

C.2.0 PROJECT DESCRIPTION

The purpose of this section is to provide a comprehensive summary of background information on the Site. The information was compiled in reasonable detail to familiarize the reviewer with the Site, with prior activities and with the RD activities.

C.2.1 SITE DESCRIPTION

The location and description of the Site is presented in Section 1.2 of the RD Work Plan.

C.2.2 SITE HISTORY

The Site history including history of operations, history of remedial actions, history of investigative programs and regulatory history is presented in Section 1.3 of the RD Work Plan.

C.2.3 PROPOSED REMEDIAL ACTION

The proposed remedial action is presented in Section 1.4 of the RD Work Plan.

C.2.4 RD OBJECTIVES

The objective of the RD is to prepare the plans and specifications for implementation of the RA for the Site in accordance with the requirements of the SOW. To achieve this objective, supplemental investigative activities to be completed during implementation of the RD consist of the following:

i) excavation of exploratory trenches in specific areas of the Site where magnetic anomalies were encountered during the RI but not investigated;

ii) characterization of the Tipple well with respect to groundwater quality and physical conditions including the collection of groundwater samples and hydrogeological measurements;

iii) collection of one round of groundwater samples and two water level measurements from the existing on-Site monitoring wells and collection of surface water samples;

iv) installation of boreholes to the depth of bedrock along the alignment of the pipe and media drain system at 100-foot centers to determine the bedrock profile;

v) collection of soil samples from each borehole location at the pipe bedding elevation and three equally spaced intervals above the

pipe bedding elevation for analysis of grain size distribution to determine the gradation requirements for the bedding and granular backfill material of the pipe and media drain;

vi) performance of a groundwater treatability study to define the parameters to be used for the final design of the groundwater treatment system components.

The field activities employed to achieve these objectives are detailed in Section 4.0 of the RD Work Plan.

C.2.5 DATA USAGE

Data generated from the various pre-design monitoring and sampling events will be used in support of the RD, as described in Section 4.0 of the RD Work Plan.

C.2.6 TARGET COMPOUNDS

The target compounds for the Site include: TCL organic and TAL inorganic parameters, general water quality inorganics and grain size distribution. Upon receipt of the results of the groundwater characterization as detailed in Section 4.8 of the RD Work Plan, a Site-specific parameter list (SSPL) will be developed for the groundwater treatability study, as detailed in Section 4.10 of the RD Work Plan. Laboratory analyses to be performed on specific sample matrices are as follows:

- Groundwater (Monitoring Wells, Tipple Well) - TCL Organics
  - TAL Inorganics
- Groundwater (Treatability Study) - SSPL
- Solids (Treatability Study) - SSPL
- Bedrock Profile - Grain Size Distribution
- Surface Water - TCL Organics
  - TAL Inorganics

C.2.7 PROJECT SCHEDULE

The proposed schedule for the RD activities is presented in Section 8.0 of the RD Work Plan.

C.3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Conestoga-Rovers & Associates (CRA), as contractor to the Summit National Facility Trust (SNFT), has overall responsibility for all phases of the RD activities at the Site. CRA will perform or supervise all field activities.

Wadsworth/Alert Laboratories, Inc. (WAL), as analytical subcontractor to CRA, will perform all chemical analyses of samples collected during the RD activities. Applied Construction Technology, Inc. (ACT) will perform all geotechnical analyses.

All firms will provide project management as appropriate to their responsibilities. CRA will provide administrative oversight and QA/QC for all deliverables. All final project deliverables will be issued by CRA.

Figure C.3.1 presents the key staff organization for the QAPP. CRA's summary of each of the key persons responsibilities is presented below:

Gary Gifford - Trust Chairperson - SNFT

- general overview of the project to ensure that the PRPs objectives are met
- participation in key negotiations with USEPA
- managerial guidance to CRA's Principal-in-Charge and Project Manager

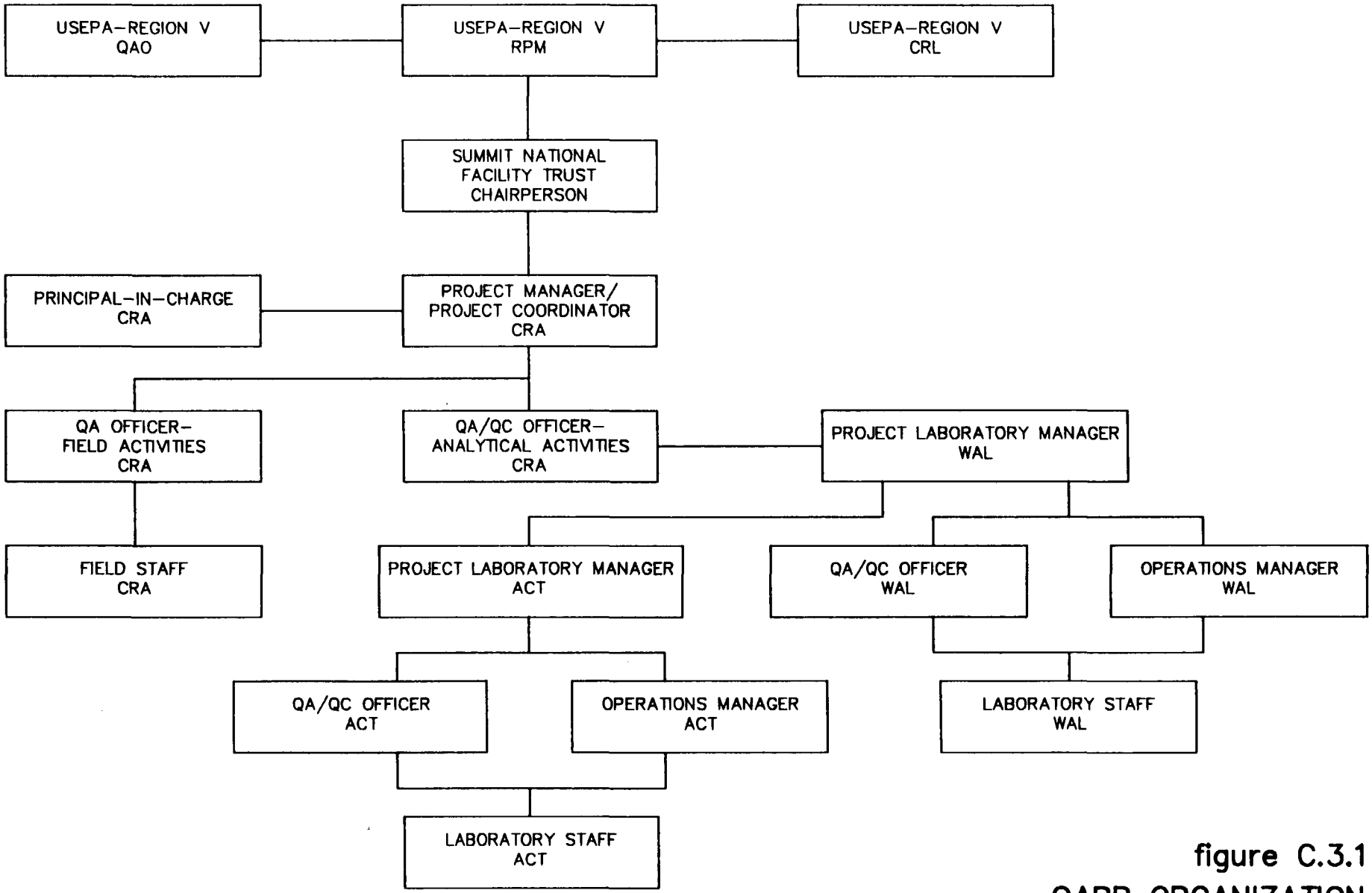

figure C.3.1

QAPP ORGANIZATION
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372–9/07/91–13–0 (C–01)

Richard Shepherd - Principal-in-Charge - CRA

- provides overall corporate management
- participation in key technical negotiations with USEPA and SNFT
- managerial and technical guidance to CRA's Project Manager

Jack Michels - Project Manager and Principal Engineer - CRA

- provides overall project management
- ensures professional services provided by CRA are cost effective and of highest quality
- ensures all resources of CRA are available on an as-required basis
- participates in key technical negotiations with USEPA
- liasion between technical group and SNFT
- liasion with USEPA and OEPA
- managerial and technical guidance to CRA's Project Coordinator

Stephen Whillier - Project Coordinator - CRA

- day-to-day project management
- managerial guidance to CRA's technical group
- overall preparation and review of RD activities report
- evidence file custodian

Steven Day - QA/QC Officer - Analytical Activities - CRA

- systems audits - laboratory activities
- overview and review field QA/QC
- coordinate supply of performance evaluation samples
- review laboratory QA/QC
- data validation and assessment

- advise on data corrective action procedures
- preparation and review of RD activities reports
- QA/QC representation of project activities

Joseph Campisi- QA Officer - Field Activities - CRA

- management of field activities and field QA/QC
- data assessment
- preparation and review of RD activities report
- technical representation of field activities
- preparation of standard operating procedures (SOPs) for field activities

Thomas Himes - Project Laboratory Manager - WAL

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- overview data review
- overview preparation of analytical reports

Mark Nebiolo - Operations Manager - WAL

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- oversee data review
- oversee preparation of analytical reports
- approve final analytical reports prior to submission to CRA

Jeff Graham - QA/QC Officer - Laboratory Activities - WAL

- overview laboratory quality assurance
- overview QA/QC documentation
- conduct detailed data review
- decide laboratory actions, if required
- technical representation of laboratory QA procedures
- preparation of laboratory SOPs
- approval of the QAPP

Alicia Danford - Sample Custodian - WAL

- receive samples and sign accompanying documents (chain-of-custody)
- notify technical staff of samples that require immediate attention
- control and regulate access to samples and log all sample movements

LeRoy Mazer - Project Laboratory Manager - ACT

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- overview data review
- overview preparation of analtyical results

George Wozniak - Operations Manager - ACT

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- oversee data review
- oversee preparation of analytical reports

- approval of final analtyical reports prior to submission to CRA

LeRoy Mazer - QA/QC Officer - Laboratory Activities - ACT

- overview laboratory quality assurance
- overview QA/QC documentation
- conduct detailed data review
- decide laboratory actions, if required
- technical representation of laboratory QA procedures
- preparation of laboratory SOPs
- approval of the QAPP

Emile Szocska - Sample Custodian - ACT

- receive samples and sign accompanying documents (chain-of-custody)
- notify technical staff of samples that require immediate attention
- control and regulate access to samples and log all sample movements

Primary responsibility for project quality rests with CRA's QA/QC Officer - Analytical Activities and QA Officer - Field Activities. Ultimate responsibility for project quality rests with the CRA's Project Manager. Independent QA will be provided by the Project Laboratory Mangers and QA/QC Officers prior to release of all data to CRA

Figure C.3.2 presents a typical flow chart for the project laboratories.

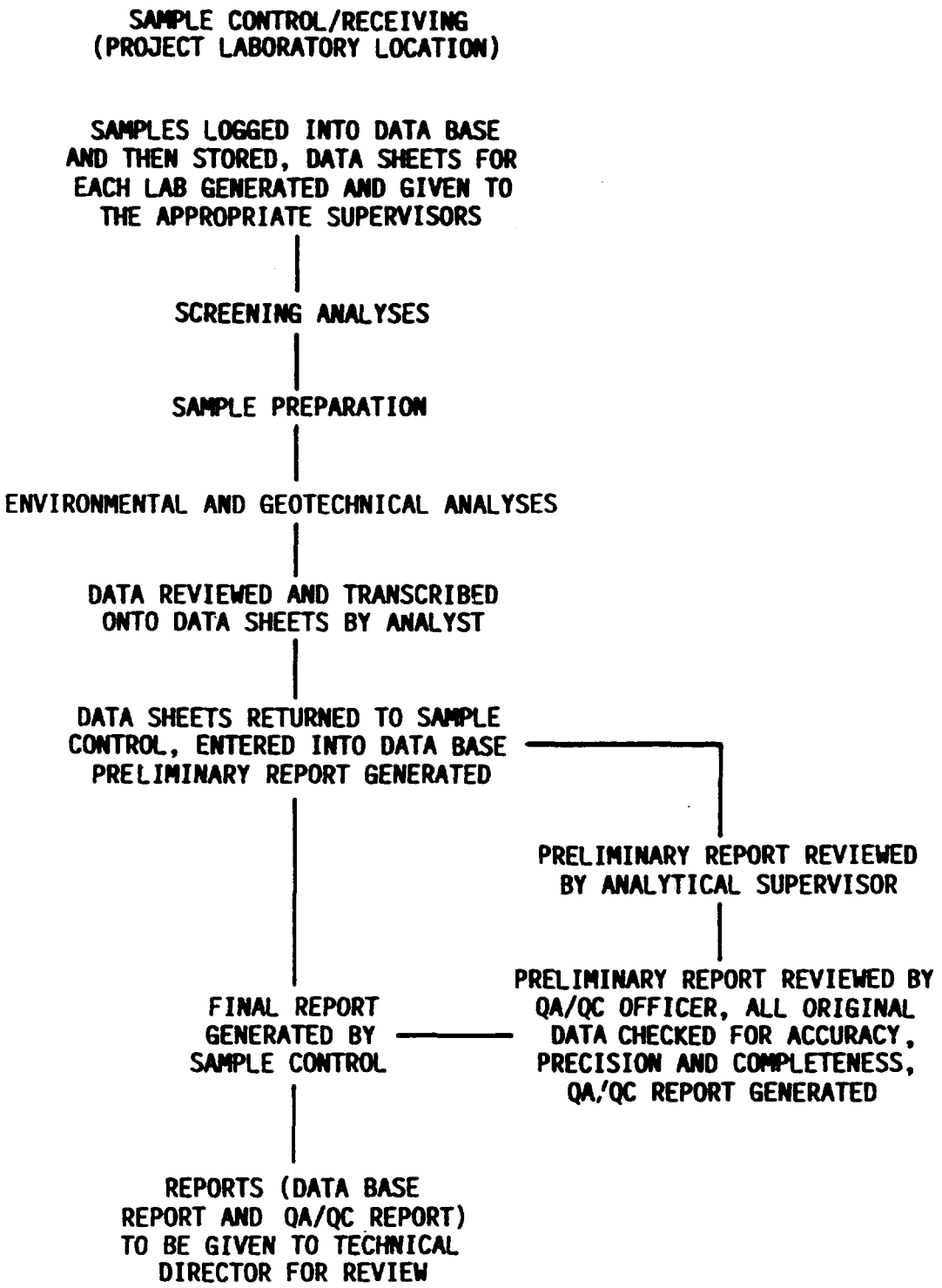

figure C.3.2

TYPICAL DATA FLOW CHART
PROJECT LABORATORY
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

USEPA RESPONSIBILITIES

The USEPA Region V Remedial Project Manger (RPM) will be responsible for overview of this project. The RPM will also be responsible for providing approval of the QAPP. Anthony Rutter is the RPM for the Site remediation activities.

The Laboratory Scientific Support Section of the Central Regional Laboratory of USEPA Region V (i.e., USEPA Region V CRL-LSS) or USEPA Central District Office (CDO) will be responsible for performance and system audits of the laboratory analyses and field activities. Charles T. Elly is Chief of the CRL section and will be responsible for scheduling the laboratory audits. Performance evaluation (PE) audits will be ordered at the discretion of the USEPA.

Additionally, the RPM and USEPA Region V Quality Assurance Officer (QAO) are responsible for reviewing and for providing final approval of the QAPP. Valerie J. Jones is Chief of the Monitoring and Quality Assurance Branch of the USEPA - Region V.

C.4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analyses and reporting that will provide accurate data for the monitoring activities. Specific procedures to be used for sampling, chain-of-custody, calibration, laboratory analyses, reporting, quality control, audits, preventive maintenance and corrective actions are presented in various sections of this QAPP.

Data quality objectives (DQO) have been established in accordance with the USEPA guidance document entitled "Data Quality Objectives for Remedial Response Activities - Development Process", dated March 1987, in conjunction with the document, "Data Quality Objectives for Remedial Response Activities - Example Scenario RI/FS Activities at a Site with Contaminated Soils and Groundwater", dated March 1987. Reference to these documents ensures that the database developed during the Site activities meets the objectives and quality necessary for its intended use.

DQOs can be classified for the measurement data by defining the level of analytical support assigned to each type of measurement data.

In general, the groundwater and treatability sample analyses will require Level III analytical support. Geotechnical analyses will require Level V analytical support. Level I support will be assigned to field measurements, such as pH, specific conductance and temperature.

The Level III support for the analyses will require that all organics and inorganics are analyzed using representative methods taken from SW-846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", 3rd edition, November 1986 (SW-846) and "Methods for the Chemcial Analysis of Water and Wastes", EPA 600/4/79-020, revised March 1983 (USEPA-WW). The Level V support will require that the geotechnical samples be analyzed using methods from the 1990 Book of ASTM Standards (ASTM). Data deliverables will consist of a full data package as prescribed in Section C.9.0 for organic and inorganics.

The use of the analytical support levels defined above will ensure that the overall objectives for the Site activities will be completed.

C.4.1 LEVEL OF QA EFFORT

To assess the quality of data resulting from the field sampling program, field duplicate samples, field rinsate samples and matrix spike samples will be collected (where appropriate) and submitted to the project laboratory.

For all field samples collected, field duplicate samples will be collected at a frequency of one per 10 investigative samples per parameter set for all sample matrices, with a minimum of one field duplicate sample submitted per sampling event. Matrix spike and matrix spike duplicate (MS/MSD) samples will be analyzed at a minimum frequency of one per 20 for each organic analysis, or a minimum of one MS/MSD set per sampling event. For the inorganics analyses, one matrix spike and laboratory replicate will be run for each set of 20 samples, or one set per matrix type.

Field rinsate samples will be submitted at a frequency of one per 10 investigative samples collected, or at a minimum of one per sampling event. Field rinsate samples will be collected by routing distilled or deionized water through decontaminated sampling equipment. Field rinsate samples are analyzed to check field decontamination procedures.

Preserved trip blank samples for volatile organic compounds (VOC) analyses (prepared by the laboratory and consisting of organic-free water poured into the sample vials) will be shipped with each shipping container of VOC sample vials by the laboratory. Trip blank samples

will be handled in a manner consistent with actual field sample handling and will be shipped back to the laboratory each day with groundwater samples. The trip blanks will provide a measure of potential cross contamination of samples during shipment and handling. However, it is noted that trip blanks will not be opened in the field.

Upon examination of the results obtained by the laboratory, if any of the aforementioned blanks are found to contain any of the target analytes, the data associated with the investigative samples and method blanks will be assessed to determine if, and to what extent, the contaminant(s) exists. Should the associated samples and method blanks be free from the contaminant(s), the investigative sample data are to remain unqualified. If the contamination in the blank can be attributed to an isolated source such as a field blank collected after a highly contaminated sample, and is not found in other investigative samples, the investigative sample data are to remain unqualified. If the contaminants are found to be in the blanks and in other investigative samples, data qualification will be instituted as described in Section C.9.0.

Field duplicate samples will be analyzed to check for sampling and analytical reproducibility. Field duplicate samples are to be used as a measure of precision throughout the sampling event. Comparison of field duplicate samples will be based upon the target analytes, both non-detected and detected, and the relative percent differences (RPD) of each analyte's concentrations. The parameters which do not meet the criteria may

only be used as qualitative measurements. Professional judgement shall determine the RPD limits on a sample-to-sample basis.

The level of QA effort required for each matrix is summarized on Table C.4.1.

TABLE C.4.1

SUMMARY OF SAMPLING AND ANALYSES PROGRAM
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Sample Matrix	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Field (1) Blanks*	*Field Duplicates*	*Matrix Spike MS/MSD(2)*	*Matrix Total Per Round*	*Frequency per Year*	*Matrix Total*
Groundwater (monitoring wells)	pH, specific Conductivity, Temperature, Water level	TCL Organics TAL Inorganics General Water Quality Parameters	34	4	4	2	44	1	44
Surface water		TCL Organics TAL Inorganics	2 4	1 1	1 1	1 1	5 7	1 1	5 7
Soil (Bedrock profile)		Grain Size Distribution	64	0	0	0	64	1	64
Treatability Study - pH Titration (Groundwater)	pH, Temperature	SSPL	1	0	0	0	1	1	1
Treatability Study - pH Tiltration (Solids)		SSPL	1	0	0	0	1	1	1
Treatability Study - Areation (Groundwater)	pH, Temperature	SSPL	1	0	0	0	1	1	1
Treatability Study - Aeration (Solids)		SSPL	1	0	0	0	1	1	1
Treatability Study - Manganese Green Sand		TSS/TDS, Selected Metals (4)	2	0	0	0	2	1	2
Treatability Study - Multi-Media Filtration (Groundwater)		TSS/TDS	2	0	0	0	2	1	2

TABLE C.4.1

SUMMARY OF SAMPLING AND ANALYSES PROGRAM
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Sample Matrix	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Field (1) Blanks*	*Field Duplicates*	*Matrix Spike MS/MSD(2)*	*Matrix Total Per Round*	*Frequency per Year*	*Matrix Total*
Groundwater (Treatability Study Test 6)		TCL Organics TAL Inorganics General Water Quality Parameters	11	0	0	0	11	1	11
Treated Groundwater (5)									

Notes:

(1) One trip blank sample will accompany each shipping container used to transport VOC samples.
(2) Matrix spike/matrix spike duplicate (MS/MSD) analyses are required for organic analyses. Samples designated for MS/MSD analyses will be collected, with extra sample volumes, at a frequency of one per sampling event. Triple the normal sample volumes will be collected for VOC and double the normal volumes will be collected for extractable organics (BNA) and PCB/Pesticides.
(3) A Site Specific Parameter List (SSPL) will be developed based upon results of the groundwater monitoring program.
(4) Select metals to include TAL metals, total and soluble manganese and total and soluble iron.
(5) Parameters numbers of samples to be determined in conjunction with USEPA and OEPA.

C.4.2 ACCURACY, PRECISION AND SENSITIVITY OF ANALYSES

The fundamental QA objective with respect to the accuracy, precision, and sensitivity of analytical data is to achieve the QC acceptance criteria of each analytical protocol. The sensitivities required for these analyses will be at least the targeted detection limits listed on Tables C.4.2 and C.4.3. These tables present targeted detection limits for all parameters. It should be noted that these limits are targeted detection limits. Actual detection limits are highly matrix dependent.

TABLE C.4.2

TARGETED DETECTION LIMITS TCL/TAL PARAMETERS[1]
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter		Targeted Detection Limits	
		Water (µg/L)	Soil/Sediment (µg/kg)
VOLATILE COMPOUNDS			
67-64-1	acetone	10	10
71-43-2	benzene	10	10
75-27-4	bromodichloromethane	10	10
75-25-2	bromoform	10	10
74-83-9	bromomethane	10	10
78-93-3	butanone	10	10
75-15-0	carbon disulfide	10	10
56-23-5	carbon tetrachloride	10	10
108-90-7	chlorobenzene	10	10
75-00-3	chloroethane	10	10
67-66-3	chloroform	10	10
74-87-3	chloromethane	10	10
10061-01-15	cis-1,3-dichloropropene	10	10
124-48-1	dibromochloromethane	10	10
75-35-3	1,1-dichloroethane	10	10
107-06-2	1,2-dichloroethane	10	10
75-35-4	1,1-dichloroethene	10	10
540-59-0	1,2-dichloroethene (total)	10	10
78-87-5	1,2-dichloropropane	10	10
100-41-4	ethylbenzene	10	10
591-78-6	2-hexanone	10	10
75-09-2	methylene chloride	10	10
108-10-1	4-methyl-2-pentanone	10	10
100-42-5	styrene	10	10
79-34-5	1,1,2,2-tetrachloroethane	10	10
127-18-4	tetrachloroethene	10	10
108-88-3	toluene	10	10
10061-02-6	trans-1,3-dichloropropene	10	10
71-55-6	1,1,1-trichloroethane	10	10
79-00-5	1,1,2-trichloroethane	10	10
79-01-6	trichloroethene	10	10
75-01-4	vinyl chloride	10	10
1330-20-7	xylenes (total)	10	10

TABLE C.4.2

TARGETED DETECTION LIMITS TCL/TAL PARAMETERS[1]
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter		Targeted Detection Limits	
		Water	Soil/Sediment
CAS No.	Compound	(µg/L)	(µg/kg)
BASE/NEUTRAL COMPOUNDS			
83-32-9	acenaphthene	10	330
208-96-8	acenaphthylene	10	330
120-12-7	anthracene	10	330
56-55-3	benzo(a)anthracene	10	330
50-32-8	benzo(a)pyrene	10	330
205-99-2	benzo(b)fluoranthene	10	330
191-24-2	benzo(g,h,i)perylene	10	330
207-08-9	benzo(k)fluoranthene	10	330
111-91-1	bis(2-chloroethoxy)methane	10	330
111-44-4	bis(2-chloroethyl)ether	10	330
108-60-1	bis(2-chloroisopropyl)ether	10	330
117-81-7	bis(2-ethylhexyl)phthalate	10	330
85-68-7	butyl benzyl phthalate	10	330
101-55-3	4-bromophenylphenyl ether	10	330
86-74-8	carbazole	10	330
106-47-8	4-chloroaniline	10	330
91-58-7	2-chloronaphthalene	10	330
7005-72-3	4-chlorophenyl phenyl ether	10	330
218-01-9	chrysene	10	330
53-70-3	dibenz(a,h)anthracene	10	330
132-64-9	dibenzofuran	10	330
95-50-1	1,2-dichlorobenzene	10	330
541-73-1	1,3-dichlorobenzene	10	330
106-46-7	1,4-dichlorobenzene	10	330
91-94-1	3,3'-dichlorobenzidine	10	330
84-66-2	diethylphthalate	10	330
131-11-3	dimethylphthalate	10	330
84-74-2	di-n-butyphthalate	10	330
117-84-0	di-n-octylphthalate	10	330
121-14-2	2,4-dinitrotoluene	10	330
606-20-2	2,6-dinitrotoluene	10	330
206-44-0	fluoranthene	10	330
86-73-7	fluorene	10	330
118-74-1	hexachlorobenzene	10	330
87-68-3	hexachlorobutadiene	10	330
77-47-4	hexachlorocyclopentadiene	10	330
67-72-1	hexachloroethane	10	330

TARGETED DETECTION LIMITS TCL/TAL PARAMETERS[1]
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter		Targeted Detection Limits	
CAS No.	Compound	Water (µg/L)	Soil/Sediment (µg/kg)
193-39-5	indeno(1,2,3-cd)pyrene	10	330
78-59-1	isophorone	10	330
91-57-6	2-methylnaphthalene	10	330
91-20-3	naphthalene	10	330
88-74-4	2-nitroaniline	50	1,700
99-09-2	3-nitroaniline	50	1,700
100-01-6	4-nitroaniline	50	1,700
98-95-3	nitrobenzene	10	330
621-64-7	N-nitroso-di-n-propylamine	10	330
86-30-6	N-nitrosodiphenylamine (diphenylamine)	10	330
85-01-8	phenanthrene	10	330
129-00-0	pyrene	10	330
120-82-1	1,2,4-trichlorobenzene	10	330
ACID COMPOUNDS			
59-50-7	4-chloro-3-methylphenol	10	330
95-57-8	2-chlorophenol	10	330
120-83-2	2,4-dichorophenol	10	330
105-67-9	2,4-dimethylphenol	10	330
51-28-5	2,4-dinitrophenol	50	1,700
534-52-1	4,6-dinitro-2-methylphenol	50	1,700
95-48-7	2-methylphenol	10	330
106-44-5	4-methylphenol	10	330
88-75-5	2-nitrophenol	10	330
100-02-7	4-nitrophenol	50	1,700
87-86-5	pentachlorophenol	50	1,700
108-95-2	phenol	10	330
95-95-4	2,4,5-trichlorophenol	50	1,700
88-06-2	2,4,6-trichlorophenol	10	330
PESTICIDES			
309-00-2	Aldrin	0.05	1.7
319-84-6	alpha-BHC	0.05	1.7
319-85-7	beta-BHC	0.05	1.7
319-84-6	alpha-Chlordane	0.05	1.7
57-74-9	gamma-Chlordane	0.05	1.7
72-54-8	4,4'-DDD	0.10	3.3
72-55-9	4,4'-DDE	0.10	3.3
50-29-3	4,4'-DDT	0.10	3.3
319-86-8	delta-BHC	0.05	1.7
60-57-1	Dieldrin	0.10	3.3

TABLE C.4.2

TARGETED DETECTION LIMITS TCL/TAL PARAMETERS[1]
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter		*Targeted Detection Limits*	
CAS No.	*Compound*	*Water (µg/L)*	Soil/Sediment *(µg/kg)*
959-98-8	Endosulfan I	0.05	1.7
33213-65-9	Endosulfan II	0.10	3.3
1031-07-8	Endosulfan sulfate	0.10	3.3
72-20-8	Endrin	0.10	3.3
53494-70-5	Endrin ketone	0.10	3.3
58-89-9	gamma-BHC (Lindane)	0.05	1.7
76-44-8	Heptachlor	0.05	1.7
1024-57-3	Heptachlor epoxide	0.05	1.7
72-43-5	Methoxychlor	0.50	17
8001-35-2	Toxaphene	5.0	170
PCBs			
12674-11-2	AROCLOR 1016	1.0	33
11104-28-2	AROCLOR 1221	1.0	33
11141-16-5	AROCLOR 1232	2.0	67
53469-21-9	AROCLOR 1242	1.0	33
12672-29-6	AROCLOR 1248	1.0	33
11097-69-1	AROCLOR 1254	1.0	33
11096-82-5	AROCLOR 1260	1.0	33

TABLE C.4.2

TARGETED DETECTION LIMITS TCL/TAL PARAMETERS[1]
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter		*Targeted Detection Limits*	
CAS No.	*Compound*	*Water* (*µg/L*)	Soil/Sediment (*µg/kg*)
Aluminum		200	40
Antimony		60	12
Arsenic		10	2
Barium		200	40
Beryllium		5	1
Cadmium		5	1
Calcium		5000	1000
Chromium		10	2
Cobalt		50	10
Copper		25	5
Iron		100	20
Lead		3	0.6
Magnesium		5000	1000
Manganese		15	3
Mercury		0.2	0.04
Nickel		40	8
Potassium		5000	1000
Selenium		5	1
Silver		10	2
Sodium		5000	1000
Thallium		10	2
Vanadium		50	10
Zinc		20	4
Total Cyanide		10	0.5

Notes:

(1) Please note that the above are practical quantitation limits (PQL). Actual quantitation limits may be higher due to matrix interferences or a high concentration of a particular analyte.

TABLE C.4.3

TARGETED DETECTION LIMITS[1]
GENERAL WATER QUALITY AND SOLIDS PARAMETERS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Parameter	*Detection Limits*	
	Water *mg/L*	*Solid* *mg/kg*
Chloride	2	40
Sulfate	1	100
Phenols	0.05	5
Manganese	5	500
Iron	0.1	500
Sodium	5	500
Total Dissolved Solids	1.0	-
Total Suspended Solids	1.0	-

Notes:

(1) Detection Limits are highly matrix dependent and may not always be achievable.

C.4.3 COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

It is expected that all analyses conducted in accordance with SW-846, USEPA-WW and ASTM methods will provide data meeting QC acceptance criteria for 95 percent of all samples tested, with field measurements requiring a 90 percent level of completeness. Any reasons for variances will be documented. The corrective actions taken if the completeness goals are not met are described in Section C.14.0 of this QAPP. WAL will conduct the analyses according to the SOPs provided in Attachment C-1.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of a sampling program and proper laboratory protocol.

The sampling networks have been designed to provide data representative of Site conditions. The procedures used to obtain the planned analytical data are documented in this QAPP. However, it may be necessary to verify similar documentation for previous analytical data to adequately establish comparability. Comparability of laboratory analyses will be ensured by the use of consistent methodology and units of measurement. Following completion of data collection, the existing data base will be evaluated for representativeness.

C.4.4 FIELD MEASUREMENTS

Measurement data will be generated during field activities. These activities include, but are not limited to, the following:

1. documenting time and weather conditions;

2. determining pH, specific conductivity, and temperature of water samples;

3. determining depths in a well;

4. verifying well development and pre-sampling purge volumes; and

5. measuring groundwater elevations in wells.

The general QA objective for such measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the use of standardized procedures.

C.5.0 SAMPLING PROCEDURES

The procedures and protocols for collecting samples and for performing all related field activities are described in Section 3.0 of the RD Work Plan.

C.6.0 SAMPLE CUSTODY AND DOCUMENT CONTROL

This section details the procedures and protocols which must be followed for the labeling and transporting of samples.

C.6.1 LABELING OF SAMPLES

A unique sample numbering system, per CRA procedures, will be used to identify each collected sample. This system will provide a tracking number to allow retrieval and cross-referencing of sample information. A listing of the sample identification numbers with written descriptions of sample location, type and date will be maintained by CRA's field personnel. A typical example of a sample numbering system to be used is described as follows:

Example:		-	GW-060191-WP-0001
where:	GW	-	Designates sample type (GW-groundwater, SW - surface water, TW - treated water, S-soil)
	060191	-	date of collection presented as month/day/year
	WP	-	sampler's initials
	0001	-	sequential number starting with 0001 at the start of the project

QC samples will also be numbered with a unique location number similar to the example above to prevent possible laboratory bias.

CRA's on-Site representative(s) will be responsible for recording the sampling activities for each day and will record in the field log book the following with respect to each sample:

i) unique sample identification number;
ii) sampling location identification;
iii) date/time of sample collection; and
iv) notes on preservation.

C.6.2 CHAIN-OF-CUSTODY

A chain-of-custody record will be maintained to document the transfer of sample containers. Each sample will be properly sealed. Sample container labels will include sample number, place of collection and date and time of collection. Samples will be placed in the shipping cooler immediately after collection. Each sample number of each sample shipped will be recorded on the record. The shipping container will then be sealed properly for shipment.

Each sample container being shipped to the laboratory will contain a chain-of-custody form. The chain-of-custody form consists of four copies which are distributed to the sampler, to the shipper, to the project laboratory and to the office file of CRA. The sampler and shipper will maintain their copies while the other two copies are placed in a waterproof enclosure within the shipping container with the samples. The laboratory, upon receiving the samples, will complete the two remaining copies. The laboratory will maintain one copy for its records. The executed original will be returned to CRA with the data deliverables package.

Upon receipt of the container at the laboratory, the shipping container will be inspected and opened by the designated sample custodian. The condition of the shipping container and sample containers will be noted on the chain-of-custody record sheet by the sample custodian. The sample custodian will document the date and time of receipt of the container, and sign the form.

If damage or discrepancies are noticed, they will be recorded in the remarks column of the record sheet, dated and signed. Any damage or discrepancies will be reported to the lab supervisor who will inform the lab manager and QA officer.

C.6.3 SAMPLE DOCUMENTATION IN THE LABORATORY

The sample custodian will assign a unique number to each incoming sample for use in the laboratory. The unique number and CRA's number will then be entered into the sample receiving log. The laboratory date of receipt will also be noted.

The laboratory will be responsible for maintaining analytical log books and laboratory data as well as a sample (on hand) inventory for submittal to CRA on an "as required" basis. Samples will be maintained by the laboratory for a period of 30 days following CRA receipt of the respective sample data for additional analyses, if necessary. Raw laboratory data files will be inventoried and maintained by the laboratory for a period of five years, at which time CRA will advise the laboratory regarding the need for additional storage.

Laboratory custody procedures and document control for those samples analyzed by the project laboratories will be carried out as specified in the appropriate SOP included in Attachment C-1.

C.6.4 STORAGE OF SAMPLES

After the sample custodian has completed the sample log-in, the chain-of-custody will be checked to ensure that all samples are stored in the appropriate locations. All samples will be stored within an access controlled location and will be maintained at 4°C until completion of all analytical work or, as a minimum, for 30 days.

C.6.5 SAMPLE DOCUMENTATION - CRA

Evidential files for the entire project will be maintained by CRA and will consist of the following:

i) project plan;
ii) project log books;
iii) field data records;
iv) sample identification documents;
v) chain-of-custody records;
vi) analytical log books, data, etc.;
vii) correspondence;
viii) sample (on-hand) inventory;
ix) report notes, calculations, etc.;
x) references, literature;
xi) data packages - check out logs;
xii) miscellaneous - photos, maps, drawings, etc.; and
xiii) final report.

The evidentiary file materials will be the responsibility of the evidentiary file custodian (Project Co-ordinator) with respect to maintenance and document removal.

C.7.0 CALIBRATION PROCEDURES AND FREQUENCY

The procedures indicated below will be performed for all samples delivered for analysis to the project laboratory. Specific instructions relevant to a particular type of analysis are given in the pertinent analytical procedures for this project, and are referenced in Section C.8.0.

For environmental analyses, the instruments will be calibrated or the continuing calibration will be checked each day that analyses will be performed. The analyst will tune and calibrate the instrument and complete calibration records for the project file.

All quality control data and records produced from calibration and tuning will be retained by the laboratory and will be made available to CRA on an "as required" basis.

The following specific analytical quality control procedures are related to each analytical batch.

C.7.1 TUNING

The following requirements will be applied to those samples analyzed by gas chromatography/mass spectrometry (GC/MS). The calibration of each of these instruments will be verified at frequencies specified in the methods referenced in Section C.8.0. Where appropriate, a new standard curve may be prepared as specified in the methods.

The tune of each GC/MS system used for the determination of organic analytes will be checked with 4-bromofluorobenzene (BFB) for determination of volatiles and with decafluorotriphenylphosphine (DFTPP) for determination of semi-volatiles. The ion abundance criteria required by the method will be met before determination of any analytes. If the system does not meet the required specification of one or more of the specified ions, the instrument will be retuned and rechecked before proceeding with sample analysis. The tune performance check criteria will be achieved on a daily basis.

C.7.2 CALIBRATION

Prior to analysis, instruments will be calibrated using the procedures specified by the SOP for each method (see Attachment C-1).

C.7.2.1 Calibration Check

Method mandated compounds will be used to check calibration of the instrument being used. All calibration checks will be conducted prior to any batch analysis.

C.7.2.2 Standard Curves for Inorganic Analysis

Standard curves used in the determination of inorganic analytes will be prepared as follows:

Typically, standard curves derived from data consisting of one reagent blank and a minimum of three concentrations will be prepared for each inorganic analyte. The standard curve will be used with each subsequent analysis, provided the standard curve is verified by using at least one reagent blank and one standard at a level normally encountered or expected in such samples. If the results of the verification are not within +/-10% of the original curve, a new standard will be prepared and analyzed. If the results of the second verification are not within +/-10% of the original

standard curve, a reference standard will be used to determine if the discrepancy is with the standard or with the instrument. New standards will also be prepared on a quarterly basis at a minimum. All data used in drawing or describing the curve will be so indicated on the curve or its description. A record will be made of the verification.

C.7.2.3 Field Instrument Calibration

Calibration of the field instruments will be done in the field prior to the collection of each water sample if well purging data indicate a change (>±10 percent) in pH and/or conductivity from the last location sampled. Calibration of field instruments will be conducted at least daily during groundwater sampling. The field equipment will be maintained, calibrated and operated in a manner consistent with the manufacturer's guidelines and USEPA standard methods. However, since the majority of field measurements will be limited to pH, conductivity, temperature and depth (water level) the following procedures will be conducted, at a minimum:

i) pH

- The pH meter will be calibrated with commercially obtained pH 7, 4 and 10 buffer solutions. The pH calibration will automatically be temperature compensated by the pH meter, and will be performed immediately before initiating a sampling event. Calibration checks will be performed with every sample collected. In the event that

the result fails to be within 0.1 pH units, the meter must be recalibrated and all samples after the last calibration must be remeasured.

- Field calibration will be performed in accordance with the following procedure:

  1. rinse the probe in deionized water;
  2. insert probe in a fresh pH 7 buffer solution;
  3. slide battery compartment cover back to the first stop exposing the adjustment potentiometers;
  4. adjust the "CAL" potentiometer such that the display reads 7.00;
  5. remove the probe and rinse in deionized water;
  6. insert probe in a fresh pH 4 or pH 10 buffer solution;
  7. adjust the slope potentiometer until the correct pH is displayed; and
  8. remove probe and rinse in deionized water.

ii) Conductivity

- Check once per sampling event against a standard solution of potassium chloride and deionized water.

- The specific conductivity meter is factory calibrated, but the calibration should be checked periodically in the field and the probe

thoroughly rinsed between samples. Field calibration of the specific conductivity meter will be performed as follows:

1. rinse probe in deionized water;
2. wipe probe and allow to dry;
3. the conductivity displayed should be zero in air;
4. adjust the zero potentiometer if necessary;
5. immerse the probe in a NBS (NIST) traceable solution of known conductivity;
6. adjust the "SPAN" potentiometer such that the correct conductivity is displayed; and
7. rinse probes thoroughly with deionized water and allow to dry.

C.7.2.4 Geotechnical Equipment

The calibration procedures and frequencies for geotechnical testing equipment is presented in Attachment C-1.

C.8.0 ANALYTICAL PROCEDURES

This section presents the analytical methods which will be employed by the project laboratories to complete all required analyses.

C.8.1 OVERVIEW

Samples collected for chemical analyses will be analyzed using SW-846 and ASTM methods. The analyses will be performed in a manner consistent with the analytical methods listed in Table C.8.1 and detailed in the respective SOPs included in Attachment C-1.

TABLE C.8.1

ANALYTICAL METHODS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Paramater	*Method*
TAL/TCL	
TCL - Volatile Organics	SW-846 8240(1)
TCL - Base Neutrals and Acids	SW-846 8270
TCL - Pesticides/PCB	SW-846 8080
TAL - Inorganics	SW-846 6010/7000 Series, 9010
General Water Quality	
Chloride	SW-846 9250/9251
Sulfate	SW-846 9035/9036
Phenols	SW-846 9065/9066
Manganese	SW-846 6010
Iron	SW-846 6010
Sodium	SW-846 6010
Total Suspended Solids (TSS)	USEPA-WW 160.2 (2)
Total Dissolved Solids (TDS)	USEPA-WW 160.1
Geotechnical Analyses	
Grain Size Distribution	ASTM D422(3)

Notes:

(1) SW-846: "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, 3rd Edition, November 1986.

(2) USEPA-WW "Methods for the Chemical Analysis of Water and Wastes" EPA 600/4/79-020, revised March 1983.

(3) ASTM "1990 Book of ASTM Standards", American Society for Testing and Materials, 1990.

C.8.2 IDENTIFICATION

Identification of all targeted analytes will be accomplished with an authentic standard of the analyte. When authentic standards are not available (i.e., for non-target compounds) identification will be considered tentative.

For gas chromatographic determinations of specific analytes, the relative retention time of the unknown will be compared with that of an authentic standard. Since a true identification by GC is not possible, an analytical run for compound confirmation will be followed according to the specifications in the methods. Peaks must elute within daily retention-time windows established for each indicator parameter to be declared a tentative or confirmed identification. Retention time windows are determined by a standard 72-hour study as defined in the respective method. Results of the study are to be filed in the laboratory and made available for inspection during a QC audit.

For GC/MS determinations of specific analytes, the spectrum of the analyte will conform to a literature representation of the spectrum or to a spectrum of the authentic standard obtained after satisfactory tuning of the mass spectrometer. The appropriate analytical methods will be consulted for specific criteria for matching the mass spectra, relative response factors, and relative retention times to those of authentic standards.

C.8.3 QUANTIFICATION

The procedures for quantification of analytes are discussed in the appropriate standard operating procedures presented in Attachment C-1.

C.8.4 DETECTION LIMIT AND QUANTIFICATION LIMIT

The data used to evaluate the Site activities will have method detection limits that are consistent with the appropriate SW-846, USEPA-WW and ASTM methods.

The targeted detection limits for chemical analyses were previously presented on Tables C.4.2 and C.4.3.

Specific detection limits are highly matrix dependent. The detection limits in Tables C.4.2 and C.4.3 are provided for guidance and may not always be technically achievable.

C.9.0 DATA REDUCTION, VALIDATION, ASSESSMENT AND REPORTING

The project laboratories will perform analytical data reduction and validation in-house under the direction of the laboratory QA/QC officers. The laboratory QA/QC officers will be responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other qualifications based on laboratory QC criteria. Data reduction, validation and reporting by each laboratory will be conducted as detailed in the following:

i) raw data produced and checked by the responsible analyst is turned over for independent review by another analyst;

ii) the area supervisor reviews the data for attainment of quality control criteria presented in the referenced analytical methods;

iii) upon completion of all reviews and acceptance of the raw data by the laboratory operations manager, a report will be generated and sent to the laboratory quality assurance officer;

iv) the laboratory quality assurance officer will complete a thorough inspection of all reports;

v) the QA officer and area supervisor will decide whether any sample re-analysis is required; and

vi) upon acceptance of the preliminary reports by the QA officer, final reports will be generated and signed by the laboratory manager;

It should be noted, however, that "sign-off" will be required following completion of each step.

CRA's QA/QC Officer - Analytical Activities will conduct an evaluation of data reduction and reporting by the laboratories. These evaluations will consider the finished data sheets, field blank data, field duplicate data and recovery data for surrogate and matrix spikes. The material will be checked for legibility, completeness, correctness and the presence of requisite dates, initials, and signatures. The results of these checks will be assessed and reported to the project managers noting any discrepancies and their effect upon the acceptability of the data. All information garnered for QA/QC checks will be discussed in the final report.

Validation of the analytical data will be performed by CRA's QA/QC Officer - Analytical Activities based on the QC criteria outlined in "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses", February 1, 1988 and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses", July 1, 1988. Assessment of analytical and field data will include checks for data consistency by looking for comparability of duplicate analyses, potential sample contamination as indicated by results of rinsate blank sample analyses, laboratory QA procedures, adherence to accuracy and precision

criteria, transmittal errors and anomalously high or low parameter values. Organic analyses will be evaluated for the following:

i) sample holding time;

ii) GC/MS tuning;

iii) calibration;

iv) blanks;

v) surrogate recoveries;

vi) matrix spike/matrix spike duplicates;

vii) field duplicates;

viii) internal standard performance; and

ix) overall assessment of data.

Inorganic analyses will be evaluated for the following:

i) holding times;

ii) calibration;

iii) blanks;

iv) ICP Interference check samples;

v) laboratory control samples;

vi) duplicate sample analyses;

vii) matrix spike sample analyses;

viii) furnace atomic absorption quality control;

ix) field duplicate; and

x) overall assessment of data.

The criteria by which the data will be evaluated will be those specified in the appropriate method. Where no criteria are established within the method, laboratory established control limits will be used. Any qualification of sample data will be based on the recommendations in the guidelines with the appropriate data qualifiers. The results of these data validations will be reported to the project managers, noting any discrepancy and their effect upon acceptability of the data.

Validation of geotechnical data will be performed by CRA's QA/QC Officer - Field Activities based on the QC requirements of the methods. The results of these checks will be assessed and reported to the project manager noting any discrepancies and their effect upon the acceptability of the data.

Raw data from field measurements and sample collection activities that are used in the project reports will be appropriately identified and appended to the report. Where data have been reduced or summarized, the method of reduction will be documented in the report. In addition, field data will be audited for anomalously high or low values that may appear to be inconsistent with other data.

The data packages supplied by the project laboratories will be consistent with the following format:

i) a case narrative that includes summary of analytical methods used and description of any unusual action or conditions;

ii) dates of sample receipt, extraction (if applicable) and analysis;

iii) method blank samples analyses summaries;

iv) tuning and calibration summaries;

v) surrogate and matrix spike/matrix spike duplicate recovery data and control limits;

vi) ICP and furnace atomic absorption spectroscopy QC;

vii) check sample data; and

viii) executed chain-of-custody forms.

C.10.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

This section presents the internal quality control checks and frequency procedures which will be employed for field and laboratory measurements.

C.10.1 FIELD QC

Quality control procedures for field measurements will be limited to checking the reproducibility of the measurement in the field by obtaining multiple readings and by calibrating the instruments (where appropriate).

Quality control of field sampling will involve collecting field duplicates and field blanks in accordance with the applicable procedures described in Sections C.4.1 and C.5.0, and the level of effort indicated in Table C.4.1.

C.10.2 LABORATORY QC

Specific procedures related to internal laboratory QC samples (namely, matrix spikes, surrogate spikes, blanks, QC check samples and matrix spike duplicates) are detailed in the following subsections.

The internal QC checks for the analyses will follow the appropriate methods specified in Table C.8.1, and criteria outlined in the applicable SOPs presented in Attachment C-1.

C.10.2.1 Blanks

A reagent blank will be analyzed by the laboratory at a frequency of one blank per 20 analyses or, in the event that an analytical batch consists of less than 20 samples, one reagent blank will be analyzed. The reagent blank, an aliquot of analyte-free water or solvent, will be carried through the entire analytical procedure.

C.10.2.2 Matrix Spike/Matrix Spike Duplicates

A matrix spike and matrix spike duplicate (MS/MSD) sample set will be analyzed at a minimum frequency of one per 20 investigative samples. Acceptable criteria and compounds that will be used for matrix spikes are identified in the appropriate methods (see Section C.8.0).

However, Table C.10.1 presents a summary of the compounds and acceptable criteria. Percent spike recoveries will be used to evaluate analytical accuracy while percent relative standard deviation or percent difference between the spike and matrix spike duplicate will be used to assess analytical precision.

Matrix spike and laboratory duplicates acceptance criteria for inorganic analyses will be those in the Functional Guidelines as referenced in Section C.9.0 on page 2 of 5.

C.10.2.3 Surrogates

Surrogates are used in all GC and GC/MS analyses. Every blank, standard, and environmental sample including MS/MSD samples will be spiked with surrogate compounds prior to purging volatiles or extracting semi-volatiles.

Surrogates will be spiked into samples according to the appropriate analytical methods. Surrogate spike recoveries will fall within the control limits set by procedures specified in the method for analytes falling within the quantification limits without dilution. Dilution of samples to bring the analyte concentration into the linear range of calibration may dilute the surrogates out of the quantification limit; assessment of analytical quality in these cases will be based in the quality control embodied in the check, matrix spike and matrix spike duplicate samples.

TABLE C.10.1

TCL ORGANIC COMPOUNDS
MATRIX SPIKE/MATRIX SPIKE DUPLICATE
PERCENT RECOVERY AND RELATIVE PERCENT
DIFFERENCE CONTROL LIMITS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

VOC (1):	*Water Recovery (%) Control Limits*		*Soil Recovery (%) Control Limits*	
1,1-Dichloroethene	61-145	(14)(2)	59-172	(22)
Trichloroethene	71-120	(14)	62-137	(24)
Chlorobenzene	75-130	(13)	60-133	(21)
Toluene	76-125	(13)	59-139	(21)
Benzene	76-127	(11)	66-142	(21)
BNA:				
1,2,4-Trichlorobenzene	39-98	(28)	38-107	(23)
Acenaphthene	46-118	(31)	31-137	(19)
2,4-Dinitrotoluene	24-96	(38)	28-89	(47)
Pyrene	26-127	(31)	35-142	(36)
N-Nitroso-Di-n-Propylamine	41-116	(38)	41-126	(38)
1,4-Dichlorobenzene	36-97	(28)	28-104	(27)
Phenol	12-89	(42)	26-90	(35)
2-Chlorophenol	27-123	(40)	25-102	(50)
4-Chloro-3 methylphenol	23-97	(42)	26-103	(33)
4-Nitrophenol	10-80	(50)	11-114	(50)
Pentachlorophenol	9-103	(50)	17-109	(47)
Pesticides/PCB:				
Lindane	56-123	(15)	46-127	(50)
Heptachlor	40-131	(20)	35-130	(31)
Aldrin	40-120	(22)	34-132	(43)
Dieldrin	52-126	(18)	31-134	(38)
Endrin	56-121	(21)	42-139	(45)
4,4-DDT	38-127	(27)	23-134	(51)

Notes:

(1) VOC = Volatile Organic Compounds.
(2) Relative percent difference (RPD) control limits in parentheses.
BNA Base/Neutral and Acid Extractable Organic Compounds.
PCB Polychlorinated Biphenyls.

Table C.10.2 presents a summary of the surrogate compounds recovery control limits as stated within the analytical methods.

C.10.2.4 Blind Check Samples

As supplied by the agencies, an analytical batch may contain a blind check sample. In general, the blind check sample will be obtained from USEPA and supplied to CRA. The analytes employed in this check sample will be a representative subset of the analytes of interest.

Standard deviations and relative standard deviations will be calculated for the percent recovery of analytes from the check samples. These are defined in Section C.13.2.

C.10.3 GEOTECHNICAL TESTING QC

Quality control procedures for geotechnical testing will be limited to those specified in ACT Quality Assurance Manual reproduced in Attachment C-1.

TABLE C.10.2

ORGANIC COMPOUNDS
SURROGATE COMPOUNDS PERCENT RECOVERY CONTROL LIMITS
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

	Control Limits	
VOC	*Soil*	*Water*
Toluene-d8	81-117	88 - 110
4-Bromo fluorobenzene	74-121	86 - 115
1,2-Dichloroethane-d4	70-121	76 - 114
BNA		
Nitrobenzene-d5	23-120	37 - 110
2-Fluorobiphenyl	18-137	29 - 107
Terphenyl-d14	30-115	10 - 133
Phenol-d5	24-113	10 - 114
2-Fluorophenol	25-121	25 - 90
2,4,6-Tribromophenol	19-122	10 - 143
Pesticides/PCB		
DBC Dibutylchlorendate (DCB)	24-150	24 - 154
TCMX Tetrachloro-m-xylene (TCMX)	60-150	60 - 150

Notes:

VOC = Volatile Organic Compounds
BNA = Base/Neutral and Acid Extractable Organic Compounds
PCB = Polychlorinated Biphenyls

C.11.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

For the purpose of external evaluation, performance evaluation check samples from the USEPA and various State agencies are analyzed periodically by the project laboratory.

Internally, the evaluation of data from these samples is done on a continuing basis over the duration of a given project.

The CRA QA/QC Officer - Analytical Activities may carry out performance and/or systems audits to ensure that data of known and defensible quality are consistently produced during a program.

Systems audits are qualitative evaluations of all components of field and laboratory quality control measurement systems. They determine if the measurement systems are being used appropriately. The audits may be carried out before all systems are operational, during the program, or after the completion of the program. Such audits typically involve a comparison of the activities given in the QA/QC plan described herein, with activities actually scheduled or performed. A special type of systems audit is the data management audit. This audit addresses only data collection and management activities.

The performance audit is a quantitative evaluation of the measurement systems used for a monitoring program. It requires testing the measurement systems with samples of known composition or behavior to

evaluate precision and accuracy. A performance/system audit may be carried out by or under the auspices of the USEPA Region V-CRL, without the knowledge of the analyst during each sampling event for this program. The Scheduling of Performance Evaluation (PE) audits will be at the discretion of USEPA.

In addition, one external QA audit may be conducted by CRA prior to the analyses of any investigatory samples. However, it should be noted that any additional external QA audits will only be performed if deemed necessary by either the SNFT or CRA project managers or the CRA QA/QC Officer - Analytical Activities. The project laboratory may also undergo PE audit(s) by the USEPA, if so requested.

C.12.0 PREVENTIVE MAINTENANCE

All analytical instruments to be used in this project will be serviced by the laboratory personnel at regulatory scheduled intervals in accordance with the manufacturers recommendations. Instruments may also be serviced at other times due to failure. Requisite servicing beyond the abilities of the laboratory personnel will be performed by the equipment manufacturer or their designated representative.

Daily checks of each instrument will be by the analyst who has been assigned responsibility for that instrument. This will include changing GC inlet liners, tuning GC/MS, checking operation of data systems, checking for leaks, etc. Manufacturer's recommended procedures will be followed in every case.

The pH and conductivity meters will be calibrated in the field as described in Section C.7.2.3. In addition, the following preventative maintenance measures will be taken in the field:

pH, Conductivity
- Keep probes clean and free of dirt by rinsing with deionized water.
- Keep deionized water around probes to prevent dehydration.

Water Level Tape - Clean probe and lower 3 feet of tape with pesticide grade isopropanol and deionized water to prevent hard water and iron buildup.

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

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy and completeness achieved during the associated sample analyses.

C.13.1 QA MEASUREMENT QUALITY INDICATORS

C.13.1.1 Precision

Precision will be assessed by comparing the analytical results between MS/MSD compounds (organics) and duplicate samples (inorganics).

C.13.1.2 Accuracy

Accuracy will be assessed by comparing a set of analytical results to the accepted or "true" values that would be expected. In general, matrix spike, matrix spike duplicates and check sample recoveries will be used to assess accuracy.

C.13.1.3 Outliers

Procedures discussed previously will be followed for documenting deviations. In the event a result deviates significantly from established control limits reported by the laboratory, this deviation will be noted and its effect on the quality of the remaining data assessed and documented.

C.13.1.4 Completeness

The required level of completeness for field measurements will be 90 percent, while the required level of data completeness for laboratory analyses will be 95 percent.

C.13.2 STATISTICAL EVALUATIONS

In examination of data and determination of its precision and accuracy, standard statistical formulae will be used.

C.13.2.1 Arithmetic Mean

The arithmetic mean is the average obtained by dividing a sum by the number of its addends. A number of recovery results are averaged together to improve the accuracy of the measurements. Figure C.13.1, equation 1, presents the formula to be used to determine the arithmetic mean.

C.13.2.2 Standard Deviation

The standard deviation is the square root of the average squared difference between the individual values and the average value. A number of recovery results are evaluated to find the numerical variation in the data which is then used in the determination of the percent relative standard deviation. Figure C.13.1 equation 2, presents the formula to be used to determine the standard deviation.

Equation 1 Determination of Arithmetic Mean ($\bar{X}$)

$$\bar{X} = \frac{\sum_{i=1}^{n} x_i}{n}$$

where n = number of measurements
x_i = value of measurements

Equation 2 Determination of Standard Deviation (σ_{n-1})

$$\sigma_{n-1} = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{X})^2}{n - 1}}$$

where n = number of measurements
x_i = value of measurements

Equation 3 Determination of Percent Relative Standard Deviation (% RSD)

$$\% \text{ RSD} = \frac{\sigma_{n-1}}{\bar{X}} \times 100$$

where σ_{n-1} = standard deviation
$\bar{X}$ = arithmetic mean

Equation 4 Determination of Percent Recovery (%R)

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

where SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

Equation 5 Determination of Relative Percent Difference (RPD)

$$RPD = \left(\frac{|R_1 - R_2|}{\frac{R_1 + R_2}{2}} \right) \times 100$$

where R_1 = value of first result
R_2 = value of second result

figure C.13.1

STATISTICAL FORMULAE
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township of Portage County, Ohio

CRA

2372-18/07/91-13-0

C.13.2.3 Percent Relative Standard Deviation

The percent relative standard deviation (%RSD) is the percentage obtained by dividing the standard deviation of the values by the arithmetic mean of the values. The %RSD is calculated on a series of measurements to evaluate an instrument's analytical precision (e.g. initial calibration). Figure C.13.1, equation 3, presents the formula to be used to determine %RSD.

C.13.2.4 Percent Recovery

The percent recovery (%R) of a parameter is the percentage obtained by dividing the amount recovered by the true amount added. The percent recoveries of spiked samples are evaluated to establish the analytical accuracy of a measurement. Figure C.13.1, equation 4, presents the formula to be used to determine the percent recovery.

C.13.2.5 Relative Percent Difference

The relative percent difference (RPD) is the percentage obtained by dividing the difference between two numbers by their arithmetic mean. The RPD is used to evaluate the analytical precision of two replicate measurements (e.g. matrix spike/matrix spike duplicate). Figure C.13.1, equation 5, presents the formula to be used to determine RPD.

C.14.0 CORRECTIVE ACTION

The need for corrective action may be identified by system or performance audits or by standard QC procedures. The essential steps in the corrective action system will be as follows:

i) checking the predetermined limits for data acceptability beyond which corrective action is required;

ii) identifying and defining problems;

iii) assigning responsibility for investigating the problem;

iv) investigating and determining the cause of the problem;

v) determination of a corrective action to eliminate the problem (this may include resampling and/or reanalyses);

vi) assigning and accepting responsibility for implementing the corrective action;

vii) implementing the corrective action and evaluating the effectiveness;

viii) verifying that the corrective action has eliminated the problem; and

ix) documenting the corrective action taken.

For each measurement system, the QA/QC Officer - Laboratory Activities will be responsible for initiating the corrective action and the laboratory supervisor will be responsible for implementing the corrective action. The corrective action taken will depend upon the QA/QC criteria that did not meet the necessary criteria, and may range from qualifying the data to resampling on the Site.

C.15.0 QUALITY ASSURANCE REPORT TO MANAGEMENT

Management will receive reports on the performance of the measurement system and data quality following each sampling round and at the conclusion of the project.

Minimally, these reports will include the following:

i) assessment of measurement quality indicators, i.e., data accuracy, precision and completeness;

ii) results of system audits; and

iii) QA problems and recommended solutions.

The CRA QA/QC Officer - Analytical Activities will be responsible within the organizational structure for preparing these reports. The final report for the project will also include a separate QA section which will summarize data quality information contained in the periodic QA/QC reports to management, and details an overall data assessment and validation in accordance with the data quality objectives outlined in this QAPP.

ATTACHMENT C-1
LABORATORY STANDARD OPERATING PROCEDURES

- WADSWORTH/ALERT LABORATORIES, INC.

- APPLIED CONSTRUCTION TECHNOLOGY, INC.

WADSWORTH/ALERT LABORATORIES

STANDARD OPERATING PROCEDURES

SAMPLE TRACKING SOP

I. The Sample Custodian will examine the shipping container and record the following information on the Cooler Receipt Form (see attachment):

A. Presence/absence of custody seal(s) on the shipping container(s)

B. Condition of custody seal (e.g. intact, broken)

II. The Sample Custodian will open the shipping container, remove the enclosed sample documents and record on the Cooler Receipt Form:

A. Presence/absence of the chain-of-custody record(s)

B. Presence/absence of airbills and/or bills of lading documenting shipment of samples

III. Remove sample containers and record on the Cooler Receipt Form:

A. Condition of the samples (e.g. intact, broken, leaking, etc.)

B. Presence/absence of sample labels and completeness

The Sample Custodian is to wear disposable latex gloves while handling samples. Dirty sample containers are to be cleaned appropriately so as not to contaminate the sample storage area.

IV. Compare the following documents to verify agreement among the information contained on them:

A. Chain-of-Custody Records

B. Sample Labels

C. Airbills or Bills of Lading

Document both agreement among the forms and any discrepancies found on the Cooler Receipt Form.

V. The Sample Custodian will measure the pH of aqueous samples with pH paper to determine whether or not the sample was properly preserved. If the sample was submitted for volatiles analysis and only one vial was received, the group leader will be notified and the pH will be taken immediately prior to sample analysis. If all samples recorded on the chain-of-custody record were received by the laboratory and there are no problems observed with the sample shipment, the Sample Custodian will sign the chain-of-custody record in the "Received for Laboratory by:" box on the document. If problems are noted, sign for shipment and note the problems in the remarks box of the chain-of-custody record. Alert the Project Manager to contact the client for all but the most minor discrepancies.

VI. The Quote/Sample Analysis Request Form (QUOTE/SAR) is reviewed to ensure that the chain-of-custody and the QUOTE/SAR agree. If they do not agree, the Project Manager is notified of the discrepancy.

VII. The samples are then logged onto the laboratory computer system. A unique identification number is assigned to each sample and worksheets are printed. The laboratory ID# assigned to each sample will be logged in the Sample Receiving Logbook.

VIII. The Sample Custodian will assure that each sample container is labeled with a unique laboratory identification number (that cannot be accidentally removed in the laboratory).

IX. The label will contain the following information:

A. Laboratory ID Number

B. Sampling Date

C. Storage Location

D. Client Code

E. Number and Size of Bottles Received

X. The Sample Custodian will put the samples into sample storage according to the Sample Storage SOP. Samples are to be placed inside of sample storage refrigerators at 4°C.

XI. The Sample Custodian will file the shipping documents, chain-of-custody, QUOTE/SAR, related correspondence, and receipt form with the analytical summary. The summary and supporting documentation are reviewed by the Sample Control Coordinator for accuracy before being submitted to Data Processing.

XII. The worksheets will be distributed to the appropriate group leaders.

XIII. A. It will be the responsibility of the Sample Custodian during normal business hours to log samples in and out of their storage locations for the appropriate analysts. Outside normal business hours, the responsibility will be that of the analyst.

B. Following the SOP assures that all samples are either in the custody of a person or are stored in a secure area during the entire time from sample receipt to completion of the analysis.

C. For the purpose of this SOP, sample custody is defined as:

1. The sample is in your actual possession.

2. The sample is in your view after being in your physical possession.

3. The sample is in a secure area.

XIV. The samples are held by the Laboratory for thirty days following the final report. Samples will be properly disposed of after thirty days, unless notified in writing. (See Chapter 28 for Laboratory Waste Disposal SOP)

XV. To ensure that holding times are not exceeded, computer-generated sheets called "Widgets" are given to each group on a daily basis. The widget contains a brief summary of each sample in-house for a given parameter. This information includes:

A. Laboratory ID Number

B. Storage Location

C. Client Code

D. Days Remaining in Holding Time

E. Days Remaining to Meet Client Deadline

F. Matrix

G. A Space for Code Denoting Analytical Process

XVI. Samples will be archived by client request for possible further analyses at a future date. Archived samples will be stored at 4°C as space permits.

Laboratory Sample Log

[illegible] LABORATORIES, INC. 890921
[illegible]

1884 [illegible] INITIALS _____ RECEIVING DATE : 890921 DATE REPORTED [illegible]
BNO. B: 31438 MATRIX: WATER DUE DATE: 891003 STOR. LOC: N/A
SAMPLE ID: AIR STRIPPED EFFLUENT 9-20-89
COMMENTS: CC OF REPORT TO D. STOTHERS.

ANALYSIS: FFH

1885 [illegible] INITIALS _____ RECEIVING DATE : 890921 DATE REPORTED [illegible]
BNO. B: 31439 MATRIX: WATER DUE DATE: 891003 STOR. LOC: W-7
SAMPLE ID: PLANT EFFLUENT GRAB
COMMENTS:

ANALYSIS: [illegible] FFH [illegible]

1886 [illegible] INITIALS _____ RECEIVING DATE : 890921 DATE REPORTED [illegible]
BNO. B: 31460 MATRIX: N/A DUE DATE: 891003 STOR. LOC: N/A
SAMPLE ID: ADDITIONAL REPORT
COMMENTS:

ANALYSIS: INVOICE

1887 [illegible] INITIALS _____ RECEIVING DATE : 890921 DATE REPORTED [illegible]
BNO. B: 31461 MATRIX: WATER DUE DATE: 891003 STOR. LOC: W6644
SAMPLE ID: INTERCEPTOR B2 9-20-89
COMMENTS:

ANALYSIS: CHLIQ FFH METALS O&G

BNO. B: 31462 MATRIX: WATER DUE DATE: 891003 STOR. LOC: W6644
SAMPLE ID: INTERCEPTOR B3 9-20-89
COMMENTS:

ANALYSIS: CHLIQ FFH METALS O&G

BNO. B: 31463 MATRIX: WATER DUE DATE: 891003 STOR. LOC: W6644
SAMPLE ID: INTERCEPTOR B4 9-20-89
COMMENTS:

ANALYSIS: CHLIQ FFH METALS O&G

1888 [illegible] INITIALS _____ RECEIVING DATE : 890921 DATE REPORTED [illegible]
BNO. B: 31464 MATRIX: WATER DUE DATE: 891003 STOR. LOC: W80X
SAMPLE ID: TEST SITE 31H00035602 9-21-89
COMMENTS:

ANALYSIS: [illegible]

DATA DELIVERABLES SOP

1.0 Scope and Application

This method lists the specific data deliverables which can be generated for an analytical report.

2.0 Summary of Method

The specific components of an analytical report may vary depending upon the requirements of a particular project. This method identifies those items which are included in a standard analytical report and those items which may be added as an optional data deliverable.

3.0 Standard Data Deliverables

Each analytical report generated by Wadsworth/ALERT Laboratories includes the following information:

General Information

1. Cover Page - which lists the site and project name
2. Project Narrative - summarizes the scope of the project including the number of samples contained in the analytical report.
3. Analytical Methods Summary - table listing the associated methods utilized during the project.
4. Copy of the Chain-of-Custody

Analytical Results

1. Analytical results for each sample
2. The parameters of interest
3. The detection limits of analysis
4. Clients sample identification
5. Laboratory sample identification number
6. Matrix identification
7. Date of sample collection

3.0 Standard Data Deliverables (continued)

Analytical Results

8. Date of sample receipt

9. Date of sample preparation/extraction

10. Date of sample analysis

11. Surrogate recoveries (where applicable by method)

Quality Control

In addition to the analytical results and general information data a quality control section is included which contains the following information:

1. Quality control definitions - identifies the basic requirements of the Wadsworth/ALERT Laboratories, Inc. QC program.

2. Quality control narrative - identifies any QA or sample problems encountered, required deviations from protocols and corrective actions taken.

3. Results of matrix spike data - includes percent recoveries and control limits.

4. Results of reagent blank data - included for each parameter analyzed.

4.0 Additional Data Deliverable Options

This section lists additional options which may be requested for inclusion in the analytical report.

Quality Control Section

1. Sample summary (summary of client ID to Lab ID)

2. QC check sample results

3. N.Y.S.D.E.C. Summary Forms

4. Instrument blank results

4.0 Additional Data Deliverable Options (continued)

Quality Control Section

5. Definition of surrogates
6. Relative Percent Deviation (RPD) for matrix spike data
7. Sample clean-up techniques employed
8. Dilution Factor Summary Table
9. Concentration of surrogates
10. Concentration of spiking compounds
11. Laboratory Chronicle (summary of preparation and analysis dates).
12. Manufacturers Lot Numbers for QC check samples and spiking solutions

GC/MS Section

1. Tuning Summary Forms
2. Tuning raw data
3. Initial and Continuing Calibration Summary Forms
4. Chromatograms for:
   a. samples
   b. standards
   c. blanks
   d. matrix spike/matrix spike duplicate
   e. check samples
5. Quantitation report
6. Mass spectral matches for target analytes
7. Summary Form for Tentatively Identified Compounds
8. Spectra for Tentatively Identified Compounds
9. Internal Standard Area Summary Form
10. Instrument detection limits

4.0 Additional Data Deliverable Options (continued)

Metals

4. Copies of instrument logbook pages
5. Copies of sample preparation logbook pages
6. Instrument detection limits
7. Dilution factors
8. Internal Chain-of Custody

Inorganics

1. Initial and Continuing Calibration Summary Form
2. Instrument printouts
3. Copies of instrument logbook pages
4. Copies of preparation logbook pages
5. Instrument detection limits
6. Dilution factors
7. Instrument blanks
8. Internal Chain-of-Custody

METHOD 8240

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS.

1.0 SCOPE AND APPLICATION

1.1 Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds may be determined by this method:

		Appropriate Technique
Analyte	CAS No.[b]	Purge and trap
Acetone	67-64-1	pp
Benzene	71-43-2	a
Bromochloromethane (I.S.)	74-97-5	a
Bromodichloromethane	75-27-4	a
4-Bromofluorobenzene(surr.)	460-00-4	a
Bromoform	75-25-2	a
Bromomethane	74-83-9	a
2-Butanone	78-93-3	pp
Carbon disulfide	75-15-0	pp
Carbon tetrachloride	56-23-5	a
Chlorobenzene	108-90-7	a
Chlorobenzene-d5 (I.S.)	108-90-7	a
Chlorodibromomethane	124-48-1	a
Chloroethane	75-00-3	a
Chloroform	67-66-3	a
Chloromethane	74-87-3	a
1,1-Dichloroethane	75-34-3	a
1,2-Dichloroethane	107-06-2	a
1,2-Dichloroethane-d4 (surr.)	107-06-2	a
1,1-Dichloroethene	75-35-4	a
1,2-Dichloroethene, total	540-59-0	a
1,2-Dichloropropane	78-87-5	a
cis-1,3-Dichloropropene	10061-01-5	a
trans-1,3-Dichloropropene	1464-53-5	a
1,4-Difluorobenzene (I.S.)	123-91-1	a
Ethylbenzene	100-41-4	a
2-Hexanone	591-78-6	pp
Methylene chloride	75-09-2	a
4-Methyl-2-pentanone	108-10-1	pp
Styrene	100-42-5	a
1,1,2,2-Tetrachloroethane	79-34-5	a
Tetrachloroethene	127-18-4	a
Toluene	108-88-3	a
Toluene-d8 (surr.)	108-88-3	a
1,1,1-Trichloroethane	71-55-6	a
1,1,2-Trichloroethane	79-00-5	a
Trichloroethene	79-01-6	a
Vinyl acetate	108-05-4	a
Vinyl chloride	75-01-4	a
Xylene (Total)	1330-20-7	a

a Adequate response by this technique.
b Chemical Abstract Services Registry Number. PPPoor purging efficiency resulting in high PQLs.
i Inappropriate technique for this analyte. PCPoor chromatographic behavior.

1.2 Method 8240 can be used to quantify most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. See Table 4 for a list of compounds, retention times, and their characteristic ions that have been evaluated on a purge and trap GC/MS system.

1.3 The practical quantitation limit (PQL) of Method 8240 for an individual compound is approximately 5 mg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 mg/l for ground water (see Table 2). PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.4 Method 8240 is based upon a purge and trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge and trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information. The chromatographic conditions, as well as typical mass spectrometer operating parameters, are given.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge and trap GC/MS following the normal water method.

2.3 The purge and trap process - An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

3.0 INTERFERENCES

3.1 Interferences purged or co-extracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, is checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A field blank prepared from ASTM Type II water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it is followed by the analysis of reagent water to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

3.4 The laboratory where volatile analysis is performed is completely free of solvents.

3.5 Impurities in the purge gas and from organic compounds out-gasing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system is demonstrated to be free from contamination under the conditions of the analysis by running calibration and reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device is avoided.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes - 10-µl, 25-µl, 100-µl, 250-µl, 500-µl, and 1,000 µl.

4.2 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Syringe - 5 ml, gas-tight with shutoff valve.

4.4 Balance - Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g.

4.5 Glass culture tubes - 20 ml, with screw caps and Teflon liners.

4.6 Volumetric flasks - 10 ml and 100 ml, class A with ground-glass stoppers.

4.7 Spatula - Stainless steel.

4.8 Disposable pipets - Pasteur.

4.9 Purge and trap device - The purge and trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber.The device is equipped with heaters capable of maintaining the purging vessels at 40°C Several complete devices are commercially available.

4.9.1 The recommended purging chamber is designed to accept 5 ml samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3-mm at the origin. The purge gas is introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be utilized, provided equivalent performance is demonstrated.

4.9.2 The trap is at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figure 2). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap is conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 ml/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap is conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column is run through the temperature program prior to analysis of samples.

4.9.3 The desorber is capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figure 2 meets these criteria.

4.9.4 The purge and trap device is coupled to a gas chromatograph, as shown in Figures 3 and 4.

4.9.5 Trap Packing Materials

4.9.5.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.9.5.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.9.5.3 Silica gel - 35/60 mesh, Davison, grade 15 or equivalent.

4.9.5.4 Coconut charcoal - Prepare from Barnebey Cheney, CA- 580-26 lot #M-2649 by crushing through 26 mesh screen.

4.10 Gas chromatograph/mass spectrometer system

4.10.1 Gas chromatograph/mass spectrometer - Finnigan GC/MS models OWA, 5100 and INCOS 50. These instruments are capable of scanning from 35-260 amu every 2 seconds or less using 70 volts (electron energy in the electron impact mode). All modes can meet the criteria in Table 3 when 50 ng of 4-bromoflurobenzene (BFB) are injected into the instrument.

4.10.2 Chromatographic columns

4.10.2.1 6 ft x 2 mm glass packed with 1% SP1000 on Carbopack-B (60-80 mesh) or equivalent.

4.10.2.2 30 m x 0.53 mm megabore column coated with appropriate siloxane capable of chromatographing the compounds listed in Table 1. Examples of such columns are DB-624 (J&W Scientific), VOCOL (Supelco) and R_{α} 502.2 (Restek).

4.10.3 Data system - A computer system, interfaced to each GC/MS, allows continuous acquisition and storage on machine-readable media, of all mass spectra obtained through the duration of the GC/MS run. The software allows searching af any data file for ions of a specified mass and plotting of ion abundances verses time or scan number. The software is capable of integrating abundances of extracted ion current profiles and has available the most recent version of the EPA/NIH Mass Spectral Library..

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified. Is free of interferences at the method detection limit (MDL) of the analytes of interest. The ASTM Type II water is further purified by purging with µltr-high purity helium. The purity is verified before sample analysis begins.

5.3 Stock solutions - Stock solutions are purchased as certified solutions from Supelco.

5.4 Working standards are prepared by appropriately diluting and compositing the purchased stack standards to achieve a solution with all non-gaseous components at 50 µg/ml. Gaseous working standards are prepared from purchased stock standards at a concentration of 100 m µg/ml. Gases are prepared weekly. All other standards are prepared monthly. Standards are stored at -10°C.

5.5 Analytical standards, those actually purged are prepared as follows:

Table 1

Preparation of Analytical Standards

Volume of 50 µg/ml non-gaseous standard (µl)	Volume of 100 µg/ml gaseous standard (µl)	Volume of 50 µg/ml Surrogate Standard (µl)	Final Concentration (µg/l)
2	1	2	100
5	2.5	5	250
10	5	10	500
15	7.5	15	750
20	10	20	1000

Note:These standard are added to 5 ml contamination-free water.

5.6 Surrogate standards - The surrogates three surrogate standards toluene-d_8, 4-bromofluorobenzene, and 1,2-dichloroethane-d_4.are prepared at a final concentration of 50 µg/ml. Each blank, sample and quality control sample is spiked with 5 µg of this solution.

5.6 Internal standards - The three internal standards bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5 are prepared at a final concentration of 50 µg/ml. Each blank, sample and quality control sample is spiked with 5 µg of this solution. For water and low level soil analysis the surrogate and internal standards may be prepared in one solution.

5.8 Bromofluorobenzene (BFB) standard - A standard solution containing 50 ng/µl of BFB in methanol is prepared.

5.9 Matrix spiking standards - Matrix spiking standards are prepared from volatile organic compounds which will be representative of the compounds being

investigated. The compounds are 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The standard is prepared in methanol, at a concentration of 50 µg/ml for each compound.

5.10 Great care is taken to maintain the integrity of all standard solutions. Standards in methanol are stored at -10°C to -20°C in screw-cap vials with Teflon liners.

5.11 Methanol, CH_3OH. Pesticide quality or equivalent. Store apart from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

6.1 Sample Preservation

6.1.1 Concentrated waste samples, solids and sludges are unpreserved and stored at 4°C.

6.1.2 Aqueous samples may be adjusted to pH 4-5 with HCl. Samples are stores at 4°C.

6.2 Sample Handling

6.2.1 Concentrated wastes samples, solids and sludge are analyzed within 14 days of sampling.

6.2.2 Unacidified aqueous samples are analyzed within 14 days of sampling.

6.2.3 Acidified aqueous samples are analyzed within 14 days of sampling.

7.0 PROCEDURE

7.1 Initial calibration for purge and trap procedure

7.1.1 Recommended GC/MS operating conditions

Electron energy	70 volts (nominal)
Mass range	35-260 amu
Scan time: To give 5 scans/peak but not to exceed 7 sec/scan	
Initial column temperature	45°C
Initial column holding time	3 minutes
Column temperature program	10°C/minutes
Final column temperature	220°C
Final column holding time	15 minutes
Injector temperature	250°C
Source temperature	80°C
Transfer line temperature	250°C
Carrier gas	Helium at 30 ml/min

The instrument temperature program may vary with the instrument and column.

7.1.2 Each GC/MS system is hardware-tuned to meet the criteria in Table 3 for a 50-ng injection 4-bromofluorobenzene (1-µl injection of the BFB standard). Analyses cannot begin until these criteria are met.

7.1.3 Assemble a purge and trap device and condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 ml/min. Prior to use, condition the trap daily for 10 min while backflushing at 180°C with the column at 220°C.

7.1.4 Prepare the calibration standards by adding the specified volumes of the contaminant-free reagent water. Add 5 µl if the external standard solution to each standard also.

7.1.5 Carry out the purge and trap analysis procedure as described in Step ?????.

7.1.6 Tabµlate the area response of the characteristic ions (see Table 4) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound is the internal standard that has a retention time closest to the compound being measured (Step 7.5.2). The RF is calculated as follows:

$$RF = (A_x c_{is})/(A_{is} C_x)$$

where-

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

7.1.7 The average RF is calculated for each compound. A system performance check is made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average RF for these compounds is 0.300 (0.250 for bromoform). These compounds typically have RFs I of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.1.7.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

7.1.7.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

7.1.7.3 Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and- trap systems and/or active sites in trapping materials.

7.1.8 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs).

$$\%RSD = \frac{SD}{X} \times 100$$

where:

RSD = relative standard deviation.

$\bar{x}$ = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

$$\text{Std. dev} = \sqrt{\frac{\sum RF_i^2 - n\,\overline{RF}^2}{n-1}}$$

The %RSD for each individual CCC is less than 30 percent. This criterion is met in order for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

7.2 Daily GC/MS calibration

7.2.1 Prior to the analysis of samples, inject or purge 50-ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 3 before sample analysis begins. These criteria is demonstrated each 12-hour shift.

7.2.2 The initial calibration curve (Step 7.1) for each compound of interest is checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC (Step 7.2.3) and CCC (Step 7.2.4).

7.2 3 System Performance Check Compounds (SPCCs) - A system performance check is made each 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system is evaluated, and corrective action is taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.2.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Step 7.1.8 are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\%\ \text{Difference} = \frac{RF_I - RF_C}{RF_I} \times 100$$

where:

RF_I = average response factor from initial calibration.
RF_C = response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory considers this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (> 25% difference), for any one CCC, corrective action Is taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration Is generated. This criterion Is met before quantitative sample analysis begins.

7.2.5 The internal standard responses and retention times in the check calibration standard is evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system is inspected for

malfunctions and corrections are, as required. If the EICP area for any of the internal standards changes by a factor of two (- 50% to + 100%) from the last daily calibration standard check, the mass spectrometer is inspected for malfunctions and corrections are made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.3 GC/MS analysis

7.3.1 Water samples

7.3.1.1 Screening of the sample prior to purge and trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge and trap system.

7.3.1.2 All samples and standard solutions are allowed to warm to ambient temperature before analysis.

7.3.1.3 Set up the GC/MS system as outlined in Step 7.1.1.

7.3.1.4 BFB tuning criteria and daily GC/MS calibration criteria are met (Step 7.2) before analyzing samples.

7.3.1.5 Adjust the purge gas (helium) flow rate to 25-40 ml/min on the purge and trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response (see Step 7.1.7).

7.3.1.6 Remove the plunger from a 5 ml syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 ml. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst fills a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20 ml syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it is analyzed within 24 hours. Care is taken to prevent air from leaking into the syringe.

7.3.1.7 The following procedure is appropriate for diluting purgeable samples. All steps is performed without delays until the diluted sample is in a gas-tight syringe.

7.3.1.7.1 Dilutions may be made in volumetric flasks (10 to 100 ml). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.3.1.7.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

7.3.1.7.3 Inject the proper aliquot of samples from the syringe prepared in Step 7.4.1.6 into the flask. Aliquots of less than 1 ml are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.3.1.7.4 Fill a 5 ml syringe with the diluted sample as in Step 7.3.1.6.

7.3.1.8 Add 5.0 µl of surrogate spiking solution (Step 5.6) and 5 µl of internal standard spiking solution (Step 5.7) through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µl of the surrogate spiking solution to 5 ml of sample is equivalent to a concentration of 50 µg/l of each surrogate standard.

7.3.1.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.3.1.10 Close both valves and purge the sample for 11.0 + 0.1 minutes at ambient temperature.

7.3.1.11 At the conclusion of the purge time, adjust the device to the purge mode and begin the gas chromatographic temperature program and GC/MS data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with inert gas between 20 and 60 ml/min for 4 minutes.

7.3.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 ml flushes of water (or methanol followed by water) to avoid carryover of pollutant compounds into subsequent analyses.

7.3.1.13 After desorbing the sample for 4 minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature is maintained at 180°C. Trap temperatures up to 220°C may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.3.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample is reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis is followed by a blank water analysis. If the blank analysis is not free of interferences, the system is decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.3.1.15 For matrix spike analysis, add 15 µl of the matrix spike solution (Step 5.9) to the 5 ml of sample purged. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/l of each matrix spike standard.

7.3.1.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Steps 7.5.1 and 7.5.2 for qualitative and quantitative analysis.

7.3.2 Water-miscible liquids are analyzed as water samples

7.3.3 Sediment/soil and waste samples - It is highly recommended that all samples of this type be screened prior to the purge and trap GC/MS analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge and trap system, and require extensive cleanup and instrument downtime.

7.3.3.1 Low-level method - This is designed for samples containing individual purgeable compounds of < 1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples. See Figure 5 for an illustration of a low soils impinger.

7.3.3.1.1 Use a 5 g sample if the expected concentration is < 0.1 mg/kg or a 1-9 sample for expected concentrations between 0.1 and 1 mg/kg.

7.3.3.1.2 The GC/MS system is set up as in Steps 7.3.1.2-7.3.1.4. This is done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve is prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature .

7.3.3.1.3 Remove the plunger from a 5 ml Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 ml. Add 5 µl each of surrogate spiking solution (Step 5.6) and internal standard solution (Step 5.7) to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together.) The addition of 10 µl of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 µg/kg of each surrogate standard.

7.3.3.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Step 7.3.3.1.1 into a tared purge device . Note and record the actual weight to the nearest 0.1 g.

7.3.3.1.5 Determine the percent moisture of the soil/sediment sample. This includes waste samples that are amenable to moisture determination. Other wastes is reported on a wet-weight basis. Immediately after weighing the sample, weigh (to 0.1 g) 5-10 g of additional sediment/soil into a tared crucible. Dry the contents of the crucibles overnight at 105°C. Allow to cool in a desiccator and reweigh the dried contents. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\% \text{ moisture} = \frac{\text{grams of sample - grams of dry sample}}{\text{grams of sample}} \times 100$$

7.3.3.1.6 Add the spiked water to the purge device, which contains the weighed amount of sample, and connect the device to the purge and trap system.

7.3.3.1.7 Heat the sample to 40°C + 1°C and purge the sample for 11.0 + 0.1 minute.

7.3.3.1.8 Proceed with the analysis as outlined in Steps 7.3.1.11-7.3.1.16. Use 5 ml of the same water as in the reagent blank. If saturated peaks occurred or would occur if a 1-9 sample were analyzed, the medium-level method is followed.

7.3.3.1.9 For low-level sediment/soils add 5 µl of the matrix spike solution (Step 5.9) to the 5 ml of water (Step 7.3.3.1.3). The concentration for a 5 g sample would be equivalent to 50 µg/kg of each matrix spike standard.

7.3.3.2 High-level method - The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/kg is analyzed by this method.

7.3.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) of sample into a tared 20 ml culture tube. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Step 7.3.3.1.5.

7.3.3.2.2 Quickly add 9.0 ml of appropriate solvent; then add 1.0 ml of the surrogate spiking solution to the vial. Cap and shake for 2 minutes.

7.3.3.2.3 Pipet approximately 1 ml of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 ml of appropriate solvent to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis. The addition of a 100-µl aliquot of each of these extracts in Step 7.3.3.2.6 will give a concentration equivalent to 6,200 µg/kg of each surrogate standard.

7.3.3.2.4 The GC/MS system is set up as in Steps 7.3.1.2-7.3.1.4. This is done prior to the addition of the solvent extract to water.

7.3.3.2.5 Remove the plunger from a 5.O ml Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 ml. Pull the plunger back to 5.0 ml to allow volume for the addition of the sample extract and of standards. Add 5 µl of internal standard solution. Add up to 100 µl of the sample extract.

7.3.3.2.6 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

7.3.3.2.7 Proceed with the analysis as outlined in Step 7.3.1.11-7.3.1.16. Analyze all reagent blanks on the same instrument as that use for the samples. The standards and blanks should also contain 100 µl of solvent to simulate the sample conditions.

7.3.3.2.8 For a matrix spike in the medium-level sediment/soil samples, add 8.0 ml of methanol, 1.0 ml of surrogate spike solution (Step 5.6), and 1.0 ml of matrix spike solution (Step 5.9) as in Step 7.3.3.2.2. This results in a 6,200 µg/kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100-µl aliquot of this extract to 5 ml of water for purging (as per Step 7.3.3.2.6).

7.4 Data interpretation

7.4.1 Qualitative analysis

7.4.1.1 An analyte (e.g. those listed in Table 4) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference is obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria is satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

7.4.1.1.1 The sample component RRT must compare within + 0.06 RRT units of the RRT of the standard component. For reference, the standard is run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT is assigned by using extracted ion current profiles for ions unique to the component of interest.

7.4.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100% is present in the sample spectrum). (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance is between 30 and 70 percent.

7.4.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) is present in the sample spectrum.

(2) The relative intensities of the major ions should agree within + 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance is between 30 and 70%).

(3) Molecular ions present in the reference spectrum is present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum is reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum is reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library

searches will the mass spectral interpretation specialist assign a tentative identification.

7.4.2 Quantitative analysis

7.4.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (e.g. see Table 5).

7.4.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste

$$\text{concentration } (\mu g/l) = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.

I_s = Amount of internal standard injected (ng).

A_{is} = Area of characteristic ion for the internal standard.

RF = Response factor for compound being measured (Step 7.2.7).

V_o = Volume of water purged (ml), taking into consideration any dilutions made.

Sediment/Soil, Sludge, and Waste

High-level

$$\text{concentration } (\mu g/kg) = \frac{(Ax)(Is)(Vt)}{(A_{is})(RF)(V_i)(W_s)}$$

Low-level

$$\text{concentration } (\mu g/kg) = \frac{(AX)(Is)}{(A_{is})(RF)(W_s)}$$

Where:

Ax, Is, Ais, RF = same as for water.

Vt = volume of total extract (µl) (use 10,000 µl or a factor of this when dilutions are made).

Vi = volume of extract added (µl) for purging.

Ws = weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

7.4.2.3 Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The % moisture of the sample (as calculated in Step 7.3.3.1.5) is reported along with the data in either instance.

7.4.2.4 Where applicable, an estimate of concentration for non-calibrated components in the sample is made. The formulas given above is used with the following modifications: The areas Ax and Ajs is from the total ion chromatograms, and the RF for the compound is assumed to be 1. The concentration obtained is reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.4.2.5 Report results without correction for recovery data. When duplicates and spiked samples are analyzed, report all data obtained with the sample results.

8.0 QUALITY CONTROL

8.1 The laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory maintains records to document the quality of the data generated. Ongoing data quality checks are compared with

established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control reference sample is analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a calibration blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank is processed as a safeguard against chronic laboratory contamination. The blank samples is carried through all stages of sample preparation and measurement.

8.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard is evaluated to determine if the chromatographic system is operating properly. Questions that are asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

8.4 Required instrument QC is found in the following section:

8.4.1 The GC/MS system is tuned to meet the BFB specifications in Step 7.1.2.

8.4.2 There is an initial calibration of the GC/MS system as specified in Step 7.1.

8.4.3 The GC/MS system must meet the SPCC criteria specified in Step 7.2.3 and the CCC criteria in Step 7.2.4, each 12 hours.

8.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.5.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 10 µg/ml in methanol. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate is made using stock standards prepared independently from those used for calibration.

8.5.2 Prepare a QC reference sample to contain 20 µg/l of each analyte by adding 200 µl of QC reference sample concentrate to 100 ml of water.

8.5.3 Four 5 ml aliquots of the well-mixed QC reference sample are analyzed according to the method beginning in Step 7.3.1.

8.5.4 Calculate the average recovery (x) in µg/l, and the standard deviation of the recovery (s) in µg/l, for each analyte using the four results.

8.5.5 For each analyte compare s and x with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and ~ for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

8.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Step 8.5.6.1 or 8.5.6.2.

8.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Step 8.5.2.

8.5.6.2 Beginning with Section 8.5.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Step 8.5.2.

8.6 The laboratory must, on an ongoing basis, analyze a reagent blank, spiked blank (check sample) and a matrix spike/matrix spike duplicate (MS/MSD) for each analytical batch.

8.6.1 The analytes and recovery criteria for the check sample are listed below. The check sample is prepared by spiking reagent blank with 2 µl of the matrix spiking solution. The final concentration of each analyte is 100 µg/l.

Analyte	Recovery Limits	
	Water	Solid
1,1-Dichloroethane	72-126	54-135
Trichloroethane	84-111	78-115
Benzene	80-119	79-116
Toluene	77-128	81-114
Chlorobenzene	91-116	86-115

8.6.1.1 If recovery limits are not met the analysis is stopped and the cause of failure is investigated.

8.6.1.2 Corrective action - Corrective actions may include recalibration, adjusting purge parameters, preparing new standards and replacing the adsorbent trap.

8.6.2 MS/MSD - The analytes concentration and recovery criteria of the MS/MSD are listed below:

Analyte	Concentration (µg/l)	Recovery Limits	
		Water	Solid
1,1-Dichloroethane	50	59-118	57-145
Trichloroethane	50	75-120	73-119
Benzene	50	77-112	73-126
Toluene	50	77-119	74-122
Chlorobenzene	50	86-115	78-121

8.6.2.1 The MS/MSD recovery limits must be met unless matrix interference is indicated.

8.6.2 2 If the recoveries fail the MS/MSD, they are reprepared.

8.6.2.3 If the reprepared MS/MSD meets the recovery limit these results are reported

8.6.2.4 If the reprepared MS/MSD fail, a matrix effect is indicated and both sets of results are reported to the quality control department.

8.6.3 For each sample analyzed, calculated the percent recovery (%R) of each surrogate in the sample:

$$\%R = \frac{\text{amount detected}}{\text{amount added}} \times 100$$

8.6.3.1 The surrogates must meet the recovery limits listed in Table 8.

8.6.3.2 Corrective action - If the recovery is not within limits, the following procedures are followed:

8.6.3.2.1 Check to be sure there are no errors in calculation , surrogate solutions and internal standards. Also check instrument performance.

8.6.3.2.2 Recaluculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

8.6.3.2.3 Re-extract and re-analyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1 were obtained using reagent water. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

10.0 DATA DELIVERABLES

10.1 Data Deliverables are addressed in the Data Deliverables Standard Operating Procedure (SOP) to determine the minimum content of the package. Expanded deliverables options are listed and may also be requested.

11.0 REFERENCES

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11.13. Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

11.14. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 4.
RETENTION TIMES AND CHARACTERISTIC IONS
FOR VOLATILE COMPOUNDS

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Ethylene oxide	1.30	44	44,43,42
Chloromethane	2.30	50	52,49
Dichlorodifluoromethane	2.47	85	85,87,101,103
Bromomethane	3.10	94	96,79
Vinyl chloride	3.80	62	64,61
Acetonitrile	3.97	41	41,40,39
Chloroethane	4.60	64	66,49
Methyl iodide	5.37	142	142,127,141
Methylene chloride	6.40	84	49,51,86
Carbon disulfide	7.47	76	76,78,44

Trichlorofluoromethane	8.30	101	103,66
Propionitrile	8.53	54	54,52,55,40
Allyl chloride	8.83	76	76, 41, 39, 78
1,1-Dichloroethene	9.00	96	61, 98
Bromochloromethane (I.S.)	9.30	128	49, 130, 51
Allyl alcohol	9.77	57	57,58,39
trans-1,2-Dichloroethene	10.00	96	61,98
1,2-Dichloroethane	10.10	62	64,98
Propargyl alcohol	10.77	55	55,39,38,53
Chloroform	11.40	83	85,47
1,2-Dichloroethane-d4(surr)	12.10	65	102
2-Butanone	12.20	72	43,72
Methacrylonitrile	12.37	41	41,67,39,52,66
Dibromomethane	12.53	93	93,174,95,172,176
2-Chloroethanol	12.93	49	49,44,43,51,80
b-Propiolactone	13.00	42	42,43,44
Epichlorohydrin	13.10	57	57,49,62,51
1,1,1-Trichloroethane	13.40	97	99,117
Carbon tetrachloride	13.70	117	119,121
1,4-Dioxane	13.70	88	88,58,43,57
Isobutyl alcohol	13.80	43	43,41,42,74
Bromodichloromethane	14.30	83	85,129
Chloroprene	14.77	53	53,88,90,51
1,2,3,4-Diepoxybutane	14.87	55	55,57,56
1,2-Dichloropropane	15.70	63	62,41
cis-1,3-Dichloropropene	15.90	75	77,39
Bromoacetone	16.33	136	43,136,138,93,95
Trichloroethene	16.50	130	95,97,132
Benzene	17.00	78	52,71
trans-1,3-Dichloropropene	17.20	75	77,39
1,1,2-Trichloroethane	17.20	97	83,85,99
3-Chloropropionitrile	17.37	54	54,49,89,91
1,2-Dibromoethane	18.40	107	107,109,93,188
Pyridine	18.57	79	79,52,51,50
2-Chloroethyl vinyl ether	18.60	63	65,106
2-Hydroxypropionitrile	18.97	44	44,43,42,53
1,4-Difluorobenzene (I.S.)	19.60	114	63,88
Malononitrile	19.60	66	66,39,65,38
Methylmethacrylate	19.77	69	69,41,100,39
Bromoform	19.80	173	171,175,252
1,1,1,2-Tetrachloroethane	20.33	131	131,133,117,119,95
1,3-Dichloro-2-propanol	21.83	79	79,43,81,49
1,1,2,2-Tetrachloroethane	22.10	83	85,131,133
Tetrachloroethene	22.20	164	129,131,166
1,2,3-Trichloropropane	22.20	75	75,77,110,112,97
1,4-Dichloro-2-butene	22.73	75	75,53,77,124,89
n-Propylamine	23.00	59	59,41,39
2-Picoline	23.20	93	93,66,92,78
Toluene	23.50	92	91,65
Ethyl methacrylate	23.53	69	69,41,99,86,114
Chlorobenzene	24.60	112	114,77
Pentachloroethanea	24.83	167	167,130,132,165,169
Ethylbenzene	26.40	106	91
1,2-Dibromo-3-chloropropane	27.23	157	157,75,155,77
4-Bromofluorobenzene (surr.)	28.30	95	174,176
Benzyl chloride	29.50	91	91,126,65,128
Styrene	30.83	104	104,103,78,51,77
Acetone	–	43	58
Acrolein	–	56	55,58
Acrylonitrile	–	53	52,51
Chlorobenzene-d5 (I.S.)	–	117	82,119
Chlorodibromomethane	–	129	208,206
1,1-Dichloroethane	–	63	65,83
Ethanol	–	31	45,27,46
2-Hexanone	–	43	58,57, 100

Iodomethane	-	142	127,141
4-Methyl-2-pentanone	-	43	58,57,100
Toluene-d8 (surr.)	-	98	70,100
Vinyl acetate	-	43	86
Xylene (Total)	-	106	91

a The base peak at m/e 117 was not used due to an interference at that mass with a nearly coeluting internal standard, chlorobenzene-d_5.

TABLE 2.
PRACTICAL QUANTITATION LIMITS (PQL) FOR VOLATILE ORGANICS[a]

Practical Quantitation Limits[b]		
Volatiles	Ground water µg/l	Low Soil/Sediment µg/kg
Acetone	100	100
Acetonitrile	100	100
Allyl chloride	5	5
Benzene	5	5
Benzyl chloride	100	100
Bromodichloromethane	5	5
Bromoform	5	5
Bromomethane	10	10
2-Butanone	100	100
Carbon disulfide	100	100
Carbon tetrachloride	5	5
Chlorobenzene	5	5
Chlorodibromomethane	5	5
Chloroethane	10	10
2-Chloroethylvinyl ether	10	10
Chloroform	5	5
Chloromethane	10	10
Chloroprene	5	5
1,2-Dibromo-3-chloropropane	100	100
1,2-Dibromoethane	5	5
Dibromomethane	5	5
1,4-Dichloro-2-butene	100	100
Dichlorodifluoromethane	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethane	5	5
1,1-Dichloroethene	5	5
trans-1,2-Dichloroethene	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
trans-1,3-Dichloropropene	5	5
Ethylbenzene	5	5
Ethylmethacrylate	5	5
2-Hexanone	50	50
Isobutylalcohol	100	100
Methacrylonitrile	100	100
Methylene chloride	5	5
Methyliodide	5	5
Methylmethacrylate	5	50
4-Methyl-2-pentanone	50	50
Pentachloroethane	10	10
Propionitrile	100	100
Styrene	5	5
1,1,1,2-Tetrachloroethane	5	5

1,1,2,2-Tetrachloroethane	5	5
Tetrachloroethene	5	5
Toluene	5	5
1,1,1-Trichloroethane	5	5
1,1,2-Trichloroethane	5	5
Trichloroethene	5	5
1,2,3-Trichloropropane	5	5
Vinyl acetate	50	50
Vinyl chloride	10	10
Xylene(Total)	5	5

a Sample PQLs are highly matrix-dependent. The PQLs listed here in are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent PQLs.
b PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, PQLs will be higher, based on the percent moisture in each sample.

Other Matrices	Factor[1]
Water miscible liquid waste	50
High-level soil & sludges	125
Non-water miscible waste	500

1PQL = [PQL for ground water (Table 2)] X [Factor] . For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 3

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent oft he base peak
95	base peak,100 percent relative abundance
96	5.0-9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0-9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

TABLE 5

INTERNAL STANDARDS WITH CORRESPONDING TARGET ANALYTES ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Vinyl Acetate	Tetrachloroethene
Chloroethene	Bromodichloromethane	1,1,2,2-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	trans-1,3-Dichloropropene	Chlorobenzene
Carbon Disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Xylene(total)
1,2-Dichloroethene(tot.)	Benzene	Bromofluorobenzene (surr)
Chloroform	cis-1,3-Dichloropropene	
1,2-Dichloroethane	Bromoform	Toluene-d_8(surr)
2-Butanone		
1,2-Dichloroethane-d_4(surr)		

(surr)=surrogate compound

TABLE 8.

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

	Low/Medium	Low/Medium
Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene	86 - 115	74 - 121
1,2-Dichloroethane-d4	76-114	70-121
Toluene-d8	88-110	81-117

SEPARATORY LIQUID-LIQUID EXTRACTION METHOD 3510

1. Scope and Application

1.1 This method describes a procedure for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2. Safety Precautions

2.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely determined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

2.2 The method analytes have been tentatively classified as hazards. Pure standard materials and stock standard solutions of those analytes that have a significant vapor pressure at room temperature should be handled in a hood.

2.3 Safety glasses, protective gloves, clothing and the appropriate use of ventilated hoods should be used throughout this method.

2.4 Treat every sample as if it were hazardous.

2.4 Treat every sample as if it were hazardous

3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. The use of high purity reagents and solvents helps minimize interference problems. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

4. Apparatus and Materials

4.1 Separatory funnel: 2-liter, with Teflon stopcock.

4.2 Pyrex glass funnel, 75 X 75.

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube: 10 ml, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.3.2 Evaporation flask : 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with clips.

4.3.3 Snyder column : Three-ball macro (Kontes K-503000-0121 or equivalent).

4.4 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.5 Water bath: Heated, with concentric ring cover, capable of temperature control (+ 5°C). The bath should be used in a hood.

4.6 Vials: Glass, 2 ml capacity with Teflon lined screw-cap.

4.7 pH indicator paper: pH range including the desired extraction pH.

4.8 Erlenmeyer flask : 250 ml.

4.9 Syringe: 5 ml.

4.10 Graduated cylinder: 1-liter.

4.11 Glass wool.

5. Reagents and Standards

5.1.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first

ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.1.3 Sodium hydroxide solution (10 N), NaOH. Dissolve 40 g NaOH in water and dilute to 100 ml.

5.1.4 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray.

5.1.5 Sulfuric acid concentrated , H_2SO_4.

5.1.6 Extraction/exchange solvent: Methylene chloride, hexane, 2-propanol, cyclohexane, acetonitrile (pesticide quality or equivalent).

5.2 Standards

5.2.1 Target Calibration Standard - not applicable.

5.2.2 Internal Standard - not applicable.

5.2.3 Surrogate Standard

5.2.3.2 Preparation for standards made up from neat sources.

5.2.3.2.1 Verify that the pure material has not exceeded its expiration date.

5.2.3. 2.2 Make sure that all glassware is cleaned according to glassware cleaning SOP prior to use.

5.2.3.2.3 Rinse all glassware three times with solvent to be used in preparation and make certain that all glassware is dry.

5.2.3.2.4 Determine amount of pure material to be added by the following formula:

Equation 1a. Pure Material Mass Needed

$$\text{Std Amt.mg} = \frac{(\text{Concentration desired mg/L})(\text{Volume of prepared standard L})}{\text{Purity (Fraction)}}$$

5.2.3.2.4.1 The purity adjustment is only necessary when the compound source purity of interest is less than 97%.

5.2.3.2.4.2 When using this equation care should be taken to make certain that all units of measurement are consistent.

5.2.3.2.5 Using an analytical balance, weigh volumetric flask and tare balance.

5.2.3.2.6 Weigh to the nearest 0.0001 g the previously determined mass in the cleaned volumetric flask.

5.2.3.2.7 Record in the standards log book weight of pure material added, and the name(s) of compound(s) of interest.

5.2.3.2.8 Fill volumetric approximately half full with desired solvent, stopper, and shake until all solid is dissolved.

5.2.3.2.9 Fill to the mark of the volumetric flask with the desired solvent, stopper, and shake vigorously inverting the volumetric flask several times.

5.2.3.2.10 Record in the standards log book; name of compound(s), concentration(s), solvent used, expiration date of neat standard, standard number, and the initials of preparer.

5.2.3.2.10.1 Transfer solution to a vial (with minimal head space) and label with the same information as was recorded in the standards log book.

5.2.3.2.11. Prior to use, have standards analyzed by the appropriate analytical group (wet chem, GC/MS or GC) to verify concentration.

5.2.3.2.12. Store at 4°C.

5.2.3.3 Preparation of standards made from dilution of stock solutions.

5.2.3.3.1 Make certain that the expiration dates of stock solutions to be used will not expire prior to the intended expiration dates of standards being prepared.

5.2.3.3.2 Make sure that all glassware is cleaned according to glassware SOP prior to use.

5.2.3.3.3 Rinse all glassware three times with solvent to be used and make certain that all glassware is dry.

5.2.3.3.4 Determine the volume of stock solution to get desired concentration by using the following equation:

Equation 1b. Volume of Stock Solution Needed

$$\text{vol. of stock solution ml} = \frac{\text{(desired Final conc µg/ml.) (Final volume ml)}}{\text{concentration of stock solution µg/ml}}$$

5.2.3.3.5.1 Make certain that mass and volume units are consistent throughout calculation.

5.2.3.3.5.2 Record the determined volume of the stock solution to be used, the name of the compound(s), concentration(s),solvent to be used, expiration date of working standard, standard number, and initials of preparer.

5.2.3.3.6. Fill volumetric flask half full with desired solvent and add previously determined volume of stock solution.

5.2.3.3.6.1 A Hamliton gas-tight syringe is suggested.

5.2.3.3.6.1.1 Prior to using syringe, rinse it at least ten times with the solvent that will be used, and at least once with the standard itself.

5.2.3.3.7 Fill to the mark of the volumetric flask with solvent in use and shake well.

5.2.3.3.8 Transfer to a vial (with a minimal head space), and label with name of compound(s), Concentration(s), solvent used, expiration date of the working standard (taking into consideration expiration date of stock solution used), standard number and initials of preparer.

5.2.3.3.9 Prior to use, have standard analyzed by appropriate analytical group (Wet Chem GC/MS or GC) to verify concentration.

5.2.3.3.10 Store at 4°C.

5.2.4 Matrix spike standard

5.2.4.1 Prepare a matrix spike standard of the appropriate concentration for each target analyte group.

6. Sample Storage

6.1 Containers used to collect samples for the determination of semivolatile organic compounds (this includes pesticides and PCBs) should be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw- top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use as a field blank.

6.2 Samples should be stored at 4°C.

7. Holding Times

7.1 Water sample shall be extracted within 7 days from sampling date.

8. Sample Preparation

8.1 Summary

A measured volume of sample, usually 1 liter, at a specified pH (see Table 1), is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step to be used.

8.2 Sample Preparation Procedure

8.2.1 Measure volume of sample by one of the methods below and transfer to the separatory funnel.

8.2.1 1 Mark the meniscus of the sample in its container and transfer to separatory funnel. To obtain initial volume, fill sample container to the mark with water and measure volume using a graduated cylinder

8.2.1.2 Using a l-liter graduated cylinder, measure 1 liter (nominal) of sample and transfer it to the separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with water to 1 liter.

8.2.3 Add 1.0 ml of the surrogate standards to all samples, spikes, and blanks (see Section 5.2 for details on the surrogate standard solution and the matrix spike solution). For the sample in each analytical batch selected for spiking, add 1.0 ml of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/µl of each base/neutral analyte and 200 ng/µl of each acid analyte in the extract to be analyzed (assuming a 1 µl injection). If Method 3640, Gel-Permeation Cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

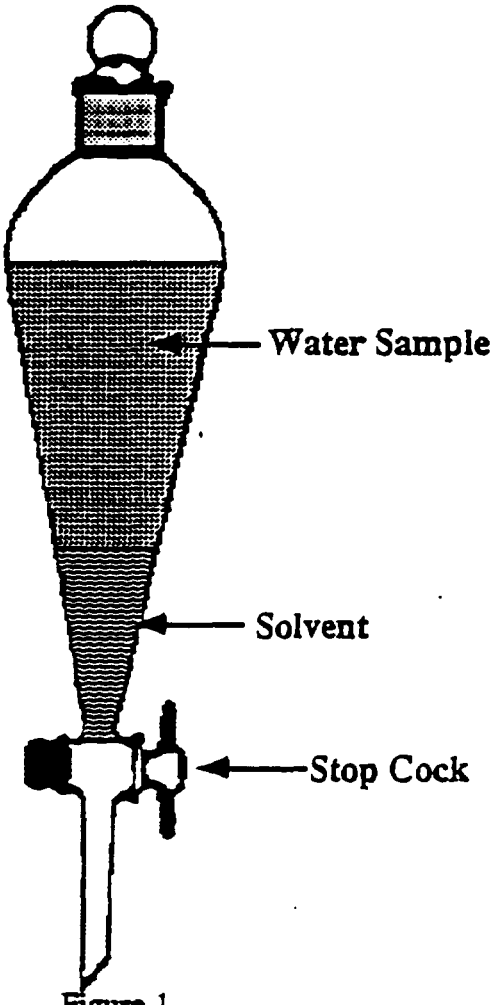

Figure 1.

8.2.2 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to that indicated in Table 1 for the specific determinative method that will be used to analyze the extract.

8.2.3 Add 60 ml of methylene chloride to the separatory funnel.

8.2.4 Seal and shake the separatory funnel vigorously for 1-2 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. Vent into hood away from other samples.

8.2.5 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 size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520.

8.2.6 Repeat the extraction two more times using fresh portions of solvent. (Steps 8.2.3 through 8.2.5). Combine the three solvent extracts.

8.2.7 If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH indicated in Table 1. Serially extract three times with 60 ml of methylene chloride, as outlined in Steps 8.2.3 through 8.2.5. Collect and combine the extracts and label the combined extract appropriately.

8.2.8 If performing GC/MS analysis (Method 8270), the acid and base/neutral extracts must be kept separate.

8.2.9 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporation flask.

8.2.10 Dry the extract by passing it through a funnel with glass wool containing about 10 - 20 g of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask which contained the solvent extract with 20-30 ml of methylene chloride and add it to the funnel to complete the quantitative transfer.

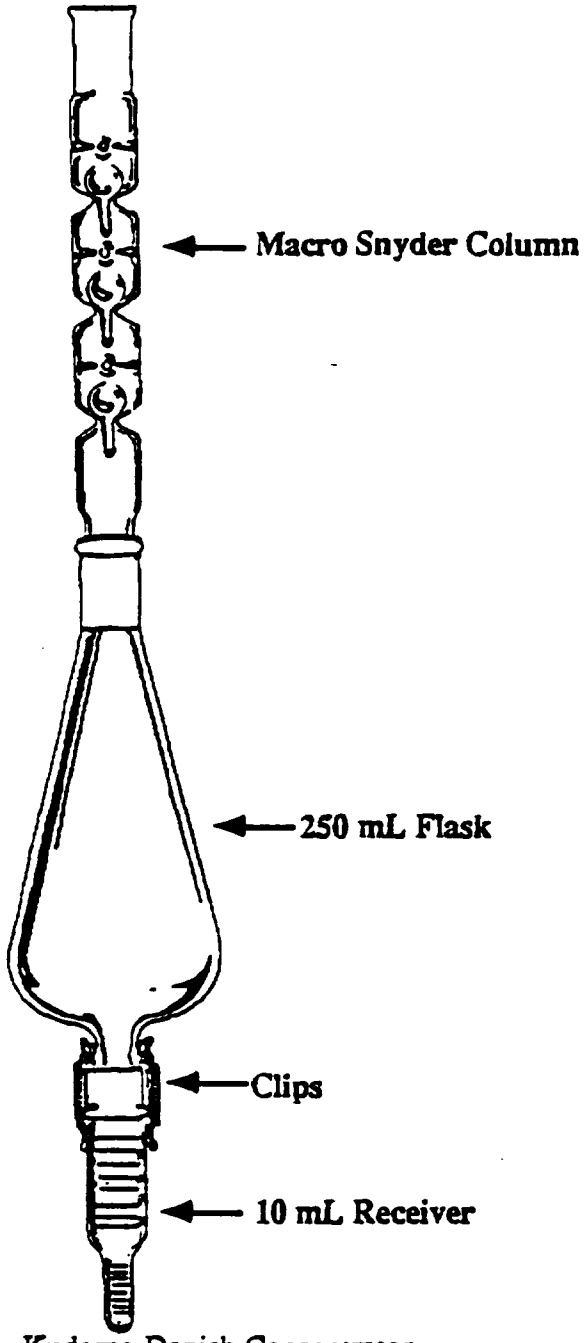

Figure 2. Kuderna-Danish Concentrator

8.2.11 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1 ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 5 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder

column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of extraction solvent.

8.2.12 If a solvent exchange is required (as indicated in Table 1), add 50 ml of the exchange solvent to the Snyder column (cooling the concentrator is not required). Concentrate the extract, as described in Step 8.2.11, raising the temperature of the water bath, if necessary, to maintain proper distillation.

8.2.13 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Step 8.2.12 or adjusted to 10.0 ml with the solvent last used.

8.2.14 Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to below 1 ml using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

8.2.14.1 Caution: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.2.15 The internal wall of the tube must be rinsed down several times with methylene chloride during the operation. During evaporation, the tube solvent level must be kept below the water level of the bath.

The extract must never be allowed to become dry

8.2.16 For BNAs evaporate extract to 1 ml, for Pest/ PCBs evaporate to 5 ml

8.2.17 The extracts obtained may now be analyzed for analyte content using a variety of organic techniques. The extract should be transferred to a vial with a Teflon lined screw-cap, labeled appropriately and stored at 4°C.

TABLE 1.
Specific Extraction Conditions For Various Determinative Methods

Determine method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (ml)	Final extract volume for analysis (ml)
8040	<2	none	2-propanol	hexane	1.0	1.0,10.0[a]
8060	as rec.	none	hexane	hexane	2.0	10.0
8080	5-9	none	hexane	hexane	10.0	10.0
8090	5-9	none	hexane	hexane	2.0	1.0
8100	as rec.	none	none	cyclohexane	2.0	1.0
8120	as rec.	none	hexane	hexane	2.0	1.0
8140	6-8	none	hexane	hexane	10.0	10.0
8250	>11	<2	none	---	---	1.0
8270	>11	<2	none	----	5.0	1.0
8310	as rec.	none	acetonitrile	---	---	1.0

[a]Phenols may be analyzed, by Method 8040, using 1.0 ml of a 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 ml hexane extract to be analyzed by GC/ECD.

[b]The specificity of GC/MS makes cleanup of the extracts less necessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

8.3 Sample Cleanup Procedures

8.3.1 The following cleanups may be applicable. Check the specifics in each particular cleanup method.

Sample Cleanup Methods

SW 846 Method 3610	Alumina Column Cleanup
SW 846 Method 3620	Florisil Column Cleanup
SW 846 Method 3630	Silica Gel Cleanup
SW 846 Method 3640	Gel-Permeation Cleanup
SW 846 Method 3650	Acid-Base Partition Cleanup
SW 846 Method 3660	Sulfur Cleanup
EPA Method 600/4-81-045	Acid Cleanup

8.4 Preparation Documentation

8.4.1 Complete the extraction portion of the Wadsworth worksheet with date and initials.

10. QA/QC requirements

10.1 Method Blank

10.1.1 A reagent blank sample should be subjected to exactly the same analytical procedures as those used on actual samples. Before processing any samples, the analyst should demonstrate, once daily, that all glassware is free of organic contamination. The reagent blank sample should be carried through all stages of the sample preparation and measurement and contains the same reagent concentrations in the final solution as in the sample solution used for analysis. If there is a change in reagents, a reagent blank should be processed as a safeguard against reagent contamination. One reagent blank should be prepared for every analytical batch on a daily basis.

10.2 Check Sample

10.2.1 The check sample,quality control reference sample concentrate should be prepared from a standard which is independent of all calibration, surrogate and matrix spike standards, but within the calibration range. A check sample is intended as an independent check of technique, methodology, and standards and should be run with every analytical batch on a daily basis and should be matrix-specific when possible. This is applicable to all organic and inorganic analyses.

10.2.2 Table 2 indicates the volume and type of check sample spiking solution to be used in each type of extraction.

Table 2

Check Sample Spike Solutions

Analyte Group	Check sample Spike Solution ID	Volume
Pest	0.2 ppm & 0.5 ppm	1 ml
PCB	20 ppm 1242	"
Pest/PCB	0.2 ppm & 0.5 ppm	"
Herb	3,1,2 ppm	"
TPH by GC	1000 ppm TPH/VOC	"
PAH	100 ppm BN spike	"
BNA	100 ppm Acid & 200 ppm Acid	"

10.3 Surrogate

10.3.1 Add an appropriate amount of surrogate compounds to each sample.

10.3.2 Table 3 indicates the volume and type of surrogate spiking solution to be used in each type of extraction.

Table 3

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume
Pest	1 ppm DBC	1 ml
PCB	NA as of 11/7/89	"
Pest/PCB	1 ppm DBC	"
Herb	NA as of 11/7/89	"
TPH by GC	NA as of 11/7/89	"
PAH	100 ppm BN	"
BNA	100 ppm Acid & 200 ppm Acid	"

10.4 Matrix Spike

10.4.1 When calculating the percent recovery of the matrix spike and matrix spike duplicate optional dilutions must be taken into account. Samples requiring optional dilutions and chosen as the matrix spike/ matrix spike duplicate samples, must be analyzed at the same dilution as the original unspiked sample. A matrix spike and matrix spike duplicate should be brought through the whole sample preparation process and subjected to exactly the same analytical procedures as those used on actual samples. A matrix spiked sample shall be analyzed with every analytical batch or every 20 samples, whichever is more frequent. The **sample to be spiked should be typical or representative of the batch.** Ideally it should be an intermediate between the cleanest and the most contaminated samples based on the best information available. It is recommended that the spike be made in a replicate of the field duplicate samples.

10.4.2 Table 4 indicates the volume and type of matrix spiking solution to be used in each type of extraction.

Table 4

Matrix Spike Solutions

Analyte Group	Check sample Spike Solution ID	Volume
Pest	0.2 ppm & 0.5 ppm	1 ml
PCB	20 ppm 1242	"
Pest/PCB	0.2 ppm & 0.5 ppm	"
Herb	3,1,2 ppm	"
TPH by GC	1000 ppm TPH/VOC	"
PAH	100 ppm BN spike	"
BNA	100 ppm Acid & 200 ppm Acid	"

10.4.2 Use an appropriate amount selected target analytes for matrix spiking.

11. Practical Quantitation Limits

11.1 Refer to the individual methods listed in Table 1 for specific analyte detection limits.

12. Recovery Limits

12.1 Check Sample

Table 5

Check Sample Recovery Limits

Type	Compound	Water Low mg/L	Water High mg/L	Solid Low mg/L	Solid High Mg/L
BNA	Total Xylenes	60	140	60	140
	1,2-Dibromi-3-chloropropane	60	140	60	140
	Ethylene dibromide	60	140	60	140
	Acenaphthene	40	94	42	96
	2,4-Dinitrotoluene	35	117	38	111
	Pyrene	44	160	45	139
	n-Nitroso-di-n-propylamine	24	117	31	118
	1,4-Dichlorobenzene	38	91	35	83
	Pentachlorophenol	4	149	20	132
	Phenol	36	88	34	96
	2-Chlorophenol	38	89	34	91
	4-Chloro-3-methylphenol	22	142	24	159
	4-Nitrophenol	1	134	22	131
GC	1,2-Dichloroethene	70	121	70	121
	Trichloroethene	65	116	65	116
	Chlorobenzene	80	114	80	114
	Toluene	75	120	75	120
	Benzene	79	117	79	117
	Trichlorotlourmethane	72	141	72	141
Pesticide	Lindane	57	127	58	121
	Heptachlor	57	127	61	122
	Aldrin	64	124	61	122
	Dieldrin	62	142	63	142
	Endrin	66	144	62	128
	4,4-DDT	59	160	61	144
Herbicide	2,4-D	35	113	23	104
	Silvex	43	111	24	105
	2,4,5-T	32	106	17	93
	n-Methyl-2-pyrrolidone				
	Sulfolane				
BTX	Benzene	75	115	75	115
	Toluene	79	118	79	118
	Xylene	80	121	80	121
PCB	Oil	70	158	70	158
	Solid	55	140	55	140
	Water	67	126	67	126
TPH	L	79	127	79	127
	S	53	120	53	120

12.2 Surrogate
Table 6

Surrogate Spike Recovery Control Limits

Type	Compounds	water Low mg/L	water High mg/L	Soil Low mg/L	Soil High mg/L
BNA	2-Fluorobiphenyl	43	116	30	115
	Nitrobenzene d5	35	114	23	120
	p-Terphenyl	33	141	18	137
	2-Fluorophenol	21	100	25	121
	Phenol D6	10	94	24	113
	2,4,6-Tribromophenol	10	123	19	122
Herb	(Water) 2,4 - DB				
	(Soil) 2,4 - DB				
PCB	DBC				
	TCMX				
Pest	(water) DBC				
	(soil) DBC				

12.3 Matrix Spike
Table 8

GC Matrix Spike Recovery Control Limits

Type	compound	Water Low mg/L	Water High mg/L	Soil Low mg/L	Soil High mg/L
Herbicides		26	134	20	104
	Potable water	35	123		
TPH's		24	99	24	99
PCB					
	Oil			59	150
	Solid			49	137
	Water	50	147		
GC					
	Benzene	65	118	70	120
	Chlorobenzene	70	112	77	120
	1,1-Dichloroethane	65	114	61	123
	Toluene	56	123	68	120
	Trichloroethene	61	116	70	118
BTX					
	Benzene	71	119	72	122
	Toluene	65	129	70	128
	Xylenes	76	124	67	127
Pesticides					
	Aldrin	45	120	41	117
	4,4-DDT	48	142	53	130
	Dieldrin	56	133	56	131
	Endrin	63	132	57	122
	Heptachlor	44	124	41	117
	Lindane	52	132	48	126

13. Waste Disposal

13.1 Generic solvent waste is transfered to the red solvent waste cans.

13.2 Rinse Freon is collected in appropriately marked containers for recycling.

13.3 Solvent saturated water is transfered to the separating container. After sufficient solvent has separated drain the water off and transfer the solvent to the red solvent waste cans.

13.4 Acid waste is collected in the acid waste container.

13.5 Solid materials (soil, gloves, soxhlet thimbles, obviously soiled paper products) are placed in the solid debris container. **Do not** put liquids in the solid waste container.

14. References and associated SOPs

14.1 References;

14.1.1 SW-846 3rd Edition Method 3510.

14.2 Associated SOPs;

14.2.1 Separatory Funnel Liquid-Liquid Extraction, Method 3520.

14.2.2 Organochlorine Pesticides and PCBs, Method 8080.

14.2.3 Organochlorine Pesticides, Method 8080.

14.2.4 PCBs, Method 8080.

14.2.5 Gas Chromatography/Mass Spectrometry for Semivolatile Organics: Capillary Column, Method 8270.

Rec'd CRA
JUL 18 1991

GC/MS SEMIVOLATILE ORGANIC COMPOUNDS

CAPILLARY COLUMN TECHNIQUE

METHOD 8270

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1. SCOPE AND APPLICATION

1.1 Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and groundwater. Direct injection of a sample may be used in limited applications.

1.2 Method 8270 can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. under the alkaline conditions of the extraction step, α-BHC, τ-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4 The practical quantitation limit (PQL) of Method 8270 for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/l for groundwater samples (see Table 10). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

Table 1 Characteristic Ions for Semivolatile Compounds

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Acenaphthene	15.13	154	153, 152
Acenaphthene-d_{10} (I.S.)	15.05	164	162, 160
Acenaphthylene	14.57	152	151, 153
Acetophenone	7.96[a]	105	77, 51
Aldrin	--	66	263, 220
Aniline	5.68	93	66, 65
Anthracene	19.77	178	176, 179
4-Aminobiphenyl	19.18[a]	169	168, 170
Aroclor-1016	--	222	260, 292
Aroclor-1221	--	190	224, 260
Aroclor-1232	--	190	224, 260
Aroclor-1242	--	222	256, 292
Aroclor-1248	--	292	362, 326
Aroclor-1254	--	292	362, 326
Aroclor-1260	--	360	362, 394
Benzidine	23.87	184	92, 185
Benzoic acid	9.38	122	105, 77
Benzo(a)anthracene	27.83	228	229, 226
Benzo(b)fluoranthene	31.45	252	253, 125
Benzo(k)fluoranthene	31.55	252	253, 125
Benzo(g,h,i)perylene	41.43	276	138, 277
Benzo(a)pyrene	32.80	252	253, 125
Benzyl alcohol	6.78	108	79, 77
α-BHC	--	183	181, 109
β-BHC	--	181	183, 109
δ-BHC	--	183	181, 109
τ-BHC (Lindane)	--	183	181, 109

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Bis(2-chloroethoxy)methane	9.23	93	95, 123
Bis(2-chloroethyl)ether	5.82	93	63, 95
Bis(2-chloroisopropyl)ether	7.22	45	77, 121
Bis(2-ethylhexyl)phthalate	28.47	149	167, 279
4-Bromophenyl phenyl ether	18.27	248	250, 141
Butyl benzyl phthalate	26.43	149	91, 206
Chlordane	--	373	375, 377
4-Chloroaniline	10.08	127	129
1-Chloronaphthalene	13.65[a]	162	127, 164
2-Chloronaphthalene	13.30	162	127, 164
4-Chloro-3-methylphenol	11.68	107	144, 142
2-Chlorophenol	5.97	128	64, 130
4-Chlorophenyl phenyl ether	16.78	204	206, 141
Chrysene	27.97	228	226, 229
Chrysene-d_{12} (I.S.)	27.88	240	120, 236
4,4'-DDD	--	235	237, 165
4,4'-DDE	--	246	248, 176
4,4'-DDT	--	235	237, 165
Dibenz(a,j)acridine	32.55[a]	279	280, 277
Dibenz(a,h)anthracene	39.82	278	139, 279
Dibenzofuran	15.63	168	139
Di-n-butyl phthalate	21.78	149	150, 104
1,3-Dichlorobenzene	6.27	146	148, 111
1,4-Dichlorobenzene	6.40	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	6.35	152	150, 115
1,2-Dichlorobenzene	6.85	146	148, 111
3,3'-Dichlorobenzidine	27.88	252	254, 126
2,4-Dichlorophenol	9.48	162	164, 98

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2,6-Dichlorophenol	10.05[a]	162	164, 98
Dieldrin	--	79	263 ,279
Diethyl phthalate	16.70	149	177, 150
p-Dimethylaminoazobenzene	24.48[a]	120	225, 77
7,12-Dimethylbenz(a)anthracene	29.54[a]	256	241, 257
α-,α-Dimethylphenethylamine	9.51[a]	58	91, 42
2,4-Dimethylphenol	9.03	122	107, 121
Dimethylphthalate	14.48	163	194, 164
4,6-Dinitro-2-methylphenol	17.05	198	51, 105
2,4-Dinitrophenol	15.35	184	63, 154
2,4-Dinitrotoluene	15.80	165	63, 89
2,6-Dinitrotoluene	14.62	165	63, 89
Diphenylamine	17.54[a]	169	168, 167
1,2-Diphenylhydrazine	--	77	105, 182
Di-n-octylphthalate	30.48	149	167, 43
Endosulfan I	--	195	339, 341
Endosulfan II	--	337	339, 341
Endosulfan sulfate	--	272	387, 422
Endrin	--	263	82, 81
Endrin aldehyde	--	67	345, 250
Endrin ketone	--	317	67, 319
Ethyl methanesulfonate	5.33[a]	79	109, 97
Fluoranthene	23.33	202	101, 203
Fluorene	16.70	166	165, 167
2-Fluorobiphenyl (Surrogate)	--	172	171
2-Fluorophenol (Surrogate)	--	112	64
Heptachlor	--	100	272, 274
Heptachlor epoxide	--	353	355, 351

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexachlorobenzene	18.65	284	142, 249
Hexachlorobutadiene	10.43	225	223, 227
Hexachlorocyclopentadiene	12.60	237	235, 272
Hexachloroethane	7.65	117	201, 199
Indeno(1,2,3-cd)pyrene	39.52	276	138, 227
Isophorone	8.53	82	95, 138
Methoxychlor	--	227	228
3-Methylcholanthrene	31.14[d]	268	253, 267
Methyl methanesulfonate	4.32[d]	80	79, 65
2-Methylnaphthalene	11.87	142	141
2-Methylphenol (o-cresol)	7.22	108	107, 79
4-Methylphenol (p-cresol)	7.60	108	107, 79
Naphthalene	9.82	128	129, 127
Naphthalene-d_8 (I.S.)	9.75	136	68
1-Naphthylamine	15.80[d]	143	115, 116
2-Naphthylamine	16.00[d]	143	115, 116
2-Nitroaniline	13.75	65	92, 138
3-Nitroaniline	15.02	138	108, 92
4-Nitroaniline	16.90	138	108, 92
Nitrobenzene	7.87	77	123, 65
Nitrobenzene-d_5 (Surrogate)	--	82	128, 54
2-Nitrophenol	8.75	139	109, 65
4-Nitrophenol	15.80	139	109, 65
N-Nitroso-di-n-butylamine	10.99[d]	84	57, 41
N-Nitrosodimethylamine	--	42	74, 44
N-Nitrosodiphenylamine	17.17	169	168, 167
N-Nitrosodipropylamine	7.55	70	42, 101, 130
N-Nitrosopiperidine	--	42	114, 55

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Pentachlorobenzene	15.64[a]	250	252, 248
Pentachloronitrobenzene	19.47[a]	295	237, 142
Pentachlorophenol	19.25	266	264, 268
Perylene-d_{12} (I.S.)	33.05	264	260, 265
Phenacetin	18.59[a]	108	109, 179
Phenanthrene	19.62	178	179, 176
Phenanthrene-d_{10} (I.S.)	19.55	188	94, 80
Phenol	577	94	65, 66
Phenol-d_6 (Surrogate)	--	99	42, 71
2-Picoline	3.75[a]	93	66, 92
Pronamide	19.61[a]	173	175, 145
Pyrene	24.02	202	200, 203
Terphenyl-d_{14} (Surrogate)	--	244	122, 212
1,2,4,5-Tetrachlorobenzene	13.62[a]	216	214, 218
2,3,4,6-Tetrachlorophenol	16.09[a]	232	230, 131
2,4,6-Tribromophenol (Surrogate)	--	330	332, 141
1,2,4-Trichlorobenzene	9.67	180	182, 145
2,4,5-Trichlorophenol	13.00	196	198, 200
2,4,6-Trichlorophenol	12.85	196	198, 200
Toxaphene	--	159	231, 233

I.S. - Internal Standard
[a]Estimated Retention Times

2. METHOD SUMMARY

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method described chromatographic conditions that will allow for the separation of the compounds in the extract.

3. SAFETY PRECAUTIONS

3.1 The majority of analytes, internal standards, and surrogates are either known or suspected carcinogens. Therefore, care must be taken to avoid inhalation and skin contact of all samples and standards.

4. INTERFERENCES

4.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

4.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. APPARATUS AND MATERIALS

5.1 Gas Chromatograph/Mass Spectrometer System

5.1.1 Gas Chromatograph: An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

5.1.2 Column: 30 m x 0.25 mm I.D. (or 0.32 mm I.D.) 1-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5 or equivalent).

5.1.3 Mass Spectrometer: Capable of scanning from 35 to 500 amu every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 2 when 1 µl of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

5.1.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

5.1.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for

ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library should also be available.

5.2 Syringe: 10 µl

Table 2 DFTPP Key Ions and Ion Abundance Criteria[a]

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

[a]J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry", Analytical Chemistry, 47, 995 (1975).

6. REAGENTS AND STANDARDS

6.1 Stock Standard Solutions, 1.00 µg/µl: Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.1.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10 ml volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.1.2 Transfer the stock standard solutions into Teflon®-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.1.3 Stock standard solutions must be replaced after one year or sooner if comparison with quality control check samples indicates a problem.

6.2 Internal Standard Solutions: The internal standards recommended are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. Other compounds may be used as internal standards as long as the requirements given in paragraph 9.3.2 are met. Dissolve 200 mg of each compound with a small volume of carbon disulfide. Transfer to a 50 ml volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene-d_{12}. The resulting solution will contain each standard at a concentration of 4,000 ng/µl. Each extract undergoing analysis should be spiked with the internal standard solution resulting in a concentration of 40 ng/µl of each internal standard. Store at 4°C or less when not being used.

6.3 GC/MS Tuning Standard: A methylene chloride solution containing 50 ng/µl of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µl each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at 4°C or less when not being used.

6.4 Calibration Standards: Calibration standards at a minimum of five concentration levels should be prepared. One of the calibration standards should be at a concentration near, but above, the method detection limit; the others should correspond to the range of concentrations found in real samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Table 1 may be included). Each calibration standard should be spiked with the internal standard solution. All standards should be stored at -10°C to -20°C and should be freshly prepared once a year, or sooner if check standards indicate a problem.

6.5 Surrogate Standards: The recommended surrogate standards are phenol-d_5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. Determine what concentration should be in the blank extracts after all extraction, clean-up, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards in all blanks, spikes, and sample extracts. Take into account all dilutions of sample extracts.

6.6 Matrix Spike Standards: Determine what concentration should be in the blank extracts after all extraction, clean-up, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards in all blanks, spikes, and sample extracts. Take into account all dilutions of sample extracts.

7. SAMPLE STORAGE

7.1 Samples and extracts are to be stored separately and should be stored in the dark at 4°C. Samples and extracts should be stored in suitable glass containers with Teflon®-lined caps.

8. HOLDING TIMES

8.1 Water samples are to be extracted within seven days of sampling and the extracts are to be analyzed within forty days of extraction.

8.2 Solids, sludges, and organic liquids are to be extracted within fourteen days of sampling and the extracts are to be analyzed within forty days of extraction.

9. PROCEDURE (TUNING, CALIBRATION, SAMPLE PREPARATION, AND ANALYSIS)

9.1 Sample Preparation: Samples must be prepared by one of the following methods prior to GC/MS analysis.

Table 3 Extraction Methods For GC/MS Analysis

Matrix	Methods
Soil/Sediment	3540, 3550
Waste	3540, 3550, 3580
Water	3510, 3520

9.1.1 Direct Injection: In very limited applications, direct injection of the sample into the GC/MS system with a 10 µl syringe may be appropriate. The detection limit is very high (approximately 10,000 µg/l); therefore, it is only permitted where concentrations in excess of 10,000 µg/l are expected. The system must be calibrated by direct injection.

9.2 Extract Clean-Up: Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

Table 4 Clean-Up Methods Used With GC/MS Analysis

Compounds	Methods
All Priority Pollutant Base, Neutral, and Acids	3640
Chlorinated Hydrocarbons	3620, 3640
Haloethers	3620, 3640
Nitroaromatics and Cyclic Ketones	3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine Pesticides & PCBs	3620, 3640, 3660
Organophosphorus Pesticides	3620, 3640
Petroleum Waste	3611, 3650
Phenols	3630, 3640, 8040[a]
Phthalate Esters	3610, 3620, 3640
Polynuclear Aromatic Hydrocarbons	3611, 3630, 3640

[a]Method 8040 includes a derivatization technique followed by GC/ECD analysis, if interferences are encountered on GC/FID.

9.3 Initial Calibration: The recommended GC/MS operating conditions:

Mass Range:	35 - 500 amu
Scan Time:	1 second/scan
Initial Column Temperature/Hold Time:	40°C for two minutes
Column Temperature Program:	40 - 300°C at 10°C/minute
Final Column Temperature Hold:	300°C (until benzo(g,h,i) perylene has eluted)
Injector Temperature:	250 - 300°C
Transfer Line Temperature:	250 - 300°C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Sample Volume:	1 - 2 µl
Carrier Gas:	Hydrogen at 50 cm/second or Helium at 30 cm/second

9.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 50 ng injection of DFTPP. Analyses should not begin until all these criteria are met. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%.

Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be visible. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6 - 12 inches of the capillary column.

9.3.2 The internal standards should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (i.e., for 1,4-dichlorobenzene-d_4, use m/z 152 for quantitation).

9.3.3 Analyze 1 µl of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). Calculate response factors (RFs) for each compound as follows.

$$RF = (A_x C_{is})/(A_{is} C_x)$$

Where:

A_x = *Area of the characteristic ion for the compound being measured*

A_{is} = *Area of the characteristic ion for the specific internal standard*

C_x = *Concentration of the compound being measured (ng/µl)*

C_{is} = *Concentration of the specific internal standard (ng/µl)*

9.3.4 The average RF should be calculated for each compound. The percent relative standard deviation (%RSD = 100[SD/$\bar{R}$F]) should also be calculated for each compound. The %RSD should be less than 30% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) <u>must</u> be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

9.3.5 For semivolatiles, the calibration check compounds are phenol, 1,4-dichlorobenzene, 2-nitrophenol, 2,4-dichlorophenol, hexachlorocyclopentadiene, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol, acenaphthene, n-nitrosodiphenylamine, pentachlorophenol, fluoranthene, di-n-octylphthalate, and benzo(a)pyrene.

9.3.6 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-d-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average $\bar{R}F$ for these compounds SPCCs is 0.050. These SPCCs typically have very low RFs (0.1 - 0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

9.4 Daily GC/MS Calibration

9.4.1 Prior to analysis of samples, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 2. These criteria must be demonstrated during each twelve-hour shift.

9.4.2 A calibration standard(s) at mid-level concentration containing all semivolatile analytes, including all required surrogates, must be performed every twelve hours during analysis. Compare the response factor data from the standards every twelve hours with the average response factor from the initial calibration for a specific instrument as per the SPCC (paragraph 9.4.3) and CCC (paragraph 9.4.4) criteria.

9.4.3 System Performance Check Compounds (SPCCs): A system performance check must be made during every twelve-hour shift. IF the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

9.4.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\%\ Difference = \frac{\bar{RF}_I - RF_c}{\bar{RF}_I} \times 100$$

Where:

$\bar{RF}_I$ = *Average Response Factor from initial calibration*

RF_c = *Response Factor from current verification check standard*

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 30%, the initial calibration is assumed to be valid. If the criterion is not met (>30% difference) for any one CCC, correction action MUST be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before sample analysis begins.

9.4.5 The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (twelve hour), the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made as appropriate.

9.5 GC/MS Analysis

9.5.1 It is highly recommended that the extract be screened on a GC/FID or GC/PID using the same type of capillary column. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

9.5.2 Just prior to analysis, spike an aliquot of the extract with internal standard so that the internal standard concentration in the extract is 40 ng/µl.

9.5.3 Analyze the extract by GC/MS using a 30 m x 0.25 mm (or 0.32 mm) silicone-coated fused-silica capillary column. The volume to be injected should ideally contain 100 ng of base/neutral and 200 ng of acid surrogates (for a 1 µl injection). The recommended GC/MS operating conditions to be used are specified in paragraph 9.3.

9.5.4 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/µl of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

9.5.5 Perform all qualitative and quantitative measurements as described in paragraph 9.6. Store the extracts at 4°C protected from light in screw-cap vials equipped with unpierced Teflon®-lined septa.

9.6 Data Interpretation

9.6.1 Qualitative Analysis

9.6.1.1 An analyte (e.g., those listed in Table 1) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the user's GC/MS. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

9.6.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same twelve hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

9.6.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) <u>must</u> be present in the sample spectrum.

9.6.1.1.3 The relative intensities of ions specified in paragraph 8.6.1.1.2 must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.)

9.6.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual

comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

9.6.2 Quantitative Analysis

9.6.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (e.g., Tables 5a and 5b).

Table 5a Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Acetophenone	Benzoic acid	Acenaphthene
Aniline	Bis(2-chloroethoxy)methane	Acenaphthylene
Benzyl alcohol	4-Chloroaniline	1-Chloronaphthalene
Bis(2-chloroethyl)ether	4-Chloro-3-methylphenol	2-Chloronaphthalene

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Bis(2-chloroisopropyl)ether	2,4-Dichlorophenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,6-Dichlorophenol	Dibenzofuran
1,2-Dichlorobenzene	α,α-Dimethylphenethylamine	Diethyl phthalate
1,3-Dichlorobenzene	2,4-Dimethylphenol	Dimethyl phthalate
1,4-Dichlorobenzene	Hexachlorobutadiene	2,4-Dinitrophenol
Ethyl methanesulfonate	Isophorone	2,4-Dinitrotoluene
2-Fluorophenol (Surrogate)	2-Methylnaphthalene	2,6-Dinitrotoluene
Hexachloroethane	Naphthalene	Fluorene
Methyl methanesulfonate	Nitrobenzene	2-Fluorobiphenyl (Surrogate)
2-Methylphenol	Nitrobenzene-d_8 (Surrogate)	Hexachlorocyclopentadiene
4-Methylphenol	2-Nitrophenol	1-Naphthylamine
N-Nitrosodimethylamine	N-Nitroso-di-n-butylamine	2-Naphthylamine
N-Nitroso-di-n-propylamine	N-Nitrosopiperidine	2-Nitroaniline
Phenol	1,2,4-Trichlorobenzene	3-Nitroaniline
Phenol-d_5 (Surrogate)		4-Nitroaniline
2-Picoline		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (Surrogate)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

Table 5b Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzo(a)anthracene	Benzo(b)fluoranthene
Anthracene	Bis(2-ethylhexyl)phthalate	Benzo(k)fluoranthene
Benzidine	Butyl benzyl phthalate	Benzo(g,h,i)perylene

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Bromophenyl phenyl ether	Chrysene	Benzo(a)pyrene
Di-n-butyl phthalate	3,3'-Dichlorobenzidine	Dibenz(a,j)acridine
4,6-Dinitro-2-methylphenol	p-Dimethylaminoazobenzene	Dibenz(a,h)anthracene
Diphenylamine	Pyrene	7,12-Dimethylbenz(a)anthracene
1,2-Diphenylhydrazine	Terphenyl-d_{14} (Surrogate)	Di-n-octylphthalate
Fluoranthene		Indeno(1,2,3-cd)pyrene
Hexachlorobenzene		3-Methylcholanthrene
N-Nitrosodiphenylamine		
Pentachlorophenol		
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

9.6.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water:

$$\text{Concentration, } \mu g/l = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_o)(V_i)}$$

Where:

A_x = *Area of characteristic ion for compound being measured*

I_s = *Amount of internal standard injected (ng)*

V_t = *Volume of total extract, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 ml extract will mean V_t = 10,000 µl. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)*

A_{is} = *Area of characteristic ion for the internal standard*

RF = *Response Factor for compound being measured (paragraph 9.3.3)*

V_o = *Volume of water extracted (ml)*

V_i = *Volume of extract injected (µl)*

Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

$$\text{Concentration, } \mu g/kg = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

Where:

A_x, I_s, V_t, A_{is}, RF, V_i = *Same as for Water*

W_s = *Weight of sample extracted or diluted in grams*

D = *(100 - % moisture in sample)/100, or 1 for a wet-weight basis*

9.6.2.3 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulas given above should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences.

9.6.2.4 Report results without correction for recovery data.

9.6.2.5 Quantitation of multicomponent compounds (e.g., Aroclors) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD by Method 8080.

10. QUALITY CONTROL

10.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

10.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent water blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent water

blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

10.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., column changed), recalibration of the system must take place.

10.4 Required instrument QC is found in the following sections:

10.4.1 The GC/MS system must be tuned to meet the DFTPP specifications in Section 9.3.1 and 9.4.1.

10.4.2 There must be an initial calibration of the GC/MS system as specified in 9.3.

10.4.3 The GC/MS system must meet the SPCC criteria specified in 9.4.3 and the CCC criteria in 9.4.4, every twelve hours.

10.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

10.5.1 A quality control (QC) check sample concentrate is required containing each analyte at a concentration of 100 µg/ml in acetone. The QC check sample concentrate may be prepared from pure standard materials or purchases as certified solutions. If prepared by the laboratory, the QC check sample concentrate must be made using stock standards prepared independently from those used for calibration.

10.5.2 Using a pipet, prepare QC check samples at a concentration of 100 µg/l by adding 1.00 ml of QC check sample concentrate to each of four 1 l aliquots of reagent water.

10.5.3 Analyze the well-mixed QC check samples according to the method beginning in Section 9.1 with the extraction of the samples.

10.5.4 Calculate the average recovery ($\bar{x}$) in µg/l and the standard deviation of the recovery (s) in µg/l for each analyte of interest using the four results.

10.5.5 For each analyte, compare s and $\bar{x}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and $\bar{x}$ for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin.

If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are analyzed.

Table 6 QC Acceptance Criteria[a]

Parameter	Test Conc. (µg/l)	Limit for s (µg/l)	Range for $\bar{X}$ (ug/l)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1 - 132.3	47 - 145
Acenaphthylene	100	40.2	53.5 - 126.0	33 - 145
Aldrin	100	39.0	7.2 - 152.2	D - 166
Anthracene	100	32.0	43.4 - 118.0	27 - 133
Benzo(a)anthracene	100	27.6	41.8 - 133.0	33 - 143
Benzo(b)fluoranthene	100	38.8	42.0 - 140.4	24 - 159
Benzo(k)fluoranthene	100	32.3	25.2 - 145.7	11 - 162
Benzo(a)pyrene	100	39.0	31.7 - 148.0	17 - 163
Benzo(g,h,i)perylene	100	58.9	D - 195.0	D - 219
Benzyl butyl phthalate	100	23.4	D - 139.9	D - 152
β-BHC	100	31.5	41.5 - 130.6	24 - 149
δ-BHC	100	21.6	D - 100.0	D - 110
Bis(2-chloroethyl)ether	100	55.0	42.9 - 126.0	12 - 158
Bis(2-chloroethoxy)methane	100	34.5	49.2 - 164.7	33 - 184
Bis(2-chloroisopropyl)ether	100	46.3	62.8 - 138.6	36 - 166
Bis(2-ethylhexyl)phthalate	100	41.1	28.9 - 136.8	8 - 158
4-Bromophenyl phenyl ether	100	23.0	64.9 - 114.4	53 - 127
2-Chloronaphthalene	100	13.0	64.5 - 113.5	60 - 118
4-Chlorophenol phenyl ether	100	33.4	38.4 - 144.7	25 - 158
Chrysene	100	48.3	44.1 - 139.9	17 - 168
4,4'-DDD	100	31.0	D - 134.5	D - 145
4,4'-DDE	100	32.0	19.2 - 119.7	4 - 136

Parameter	Test Conc. (µg/l)	Limit for s (µg/l)	Range for $\bar{X}$ (ug/l)	Range p, p_s (%)
4,4'-DDT	100	61.6	D - 170.6	D - 203
Dibenzo(a,h)anthracene	100	70.0	D - 199.7	D - 227
Di-n-butyl phthalate	100	16.7	8.4 - 111.0	1 - 118
1,2-Dichlorobenzene	100	30.9	48.6 - 112.0	32 - 129
1,3-Dichlorobenzene	100	41.7	16.7 - 153.9	D - 172
1,4-Dichlorobenzene	100	32.1	37.3 - 105.7	20 - 124
3,3'-Dichlorobenzidine	100	71.4	8.2 - 212.5	D - 262
Dieldrin	100	30.7	44.3 - 119.3	29 - 136
Diethyl phthalate	100	26.5	D - 100.0	D - 114
Dimethyl phthalate	100	23.2	D - 100.0	D - 112
2,4-Dinitrotoluene	100	21.8	47.5 - 126.9	39 - 139
2,6-Dinitrotoluene	100	29.6	68.1 - 136.7	50 - 158
Di-n-octylphthalate	100	31.4	18.6 - 131.8	4 - 146
Endosulfan sulfate	100	16.7	D - 103.5	D - 107
Endrin aldehyde	100	32.5	D - 188.8	D - 209
Fluoranthene	100	32.8	42.9 - 121.3	26 - 137
Fluorene	100	20.7	71.6 - 108.4	59 - 121
Heptachlor	100	37.2	D - 172.2	D - 192
Heptachlor epoxide	100	54.7	70.9 - 109.4	26 - 155
Hexachlorobenzene	100	24.9	7.8 - 141.5	D - 152
Hexachlorobutadiene	100	26.3	37.8 - 102.2	24 - 116
Hexachloroethane	100	24.5	55.2 - 100.0	40 - 113
Indeno(1,2,3-cd)pyrene	100	44.6	D - 150.9	D - 171
Isophorone	100	63.3	46.6 - 180.2	21 - 196
Naphthalene	100	30.1	35.6 - 119.6	21 - 133
Nitrobenzene	100	39.3	54.3 - 157.6	35 - 180
N-Nitrosodi-n-propylamine	100	55.4	13.6 - 197.9	D - 230
PCB-1260	100	54.2	19.3 - 121.0	D - 164

Parameter	Test Conc. (µg/l)	Limit for s (µg/l)	Range for $\bar{x}$ (ug/l)	Range p, p_s (%)
Phenanthrene	100	20.6	65.2 - 108.7	54 - 120
Pyrene	100	25.2	69.6 - 100.0	52 - 115
1,2,4-Trichlorobenzene	100	28.1	57.3 - 129.2	44 - 142
4-Chloro-3-methylphenol	100	37.2	40.8 - 127.9	22 - 147
2-Chlorophenol	100	28.7	36.2 - 120.4	23 - 134
2,4-Chlorophenol	100	26.4	52.5 - 121.7	39 - 135
2,4-Dimethylphenol	100	26.1	41.8 - 109.0	32 - 119
2,4-Dinitrophenol	100	49.8	D - 172.9	D - 191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0 - 100.0	D - 181
2-Nitrophenol	100	35.2	45.0 - 166.7	29 - 182
4-Nitrophenol	100	47.2	13.0 - 106.5	D - 132
Pentachlorophenol	100	48.9	38.1 - 151.8	14 - 176
Phenol	100	22.6	16.6 - 100.0	5 - 112
2,4,6-Trichlorophenol	100	31.7	52.4 - 129.2	37 - 144

s = Standard Deviation for four recovery measurements
$\bar{x}$ = Average recovery for four recovery measurements
p, p_s = Percent recovery measured
D = Detected; result must be greater than zero

[a]Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations.

10.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 10.5.6.1 or 10.5.6.2.

10.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with Section 10.5.2.

10.5.6.2 Beginning with Section 10.5.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 10.5.2.

10.6 The laboratory must, on an ongoing basis, analyze a reagent blank, a matrix spike, and a matrix spike duplicate/duplicate for each analytical batch (up to a maximum of twenty samples per batch) to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

10.6.1 A matrix spike and matrix spike duplicate must be performed for each group of twenty samples of a similar matrix.

10.6.2 Use the compounds listed in Table 7 to spike samples with 100 µg of base/neutral and 200 µg of acid compounds.

Table 7 Matrix Spiking Solutions

Base/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

10.6.3 Individual component recoveries of the matrix spike are calculated using the following equation.

$$\textit{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike sample results

SR = Sample result

SA = Spike added from spiking mix

10.6.4 Relative Percent Difference (RPD)

The laboratory is required to calculate the relative percent difference between the matrix spike and matrix spike duplicate. The relative percent difference (RPD) for each component are calculated using the following equation:

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

Where:

RPD = Relative Percent Difference

D_1 = *First sample value*

D_2 = *Second sample value (duplicate)*

10.7 Corrective action must be taken when matrix spike recoveries and RPDs fall out of the established control limits (see Table 8).

10.7.1 If an analyte fails the recovery criteria in Table 8, then a check sample must be prepared and analyzed. If the check sample recoveries are in control, then a matrix effect can account for the out-of-control matrix spike recoveries and no further action is necessary.

10.7.2 If the MS/MSD RPD exceeds the control limit and/or if the recoveries of one matrix spike are in control while the other is out-of-control, then it is possible an error was made in the extraction process or the sample may not have been homogenized properly. Then it is necessary to reextract the sample, matrix spike, and matrix spike duplicate.

Table 8 Matrix Spike Control Limits[a]

Fraction	Matrix Spike Compound	Water Recovery	Soil/Sediment Recovery	RPD
BN	1,2,4-Trichlorobenzene	39 - 98	38 - 107	23
BN	Acenaphthene	46 - 118	31 - 137	19
BN	2,4-Dinitrotoluene	24 - 96	28 - 89	47
BN	Pyrene	26 - 127	35 - 142	36
BN	N-Nitroso-di-n-propylamine	41 - 116	41 - 126	38
BN	1,4-Dichlorobenzene	36 - 97	28 - 104	27
Acid	Pentachlorophenol	9 - 103	17 - 109	47

Fraction	Matrix Spike Compound	Water Recovery	Soil/Sediment Recovery	RPD
Acid	Phenol	12 - 89	26 - 90	35
Acid	2-Chlorophenol	27 - 123	25 - 102	50
Acid	4-Chloro-3-methylphenol	23 - 97	26 - 103	33
Acid	4-Nitrophenol	10 - 80	11 - 114	50

[a]These limits are taken from the CLP SOW. Alternately each laboratory may determine their own limits from actual recovery data.

10.8 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix) as in Section 11.6, calculate the average percent recovery ($\bar{p}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{p} - 2s_p$ to $\bar{p} + 2s_p$. If $\bar{p}$ = 90% and s_p = 10%, for example, the accuracy interval is expressed as 70 - 110%. Update the accuracy assessment for each analyte on a regular basis (e.g., after each five to ten new accuracy measurements).

10.9 To determine acceptable accuracy and precision limits for surrogate standards, the following procedure should be performed.

10.9.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

10.9.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (P) and standard deviation of the percent recovery (s) for each of the surrogates.

10.9.3 For a given matrix, calculate the upper and lower control limits for method performance for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = P + 3s
Lower Control Limit (LCL) = P - 3s

10.9.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Table 9. The limits given in Table 9 are multi-laboratory performance based limits for soil and aqueous samples and, therefore, the single laboratory limits established in paragraph 11.9.3 must fall within those given in Table 9 for these matrices.

Table 9 Surrogate Spike Recovery Limits for Water and Soil/Sediment Samples

Surrogate	Water Low/Medium	Soil/Sediment Low/Medium
Nitrobenzene-d_5	35 - 114	23 - 120
2-Fluorobiphenyl	43 - 116	30 - 115
p-Terphenyl-d_{14}	33 - 141	18 - 137
Phenol-d_6	10 - 94	24 - 113
2-Fluorophenol	21 - 100	25 - 113
2,4,6-Tribromophenol	10 - 123	19 - 122

10.9.5 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions, and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration".

10.9.6 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

10.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11. PRACTICAL QUANTITATION LIMITS

Table 10 Practical Quantitation Limits (PQL) For Semivolatile Organics[a]

Semivolatiles	CAS Number	Practical Quantitation Limits[b]	
		Groundwater µg/l	Low Soil/Sediment[c] µg/kg
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
Bis(2-chloroisopropyl)ether	39638-32-9	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic Acid	65-85-0	50	1600
Bis(2-Chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330

Semivolatiles	CAS Number	Practical Quantitation Limits[b]	
		Groundwater µg/l	Low Soil/Sediment[c] µg/kg
Hexachlorocyclopentadiene	77-47-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330

Semivolatiles	CAS Number	Practical Quantitation Limits[b]	
		Groundwater µg/l	Low Soil/Sediment[c] µg/kg
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330

Other Matrices	Factor[d]
Medium-level soil and sludges by sonicator (mg/kg)	2

[a]These practical quantitation limits are highly matrix-dependant. They are listed here for guidance and may not always be achievable.

[b]PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis, therefore, PQLs will be higher based on the % moisture in each sample. This is based on a 30 g sample and gel permeation chromatography cleanup.

[c]Laboratories performing GPC clean-up must multiply the PQL by 2.

[d]PQL = (Groundwater PQL) x (Factor)

12. TARGET CALIBRATION

12.1 See Sections 9.3 and 9.4

13. QUALITATIVE IDENTIFICATION INFORMATION

13.1 See Section 9.6

14. RECOVERY LIMITS

14.1 Recovery limits are established by each laboratory as stated in the check sample, surrogate spike, and matrix spike QC SOPs.

15. WASTE DISPOSAL - Not Applicable

16. TROUBLESHOOTING GUIDE - Not Applicable

17. GLOSSARY OF TERMS - Not Applicable

18. REFERENCES AND ASSOCIATED SOPS

18.1 Reference

18.1.1 SW846 Third Edition, Gas Chromatography/Mass Spectrometry for Semivolatile Organics: Capillary Column, Method 8270

18.1.2 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984

18.2 Associated SOPs

18.2.1 Separatory Funnel Liquid/Liquid Extraction, Method 3510

18.2.2 Continuous Liquid/Liquid Extraction, Method 3520

18.2.3 Soxhlet Extraction, Method 3540

18.2.4 Sonication Extraction, Method 3550

18.2.5 Waste Dilution, Method 3580

PESTICIDE/PCB

BY METHOD 8080

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1. SCOPE AND APPLICATION

1.1 Method 8080 is used to determine the concentration of certain organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater and solid sample matrices. Specifically, Method 8080 may be used to detect the following substances.

Table 1 Pesticide and Polychlorinated Biphenyls Analyzed by Method 8080

Common Name	Chemical Abstract Service Registry Number (CAS)
Aldrin	309-00-2
alpha-BHC	319-84-6
alpha-Chlordane[a]	5103-71-9
beta-BHC	319-85-7
Chlordane	57-74-9
delta-BHC	319-86-8
gamma-BHC (Lindane)	58-89-9
gamma-Chlordane[a]	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-47
Endrin Ketone[a]	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Aroclor-1016	12674-11-2

Common Name	Chemical Abstract Service Registry Number (CAS)
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

[a]These compounds are not required by Method 8080.

1.2 Method 8080 is recommended for use only by, or under close supervision of, experienced residue analysts.

1.3 Method detection limits for organochlorine pesticides and polychlorinated biphenyls (PCBs) are listed in Table 2.

Table 2 Method Detection Limits for Organochlorine Pesticides and PCBs

Compound	Method Detection Limit (µg/l)
Aldrin	0.0011
alpha-BHC	0.0011
alpha-Chlordane	0.0033
beta-BHC	0.004
delta-BHC	0.0022
Chlordane	0.019
gamma-BHC (Lindane)	0.004
gamma-Chlordane	0.0032
4,4'-DDD	0.0040
4,4'-DDE	0.0047
4,4'-DDT	0.0042
Dieldrin	0.0026
Endosulfan I	0.0020
Endosulfan II	0.0027
Endosulfan Sulfate	0.0051

Compound	Method Detection Limit (µg/l)
Endrin	0.0048
Endrin Aldehyde	0.0052
Endrin Ketone	0.0068
Heptachlor	0.0025
Heptachlor Epoxide	0.0081
Methoxychlor	0.014
Toxaphene	0.030
Aroclor-1016	0.006
Aroclor-1221	0.022
Aroclor-1232	0.0015
Aroclor-1242	0.0217
Aroclor-1248	0.0179
Aroclor-1254	0.0127
Aroclor-1260	0.0148

2. METHOD SUMMARY

2.1 This method provides gas chromatographic conditions for detection and identification of organochlorine pesticides and polychlorinated biphenyls (PCBs).

2.2 An aliquot of prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by an electron capture detector (ECD).

2.3 The level of quality control needed to validate the method is described later in this SOP.

3. SAFETY PRECAUTIONS

3.1 The toxicity or carcinogenicity of the chemicals used in this method has not been precisely determined; each chemical is treated as a potential health hazard, and exposure to these chemicals is to be minimized.

3.2 The laboratory maintains a current awareness file containing material safety data sheets (MSDS) for each chemical used in this method. The file is easily accessible to all employees.

3.3 Safety glasses and protective gloves and clothing must be worn. Manipulation of the samples and sample extracts are performed in a safety hood.

4. INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baseline, causing misinterpretations of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis.

4.2 Contamination by carryover may occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of hexane to check for cross-contamination.

5. APPARATUS AND MATERIALS

5.1 Gas Chromatographic System

5.1.1 Gas Chromatograph: Modified to accept megabore columns

5.1.2 Data System or Integrator: Capable of peak integration

5.1.3 Gas chromatographic columns:

Table 3 Gas Chromatographic Columns

Column	Length	Internal Diameter	Film Thickness
DB-608	30 mm	0.53 mm	1 micron
DB-5	30 mm	0.53 mm	1 micron
DB-1701	30 mm	0.53 mm	1 micron

5.1.3.1 Since confirmation runs are required for organochlorine pesticides and PCBs, a Restek press fit "Y" splitter can be used to allow two columns to be utilized with one autosampler injector.

5.1.4 Autosampler: Capable of reproducible injections

5.1.5 Carrier Gas: Helium, ultra high purity (99.999%)

5.1.6 Make-up Gas: Nitrogen, ultra high purity (99.999%)

5.1.7 Detector: Electron Capture Detector (ECD)

5.2 Pipettes: Disposable , 10 - 20 µl, 25 - 50 µl, 50 - 100 µl, 100 - 200 µl Pasteur, 5 3/4 inch

5.3 Syringes: Gas-tight, 250 µl, 500 µl, and 1 ml

5.4 Glassware: Beakers, 100 ml and 200 ml; volumetric flasks with ground-glass stoppers; and amber jars with Teflon®-lined screw caps.

5.5 Autosampler Vials: 1 ml with 11 mm crimp cap, Teflon®/silicon septum liner

5.6 Balance: Analytical, capable of accurately weighing ± 0.0001 g

5.7 Test Tubes: 10 x 100 mm culture tubes with Teflon®-lined screw caps

5.8 Test Tube Racks

6. REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Hexane: Pesticide residue grade

6.1.2 Isopropyl Alcohol: reagent grade

6.1.3 Reagent Water: Reagent water is defined as a water in which an interferant is not observed at or above the detection limits of each parameter of interest.

6.1.4 Tetrabutylammonium Hydrogen Sulfate (TBA): 96% purity or greater

6.1.4.1 TBA Solution Preparation

6.1.4.1.1 Into a 100 ml volumetric flask, add 3.39 g of tetrabutylammonium hydrogen sulfate and dilute to the mark with reagent water.

6.1.4.1.2 Invert the flask several times until the TBA has dissolved.

6.1.4.1.3 Add 2 ml of hexane and shake the flask for approximately one minute. Remove and dispose of the hexane layer (this helps to remove organic contaminations).

6.1.4.1.4 Repeat Step 6.1.4.1.3.

6.1.4.1.5 Into an amber 200 ml jar with a Teflon®-lined screw cap, add 25 g of anhydrous sodium sulfite.

6.1.4.1.6 Slowly add solution of TBA and shake until the sodium sulfite has dissolved.

6.1.4.1.7 This solution can be stored at room temperature for at least a month.

6.2 Standards

6.2.1 The organochlorine pesticides and polychlorinated biphenyls (PCBs) standards are purchased from chemical suppliers with results traceable to the National Bureau of Standards.

6.2.2 Stock Standard Solution

6.2.2.1 All individual organochlorine pesticides and polychlorinated biphenyls (PCBs) are prepared in hexane. The solutions are stored at less than 0°C for up to one year.

6.2.3 Working Standard

6.2.3.1 Working standards are prepared in hexane at the concentrations listed in Table 4. These solutions are stored at 4°C for up to six months. They are replaced sooner if comparisons with check standards indicate a problem.

6.2.3.1.1 The single peak responding pesticides are separated into IND A (Individual Mix A) and IND B (Individual Mix B) and contain the following components.

Table 4 Working Standard Concentrations (µg/l)

Compound	C_1	C_2	C_3	C_4	C_5
Individual Mix A					
alpha-BHC	0.005	0.01	0.0125	0.025	0.05
gamma-BHC	0.005	0.01	0.0125	0.025	0.05
Heptachlor	0.005	0.01	0.0125	0.025	0.05
Endosulfan I	0.005	0.01	0.0125	0.025	0.05
Dieldrin	0.01	0.02	0.025	0.05	0.1
Endrin	0.01	0.02	0.025	0.05	0.1
4,4'-DDD	0.01	0.02	0.025	0.05	0.1
Endrin Aldehyde	0.01	0.02	0.025	0.05	0.1
Endosulfan Sulfate	0.01	0.02	0.025	0.05	0.1
Endrin Ketone	0.01	0.02	0.025	0.05	0.1

Compound	C_1	C_2	C_3	C_4	C_5
Dibutylchlorendate[a]	0.025	0.05	0.0625	0.125	0.25
Individual Mix B					
beta-BHC	0.005	0.01	0.0125	0.025	0.05
delta-BHC	0.005	0.01	0.0125	0.025	0.05
Aldrin	0.01	0.02	0.025	0.05	0.1
Heptachlor Epoxide	0.005	0.1	0.0125	0.025	0.05
gamma-Chlordane	0.005	0.01	0.0125	0.025	0.05
alpha-Chlordane	0.005	0.01	0.0125	0.025	0.05
4,4'-DDE	0.01	0.02	0.025	0.05	0.1
Endosulfan II	0.01	0.02	0.025	0.05	0.1
Multi-Response Standards					
Chlordane	0.05	0.1	0.2	0.25	0.5
Toxaphene	0.25	0.5	1.0	1.25	2.5
Aroclor-1016	0.05	0.1	0.2	0.4	0.5
Aroclor-1221	0.05	0.1	0.2	0.4	0.5
Aroclor-1232	0.05	0.1	0.2	0.4	0.5
Aroclor-1242	0.05	0.1	0.2	0.4	0.5
Aroclor-1248	0.05	0.1	0.2	0.4	0.5
Aroclor-1254	0.1	0.2	0.5	0.8	1.0
Aroclor-1260	0.1	0.2	0.5	0.8	1.0

[a]Surrogate

6.2.4 A system evaluation mix (Eval B) is prepared in hexane at the following concentrations.

Table 5 System Evaluation Mix Concentrations

Compound	Concentration, µg/ml
Aldrin	0.01
Endrin	0.02
4,4'-DDT	0.02
Dibutylchlorendate	0.05

6.2.5 Check sample and MS/MSD acid organochlorine pesticides and PCBs spiking solutions are prepared in acetone at the following concentrations.

Table 6 Check Sample and MS/MSD Pesticide/PCB Concentrations

Compound	Water, µg/ml	Low-Level Solid, µg/ml	Medium-Level Solid, µg/ml
gamma-BHC (Lindane)	0.2	2	2
Heptachlor	0.2	2	2
Aldrin	0.2	2	2
Dieldrin	0.5	5	5
Endrin	0.5	5	5
4,4'-DDT	0.5	5	5

6.2.5 The surrogate spiking solutions are prepared in acetone at the following concentrations.

Table 7 Surrogate Spike Pesticide/PCB Concentrations

Surrogate	Water, µg/ml	Low-Level Solid, µg/ml	Medium-Level Solid, µg/ml
Dibutylchlorendate	1	5	20
2,4,5,6-Tetrachloro-m-xylene	0.1	0.5	2

7. SAMPLE STORAGE AND PRESERVATION

7.1 Water and solid samples are stored in glass containers with Teflon®-lined caps at 4°C until the time of extraction.

7.2 Sample extracts and dilutions are stored in test tubes and vials at 4°C until the time of analysis.

8. HOLDING TIMES

8.1 Aqueous samples are extracted within seven days of sampling.

8.2 Solid samples are extracted within fourteen days of sampling.

8.3 Oils are prepared within fourteen days of sampling.

8.4 Extracts and preparative dilutions are analyzed within forty days of extraction.

9. TUNING AND CALIBRATION

9.1 Initial Calibration

9.1.1 Recommended Instrument Conditions

9.1.1.1 Helium carrier gas flow rate: 5 - 6 ml/minute

9.1.1.2 Nitrogen make-up gas flow rate: 30 - 35 ml/minute

9.1.1.3 GC Conditions

9.1.1.3.1 Isothermal

Oven Temperature:	220°C
Detector Temperature:	325°C
Injector Temperature:	250°C
Analysis Time:	30 minutes

9.1.1.3.2 Temperature Program

Initial Temperature:	150°C
Initial Hold Time:	0.5 minutes
Rate:	5°C/minute
Final Temperature:	255°C
Final Hold Time:	12 minutes
Detector Temperature:	325°C
Injector Temperature:	250°C

9.1.1.4 Two columns of dissimilar phase are required (see Section 5.1.3 for column selection).

9.1.1.5 Injection volume: 1 µl

9.1.2 If the injection port or front of the column is dirty, 4,4'-DDT and endrin are easily degraded in the injection port . This is the result of buildup of high boiling residue from sample injection. Check for degradation problems by injecting 1 µl of the Eval B mix. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). If degradation of either DDT or endrin exceeds 20%, replace the glass injection insert with a cleaned and deactivated insert. If this procedure fails to eliminate degradation problem, clip off up to one foot of the injection port side of the column.

Calculate the percent breakdown as follows:

$$\text{\% Breakdown from 4,4'-DDT} = \frac{\text{Total DDT Degradation Peak Area (DDE + DDD)}}{\text{Total DDT Peak Area (DDT + DDE + DDD)}} \times 100$$

$$\text{\% Breakdown from Endrin} = \frac{\text{Total Endrin Degradation Peak Area (Endrin Aldehyde + Endrin Ketone)}}{\text{Total Endrin Peak Area (Endrin + Endrin Aldehyde + Endrin Ketone)}} \times 100$$

9.1.3 For each organochlorine pesticide, polychlorinated biphenyl (PCB), and surrogate standard, analyze five calibration standards utilizing the GC conditions in Section 9.1.1.

9.1.3.1 One of the standards is analyzed at or near the concentration which corresponds to the detection limit. The concentration of each organochlorine pesticide and polychlorinated biphenyl (PCB) standard is listed in Section 6.2.3.

9.1.4 Each calibration standard is injected into the GC with the same technique used to inject the samples (i.e., autosampler injection, manual injection).

9.1.5 The calibration factor for each pesticide is calculated as follows (for multi-peak responding components, 3 - 5 peaks are chosen and the corresponding heights/areas are summed).

$$Cf = \frac{\text{Height/Area}}{\text{Weight Injected (ng)}}$$

9.1.6 The average calibration factor for each organochlorine pesticide and polychlorinated biphenyl (PCB) is determined using the five calibration factors from the initial calibration curve as follows.

$$Cf\ average = \frac{Cf_1 + Cf_2 + Cf_3 + Cf_4 + Cf_5}{5}$$

9.1.7 The percent relative standard deviation (%RSD) is calculated from the initial calibration as follows.

$$\%RSD = \frac{SD}{Cf\ avg} \times 100$$

Where:

% RSD = Percent Relative Standard Deviation

SD = Standard Deviation for a small population (On - 1)

CF = Average Calibration Factor

9.1.8 The % RSD for each organochlorine pesticide and polychlorinated biphenyl (PCB) must be 20% or less over the working range of the curve before any samples can be analyzed.

9.1.9 Current initial calibration data and original chromatograms are retained while in use. Once a new initial calibration is generated, the prior initial calibration is then filed and stored for future reference and recordkeeping.

9.2 Continuing Calibration

9.2.1 For recommended instrument conditions see Section 9.1.1.

9.2.2 The continuing calibration curve, for each organochlorine pesticide and polychlorinated biphenyl (PCB) is verified at the beginning of an analytical sequence, after every ten samples, and at the end of an analytical sequence by running a mid-range continuing calibration standard.

9.2.3 The continuing calibration Cf (calibration factor) for each organochlorine pesticide and polychlorinated biphenyl (PCB) is calculated using the equation in Section 9.1.5.

9.2.4 The percent difference (%D) between the continuing calibration factor and the average calibration is calculated as follows.

$$\%D = \frac{Cf\ avg - Cf_c}{Cf\ avg} \times 100$$

Where:

%D = *Percent Difference*

Cf avg = *Average Calibration Factor from the initial calibration*

Cf_c = *Calibration Factor from the continuing calibration standard*

9.2.5 The continuing calibration factors for each single component pesticide may vary by no more than ± 15 percent difference on the primary column.

9.2.6 All multi-response product continuing calibrations may vary by no more than ± 15 percent difference on the primary column.

9.2.7 The analyst may use both columns to meet criteria (i.e., if one compound is greater than 15 percent difference on the primary column, then the analyst must use the secondary column to meet criteria).

9.2.8 The secondary or confirmation column for organochlorine pesticides and polychlorinated biphenyls (PCBs) continuing calibrations can vary by no more than ± 25% in order to confirm the presence of a pesticide.

9.2.9 If all continuing calibration criteria are not met and the instrument is in optimum operating condition, a new initial calibration is generated for all failed compounds. All samples associated with a failed continuing calibration are not valid, and must be reanalyzed by a system that passes criteria.

9.2.10 All continuing calibration data and original chromatograms are filed and stored for future reference and recordkeeping.

9.3 Retention Time Windows

9.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all pesticide and PCB standard mixtures throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

9.3.2 Calculate the standard deviation of the three absolute retention times for each single component standard. For multi-response products (i.e., PCBs), chose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

9.3.2.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multi-responding products, the analyst may reference the retention time window, but should primarily rely on pattern recognition.

9.3.2.2 In those cases where the standard deviation for a particular standard is zero, the analyst must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

9.3.3 The analyst must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed, flows rates are readjusted or GC conditions are changed. The data must be retained by the laboratory.

10. SAMPLE PREPARATION

10.1 Appropriate extraction techniques are used before sample analysis.

10.1.1 Medium-Level Soil Extraction Amounts

- Initial - 30 g
- Final - 100 ml

(Unless insufficient sample or other matrix constraints)

10.1.2 Low-Level Soil Extraction Amounts

- Initial - 30 g
- Final - 20 ml

10.1.3 Water Extraction Amounts

- Initial - 1 l
- Final - 5 ml

(Unless insufficient sample)

10.2 The following clean-up procedures may be used when sulfur interferences are present.

10.2.1 TBA Solution Clean-Up Procedure

Into a 40 ml volume bottle, add 2 ml of sample, 1 ml TBA solution, and 1 ml of isopropyl alcohol. Shake vigorously for one minute after which a precipitation will form. Next, add 5 ml of reagent water and again shake vigorously for one minute. Allow the phases to separate, the top layer is the sample extract.

10.2.2 Mercury Clean-Up Procedure

Into a 5 ml culture tube, add about 2 ml of the sample extract. Using a disposable pipette, pipette a small amount of mercury from the container and add it to the sample extract. Vortex this mixture for about one minute. If the mercury has turned black, the procedure may be repeated to ensure the removal of sulfur interferences.

10.3 Gel permeation chromatography (GPC) may be employed to eliminate lipids, polymers, copolymers, natural resins and polymers, cellular components, viruses steroids, and dispersed high-molecular-weight compounds that may interfere with the target compounds.

10.4 Florisil cartridge clean-up may be used to reduce matrix interferences caused by polar compounds.

10.5 The sample extract may be screened to determine if the level of the organochlorine pesticides and polychlorinated biphenyls (PCBs) exceeds the working range of the calibration curve, an appropriate dilution is performed to maintain a level within the initial calibration curve.

11. SAMPLE ANALYSIS

11.1 A method blank and check sample must be analyzed and pass criteria established in Section 12 before proceeding with sample analysis.

11.2 For recommended instrument conditions, see Section 9.1.1.

11.3 The samples must be analyzed as part of an analytical sequence as follows.

- Evaluation Mix B
- Individual Mix A
- Individual Mix B
- Toxaphene
- Chlordane
- Aroclor-1242
- Aroclor-1248
- Aroclor-1254
- Aroclor-1260
- 10 samples (QC or Production)
- One Multi-response Product
- Individual Mix A

- 10 samples (QC or Production)
- One of the other Multi-Response Products
- Individual Mix B

This analytical sequence may be continued as long as the sequence continues to pass established criteria.

11.4 Inject 1 µl of the sample extract or diluted sample into the GC using the same operating conditions and techniques as those used in the calibration of the instrument.

11.5 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) Identification

11.5.1 Organochlorine pesticide and polychlorinated biphenyl (PCB) identification requires the use of two columns with dissimilar phase. One of the columns used is the primary or quantitation column while the other is the secondary or confirmation column.

11.5.2 On the primary or quantitation column, compare the samples chromatographic peak retention times to the retention time windows established for the organochlorine pesticides and polychlorinated biphenyls (PCBs) of interest. For PCB and multi-response pesticides the analyst should rely heavily on pattern recognition.

11.5.3 If the retention time for an unknown peak yields a positive match on the primary column, then the secondary or confirmation column must also yield a positive match for a organochlorine pesticide or polychlorinated biphenyl (PCB) to be detected.

11.5.4 If a positive match is not established on both columns, then no organochlorine pesticides and polychlorinated biphenyls (PCBs) of interest are detected in the sample.

11.5.5 Although retention time windows are established to assist the analyst, sound analytical judgement must also be used while interpreting chromatograms. The experience of the analyst is critical to properly identify organochlorine pesticides and polychlorinated biphenyls (PCBs).

11.6 Sample Quantification

11.6.1 The calibration factor of the continuing calibration standard is run immediately prior to the sample is used to quantify the organochlorine pesticide or polychlorinated biphenyl (PCB). If samples are run immediately after the initial calibration, then the average calibration factor is used.

11.6.2 Calculate the weight of the analyte present in the sample as follows

$$Amt = \frac{Ht}{Cf}$$

Where:

Amt = Weight of analyte present in the sample

Ht = Height/Area of the analyte present in the sample

Cf = Calibration Factor of the analyte of interest

11.6.3 If the weight (ng) of the analyte present in the sample exceeds the working range of the calibration curve, an appropriate dilution is performed and the sample is reanalyzed. All dilutions are prepared with hexane as the diluent.

11.6.4 Calculate the sample results as follows.

$$Conc,\ Pest/PCB = \frac{Ht \times V_f}{V_i \times Cf \times INJ_v} \times D$$

Where:

Ht = Height/Area of the analyte present in the sample

V_f = Final extract volume

V_i = Initial extraction sample volume or weight

Cf = Calibration Factor

INJ_v = Injection Volume

D = Dilution Factor, if appropriate (Example: If a 1:10 dilution was performed on the sample, D = 10)

11.6.5 Water and soil unit determinations are derived as follows.

Water Units

$$Conc.,\ Pest = \frac{Ht\ (height) \times V_f\ (ml) \times D\ (unitless)}{V_i\ (L) \times Cf\ \frac{height}{ng} \times INJ_v\ (\mu l)}$$

Solid Units

$$Conc., Pest = \frac{Ht\ (height) \times V_i\ (ml)}{V_i\ (g) \times Cf\ \frac{height}{ng} \times INJ_V\ (\mu l)} \times D\ (unitless)$$

11.6.6 Detection limits or practical quantitation limits (PQLs) for organochlorine pesticides and polychlorinated biphenyls (PCBs) are calculated as follows.

Table 8 Practical Quantitation Limit Calculations

Compound	Water µg/l	Low-Level µg/kg	Medium-Level mg/kg
Aldrin alpha-BHC beta-BHC delta-BHC gamma-BHC (Lindane) Heptachlor Heptachlor Epoxide Endosulfan I	$\frac{0.01\ ng \times 5\ ml}{11 \times 1\ \mu l}$ = 0.05 µg/l	$\frac{0.01\ ng \times 20\ ml}{30\ g \times 1\ \mu l}$ = 6.7 µg/kg Which is rounded to 8 µg/kg[a]	$\frac{0.01\ ng \times 100\ ml}{30\ g \times 1\ \mu l}$ = 0.033 mg/kg Which is rounded to 0.05 mg/kg
Alachlor Dieldrin 4,4'-DDE 4,4'-DDD 4,4'-DDT Endosulfan II Endosulfan Sulfate Endrin Endrin Aldehyde	$\frac{0.02\ ng \times 5\ ml}{11 \times 1\ \mu l}$ = 0.1 µg/l	$\frac{0.02\ ng \times 20\ ml}{30\ g \times 1\ \mu l}$ = 13.3 µg/kg Which is rounded to 16 µg/kg[a]	$\frac{0.02\ ng \times 100\ ml}{30\ g \times 1\ \mu l}$ = 0.067 mg/kg Which is rounded to 0.1 mg/kg

Compound	Water µg/l	Low-Level µg/kg	Medium-Level mg/kg
Aroclor-1016 Aroclor-1221 Aroclor-1232 Aroclor-1242 Aroclor-1248 Chlordane Methoxychlor	0.1 ng x 5 ml / 11 x 1 µl = 0.5 µg/l	0.1 ng x 20 ml / 30 g x 1 µl = 16 µg/kg Which is rounded to 80 µg/kg[a]	0.1 ng x 100 ml / 30 g x 1 µl = 0.33 mg/kg Which is rounded to 0.5 mg/kg
Aroclor-1254 Aroclor-1260 Toxaphene	0.2 ng x 5 ml / 11 x 1 µl = 1 µg/l	0.2 ng x 20 ml / 30 g x 1 µl = 133 µg/kg Which is rounded to 160 µg/kg[a]	0.2 ng x 100 ml / 30 g x 1 µl = 0.67 mg/kg Which is rounded to 1 mg/kg

[a]CLP Detection Limit

11.7 The results are then reported on the sample worksheet along with any organochlorine pesticide and polychlorinated biphenyl (PCB) identifications, detection limits, and surrogate recoveries.

11.8 Report the run and all related information in the logbooks of each GC used for analysis.

11.9 A file folder is created for samples which are grouped according to lot number.

11.9.1 Information listed on the folder will include:

- Client Code
- Due Date
- Analysis Fraction (i.e., Pesticide/PCB)
- Sample Numbers

11.9.2 Upon completion of analysis, the following will be contained in each folder, which is then handed in to the group leader.

- Completed Worksheet
- Sample Chromatograms
- Calculation Sheet
- Copies of Standard Chromatograms
- Copies of Initial Calibrations
- Copies of Continuing Calibrations

12. QA/QC REQUIREMENTS

12.1 Calibration requirements stated in Section 9 are met before sample analysis proceeds.

12.2 Surrogate

12.2.1 Dibutylchlorendate (DBC) and 2,4,5,6-Tetrachloro-m-xylene (TCMX) are spiked into every method blank, check sample, matrix spike, and matrix spike duplicate.

12.2.2 Add 1 µl of the appropriate surrogate spiking solution (Section 6.2.6) to each method blank, check sample, sample, and MS/MSD.

12.2.3 The theoretical surrogate concentration is calculated as follows.

$$Tconc_{ss} = \frac{V_{ss} \times Conc_{ss}}{V_i}$$

Where:

$Tconc_{ss}$ = *Theoretical concentration of each surrogate*

V_{ss} = *Volume of surrogate spiking solution*

$Conc_{ss}$ = *Calculated surrogate analyte concentration*

V_i = *Initial volume*

12.2.4 After each sample has been analyzed, the surrogate result is calculated (Section 11.6.4).

12.2.5 The percent recovery (%R) for each surrogate is calculated as follows.

$$\%R = \frac{Conc_{ss}}{TConc_{ss}} \times 100$$

Where:

$\%R$ = *Percent Recovery*

$Conc_{ss}$ = *Calculated surrogate analyte concentration*

$TConc_{ss}$ = *Theoretical surrogate analyte concentration*

12.2.6 Dibutylchlorendate and 2,4,5,6-Tetrachloro-m-xylene surrogate recovery limits are be updated periodically; therefore, compare the results to the most recent control limit criteria generated by the Quality Control Department.

12.2.7 Dibutylchlorendate is the primary surrogate and should used whenever possible. However, if DBC recovery is low or compounds interfere with DBC, then the TCMX should be evaluated for acceptance.

12.2.8 If DBC and TCMX surrogate recoveries fail for the method blank, data must be evaluated in the following manner.

12.2.8.1 If sample surrogate results meet the recovery limits and no pesticides or PCBs are present above the detection limit, a sample may serve as a blank. A sample can to serve as its own blank.

12.2.8.2 If sample surrogate results meet the recovery limit, but pesticides and PCBS are present above the detection limit, the samples are reextracted along with all quality control samples in the batch until all blank criteria are met.

12.2.9 If DBC and TCMX sample surrogate recoveries fail, then the sample is reextracted.

12.2.9.1 If the reextracted sample surrogate results meet the recovery limits, then the reextracted results are reported.

12.2.9.2 If the reextracted sample surrogate results fail and are similar to the surrogate recoveries originally detected, then a matrix effect is indicated and the original results are reported. A note indicating the presence of matrix interference accompanies the sample worksheet.

12.2.10 If the sample is diluted such that DBC and TCMX can no longer be quantitated, then the surrogate results are considered diluted out and is reported as such on the sample worksheet.

12.2.11 The surrogate recovery for each sample is reported on the sample worksheet.

12.3 Method Blank

12.3.1 Each day samples are extracted, a method blank is extracted using the same procedure.

12.3.2 The method blank is analyzed before sample analysis begins.

12.3.3 The method blank cannot contain any analyte of interest above the detection limit.

12.3.4 If blank contaminants exceed the detection limit, all samples and quality control samples associated with the contaminated method blank are reextracted and reanalyzed.

12.3.5 The blank results along with its surrogate recoveries are reported on the corresponding blank worksheet. Once completed, the worksheet is then turned in to the Quality Control Department.

12.3.6 All original method blank chromatograms are filed and stored for future reference and recordkeeping.

12.4 Check Sample

12.4.1 Each time samples are extracted, a check sample is extracted using the same procedure.

12.4.2 The check sample is analyzed after the method blank and before the samples.

12.4.3 The solution used to spike the check sample is prepared separately from that used to standardize the instrument and serves to monitor the extraction and calibration standards. The solution is prepared in acetone so that it is soluble in water matrices.

12.4.4 The check sample is spiked before preparation with 1 ml of the check sample spiking solution (Section 6.2.5) and 1 ml of the appropriate surrogate spiking solution (Section 6.2.6).

12.4.5 Calculate the theoretical concentration for each analyte in the check sample as follows.

$$Tconc_{cs} = \frac{V_{css} \times Conc_{css}}{V_i}$$

Where:

$Tconc_{cs}$ = *Check sample analyte theoretical concentration*

V_{css} = *Volume of check sample spiking solution*

$Conc_{css}$ = *Check sample spiking solution concentration*

V_i = *Initial volume*

12.4.6 After the check sample has been analyzed, calculate the check sample results as described in Section 11.6.4.

12.4.7 The percent recovery (%) of the check sample is calculated as follows.

$$\%R = \frac{Conc_{CS}}{TConc_{CS}} \times 100$$

Where:

$\%R$ = *Percent Recovery*

$Conc_{CS}$ = *Calculated check sample analyte concentration*

$TConc_{CS}$ = *Theoretical check sample analyte concentration*

12.4.8 Check sample recovery limits are updated periodically; therefore, compare the results to the most recent control limit criteria generated by the Quality Control Department.

12.4.9 If the check sample fails to meet the recovery limits, all samples associated with the check sample must be reextracted and reanalyzed.

12.4.10 Check sample results along with its surrogate recoveries are reported on the corresponding check sample worksheet. Once completed, the worksheet is then turned into the Quality Control Department.

12.4.11 All original check sample chromatograms are filed and stored for future reference and recordkeeping.

12.5 Matrix Spike/Matrix Spike Duplicate

12.5.1 Every twentieth sample of a similar matrix is split into an original sample, a matrix spike (MS) subsample, and a matrix spike duplicate (MSD) subsample if sufficient sample is provided.

12.5.2 The MS/MSD samples are analyzed immediately following the original sample.

12.5.3 The MS/MSD is spiked with the same amounts of spiking solutions as the check sample (Section 12.4.4).

12.5.4 The theoretical MS/MSD spike amounts for each analyte are calculated as follows.

$$Tconc_{MS} = \frac{V_{MSS} \times Conc_{MSS}}{V_1}$$

Where:

$Tconc_{MS}$ = *Theoretical concentration of each spiking analyte*

V_{MSS} = *Volume of matrix spike solution*

$Conc_{MSS}$ = *Concentration of each spiking analyte in the matrix spike solution*

V_1 = *Initial volume*

12.5.5 After the MS/MSD samples have been analyzed, calculate the true amount of each analyte in the MS/MSD sample (Section 11.6.4).

12.5.6 The percent recovery (%R) for each analyte in the MS/MSD sample is calculated as follows.

$$\%R = \frac{Conc_{MS} - Sample\ Amt}{TConc_{MS}} \times 100$$

Where:

%R = *Percent Recovery*

$Conc_{MS}$ = *Calculated analyte concentration in the MS/MSD sample*

Sample Amt = *Concentration of the analyte found in the original sample*

$TConc_{MS}$ = *Theoretical MS/MSD analyte concentration*

12.5.7 All original MS/MSD sample chromatograms are put in folders according to lot number that the MS/MSD sample was associated with.

12.5.8 MS/MSD recovery limits are updated periodically; therefore, compare the results to the most recent control limit criteria generated by the Quality Control Department.

12.5.9 If the MS/MSD samples do not meet analyte recovery limits, a different sample is then chosen to be spiked and replaces the original MS/MSD sample.

12.5.10 The MS/MSD sample results and surrogate recoveries are then recorded on the MS/MSD sample result sheet. Once completed, this worksheet is turned in to the Quality Control Department.

13. PRACTICAL QUANTITATION LIMITS

13.1 See Table 8

14. TARGET CALIBRATION - Not Applicable

15. QUALITATIVE IDENTIFICATION INFORMATION - Not Applicable

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Consult facility Quality Control Department.

16.2 Matrix Spike

16.2.1 Consult facility Quality Control Department.

16.3 Surrogate

16.3.1 Consult facility Quality Control Department.

17. WASTE DISPOSAL - Not Applicable

18. TROUBLESHOOTING GUIDE - Not Applicable

19. GLOSSARY OF TERMS - Not Applicable

20. REFERENCES AND ASSOCIATED SOPS

20.1 References

20.1.1 SW846, Method 8080

20.2 Associated SOPs

20.2.1 Separatory Funnel Liquid/Liquid Extraction, Method 3510

20.2.2 Continuous Liquid/Liquid Extraction, Method 3520

20.2.3 Soxhlet Extraction, Method 3540 Low-Level

20.2.4 Soxhlet Extraction, Method 3540 Medium-Level

20.2.5 Sonication Extraction, Method 3550 Low-Level

Total Metals Analysis
by
Inductively Coupled Plasma
Atomic Emission Spectroscopy

6010

1. Scope and Application

1.1 Inductively coupled plasma-atomic emission spectroscopy (ICP) determines trace elements including metals in solution. The method is applicable to a large number of metals and wastes. All matrices, including ground water, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

1.2 Elements for which Method 6010 is applicable are listed in Table 2. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. The data shown in Table 2 provide concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2. Safety Precautions

2.1 The toxicity or carcinogenicity of chemicals used in this method has not been precisely determined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

2.2 The method analytes have been tentatively classified as hazards. Pure standard materials and stock standard solutions of those analytes that have a significant vapor pressure at room temperature should be handled in a hood.

2.3 Safety glasses, protective gloves, clothing and the appropriate use of ventilated hoods should be used throughout this method.

2.4 CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.1.1 In the determination of trace metals, containers can introduce either positive or negative errors in the measurement of trace metals by (a) contributing contaminants through leaching or surface desorption, and (b) depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis require particular attention. The following cleaning treatment sequence has been determined to be adequate to minimize contamination in the sample bottle, whether borosilicate glass, linear polyethylene, polypropylene, or Teflon: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and Type II water.

NOTE: Chromic acid should not be used to clean glassware, especially if chromium is to be included in the analytical scheme. Commercial, non-chromate products (e.g., Nochromix) may be used in place of chromic acid if adequate cleaning is documented by an analytical quality control program. (Chromic acid should also not be used with plastic bottles.)

3.2 Spectral interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements.

3.2.1 Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

3.2.2 Users of simultaneous multi-element instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this

purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

3.2.3 Spectral interferences were evaluated at Wadsworth/ALERT Laboratories for both the Lehman Plasma Spec ICP 2.5 and the Perkin Elmer Plasma II Emission Spectrometer. The high resolution of the Lehman's escelle grating completely eliminated all interelement spectral interferences for elements listed in Table 1. When spectral interferences occur on the PE Plasma II a secondary wavelength will be used.

3.3 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

3.4 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

3.5 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 3.5.1 and 3.5.2, will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

3.5.1 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within + 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

3.5.2 Matrix spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

4. Apparatus and Materials

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 Computer-controlled emission spectrometer with background correction.

4.1.2 Radio frequency generator.

4.1.3 Argon gas supply - Welding grade or better.

5. Reagents and Standards

5.1 Reagents

5.1.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.2 Hydrochloric acid (conc), HCl.

5.1.3 Hydrochloric acid (1:1), HCl. Add 500 ml concentrated HCl to 400 ml water and dilute to 1 liter.

5.1.4 Nitric acid (conc), HNO_3.

5.1.5 Nitric acid (1:1), HNO_3. Add 500 ml concentrated HNO_3 to 400 ml water and dilute to 1 liter.

5.1.6 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.2 Standards

Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99 to 99.999% pure).

5.2.1 Target Calibration Standards

5.2.1.1 Multi-element calibration standards will be prepared or purchased for the following mixtures in Table 1. Prepare them at concentrations appropriate to cover the linear calibration range.

TABLE 1.

Solution	Elements
I	Al, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, Tl, Zn
II	Ca, K, Mg, Na
III	Ag
IV	V

Prepare the following single element solutions as needed.

B
Bi
Li
Mo
P
Si
Sn
Sr
Ti
W

5.2.1.2 The instrument check standard is prepared by the analyst by combining compatible elements at concentrations equivalent to the midpoint of their respective calibration curves (see Section 8.4 for use).

5.2.1.3 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at approximate concentrations of 10 times the instrumental detection limits. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

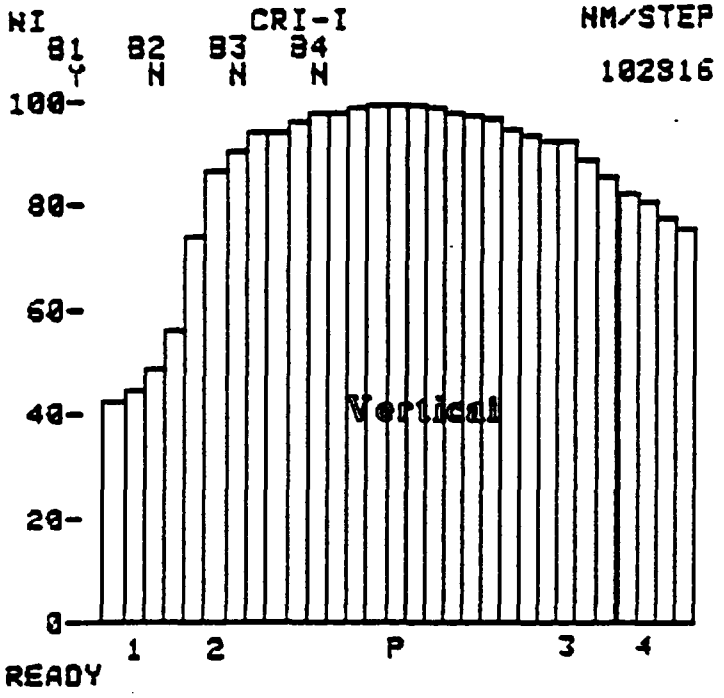
5.2.2 Matrix Spike Standard - a mid-range standard is used for spiking the matrix spike/matrix spiked duplicate.

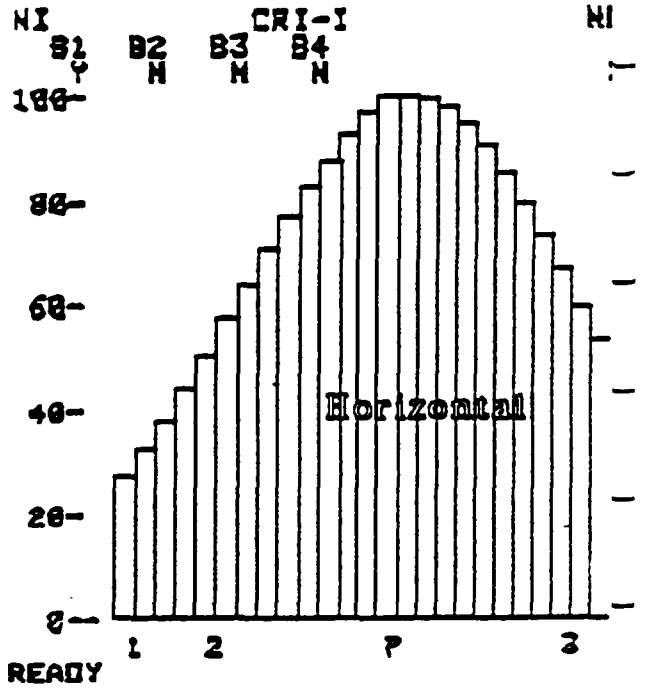
5.2.3 Tuning Standard

5.2.3.1 The tuning standard consist of one or more elements at the appropriate concentration necessary to optimize the optical hardware and software.

5.2.5.2 For the Leemen ICP prepare a 10 ppm solution of Ni. This will be used to align to optical hardware for both the vetical and horizontal axes. Show below are two typical printouts.

PEAK 329536 7:47 27 APR 89
APER X 248
APER Y 141





6. Sample Storage

6.1 Sample Handling and Preservation. Suggested collection handling is listed in Table 3.

Table 2.

Measurement	Preservative[a]
Metals (except hexavalent chromium and mercury):	
Total recoverable	HN03 to pH <2
Dissolved	Filter on site; HN03 to pH <2
Suspended	Filter on site Total HN03 to pH <2
Chromium VI:	Cool, 4°C
Mercury:	
Total	HN03 to pH <2
Dissolved	Filter; HN03 to pH <2

[a]Solid samples usually require no preservation other than storing at 4°C until analyzed.

6.2 Either plastic or glass containers which have been properly cleaned may be used for sample storage. See section 3.1 above.

7. Holding Times

7.1 Table 3 contains the sample holding times for properly preserved samples.

Table 3

Measurement	Holding Time
Metals (except hexavalent chromium and mercury):	6 months
Chromium VI:	24 hours
Mercury:	28 days

8. Tuning and Calibration

8.1 Summary

The ICP instrument should be tuned following the manufacturers recommendations. An initial calibration of a blank, calibration standards and an interference check standard. is followed by the analysis of 10 samples. This is followed by the continuing calibration of a check standard and a calibration blank. The cycle of 10 samples and continuing calibration is repeated.

8.2 Instrument Tuning

8.2.1. For Perkin-Elmer Plasma II Emission Spectrometer, tune instrument by doing a WCAL (wave calibration) using the solution from 5.2.5.3.

8.2.2 For the Lehman ICP, tune the optics with the Nickel peaking routine using the solution from section 5.2.5.2.

8.3 Initial Calibration

8.3.1 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 5.2.

8.3.2 Check instrument calibration using a calibration blank and at least two appropriate standards.

8.3.3 Flush the system with the calibration blank (Section 11.1) between each standard. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.)

8.4 Continuing Calibration

8.4.1 Verify calibration every 10 samples and at the end of the analytical run, using a calibration blank (Section 11.1.3) and a check standard (Section 5.2.1.2).

8.4.2 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.4.3 The results of the calibration blank are to agree within three standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

8.4.4 Verify the interelement and background correction factors at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check sample (Section 5.2.1.3). Results should be within + 20% of the true value obtained in Section 8.4.2.

9. Sample Preparation

9.1 Summary

Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices. Solubilization and digestion procedures are presented in Sample Preparation Methods.

9.2 Sample Preparation Procedure

Samples will have been previously prepared. In some cases when the analyte concentration exceeds the linear range of the instrument it may be necessary to dilute the sample with water.

9.3 Sample Cleanup Procedures

9.3.1 On some occasions it may it may be necessary to filter out undigested solids prior to analysis to prevent nebulizer clogging.

9.4 Preparation Documentation

9.4.1 All dilutions or filtrations must be recorded in the appropriate laboratory log book.

WADSWORTH TESTING LABORATORIES / ALERT INC.

ICP INSTRUMENT LOGSHEET

SAMPLE NUMBER	CUSTOMER CODE	MO.	DAY	YEAR	TIME	ELEMENT FILE	SEQUENCE FILE	SOURCE FILE	METHOD FILE	POSITION	Signal Comp. Y	Signal Comp. N	Internal Std. Y	Internal Std. N	PERFORMANCE STD.	STDS. USED FOR ANALYSIS	ANALYST	COMMENTS
BIK 5-12	WADQC	5	15	89		HSL						✓		✓		KWATK KWA2A	JAE	H_2O
CAL 5-12	WADQC																JAE	H_2O
EP BIK5-12	WADQC					HSL										KWATK KWA2A	JAE	EP Tox
BOL 5-11	WADQC					HSL	MoW									KWATK KWA2A MoW	JAE	H_2O
CAL 5-11	WADQC					HSL	MoW										JAE	H_2O
17510	GECO1					HSL	MoW										JAE	H_2O
17511	GECO1					HSL	MoW									KWATK KWA2A MoW	JAE	H_2O
17496	WADO2					Ag Ba Fe	Cd Ca	Cu Mg Mn	Na Ni Pb	Sb Zn						KWATK KWA2A	JAE	H_2O NS/NSD
17497	WADO2					Ag Ba Fe	Cd Ca	Cu Mg Mn	Na Ni Pb	Sb Zn							JAE	H_2O
17498	WADO2					Ag Ba Fe	Cd Ca	Cu Mg Mn	Na Ni Pb	Sb Zn							JAE	H_2O
17382	GYT19					Ag Ba	Cd Ca	Pb									JAE	EP Tox
17383	GYT19				HSL	Ag Ba	Cd Ca	Pb									JAE	EP Tox
17384	GYT19					Ag Ba	Cd Ca	Ni Pb									JAE	EP Tox
17638	HARO1					Ba Ca	Cu Fe	Pb Zn									JAE	H_2O
17639	HARO1					Ba Ca	Cu Fe	Pb Zn									JAE	H_2O
17641	HARO1					Ba Ca	Cu Fe	Pb Zn									JAE	H_2O
17642	HARO1					Ba Ca	Cu Fe	Pb Zn									JAE	H_2O
17535	CBEO1					Ca Pb	Y										JAE	H_2O
17540	KRCO1					Cd Ca	Cu Ni	Pb Zn									JAE	H_2O
17542	LEMO1					Cd Ca	Cu Ni	Pb Zn									JAE	H_2O
17495	SCSC1					Ba Pb	Zn										JAE	EP Tox
17624	WADO2					Ag Ba Ca	Cd Ca Cu	Mg Mn Na	Ni Pb Sb	Zn							JAE	H_2O
17625	WADO2					Ag Ba Ca	Cd Ca Cu	Mg Mn Na	Ni Pb Sb	Zn							JAE	H_2O
17468	TKNO1					Ag Ba	Cd Ca	Pb Ni									JAE	EP Tox
17469	TKNO1	5	15	89		Ag Ba	Cd Ca	Ni Pb								KWATK KWA2A	JAE	EP Tox

Form 566

№ ...599

10. Sample Analysis

10.1 Summary

Once the instrument has been successfully calibrated 10 samples may be analyzed before a continuing calibration is required. Calculate the sample concentration taking any dilutions into account.

10.2 Recommended Instrument Conditions

10.2.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2.2 The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

10.3 Sample Analysis Procedure

10.3.1 Preliminary Evaluation

10.3.1.1 Flush the system with the calibration blank solution for at least 1 minute (Section 11.1) before the analysis of each sample.

10.3.1.2 Analyze the instrument check standard (Section 5.2.1.2) and the calibration blank (Section 11.1) after each 10 samples. See section 8.4 for more details on this continuing calibration.

10.3.1.3 Dilute and reanalyze samples that are more concentrated than the linear calibration limit or use an alternate, less sensitive line for which quality control data is already established.

10.3.2 Confirmation - not applicable.

10.3.3 Calculations

10.3.3.1 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All water results should be reported in mg/L with two significant figures. All soil results should be reported in mg/kg with two significant figures after the amount of sample has been taken into account.

Equation 2. (water sample)

Element mg/L = L.S. conc. mg/L X D.F.

where:

L.S. conc = the least squares value for concentration calculated by the ICP instrument computer.

D.F. = dilution factor

Equation 3. (solid sample)

Element mg/kg = L.S. conc. mg/L X W.F. X D.F.

where:

L.S. conc = the least squares value for concentration calculated by the ICP instrument computer.

W.F. = weight factor (g solid/ml final digestate volume)

D.F. = dilution factor

10.4 Report Documentation

10.4.1 Complete the analysis portion of the Wadsworth worksheet. Be sure to include concentration, units, detection limit, date and analyst initials.

I. C. P.

Analyst's Initials KG JAI

CHECK SAMPLE WORKSHEET

Client Code: WADOC
Sample Number: 9088-840525
Matrix: Water

Preparation Date: 5-25-84
Analysis Date: 5-26-84
QC Group Number: 26
5-30-84

Element	True Spike Amount	Measured Spike Amount	Percent Recovery	Element	True Spike Amount	Measured Spike Amount	Percent Recovery
Ag	2000	1834	92	Mn	2000	1913	96
Al	2000	1891	95	Mo			
As				Na	4000	3960	99
B	5000	4904	98	Ni	2000	1893	95
Ba	2000	1799	90	Pb	2000	1859	93
Be	2000	1937	97	S			
Ca	4000	3895	97	Sb	2000	1836	94
Cd	2000	1902	95	Se			
Co	2000	1842	92	Si			
Cr	2000	1816	91	Sn	10,000	9741	97
Cr+				Sr			
Cu	2000	1782	89	Ti	2,000	1862	93
Fe	2000	1991	100	Tl	2000	1660	83
Hg				V	2000	1736	87
K	4000	3792	95	Zn	2000	1950	98
Mg	4000	3536	88	Zr			

METALS WORKSHEET - CANTON

890525

PROJECT MGR. : Customer Service
CLIENT CODE : MTE02

TIME : 10:55:28
LAB NUMBER : 5653-18919
RECEIVING DATE : 890525
REQUEST DATE :890525
HOLDING EXP. DATE:890623
DUE DATE :890527*
** NOON **
PRIORITY :72HR

RUSH

SAMPLE MATRIX : SOLID
SITE : PS2A/DETROIT WWTP
SAMPLING DATE: 890524

AMOUNT RECEIVED : 1X250/1X100ML
SAMPLE ID : SS-14 9.0 TO 11.0' 5-24-89 16:33
STORAGE LOCATION : B1GCMS
COMMENTS
MS REQUIRED. REFER TO SPECIAL QC REQUIREMENTS.

VOC : Y BNA : Y PEST : N METAL : Y WET : Y

PREPARATION DATE : 5-25
SAMPLE WEIGHT : ~~202~~ 201

PREPARATION : TOTAL
PREP CHARGE : _____

ELEMENT	RESULT	UNITS	LDL	PREPARATION ANALYSIS DATE	INITIALS	
Ag	<.5	mg/kg	.5	5-25/5-26	JAE	A (I) F H
As	2.0		.5	5-25/5-26	SM	A I (F) H
Ba	~~57~~ 11		.5	5-25/5-26	JAE	A (I) F H
Cd	<.5		.5		JAE	A (I) F H
Cr	5.6		1.0		JAE	A (I) F H
Hg	<.25		.25	5-25/5-26	[illegible]	A I F (H)
Pb	<2.5		2.5	5-25/5-26	JAE	A (I) F H
Se	<.25		.25	5-25/5-26	[illegible]	A I (F) H
TS	82	%	.5	5-26/5-30	DN/DH/JAE	A I F H

MATRIX SPIKE/DUPLICATE RECOVERY WORKSHEET - CANTON

CLIENT CODE : OPC01
SAMPLE NUMBER : 5596-18547
RECEIVING DATE : 890520
MATRIX : Water

PREPARATION DATE : 5-22-89
ANALYSIS DATE : 5-23-89

ANALYSIS	MEASURED SAMPLE AMOUNT, UG	TRUE SPIKE AMOUNT, UG	MEASURED SPK AMOUNT, UG	MEASURED SPK/DUP AMOUNT, UG	SPK PERCENT RECOVERY	SPK/DUP PERCENT RECOVERY
METALS						
KL Ag	ND	2000	2198	2177	~~2198~~ 110	109
KL Al	~~3314~~ 464	2000	2937	2876	124	121
SM As	ND	100	102	102	102	102
* B						
KL Ba	43	2000	2149	2150	105	105
KL Be	ND	2000	2064	2090	103	105
SM Cd	ND	5	4.3	4.3	86	86
KL Co	ND	2000	2080	~~2146~~ 2063	104	~~107~~ 103
KL Cr	ND	2000	2088	2116	104	106
KL Cu	ND	2000	~~2133~~ 2042	~~2139~~ 2064	102	103
KL Fe	972	2000	3237	3134	113	108
JK Hg	ND	5	5.2	5.2	104	104
KL Mg	7863	4000	11440	12,048	89	105

* No SpK/Dup for B, Ti, Sn.

MATRIX SPIKE/DUPLICATE RECOVERY WORKSHEET - CANTON

CLIENT CODE : OPC01
SAMPLE NUMBER : 5596-18547
RECEIVING DATE : 890520
MATRIX : Water

PREPARATION DATE : 5-22-89

ANALYSIS DATE : 5-23-89

	ANALYSIS	MEASURED SAMPLE AMOUNT, UG	TRUE SPIKE AMOUNT, UG	MEASURED SPK AMOUNT, UG	MEASURED SPK/DUP AMOUNT, UG	SPK PERCENT RECOVERY	SPK/DUP PERCENT RECOVERY
	METALS						
KG	Mn	293	2000	2333	2338	~~2335~~ 102	102
KG	Mo	ND	5000	5078	5172	102	104
KG	Ni	ND	2000	2032	2078	102	104
KG	Pb	ND	2000	2118	2118	106	106
KG	Sb	ND	2000	2043	1913	102	96
Sm	Se	ND	100	98	99	98	99
*	Sn						
*	Ti						
KG	Tl	ND	2000	2162	2177	108	109
KG	Zn	36	2000	2073	2076	102	102
*	B						

MATRIX SPIKE/DUPLICATE RECOVERY WORKSHEET - CANTON

CLIENT CODE : PPG01 ~~SH~~ SH SUS
SAMPLE NUMBER : 5654-~~18704~~ 18549
RECEIVING DATE : 5-23-89
MATRIX : WATER

PREPARATION DATE : 5-25-89
ANALYSIS DATE : 5-30-89

C.A. for 18547

ANALYSIS	MEASURED SAMPLE AMOUNT, UG	TRUE SPIKE AMOUNT, UG	MEASURED SPK AMOUNT, UG	MEASURED SPK/DUP AMOUNT, UG	SPK PERCENT RECOVERY	SPK/DUP PERCENT RECOVERY
METALS						
Sn	ND	10,000	9850	10045	99	100
Ti	ND	2,000	1845	1876	92	94
B	ND	5,000	4840	4937	97	99

11. QA/QC requirements

11.1 Method and Calibration Blanks

Two types of blanks are required for the analysis. The method or reagent blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing, and the calibration blank is used in establishing the analytical curve.

11.1.1 A reagent blank sample should be subjected to exactly the same analytical procedures as those used on actual samples. Before processing any samples, the analyst should demonstrate, at least once daily, that all glassware is free of organic contamination. The reagent blank sample should be carried through all stages of the sample preparation and measurement and contains the same reagent concentrations in the final solution as in the sample solution used for analysis. If there is a change in reagents, a reagent blank should be processed as a safeguard against reagent contamination. One reagent blank should be prepared for every analytical batch or for every 20 samples, whichever is greater.

11.1.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried

through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

11.1.3 Employ a minimum of one reagent blank per sample batch to determine if contamination or any memory effects are occurring.

11.1.4 The calibration blank is prepared by diluting 2 ml of (1:1) HN03 and 10 ml of (1:1) HCl to 100 ml with water. Prepare a sufficient quantity to flush the system between standards and samples.

11.2 Check Sample

11.2.1 The check sample,quality control reference sample concentrate should be prepared from an independent standard at a concentration other than that used for calibration, but within the calibration range. A check sample is intended as an independent check of technique, methodology, and standards and should be run with every analytical batch or every 20 samples, whichever is greater. This is applicable to all organic and inorganic analyses.

11.3 Matrix Spike

11.3.1 When calculating the percent recovery of the matrix spike and matrix spike duplicate optional dilutions must be taken into account. Samples requiring optional dilutions and chosen as the matrix spike/ matrix spike duplicate samples, must be analyzed at the same dilution as the original unspiked sample. A matrix spike and matrix spike duplicate should be brought through the whole sample preparation process and subjected to exactly the same analytical procedures as those used on actual samples. A matrix spiked sample shall be analyzed with every analytical batch or every 20 samples, whichever is greater. The sample to be spiked should be typical or representative of the batch. Ideally it should be an intermediate between the cleanest and the most contaminated samples based on the best information available. It is recommended that the spike be made in a replicate of the field duplicate samples. This is applicable to all organic or inorganic chemical analyses.

11.3.2 Analyze one replicate sample for every 20 samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process.

11.3.2.1 The relative percent difference between replicate determinations is to be calculated as follows:

Equation 4.

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100$$

RPD = relative percent difference.
D1 = first sample value.
D2 = second sample value (replicate).

(A control limit of + 20% for RPD shall be used for sample values greater than 10 times the instrument detection limit.)

11.3.2.2 The spiked replicate sample recovery is to be within + 20% of the actual value.

12. Practical Quantitation Limits

TABLE 4

Element	Symbol	CAS #	Quantitation Limits water(µg/L)	soil(mg/kg)
Aluminum	Al	7429-90-5	100	5
Antimony	Sb	7440-36-0	200	10
Arsenic	As	7440-38-2	200	10
Barium	Ba	7440-39-3	10	0.5
Beryllium	Be	7440-41-7	5	0.25
Boron	B	7440-42-8	100	5
Bismuth	Bi	7440-69-9	500	25
Cadmium	Cd	7440-43-9	10	0.5
Calcium	Ca	7440-70-2	10	0.5
Chromium	Cr	7440-47-3	20	1
Cobalt	Co	7440-48-4	50	2.5
Copper	Cu	7440-50-8	10	0.5
Iron	Fe	7439-89-6	50	2.5
Lead	Pb	7439-92-1	50	2.5
Lithium	Li	7439-93-2	100	5
Magnesium	Mg	7439-95-4	10	0.5
Manganese	Mn	7439-96-5	10	0.5
Molybdenum	Mo	7439-98-7	500	25
Nickel	Ni	7440-02-0	40	2
Phosphorus	P	7723-14-0	1000	50
Potassium	K	7440-09-7	500	25
Selenium	Se	7782-49-2	500	25
Silicon	Si	7440-21-2	1000	50
Silver	Ag	7440-22-4	10	0.5
Sodium	Na	7440-23-5	10	0.5
Strontium	Sr	7440-24-6	10	0.5
Thallium	Tl	7440-28-0	100	5
Tin	Sn	7440-31-5	1000	50
Titanium	Ti	7440-32-6	10	0.5
Tungsten	W	7440-33-7	500	25
Vanadium	V	7440-62-2	50	2.5
Zinc	Zn	7440-66-6	10	0.5

13. Target Calibration

13.1 Working Linear Ranges

TABLE 5

Element	Symbol	Working Linear Ranges (mg/L) daily cal	quarterly calibration (maximum)
Aluminum	Al	1 - 10	500,000
Antimony	Sb	1 - 10	100,000
Arsenic	As	1 - 10	500,000
Barium	Ba	1 - 10	50,000
Beryllium	Be	1 - 10	50,000
Boron	B		50,000
Bismuth	Bi		50,000
Cadmium	Cd	1 - 10	100,000
Calcium	Ca	10 - 100	500,000
Chromium	Cr	1 - 10	100,000
Cobalt	Co	1 - 10	100,000
Copper	Cu	1 - 10	100,000
Iron	Fe	1 - 10	500,000
Lead	Pb	1 - 10	500,000
Lithium	Li		
Magnesium	Mg	10 - 100	500,000
Manganese	Mn	1 - 10	500,000
Molybdenum	Mo		
Nickel	Ni	1 - 10	100,000
Phosphorus	P		
Potassium	K	10 - 100	500,000
Selenium	Se	1 - 10	500,000
Silicon	Si		100,000
Silver	Ag	0.1 - 2	2,300
Sodium	Na	10 - 100	500,000
Strontium	Sr		
Thallium	Tl	1 - 10	500,000
Tin	Sn		100,000
Titanium	Ti		50,000
Tungsten	W		100,000
Vanadium	V	1 - 10	100,000
Zinc	Zn	1 - 10	100,000

14. Qualitative Identification Information

14.1 Wavelengths

TABLE 6

Element	Wavelengths (nm)	
	Primary	Secondary
Aluminum	308.215	396.152
Antimony	206.833	217.581
Arsenic	193.696	189.042
Barium	455.403	493.409
Beryllium	234.86	313.04
Bismuth	223.061	306.772
Boron	249.773	208.959
Cadmium	214.438	226.50
Calcium	317.933	393.366
Chromium	267.716	205.552
Cobalt	228.616	238.892
Copper	324.754	224.700
Iron	259.940	238.204
Lead	220.353	216.999
Lithium	670.784	610.781
Magnesium	279.079	279.553
Manganese	257.610	259.610
Molybdenum	202.030	203.844
Nickel	231.604	232.003
Phosphorus	213.618	214.914
Potassium	766.491	769.896
Selenium	196.026	203.985
Silicon	251.611	212.412
Silver	328.068	338.289
Sodium	589.50	588.995
Strontium	407.771	421.552
Thallium	190.864	351.924
Tin	189.989	235.484
Titanium	334.941	336.121
Tungsten	207.911	224.875
Vanadium	310.20	292.402
Zinc	213.856	202.548

15. Recovery Limits
15.1 Check Sample

TABLE 7.

Element	% Recovery Limits	
	Water	Solid
Aluminum	80 - 121	NA
Antimony	69 -121	NA
Arsenic	77 - 125	NA
Barium	85 -108	NA
Beryllium	83 -112	NA
Bismuth		
Boron		
Cadmium	83 -112	NA
Calcium	86 - 118	NA
Chromium	77 - 117	NA
Cobalt	84 - 110	NA
Copper	82 - 110	NA
Iron	77 - 117	NA
Lead	78 - 115	NA
Lithium		
Magnesium	84 - 110	NA
Manganese	84 - 109	NA
Molybdenum	78 - 108	NA
Nickel	82 - 108	NA
Phosphorus		
Potassium	76 - 124	NA
Selenium	69 - 119	NA
Silicon	0 - 999	NA
Silver	68 - 117	NA
Sodium	63 - 148	NA
Strontium	68 - 106	NA
Thallium	75 - 114	NA
Tin	55 - 137	NA
Titanium		
Tungsten	73 - 108	NA
Vanadium	82 - 106	NA
Zinc	81 - 114	NA

15.2 Matrix Spike

TABLE 8.

Element	% Recovery Limits Water	Solid
Aluminum	70 - 129	55 - 143
Antimony	43 - 122	43 - 122
Arsenic	60 - 137	60 - 137
Barium	72 - 112	63 - 122
Beryllium	75 - 108	67 - 116
Bismuth		
Boron		
Cadmium	74 - 108	66 - 116
Calcium	73 - 121	61 - 133
Chromium	74 - 110	65 - 118
Cobalt	73 - 106	64 - 115
Copper	74 - 106	67 - 114
Iron	71 - 113	60 - 124
Lead	72 - 113	61 - 123
Lithium		
Magnesium	65 - 129	65 - 129
Manganese	72 - 112	62 - 122
Molybdenum	73 - 118	62 - 129
Nickel	72 - 106	64 - 115
Phosphorus		
Potassium	70 - 127	56 - 141
Selenium	45 - 106	45 - 106
Silicon	83 - 112	76 - 120
Silver	67 - 110	67 - 110
Sodium	65 - 123	50 - 137
Strontium	82 - 113	74 - 121
Thallium	67 - 109	67 - 109
Tin	65 - 128	65 - 128
Titanium	86 - 108	80 - 114
Tungsten	72 - 115	62 - 126
Vanadium	75 - 109	67 - 118
Zinc	74 - 107	65 - 116

16. Waste Disposal

16.1 All wastes generated from the use of this method (nebulizer waste, leftover digested sample and expired standards) should be flushed down the drain with large amounts of water. The laboratory neutralization tank will take care of the acids contained in this waste.

17. Trouble Shooting Guide

17.1 Sample introduction is the most troublesome part of an inductively coupled plasma optical emission spectroscopy analysis. Sample precision is highly dependent on sample transport to the plasma.

17.1.1 Check pumb tubing daily; replace if worn or stretched. It should be replaced at least once a week.

17.1.2 Make sure all solutions are matrix matched, including the rinse water.

17.1.3 For sample introduction, the use of tygon tubing should be minimized. Metals tend to adhere to the inside tube walls and lead to carryover problems. Polyethylene and Teflon are more preferable tube materials.

17.1.4 The nebulizer should be clean at all times. To avoid clogging the nebulizer, all samples high in dissolved solids should be filtered.

17.1.5 The sample injector tube (the center part of the plasma torch) should be clean at all times. If not remove, disassemble and clean the torch.

18. References and associated SOPs

19.1 SW-846 method 6010.

ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY
METHOD 3010

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

1.2 Samples prepared by Method 3010 may be analyzed by FLAA or ICP for the following:

Aluminum	Magnesium
*Arsenic	Manganese
Barium	Molybdenum
Beryllium	Nickel
Cadmium	Potassium
Calcium	*Selenium
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

* Analysis by ICP

NOTE: See Method 7760 for FLAA preparation for Silver.

1.3 This digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interferences during furnace atomization.

2.0 SUMMARY OF METHOD

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

3.0 INTERFERENCES

3.1 Interferences are discussed in the referring analytical method.

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers: 150-mL.
4.2 Watch glasses: Ribbed and plain.
4.3 Quantitative filter paper or centrifugation equipment.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Nitric acid (concentrated), HNO_3. Acid should be analyzed to determine levels of impurities. If method blank is < MDL, the acid can be used.

5.4 Hydrochloric acid (1:1), HCl. Prepared from water and hydrochloric acid. Hydrochloric acid should be analyzed to determine level of impurities. If method blank is < MDL, the acid can be used.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and water. Plastic and glass containers are both suitable. See Chapter Three, Step 3.1.3, for further information.

6.3 Aqueous wastewaters must be acidified to a pH of < 2 with HNO_3.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a 150-mL Griffin beaker and add 3 mL of concentrated HNO_3. Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and cautiously evaporate to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 3-mL portion of concentrated HNO_3. Cover the beaker with a nonribbed watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and reprepare.

7.2 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, uncover the beaker or use a ribbed watch glass, and evaporate to a low volume (3 mL), not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add a small quantity of 1:1 HCl (10 mL/100 mL of final solution), cover the beaker, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.
7.3 Wash down the beaker walls and watch glass with water and, when necessary, filter or centrifuge the sample to remove silicates and other insoluble material that could clog the nebulizer. Filtration should be done only if there is concern that insoluble materials may clog the nebulizer. This additional step can cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and prerinsed with dilute HNO_3. Adjust to the final volume of 100 mL with water. The sample is now ready for analysis.

8.0 QUALITY CONTROL
8.1 All quality control measures described in Chapter One should be followed.
8.2 For each analytical batch of samples processed, blanks (calibration and reagent) should be carried throughout the entire sample-preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.
8.3 Replicate samples should be processed on a routine basis. A replicate sample is a sample brought through the whole sample preparation and analytical process. A replicate sample should be processed with each analytical batch or every 20 samples, whichever is greater.
8.4 Spiked samples or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.
8.5 The method of standard addition shall be used for the analysis of all EP extracts (see Method 7000, Step 8.7).

9.0 METHOD PERFORMANCE
9.1 No data provided.

10.0 REFERENCES
1. Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.
2. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY GFAA SPECTROSCOPY
METHOD 3020

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by furnace atomic absorption spectroscopy (GFAA) for the metals listed below. The procedure is used to determine the total amount of the metal in the sample.

1.2 Samples prepared by Method 3020 may be analyzed by GFAA for the following metals:

Beryllium	Lead
Cadmium	Molybdenum
Chromium	Thallium
Cobalt	Vanadium

NOTE: For the digestion and GFAA analysis of arsenic and selenium, see Methods 7060 and 7740. For the digestion and GFAA analysis of silver, see Method 7761.

2.0 SUMMARY OF METHOD

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is cooled and brought up in dilute nitric acid such that the final dilution contains 3% (v/v) nitric acid. If the sample contains suspended solids, it must be centrifuged, filtered, or allowed to settle.

3.0 INTERFERENCES

3.1 Interferences are discussed in the referring analytical method.

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers: 150-mL,

4.2 Watch glasses.

4.3 Qualitative filter paper or centrifugation equipment.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.
5.3 Nitric acid (concentrated), HNO_3. Acid should be analyzed to determine levels of impurities. If method blank is < MDL, the acid can be used.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING
6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.
6.2 All sample containers must be prewashed with detergents, acids, and water. Plastic and glass containers are both suitable. See Chapter Three, Step 3.1.3, for further information.
6.3 Aqueous wastewaters must be acidified to a pH of < 2 with HNO_3.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a 150-mL Griffin beaker and add 3 mL of concentrated HNO_3. Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and cautiously evaporate to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 3-mL portion of concentrated HNO_3. Cover the beaker with a non-ribbed watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.
7.2 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). When the digestion is complete, evaporate to a low volume (3 mL). Use a ribbed watch glass, not allowing any portion of the bottom of the beaker to go dry. Remove the beaker and add approximately 10 mL of water, mix, and continue warming the beaker for 10 to 15 minutes to allow additional solubilization of any residue to occur.
7.3 Remove the beaker from the hot plate and wash down the beaker walls and watch glass with water. When necessary, filter or centrifuge the sample to remove silicates and other insoluble material that may interfere with injecting the sample into the graphite atomizer. (This additional step can cause sample contamination unless the filter and filtering apparatus are

thoroughly cleaned and prerinsed with dilute HNO_3.) Adjust to the final volume of 100 mL with water. The sample is now ready for analysis.

8.0 QUALITY CONTROL

8.1 All quality control measures described in Chapter One should be followed.

8.2 For each group of samples processed, preparation blanks (water and reagent) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

8.3 Replicate samples should be processed on a routine basis. Replicate samples will be used to determine precision. The sample load will dictate the frequency, but 20% is recommended.

8.4 Spiked samples or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

8.5 The concentration of all calibration standards should be verified against a quality control check sample obtained from an outside source.

8.6 The method of standard addition shall be used for the analysis of all EP extracts. See Method 7000, Step 8.7, for further information.

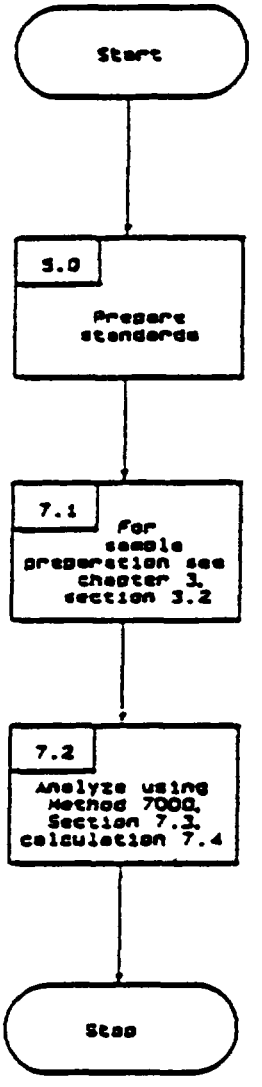
9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

1. Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

2. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.


Start
5.0
Prepare standards
7.1
For sample preparation see chapter 3, section 3.2
7.2
Analyze using Method 7000, Section 7.3, calculation 7.4
Stop

LEAD (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000.

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 See Section 3.0 of Method 7000 if interferences are suspected.

3.2 Background correction is required.

3.3 If poor recoveries are obtained, a matrix modifier may be necessary. Add 10 uL of phosphoric acid (Paragraph 5.3) to 1 mL of prepared sample in the furnace sampler cup and mix well.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000.

4.2 Instrument parameters (general):

4.2.1 Drying time and temp: 30°sec at 125°C.
4.2.2 Ashing time and temp: 30°sec at 500°C.
4.2.3 Atomizing time and temp: 10 sec at 2700°C.
4.2.4 Purge gas: Argon.
4.2.5 Wavelength: 283.3 nm.
4.2.6 Background correction: Required.
4.2.7 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE: The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-uL injection, continuous-flow purge gas, and nonpyrolytic graphite. Smaller sizes of furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 Preparation of standards:

5.2.1 Stock solution: Dissolve 1.599 g of lead nitrate, $Pb(NO_3)_2$ (analytical reagent grade), in Type II water, acidify with 10 mL redistilled HNO_3, and dilute to 1 liter with Type II water. Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.

5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentrations as in the sample after processing (0.5% v/v HNO_3).

5.3 Phosphoric acid: Reagent grade.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

7.1 Sample preparation: The procedures for preparation of the sample are given in Chapter Three, Section 3.2.

7.2 See Method 7000, Paragraph 7.3, Furnace Procedure. The calculation is given in Method 7000, Paragraph 7.4.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 239.2 of Methods for Chemical Analysis of Water and Wastes.

9.2 The performance characteristics for an aqueous sample free of interferences are:

Optimum concentration range: 5-100 ug/L.
Detection limit: 1 ug/L.

9.3 The data shown in Table 1 were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

10.0 REFERENCES

1. Lead by Flameless Atomic Absorption with Phosphate Matrix Modification, Atomic Spectroscopy, 1 (1980), no. 3, pp. 80-81.

2. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Contaminated soil	3050	163, 120 mg/g
Paint primer	3050	0.55, 0.63 mg/g
Lagoon soil	3050	10.1, 10.0 ug/g
NBS SRM 1646 Estuarine sediment	3050	23.7 ug/g[a]
NBS SRM 1085 Wear metals in lubricating oil	3030	274, 298 ug/g[b]
Solvent extracted oily waste	3030	9, 18 ug/L

[a]Bias of -16% from expected.

[b]Bias of -10 and -2% from expected, respectively.

MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

2.2 Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/L.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from Type II water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before adding stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer or equivalent: Any atomic absorption unit with an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed. Instruments designed specifically for the measurement of mercury using the cold-vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

4.2 Mercury hollow cathode lamp or electrodeless discharge lamp.

4.3 Recorder: Any multirange variable-speed recorder that is compatible with the UV detection system is suitable.

4.4 Absorption cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells may be constructed from Plexiglas tubing, 1 in. O.D. x 4.5 in. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2-in. x 2-in. cards. One-in.-diameter holes are cut in the middle of each card. The cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter/min.

4.7 Aeration tubing: A straight glass frit with a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.8 Drying tube: 6-in. x 3/4-in.-diameter tube containing 20 g of magnesium perchlorate or a small reading lamp with 60-W bulb which may be used to prevent condensation of moisture inside the cell. The lamp should be positioned to shine on the absorption cell so that the air temperature in the cell is about 10°C above ambient.

4.9 The cold-vapor generator is assembled as shown in Figure 1.

4.9.1 The apparatus shown in Figure 1 is a closed system. An open system, where the mercury vapor is passed through the absorption cell only once, may be used instead of the closed system.

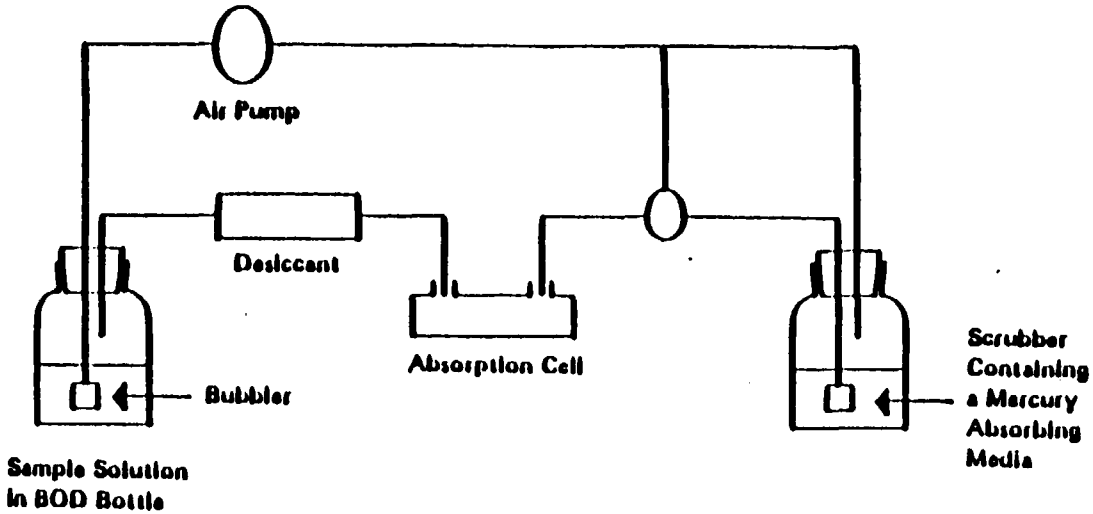


Figure 1. Apparatus for flameless mercury determination.

4.9.2 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system either to vent the mercury vapor into an exhaust hood or to pass the vapor through some absorbing medium, such as:

1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4; or
2. 0.25% Iodine in a 3% KI solution.

A specially treated charcoal that will adsorb mercury vapor is also available from Barnebey and Cheney, East 8th Avenue and North Cassidy Street, Columbus, Ohio 43219, Cat. #580-13 or #580-22.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Sulfuric acid (H_2SO_4), concentrated: Reagent grade.

5.3 Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.

5.4 Nitric acid (HNO_3), concentrated: Reagent grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

5.5 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N H_2SO_4. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

5.6 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in Type II water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

5.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of Type II water.

5.8 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of Type II water.

5.9 Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of Type II water. Add 10 mL of concentrated HNO_3 and adjust the volume to 100.0 mL (1 mL = 1 mg Hg).

5.10 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 g per mL. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask, as needed, before addition of the aliquot.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH <2 with HNO_3. The suggested maximum holding times for these samples are 38 days in glass containers and 13 days in plastic containers.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Sample preparation: Transfer 100 mL, or an aliquot diluted to 100 mL, containing <1.0 g of mercury, to a 300-mL BOD bottle. Add 5 mL of H_2SO_4 and 2.5 mL of concentrated HNO_3, mixing after each addition. Add 15 mL of potassium permanganate solution to each sample bottle. Sewage samples may require additional permanganate. Ensure that equal amounts of permanganate are added to standards and blanks. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. After a delay of at least 30 sec, add 5 mL of stannous sulfate, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.2 Standard preparation: Transfer 0-, 0.5-, 1.0-, 2.0-, 5.0-, and 10.0-mL aliquots of the mercury working standard, containing 0-1.0 ug of mercury, to a series of 300-mL BOD bottles. Add enough Type II water to each bottle to make a total volume of 100 mL. Mix thoroughly and add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to each bottle. Add 15 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized, wait 30 sec, add 5 mL of the stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.

7.3 Analysis: At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously. The absorbance will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and

continue the aeration until the absorbance returns to its minimum valve. Close the bypass valve, remove the stopper and frit from the BOD bottle, and continue the aeration.

7.4 Construct a calibration curve by plotting the absorbances of standards versus micrograms of mercury. Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

7.5 Analyze all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences by the method of standard additions.

7.6 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.7 Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 ug/g dry weight).

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process.

8.7 The method of standard additions (see Method 7000, Section 8.7) shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are available in Method 245.1 of Methods for Chemical Analysis of Water and Wastes.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 245.1.

PHENOLICS

(TRAACS 800)

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1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of Total Recoverable Phenolics in waters, liquids, solids, and sludges.

2. METHOD SUMMARY

2.1 This automated method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex.

3. SAFETY PRECAUTIONS

3.1 Standard safety practices should be followed. The analyst should wear a laboratory coat, safety glasses, and disposable gloves.

4. INTERFERENCES

4.1 Sulfur compounds interfere, but can be eliminated by the addition of phosphoric acid and copper sulfate upon sampling.

4.2 Oxidizing agents, such as chlorine, cause negative results but can be eliminated by the addition of ferrous ammonium sulfate during sampling.

5. APPARATUS AND MATERIALS

5.1 Distillation Apparatus: All glass consisting of a 1 liter Pyrex distilling flask and Graham condenser.

5.2 Glass Funnels

5.3 Volumetric Flasks: 100 ml and 500 ml

5.4 Volumetric Pipets: Various

5.5 Graduated Cylinders: Various

5.6 TRAACS 800 Auto Analyzer (See Operations Manual for requirements.)

5.7 Erlenmeyer Flasks: 125 ml

6. REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Copper Sulfate Solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ to a 1000 ml volumetric flask and dilute to volume with deionized water.

6.1.2 Phosphoric Acid Solution: Add 50 ml of conc. H_3PO_4 to 450 ml of deionized water. Store in a squirt bottle.

6.1.3 Methyl Orange Indicator: Add 0.25 g of methyl orange to 500 ml of deionized water and mix.

6.1.4 Sodium Hydroxide, 1 N: Add 40 g of NaOH to 1000 ml of deionized water, mix well, and store in a squirt bottle.

6.1.5 Triton Solution, 50/50: Add 1 ml of 50% triton (1/2 triton X-100 and 1/2 isopropyl alcohol) to 1000 ml of deionized water and mix.

6.1.6 Triton Solution, 3 ml: Add 3 ml of triton X-100 to 1000 ml of deionized water and mix well.

6.1.7 4-Aminoantipyrine Solution: Add 0.23 g of 4AAP to a 100 ml volumetric flask, dilute to volume with deionized water, and mix.

6.1.8 Potassium Ferricyanide Buffer: Add 4.83 g of ammonium chloride (NH_4Cl) to a 1000 ml volumetric flask containing 500 ml of deionized water. Mix and add 40.7 ml of ammonium hydroxide (NH_4OH). Add 5.50 g of potassium ferricyanide ($K_3Fe(CN)_6$) and mix until dissolved. Bring to volume with deionized water, transfer to an amber storage container, and add 6 ml of triton X-100 50%.

6.2 Standards

6.2.1 Internal Standard

6.2.1.1 Phenol Standard, 1000 mg/l (Purchased): From the 1000 mg/l standard, prepare the following standards diluting to volume with deionized water.

Table 1 Standards

Conc. of Standard (mg/l)	ml of Standard	Conc. of Standard Used (mg/l)	Final Volume (ml)
10	5	1000	500
1.0	10	10	100
0.5[1]	5	10	100
0.25[1]	25	1	100
0.1[1]	10	1	100
0.05[1]	10	0.5	100
0.025[1]	10	0.25	100

[1]Denotes Calibration Standard

6.2.2 Matrix Spike Standard

6.2.2.1 See Section 6.2.1 for preparation of the 1.0 mg/l standard.

7. SAMPLE STORAGE

7.1 Water samples are preserved with H_2SO_4 and $CuSO_4$ to a pH <2. Non-waters are unpreserved. All matrices are stored in glass containers at 4°C.

8. HOLDING TIMES

8.1 The holding time is twenty-eight days from sampling to analysis.

9. TUNING AND CALIBRATION

9.1 Summary

9.1.1 The TRAACS 800 Auto Analyzer is calibrated using five standards at the time of sample analysis.

9.2 Initial Calibration

9.2.1 Calibrate the instrument according to the manufacturer's instructions using five calibration standards. See Section 6.2.1 for standard concentrations and preparation.

9.3 Continuing Calibration

9.3.1 The distilled check sample (0.05 mg/l) is analyzed at the beginning, every ten samples, and at the end of the run to insure continued linearity.

10. SAMPLE PREPARATION

10.1 Summary

10.1.1 An aliquot of sample is distilled and the distillate is collected quantitatively.

10.2 Sample Preparation Procedure

10.2.1 Waters

10.2.1.1 Transfer 500 ml of sample to the distilling flask and add 5 - 10 boiling beads. At this time, add any standards or spikes required.

10.2.1.2 Add 5 ml of copper sulfate solution and 1 squirt of methyl orange. The solution should turn pink, if not, add phosphoric acid solution until a pink color develops.

10.2.1.3 Close the vessel and place a 500 ml volumetric flask under the condenser.

10.2.1.4 Begin distilling until the volumetric is half to three quarters full. Allow the sample to cool. Add 200 - 250 ml of deionized water and continue distilling until the volumetric reaches volume.

10.2.1.5 Stopper the volumetric and label.

10.2.2 Non-Waters

10.2.2.1 Transfer 5 g of sample to the distilling flask and add 250 - 300 ml of deionized water. At this time, add any required standards or spikes.

10.2.2.2 Add 5 - 10 boiling beads, 5 ml of copper sulfate solution and 1 squirt of methyl orange. The solution should turn pink, if not, add phosphoric acid solution until a pink color develops.

10.2.2.3 Place a 100 ml volumetric flask under the condenser and begin distilling until the volume is reached on the volumetric.

10.2.2.4 Stopper the volumetric and label.

10.3 Preparation Documentation

10.3.1 Record all the data from distillation in the Phenol Distillation Logbook including sample volume, spike used, and final volume of distillate.

11. SAMPLE ANALYSIS

11.1 Summary

11.1.1 The distillate is analyzed for phenol on the TRAACS 800 Auto Analyzer.

11.2 Recommended Instrument Conditions

11.2.1 An instrument base and gain should be performed prior to running analysis. See the manufacturer's instructions for the procedure.

11.3 Sample Analysis Procedure

11.3.1 Set up and run the instrument according to the manufacturer's instructions. The phenol program is set up to analyze five calibration standards with verification at the beginning and end of the run. It is recommended that a blank and check sample are

analyzed every ten samples to insure continued linearity throughout the run.

11.3.2 Preliminary Evaluation

11.3.2.1 A phenol screen can be performed prior to analysis using the reagents from the Phenol - Manual Spectrophotometer. Add a few drops of each reagent (in order) and if a red or red/brown color develops, phenol is present. Make the appropriate dilutions with deionized water prior to analyzing.

11.3.3 Confirmation

11.3.3.1 When the respective phenol run is completed, the calibration curve must be retrieved to verify calibration linearity. A non-linear curve suggests that the standards or reagents are bad and that the data should not be used.

11.3.4 Calculations

Waters

Phenol, mg/l = Instrument Value

Non-Waters

Phenol, mg/kg = Instrument Value x 20

11.4 Report Documentation

11.4.1 All distillation data and instrument data must be recorded in the Auto Analyzer Logbook.

11.4.2 All hard copies, including the calibration curve, should be filed and stored indefinitely.

12. QA/QC REQUIREMENTS

12.1 Method Blank

12.1.1 A distilled blank is required per batch of twenty samples using 500 ml of deionized water.

12.2 Check Sample

12.2.1 A distilled check sample is required per batch of twenty samples using 450 ml of deionized water and 25 ml of a 1.0 mg/l standard.

12.3 Matrix Spike

12.3.1 A distilled matrix spike/matrix spike duplicate is required per batch of twenty samples.

12.3.1.1 For waters, use 50 ml of sample, 25 ml of 1.0 mg/l standard, and 400 ml of deionized water into 500 ml.

12.3.1.2 For non-waters, use 5 g of sample, 5 ml of 1.0 mg/l standard, and 300 ml of deionized water into 100 ml.

13. PRACTICAL QUANTITATION LIMITS

13.1 The lower detection limit for waters is 0.01 mg/l and for non-waters is 0.2 mg/kg.

14. TARGET CALIBRATION

14.1 Working Linear Range

14.1.1 The phenol working range for waters is 0.01 - 0.5 mg/l and for non-waters is 0.2 - 10 mg/kg.

15. QUALITATIVE IDENTIFICATION INFORMATION

15.1 Wavelengths

15.1.1 The wavelength used is 510 nm.

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Refer to laboratory-specific check sample recovery limits.

16.2 Matrix Spike

16.2.1 Refer to laboratory-specific matrix spike recovery limits.

17. WASTE DISPOSAL

17.1 Most of the distillation flask contents and distillates can be rinsed down the drain with large amounts of water.

17.2 Excess solid wastes should be disposed of in the solid waste container.

18. TROUBLESHOOTING GUIDE

18.1 Consult the TRAACS 800 Operations Manual for any instrument problems and routine maintenance.

18.2 Oil and particulate in the distillate will interfere. Filter or remove such interferences from an aliquot of distillate prior to analyzing.

19. GLOSSARY OF TERMS - Not Applicable

20. REFERENCES AND ASSOCIATED SOPS

20.1 SW846, Test Methods for Evaluating Solid Wastes, Method 9065 and 9066 for Phenolics

20.2 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 420.1 and 420.2 for Phenolics

PHENOLICS

(MANUAL SPECTROPHOTOMETER)

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Table 1 Calibration Standards 4

1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of Total Recoverable Phenolics in drinking, surface, and saline waters, solid and liquid, domestic, and industrial wastes.

2. METHOD SUMMARY

2.1 Phenolic distillates react with 4-aminoantipyrine at pH 10.0 (±0.2) in the presence of potassium ferricyanide to form a colored antipyrine dye. The amount of color produces is a function of the concentration of phenolic material.

3. SAFETY PRECAUTIONS

3.1 Standard safety practices should be followed. The analyst should wear a laboratory coat, safety glasses, and disposable gloves.

4. INTERFERENCES

4.1 Oxidizing agents, such as chlorine, cause negative results but can be eliminated by the addition of ferrous ammonium sulfate during sampling.

5. APPARATUS AND MATERIALS

5.1 Distillation Apparatus: All glass consisting of a 1 liter Pyrex distilling flask and Graham condenser.

5.2 Graduated Cylinders: 250 ml

5.3 Spectrophotometer: for use at 510 nm

5.4 Flasks: 100 ml and 500 ml, Volumetric

5.5 Pipette: Graduated and Volumetric

5.6 Cells: 1 cm

5.7 Boiling Beads: Glass

6. REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Phosphoric Acid Solution: Add 10 ml of 85% H_3PO_4 to 100 ml of deionized water.

6.1.2 Copper Sulfate Solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in deionized water and dilute to 1000 ml.

6.1.3 Buffer Solution: Dissolve 16.9 g ammonium chloride (NH_4Cl) in 143 ml conc. NH_4OH and dilute to 250 ml with deionized water. Two ml should adjust 100 ml of distillate to pH 10.

6.1.4 4-Aminoantipyrine Solution: Dissolve 2 g of 4AAP in deionized water and dilute to 100 ml. (Should be made within twenty-four hours of analysis).

6.1.5 Methyl Orange Indicator: Dissolve 0.25 g methyl orange in 500 ml of deionized water.

6.1.6 Potassium Ferricyanide Solution: Dissolve 8 g of $K_3Fe(CN)_6$ in deionized water and dilute to 100 ml. (Should be made at least four hours before analysis).

6.2 Standards

6.2.1 Calibration Standard

6.2.1.1 Stock Standard (1000 mg/l): Dissolve 1000 g of phenol in deionized water and dilute to 1000 ml. Store in a refrigerator.

6.2.1.2 Intermediate Standard (10 mg/l): Dilute 10 ml of stock phenol standard to 1000 ml with deionized water. Make up fresh daily.

6.2.1.3 Working Calibration Standards: Prepare the following standards in 100 ml volumetric flasks.

Table 1 Calibration Standards

Volume of 10 mg/l Standard (ml)	Concentration (mg/l)
0	0
0.5	0.05
1.0	0.10
2.0	0.20
5.0	0.50
10.0	1.00

6.2.2 Internal Standard

6.2.2.1 Prepare a 1.0 mg/l standard as in 6.2.1.3 to use as a check standard.

6.2.3 Matrix Spike Standard

6.2.3.1 Prepare a 1.0 mg/l standard as in 6.2.1.3 to use as a MS/MSD standard.

7. SAMPLE STORAGE

7.1 Samples are preserved to a pH of <2 with sulfuric or phosphoric acid and stored in glass (only) containers at 4°C. The addition of copper sulfate helps minimize biodegradation.

8. HOLDING TIMES

8.1 The holding time is twenty-eight days from sampling to analysis.

9. TUNING AND CALIBRATION

9.1 Summary

9.1.1 The spectrophotometer is set at a wavelength of 510 nm and calibrated to read absorbance or concentration of the distillates.

9.2 Initial Calibration

9.2.1 Calibrate with the applicable standards for the desired curve (see Section 6.2.1 for calibration standards).

9.3 Continuing Calibration

9.3.1 Every ten samples the calibration curve must be verified with a midrange standard (0.1 mg/l).

10. SAMPLE PREPARATION

10.1 Summary

10.1.1 A known volume of sample (500 ml or 5 - 20 g) is distilled under acidic conditions and condensed to a final volume of 100 - 500 ml in a volumetric flask.

10.2 Sample Preparation Procedure

10.2.1 For waters, pour 500 ml of the sample into the 1000 ml distilling flask.

10.2.2 For solids (and non-waters), add 5 - 20 g of the sample and 200 - 350 ml deionized water to the distilling flask.

10.2.3 To each flask, add 5 - 7 boiling beads, 5 ml $CuSO_4$ solution, 5 - 10 drops of methyl orange indicator, and H_3PO_4 solution until the sample is acidic (turns a pink-red color).

10.2.4 Place a volumetric flask (500 ml for waters, 100 ml for 5 g of solid and 250 ml for 20 g of solid) under the condenser.

10.2.5 Distill the sample into volumetric flasks. For solids, distill to volume in the 100 and 250 ml flasks. For waters, distill until the volumetric flask is half to three-quarters full. Cool distilling flask, add approximately 200 ml deionized water, and continue distilling into the 500 ml volumetric flasks until the volume is reached.

10.2.6 Cap volumetric flasks and analyze as soon as possible.

10.3 Preparation Documentation

10.3.1 Record all the data from distillation in the Phenol Distillation Logbook.

11. SAMPLE ANALYSIS

11.1 Summary

11.1.1 After the sample has been distilled, the distillate is read against the calibration curve on the spectrophotometer at a wavelength of 510 nm using a standard 1 cm cell.

11.2 Recommended Instrument Conditions

11.2.1 Allow the spectrophotometer to warm up sufficiently prior to calibration.

11.3 Sample Analysis Procedure

11.3.1 To 100 ml of distillate or an aliquot diluted to 100 ml and all calibration standards, add 2 ml of buffer solution and mix. Add 2 ml aminoantipyrine solution and mix. Add 2 ml potassium ferricyanide solution and mix. After fifteen minutes, analyze using absorbance or concentration at 510 nm.

11.3.2 Preliminary Evaluation

11.3.2.1 A quick spot test can be performed to screen for possible high samples. Add 1 squirt of each reagent in Step 11.3.1 in the same order to a small amount of distillate and look for a pink to red to blood red color. Color intensity helps with dilution making decisions.

11.3.3 Confirmation

11.3.3.1 A midrange calibration standard is analyzed every ten samples to insure linearity of the calibration curve.

11.3.4 Calculation

11.3.4.1 If the spectrophotometer is calibrated in the concentration, the result is read directly off of the instrument (multiplied times the dilution). If the spectrophotometer reads absorbance, prepare a linear regression curve (abs. vs. conc.) to determine the concentration, multiply any dilutions by the concentration.

11.4 Report Documentation

11.4.1 All data must be recorded in the Phenolics Logbook.

12. QA/QC REQUIREMENTS

12.1 Method Blank

12.1.1 A blank is prepared and analyzed per batch of twenty samples using deionized water.

12.2 Check Sample

12.2.1 A check sample is prepared and analyzed per batch of twenty samples using a midrange calibration standard (0.1 mg/l).

12.3 Matrix Spike

12.3.1 A one in ten sample matrix spike/matrix spike duplicate is required using a known amount of a midrange spike (0.05 - 0.1 mg/l).

13. PRACTICAL QUANTITATION LIMITS

13.1 The lower detection limit is 0.01 - 0.05 mg/l or 0.1 - 0.2 mg/kg (depending on volumes and instruments used).

14. TARGET CALIBRATION

14.1 Working Linear Range

14.1.1 The phenol working range is 0.01 - 0.5 or 0.05 - 1.0 mg/l (0.2 - 10 or 0.1 - 5 mg/kg) depending on volumes and instruments used.

15. QUALITATIVE IDENTIFICATION INFORMATION

15.1 Wavelengths

15.1.1 The wavelength used is 510 nm.

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Refer to laboratory-specific check sample recovery limits.

16.2 Matrix Spike

16.2.1 Refer to laboratory-specific matrix spike recovery limits.

17. WASTE DISPOSAL

17.1 All distilled samples and distillates can be rinsed down the drain with large amounts of water.

18. TROUBLESHOOTING GUIDE

18.1 Foaming during distillation can be prevented by adding a few drops of silicon emulsion defoamer prior to distillation.

19. GLOSSARY OF TERMS - Not Applicable

20. REFERENCES AND ASSOCIATED SOPS

20.1 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 420.1

20.2 Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, Method 510 through 510C.

20.3 SW846, Test Methods for Evaluating Solid Wastes, Method 9065.

SULFATE

(TRAACS 800)

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1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of sulfate in surface and drinking waters and domestic and industrial wastes.

2. METHOD SUMMARY

2.1 The sample passes through a cation exchange column to remove multivalent metal ions and then is reacted with an alcohol solution of barium chloride and methylthymol blue (MTB) to form barium sulfate which is a blue color. The amount of sulfate present is measured spectrophotometrically.

3. SAFETY PRECAUTIONS

3.1 Standard safety practices should be followed. The analyst should wear a laboratory coat, safety glasses, and disposable gloves.

4. INTERFERENCES

4.1 High concentrations of calcium and other multivalent cations may interfere with the ion exchange capacity of the column. These samples should be analyzed using the turbidimetric method.

4.2 Turbid samples should be filtered.

4.3 Samples with a pH <2 should be neutralized prior to analyzing.

5. APPARATUS AND MATERIALS

5.1 TRAACS 800 Auto Analyzer (See Operations Manual for requirements.)

5.2 Erlenmeyer Flasks: 125 ml

6. REAGENTS AND STANDARDS

6.1 Reagents

6.1.1 Triton Solution, 50/50: Add 10 ml of isopropyl alcohol to 10 ml of Triton X-100. Store in an eyedropper bottle.

6.1.2 Triton Solution, 50/50: Add 1 ml of Triton 50/50 to 1000 ml of deionized water.

6.1.3 Stock Barium Chloride Solution: Add 0.80 g of $BaCl_2 \cdot 2H_2O$ to 400 ml of deionized water in a 500 ml volumetric flask. Dilute to volume with deionized water and mix well.

6.1.4 Stock Hydrochloric Acid, 0.25 N: Add 21 ml of concentration HCl to 900 ml of deionized water in a 1000 ml volumetric flask. Bring to volume with deionized water and mix well.

6.1.5 Methylthymol Blue (MTB) Solution: Add 0.036 g of Methylthymol Blue, 6.0 ml of stock barium chloride solution, 10.0 ml of 0.25 N HCl solution, and 34 ml of deionized water to a 250 ml volumetric flask. Dilute to volume using isopropyl alcohol. Mix thoroughly and deaerate. Prepare fresh daily.

6.1.6 Sodium Hydroxide Solution, 0.4 N: Add 13 g of NaOH pellets to a 1000 ml volumetric flask and dilute to volume using deionized water.

6.1.7 Working Sodium Hydroxide Solution, 0.06 N: Add 150 ml of 0.4 N NaOH solution to 400 ml of isopropyl alcohol in a 1000 ml volumetric flask and dilute to volume with deionized water.

6.1.8 Diluent: Add 0.10 g sodium chloride (NaCl) to a 1000 ml volumetric flask and dilute to volume. Mix and add 3 ml of Triton 50/50.

6.1.9 System Wash Solution: Add 50 g Tetrasodium EDTA to 600 ml of deionized water in a 1000 ml volumetric flask. Dilute to volume with deionized water and mix well.

6.2 Standards

6.2.1 Target Calibration Standards

6.2.1.1 Prepare the following standards from a 1000 mg/l sulfate standard using volumetric pipets and diluting all volumes to 100 ml in volumetric flasks.

Table 1 Standards

Final Conc. Std. (mg/l)	ml of Std. Used	Conc. Std. Used (mg/l)	Final Volume
100	10	1000	100
50	5	1000	100
25	25	100	100
10	10	100	100
5	5	100	100

6.2.2 Check Standard

6.2.2.1 For an check sample, prepare a 50 mg/l standard (as in Section 6.2.1.1).

6.2.3 Matrix Spike Standard

6.2.3.1 Prepare a 50 mg/l standard as in Section 6.2.1.1.

7. SAMPLE STORAGE

7.1 The samples are unpreserved and stored in plastic or glass containers at 4°C.

8. HOLDING TIMES

8.1 The holding time is twenty-eight days from sampling to analysis.

9. TUNING AND CALIBRATION

9.1 Summary

9.1.1 The TRAACS 800 Auto Analyzer is calibrated at the beginning of each run using five calibration standards.

9.2 Initial Calibration

9.2.1 Calibrate the instrument according to the manufacturer's instructions using 100, 50, 25, 10, and 5 mg/l standards.

9.3 Continuing Calibration

9.3.1 The instrument linearity is checked at the beginning of each run, every ten samples, and at the end of each run with a midrange standard (50 mg/l standard is recommended).

10. SAMPLE PREPARATION

10.1 Summary

10.1.1 No preparation is necessary for water samples. For solid samples, a deionized water extract is necessary prior to analysis.

10.2 Sample Preparation Procedure

10.2.1 Follow the Solid Extraction for Wet Chemistry Parameters SOP for solid preparations.

10.3 Preparation Documentation

10.3.1 All preparation information for each extraction is to be recorded in the Deionized Extraction Logbook.

11. SAMPLE ANALYSIS

11.1 Summary

11.1.1 The sample is analyzed on the TRAACS 800 Auto Analyzer for sulfate using the Methylthymol Blue Method.

11.2 Recommended Instrument Conditions

11.2.1 An instrument base and gain should be performed daily prior to analysis. See the manufacturer's instructions for the procedure.

11.3 Sample Analysis Procedure

11.3.1 Set up and run the instrument according to the manufacturer's instructions. The sulfate program is set up to analyze five calibration standards with verification at the beginning and end of the run using midrange standards. A blank and a midrange check sample should be analyzed every ten samples to verify continued linearity throughout the run.

11.3.2 Confirmation

11.3.2.1 When the run is completed, the calibration curve should be retrieved and reviewed to verify calibration linearity. A non-linear curve suggests that the standards or reagents are bad and that the data should not be used.

11.3.3 Calculations

$$\textit{Sulfate, mg/l} = \textit{Instrument Value}$$

11.4 Report Documentation

11.4.1 Record all the information in the Auto Analyzer Logbook including the instrument and calculated values.

11.4.2 Record all necessary information on the printed hard copy along with the calibration curve. These copies should be filed indefinitely.

12. QA/QC REQUIREMENTS

12.1 Method Blank

12.1.1 A blank is required per batch of twenty samples using deionized water.

12.2 Check Sample

12.2.1 A midrange check sample (50 mg/l) is required per batch of twenty samples.

12.3 Matrix Spike

12.3.1 A midrange matrix spike/matrix spike duplicate is required per batch of twenty samples using equal parts of sample and a midrange standard (2.0 ml sample and 2.0 ml 50 mg/l sulfate is recommended).

13. PRACTICAL QUANTITATION LIMITS

13.1 The lower detection limit is 5 mg/l.

14. TARGET CALIBRATION

14.1 Working Linear Range

14.1.1 The working range is 5 - 100 mg/l.

15. QUALITATIVE IDENTIFICATION INFORMATION

15.1 Wavelengths

15.1.1 The required wavelength for sulfate is 460 nm.

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Refer to laboratory-specific check sample recovery limits.

16.2 Matrix Spike

16.2.1 Refer to laboratory-specific matrix spike recovery limits.

17. WASTE DISPOSAL

17.1 All wastes can be rinsed down the drain with large amounts of water.

18. TROUBLESHOOTING GUIDE

18.1 Consult the TRAACS 800 Operations Manual for any instrument problems and routine maintenance.

18.2 Consult the sulfate method (TRAACS Instrument Method) for care and use of the sulfate manifold and cation exchange column.

19. GLOSSARY OF TERMS - Not Applicable

20. REFERENCES AND ASSOCIATED SOPS

20.1 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Sulfate (Automated Methylthymol Blue) Method 375.2

CYANIDE

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1. PARAMETER

   1.1 This method is used for the determination of cyanide.

2. RANGE OF MEASUREMENT

   2.1 The working range of the method for aqueous samples is 10 to 100 µg/l.

   2.2 The working range of the method for solid samples is 0.5 mg/kg to 5 mg/kg.

3. LIMIT OF DETECTION

   3.1 The limit of detection for waters is 10 µg/l.

   3.2 The limit of detection for solids is 0.5 mg/kg.

4. SAMPLE MATRIX

   4.1 This method is applicable for the determination of total cyanide in solid, liquid, and aqueous matrices.

5. PRINCIPLE, SCOPE, AND APPLICATION

   5.1 The cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

   5.2 The sodium hydroxide solution is analyzed colorimetrically on an auto analyzer using the pyridine-barbituric acid method.

6. INTERFERENCES AND CORRECTIVE ACTIONS

   6.1 Nitrites and nitrates interfere, but are eliminated by the addition of sulfamic acid.

   6.2 Chloride interferes, but can be eliminated by the addition of ascorbic acid prior to distillation.

   6.3 Sulfides interfere, but can be eliminated by treating sodium hydroxide distillate with cadmium carbonate prior to analysis.

7. SAFETY PRECAUTIONS

   7.1 Standard safety practices should be followed. Analysts should wear a laboratory coat, safety glasses, and disposable gloves.

8. SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING

   8.1 A minimum of 1 liter of water is collected for this analysis.

   8.2 A minimum of 50 g of a solid sample is collected for this analysis.

8.3 Aqueous samples are preserved with NaOH to a pH of >12 and are stored at 4°C until the time of analysis.

8.4 Solids are not chemically preserved and are stored at 4°C until the time of analysis.

8.5 The holding time for aqueous and solid samples is fourteen days from sampling to analysis.

9. APPARATUS AND MATERIALS

9.1 Cyanide Distillation Apparatus

9.2 Bunsen Burners

9.3 Vacuum Pump

9.4 Graduated Cylinders: Various sizes

9.5 Volumetric Flasks: Various sizes

9.6 Volumetric Pipets: Various sizes

9.7 Balance: Top loading

9.8 TRAACS 800 Auto Analyzer: See manufacturer for requirements

10. ROUTINE PREVENTIVE MAINTENANCE

10.1 The manufacturer's recommendations are followed for routine preventive maintenance.

11. REAGENTS AND CALIBRATION STANDARDS

11.1 Reagents

11.1.1 Sulfamic Acid: Crystals

11.1.2 Ascorbic Acid: Crystals

11.1.3 Cadmium Carbonate: Powder

11.1.4 Sodium Hydroxide, 1.25N: Add 50 g of sodium hydroxide pellets (NaOH) to a 1000 ml volumetric flask and dilute to volume with deionized water.

11.1.5 Sulfuric Acid Solution, (1:1): Slowly add 500 ml conc. H_2SO_4 to 500 ml of deionized water. Cool and dilute to 1000 ml with deionized water.

11.1.6 Magnesium Chloride Solution: Add 510 g $MgCl_2 \cdot 6H_2O$ to a 1000 ml volumetric flask and dilute to volume with deionized water.

11.1.7 Phosphate Buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate - dibasic (Na_2HPO_4) to 800 ml of deionized water in a 1000 ml volumetric flask. Mix, bring to volume with deionized water, and add 1 ml Brig 35 Mix.

11.1.8 Chloramine-T Reagent: Add 1.0 g of chloramine-T to a 250 ml volumetric flask and dilute to volume with deionized water. Prepare fresh daily.

11.1.9 Pyridine Reagent: Add 15.0 g of barbituric acid to a 1000 ml volumetric flask. Add 75 ml of pyridine and 15 ml of conc. HCl and mix. Bring to volume with deionized water and store at 4°C in an amber glass bottle.

11.1.10 Sodium Hydroxide, 0.25 N: Add 200 ml of 1.25 N NaOH to a 1000 ml volumetric flask and dilute to volume with deionized water.

11.2 Standards

11.2.1 Cyanide Stock Standard, 1000 ppm: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 ml volumetric flask and dilute to volume with deionized water. Mix well.

11.2.2 Prepare the following working standards and bring up to volume with deionized water.

Table 1 Standards

Standard ml	Conc. Standard mg/l	1.25 NaOH ml	Final Volume	Final Conc. mg/l
10	1000	10	100	100
10	100	10	100	10
10	10	10	100	1.0
10	1	20	100	0.1[d]
5	1	20	100	0.05[d]
25	0.1	20	100	0.025[d]
10	0.1	20	100	0.01[d]
10	0.05	20	100	0.005[d]

[d]Denotes calibrating standards

11.2.3 Check and Matrix Spike Standard: Prepare a 1.0 mg/l standard as in Table 1.

12. CALIBRATION PROCEDURES

12.1 The instrument is calibrated using five cyanide standards (see Table 1) at the time of analysis.

12.2 The calibration is checked every ten samples and at the beginning and end of each run using a midrange check standard.

13. SAMPLE PREPARATION

13.1 Total Cyanide Analysis

13.1.1 Add 500 ml or 10 g of the sample to the distillation flask. Bring all volumes to 500 ml with deionized water. Add 50 ml of 1.25 N NaOH solution to the absorber tube and assemble the scrubber. Assemble the cyanide distillation apparatus.

13.1.2 Turn of the vacuum source and adjust the flow such that an even stream of air bubbles in the scrubber tube. At this time add any standard (check or MS/MSD) to the appropriate sample through the inlet tube. Rinse the inlet with deionized water. Through the inlet tube, add 1 scoop (0.5 - 1.0 g) of sulfamic acid, 50 ml of 1:1 sulfuric acid, and 20 ml magnesium chloride solution. Be sure to rinse the inlet tube with deionized water between and after reagent additions.

13.1.3 Turn on the cooling water and the bunsen burners. Heat the sample for one hour and fifteen minutes. Be sure to adjust the air flow as necessary. After the heating period, turn off the heat and allow to cool for fifteen minutes. Keep the air on.

13.1.4 Disconnect the scrubber and pour the sodium hydroxide solution into a 250 ml volumetric flask. Bring the flask up to volume with deionized water rinsings from the absorber tube. Be sure to properly label the flask.

13.2 Preparation Documentation

13.2.1 Record all the information in the Cyanide Distillation Logbook.

14. ANALYTICAL MEASUREMENT

14.1 Recommended Instrument Conditions

14.1.1 An instrument base and gain must be performed each day and the respective values should be on or near: Base = 75, Gain = 150. See the manufacturer's instructions on how to perform a base and gain.

14.2 Sample Analysis Procedure

14.2.1 Set up and run the instrument according to the manufacturer's specifications. The required wavelength for cyanide on the TRAACS 800 is 570 nm. The program is set up to analyze five calibration standards with verification at the beginning and the end of the run. It is recommended that a blank and a check sample are analyzed every ten samples to verify linearity throughout the run.

14.2.2 Confirmation

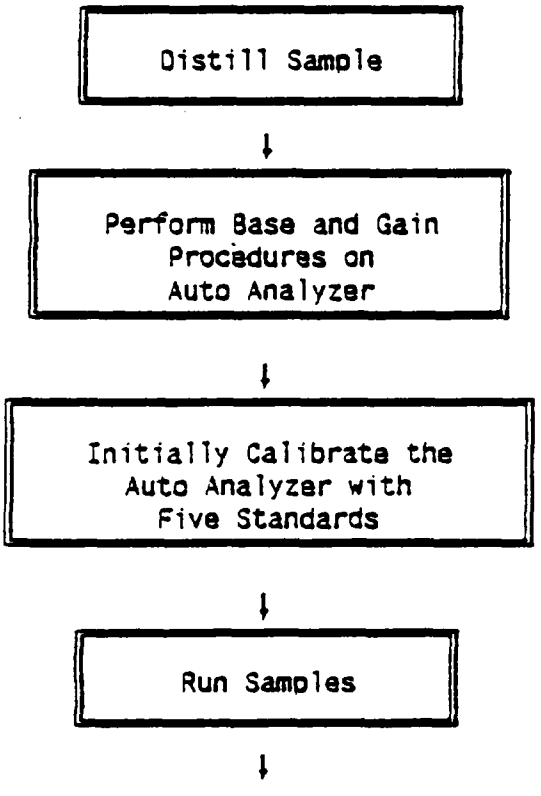
14.2.2.1 When the respective cyanide run is completed, the calibration curve must be retrieved to verify calibration linearity. A non-linear curve suggests that the standards or reagents are bad and that the data should not be used.

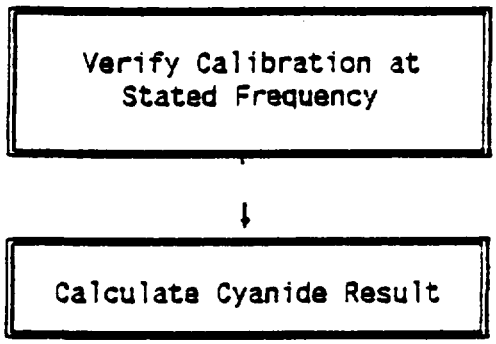
14.3 Report Documentation

14.3.1 All necessary information must be recorded on the hard copy printout and filed accordingly. This document must be kept indefinitely. Be sure a copy of the calibration curve is included.

14.3.2 All instrument data and calculated data must be recorded in the TRAACS 800 Logbook.

15. FLOWCHART OF ANALYTICAL PROCESS





16. DATA TREATMENT

16.1 Calculations

$$\text{Cyanide, mg/l} = \frac{\text{Instrument Value} \times 250}{\text{ml of sample used}}$$

$$\text{Cyanide, mg/kg} = \frac{\text{Instrument Value} \times 250}{\text{g of sample used}}$$

17. DATA DELIVERABLES

17.1 The client sample report contains the following information.

17.1.1 Case narrative briefly describing the methods used for sample analysis.

17.1.2 Sample results

17.1.3 Quality control report which contains:

17.1.3.1 Method blank analysis results

17.1.3.2 Check sample data along with laboratory control limits

17.1.3.3 Matrix spike/matrix spike duplicate data along with laboratory control limits

17.2 All associated data (logbooks, raw data, etc.) will be kept at the laboratory and will be available upon request.

18. QUALITY CONTROL REQUIREMENTS

18.1 Method Blank

18.1.1 A deionized water blank (500 ml) must be distilled and analyzed per rack (batch of sixteen).

18.2 Check Sample

18.2.1 A midrange check sample (500 ml of deionized water and 10 ml of 1.0 ppm - 0.04 ppm true value) must be distilled and analyzed per rack (batch of sixteen).

18.3 Matrix Spike

18.3.1 A matrix spike/matrix spike duplicate is required per batch of twenty samples using a midrange spike (250 ml or 10 g of sample and 10 ml of 1.0 ppm - 0.04 ppm true value) which is distilled and analyzed.

19. REFERENCES

19.1 SW846, Method 9012 A

19.2 EPA 600, Methods 335.1 and 335.2

CHLORIDE (TRAACS 800)

I. SCOPE AND APPLICATION

1. Applicable to waters and drinking waters. Solids may be analyzed following the digestion in Nitrite Comment (#3) Section, however, it is not recommended.

II. SAMPLE HANDLING

1. Unpreserved samples in plastic or glass containers. Holding time is 28 days.

III. REAGENTS AND EQUIPMENT

1. Traacs 800 (See Traacs Operation)
2. 125ml Erlenmeyer Flasks and Holed Stoppers
3. Probe .027"
4. Flowcell 10mm
5. Wavelength Cell 480nm
6. Manifold - Multitest Cartridge with Normal Tubes
7. Reagents
   a. Color Reagent (Alpkem) with 5-6 drops Brij-35 per 150ml.
   b. Chloride Standard - 1000ppm (Hach).
   c. Sulfate Standard - 1000ppm (Hach).
   d. Chloride/Sulfate Calibration Standards: Add appropriate volume of chloride standard (1000ppm) and sulfate standard (1000ppm) using volumetric pipets (or autopipettor) to 100ml volumetrics and bring to volume with DI.

Conc. Cl/SO4	mlCl	mlSO4	Final Volume
10/100ppm	10	10	100
50/50ppm	5	5	100
25/25ppm	2.5	2.5	100
10/10ppm	1.0	1.0	100
2/5ppm	.2	.5	100

NOTE: Prepare an extra 50/50 standard to use as a check standard. All standards good for 1 month.

IV. PROCEDURE

Chloride is usually analyzed as a dual channel with sulfate. Also see that Method in this Manual.

1. Specifications
   a. Code = CLSO4 (or Cl for chloride only)
   b. Number of Analysis = 2 (or 1 with chloride alone)
   c. Channel = 1
   d. Manifold = Multitest Board
   e. Probe = .027" (large - with splitter for sulfate)
   f. Flowcell = 10mm
   g. Wavelength Cell = 480nm
   h. Range = 2-100mg/l
   i. Standards:
      a. 100ppm
      b. 50ppm
      c. 25ppm
      d. 10ppm
      e. 2ppm

2. Start Up Solutions (and Wash Solution)
   a. All 5 lines go into DI water (NO wetting reagents)
3. Running Solutions
   a. GRY/GRY line (#1) in color reagent.
   b. All other lines (#2-5) in DI water.
4. Base and Gain
   a. Performed on the 100/100ppm standard.
   b. Normal Values
      1. Base = 70
      2. Gain = 20

   NOTE: These values are for reference only and are subject to change because of new reagents, standards, tubing or manifold cleaning.

5. Normal Tray Protocol
   a. P01, SC>1, H01, 2L04, 3I03, #5>6, H01, 2L04, G01, E.
6. Running Samples
   a. Follow the normal tray protocol in the Lab Procedures Section of this Manual.
   b. Dilutions may be done using DI water.
   c. Blanks are DI water analyzed directly.
   d. Check samples are the 50/50 standard analyzed directly.
   e. MS/MSD's are 2.0ml sample and 2.0ml 50/50 standard. Mix and run directly.
7. Shut Down
   a. See Start Up Section. Run for at least 5 minutes on high.
   b. Be sure to dump out used reagents, standards and wash solutions to prevent contamination.

V. CALCULATIONS

1. Chloride mg/l = inst. value (x any dilutions)
2. Check sample % recovery = $\frac{\text{inst. value}}{50 \text{ (true)}} \times 100$
3. MS/MSD % recovery = $\frac{A-B}{100 \text{ (true)}} \times 100$

   A = Inst. value MS/MSD x 4
   B = Inst. value sample x 2

4. Units = mg/l
5. LDL = <2mg/l

VI. COMMENTS

1. Method Reference is EPA 600 Method 325.2.
2. Be sure to filter out any particulates prior to analyzing.
3. Low level chloride can be run. Range is 1-10mg/l. Code is "Chloride".
4. Be sure a certified analyst analyzes drinking waters.

CHLORIDE-TITRIMETRIC

CM-1

I. SCOPE AND APPLICATION

1. Applicable to waters only.

II. SAMPLE HANDLING

1. Unpreserved samples in plastic or glass containers. Holding time is 28 days.

III. REAGENTS AND EQUIPMENT

1. 125ml Erlenmeyer Flasks
2. 50ml Graduated Cylinders
3. Disposable Pipets, 5ml Volumetric Pipet
4. Stir Plate and Stir Bars
5. 25ml or 10ml Micro Buret
6. Reagents
   a. Nitric acid solution: add 3ml conc. HNO_3 to 97ml DI water. Mix and store in a plastic container.
   b. Mixed Indicator: Transfer 0.5 grams Diphenolcarbizone and 0.05 grams Bromophenol Blue powder to a 100 ml volumetric using 95% Methanol. Dilute to the mark with Methanol. Store in a dark, glass bottle.
   c. Mecuric Nitrate Titrant (0.025N): Dissolve 4.2930g $HgNO_3$ in 50ml DI water and 0.5ml conc. HNO_3 in a 1000ml volumetric flask. Bring to volume with DI water. Mix and store in a dark bottle.
   d. 50ppm Chloride Standard: Pipet 5.0ml 1000ppm Chloride Standard into a 100ml volumetric and bring to volume with DI water.

IV. PROCEDURE

1. Pour 50ml sample or an aliquot of sample brought up to 50ml into an Erlenmeyer flask (5ml sample and 45ml DI is a 1:10).
2. Add 10 drops mixed indicator and begin stirring.
3. Add Nitric acid solution drop by drop until a yellow color develops, add a couple of drops more.
4. Fill buret with DI water and with titrant one time each. Drain and fill with titrant. Be sure to remove all air bubbles and fill to the zero mark.
5. Titrate with 0.025 Titrant until a violet color persists throughout the solution. Record amount of titrant used in Chloride Titrimetric Logbook.
6. Run a blank using 50ml DI water.
7. Run a Check Standard using 50ml of a 50ppm Chloride Standard.
8. Run MS/MSD's using 25ml sample and 25ml 50ppm Chloride Standard.

V. CALCULATIONS

1. mg/l Chloride = (ml tit. x 0.025 x 35,450) divided by ml sample

2. Check Std. % Recovery =

$$(\text{ml tit.} \times 0.025 \times 35{,}450) / \underset{(\text{true})}{2500} \times 100$$

3. MS/MSD % Recovery =

$$((\text{ml tit.} \times 0.025 \times 35{,}450) - (\text{mg/l sample} \times 25^{*})) / \underset{(\text{true})}{1250} \times 100$$

* If sample is ND, subtract 0.

4. LDL = 2 mg/l
5. Units are mg/l

VI. COMMENTS

1. Method Reference is EPA 600 Method 325.3.
2. Samples that do not turn colors upon addition of reagents need to be diluted.
3. For interferences, refer to method reference.
4. A 1mg/l LDL can be achieved by using .0141N Titrant. Refer to method reference for preparation of titrant.
5. OHIO DRINKING WATERS MUST BE ANALYZED BY A CERTIFIED ANALYST.

TOTAL DISSOLVED SOLIDS (TDS)

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1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of Total Dissolved Solids in drinking, surface, saline, domestic and industrial waters and wastes.

1.2 The normal range of the determination is 5 mg/l to 20,000 mg/l.

2. METHOD SUMMARY

2.1 The mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to a constant weight at 180°C.

2.2 If the total suspended solids is being determined, the filtrate from that method may be used to determine the total dissolved solids.

3. SAFETY PRECAUTIONS

3.1 Standard safety practices should be followed. Analysts should wear a laboratory coat, safety glasses, and disposable gloves.

4. INTERFERENCES

4.1 Mineralized waters may be hygroscopic and require extra drying time, desiccation, and rapid weighing.

4.2 Samples containing high concentrations of bicarbonate will require prolonged drying at 180°C to complete the bicarbonate to carbonate conversion.

4.3 Attempt to limit to the dried residue to 200 mg to avoid water being trapped in a thick crust of dried sample.

5. APPARATUS AND MATERIALS

5.1 Drying Oven: Capable of maintaining 180°C (± 2°C)

5.2 Desiccator(s)

5.3 Whatman Glass Fiber Filter Discs (934-AH) or Comparable Filtering Media

5.4 Analytical Balance: Capable of weighing to the nearest 0.1 mg

5.5 Graduated Cylinders: 100 ml

5.6 Vacuum Pump

5.7 Glass Beakers: 150 ml - 200 ml or similar capacity porcelain evaporating dishes

5.8 Steam Bath or Heating Mantle: Located in a hood

5.9 Vacuum Flask and Filtering Apparatus

6. REAGENTS AND STANDARDS

6.1 Total Dissolved Solids Standard

6.1.1 Transfer 0.520 g of potassium chloride to a 100 ml volumetric flask, bring to volume with deionized water and mix well.

7. SAMPLE STORAGE

7.1 Samples are to be stored at 4°C.

7.1.1 This should keep microbiological decomposition of solids to a minimum.

8. HOLDING TIMES

8.1 Sample preparation and analysis must be completed within seven days of the sampling date.

9. TUNING AND CALIBRATION - Not Applicable

10. SAMPLE PREPARATION

10.1 Summary

10.1.1 Mixed sample is filtered using a prepared disc and filtrate is taken to dryness. The dried residue is maintained at the temperature 180°C for two hours.

10.2 Sample Preparation

10.2.1 Prepare filters by placing on the filtering apparatus (rough side up) and filter 100 ml of deionized water through it. Remove the filter, place in the drying oven at 103 - 105°C for one hour, desiccate, and weigh if total suspended solids are requested.

10.2.2 Make sure the samples are at room temperature before starting the analysis.

10.2.3 Accurately weigh the beaker on the analytical balance, record the weight in the Total Dissolved Solids Logbook, and label the beaker with the sample ID.

10.2.4 Filter 100 ml of well-mixed sample through the filter (rough side up), add rinsate from graduate into the apparatus, and rinse the filtering device.

10.2.5 Take the sample to dryness either on a steam bath or in the drying oven (103 - 105°C).

10.2.6 When the sample appears to be dry, place in the drying oven at 180°C for two hours. If the drying oven was used to remove moisture, elevate the temperature on the drying oven to 180°C; maintain sample residues at this temperature for two hours.

10.2.7 Remove beaker(s) and place in desiccator(s) until cool (at least one hour).

10.2.8 Weigh the beaker with the residue on the analytical balance and record the value in the Total Dissolved Solids Logbook.

11. SAMPLE ANALYSIS

11.1 Summary

11.1.1 After the sample has been filtered, the residue dried, cooled, and weighed. The TDS is calculated.

11.2 Sample Calculation

$$\text{Total Dissolved Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:

A = Weight of dried residue and dish in mg

B = Weight of dish in mg

C = Volume of sample used in ml

11.3 Check Sample Calculation

$$\%\ \text{Recovery} = [(\text{mg/l} \times 1000) \div 52{,}000^{d}] \times 100$$

[d] *True Concentration*

12. QA/QC REQUIREMENTS

12.1 Method Blank

12.1.1 A blank of 100 ml of deionized water is analyzed per batch of twenty samples.

12.2 Check Sample

12.2.1 A check sample of 100 ml spiked with 1.0 ml of TDS standard is analyzed per batch of twenty samples.

13. PRACTICAL QUANTITATION LIMITS

13.1 The lower detection limit is 5 mg/l and the suggested upper limit is 20,000 mg/l.

14. TARGET CALIBRATION - Not Applicable

15. QUALITATIVE IDENTIFICATION INFORMATION - Not Applicable

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Refer to laboratory-specific check sample recovery limits.

17. WASTE DISPOSAL

17.1 Residues are placed in the appropriate waste containers.

18. TROUBLESHOOTING GUIDE

18.1 Check client histories whenever possible to avoid repeat analysis due to large residues (residues greater than 200 mg).

19. GLOSSARY OF TERMS

19.1 Total Dissolved Solids is also referenced as Filterable Residue.

20. REFERENCES AND ASSOCIATED SOPS

20.1 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 160.1

20.2 Standard Methods for the Examination of Water and Wastewater, Sixteenth Edition 1985, Method 209B

TOTAL SUSPENDED SOLIDS (TSS)

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1. SCOPE AND APPLICATION

1.1 This method is applicable for the determination of Total Suspended Solids in drinking, surface, saline, domestic and industrial waters and wastes.

2. METHOD SUMMARY

2.1 The well mixed sample is filtered through a standard glass fiber filter and the residue retained is quantified.

3. SAFETY PRECAUTIONS

3.1 Standard safety practices should be followed. Analysts should wear a laboratory coat, safety glasses, and disposable gloves.

4. INTERFERENCES

4.1 Samples with high total dissolved solids may interfere. Proper post-filter washing will help minimize this interference.

5. APPARATUS AND MATERIALS

5.1 Drying Oven: 103 - 105°C

5.2 Desiccator(s)

5.3 Whatman Glass Fiber Filter Discs (934-AH) or Comparable Filtering Media

5.4 Analytical Balance: Capable of weighing to the nearest 0.1 mg

5.5 Graduated Cylinders: 100 ml

5.6 Vacuum Pump

5.7 Watch Glass: 65 mm

5.8 Volumetric Pipet: 5 ml

5.9 Filtering Apparatus

5.10 Tweezers

6. REAGENTS AND STANDARDS

6.1 Standards

6.1.1 Check Standard

6.1.1.1 Add 12.50 g potassium bromide (KBr) and 1.00 g Kaolin to a 250 ml volumetric flask. Add approximately 200 ml of deionized water and place on a stirring hot plate. Stir

and warm gently for fifteen minutes. Cool and dilute to volume with deionized water. The true value is 800 mg/l.

7. SAMPLE STORAGE

7.1 Unpreserved samples are stored in plastic or glass containers at 4°C.

8. HOLDING TIMES

8.1 The holding time is seven days from sampling to analysis.

9. TUNING AND CALIBRATION - Not Applicable

10. SAMPLE PREPARATION

10.1 Summary

10.1.1 First, the filters are prepared and weighed and then a known volume of sample is filtered through each specified filter.

10.2 Filter Preparation

10.2.1 Place a new 934-AH filter on the filtering apparatus and filter 100 ml of deionized water through it. Place the filter on a clean, dry watch glass. Prepare one for each sample.

10.2.2 Place the washed filters in the drying oven for at least one hour and in a desiccator for at least one hour.

10.2.3 Weigh the filter on an analytical balance and record the initial weight in the Total Suspended Solids Logbook.

10.2.1.4 Record the associated sample number on the watch glass.

10.3 Sample Preparation

10.3.1 Shake the sample vigorously and transfer 100 ml into a graduated cylinder.

10.3.2 Place the pre-weighed filter on the filtering apparatus and apply the vacuum.

10.3.3 Pour the sample through the filter. Rinse the graduated cylinder, funnel walls, and the filter with deionized water. Total washings should be between 25 - 30 ml.

10.3.4 Place the filter back on the watch glass and put it in the drying oven at 103 - 105°C for at least one hour.

10.3.5 Place the filter in the desiccator for at least one hour.

10.4 Preparation Documentation

10.4.1 Record all necessary information in the Total Suspended Solids Logbook.

11. SAMPLE ANALYSIS

11.1 Summary

11.1.1 The glass fiber filter is weighed and the amount of the total suspended solids is calculated.

11.2 Sample Analysis Procedure

11.2.1 Weigh the filter and record the final weight in the Total Suspended Solids Logbook. Save the filter until all the calculations are performed.

11.3 Sample Calculations

$$\textit{Total Suspended Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:

A = Final weight, filter and sample

B = Initial weight, filter only

C = ml of sample filtered

11.4 Report Documentation

11.4.1 Record all the information in the Total Suspended Solids Logbook.

12. QA/QC REQUIREMENTS

12.1 Method Blank

12.1.1 A blank of 100 ml of deionized water is analyzed per batch of twenty samples.

12.2 Check Sample

12.2.1 A check sample is analyzed per batch of twenty samples using 95 ml of deionized water and 5.0 ml of 800 mg/l check standard.

13. PRACTICAL QUANTITATION LIMITS

13.1 The lower detection limit is 5 mg/l.

14. TARGET CALIBRATION

14.1 Working Linear Range

14.1.1 The working linear range is 5 - 20,000 mg/l.

15. QUALITATIVE IDENTIFICATION INFORMATION - Not Applicable

16. RECOVERY LIMITS

16.1 Check Sample

16.1.1 Refer to laboratory-specific check sample recovery limits.

17. WASTE DISPOSAL

17.1 Water wastes can be rinsed down the drain with large quantities of water.

17.2 Filters can be disposed of in the solid waste receptacle.

18. TROUBLESHOOTING GUIDE - Not Applicable

19. GLOSSARY OF TERMS - Not Applicable

20. REFERENCES AND ASSOCIATED SOPS

20.1 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 160.2

20.2 Standard Methods for the Examination of Water and Wastewater, Sixteenth Edition 1985, Method 209C

APPLIED CONSTRUCTION TECHNOLOGY, INC.

STANDARD OPERATING PROCEDURES

APPLIED CONSTRUCTION TECHNOLOGY, INC.
QUALITY ASSURANCE MANUAL

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1. INTRODUCTION

1.1 The PURPOSE of this Manual is to describe the Quality Control system of Applied Construction Technologies, Inc.

1.2 The function of the Quality Control System is to present a STANDARD whereby Independent Commercial Laboratories can evaluate and attest to their reliability in making precision measurements in testing and/or anlyzing materials, products, systems and/or performing research.

1.3 The Manual outlines and describes the procedures for establishing and maintaining the quality of analyses, research, inspection and testing within the Independent Laboratory.

The control of quality is a continuing process; consequently, this manual provides not only for the formation of a program, but for it's continuation through calibration and/or standardization systems and/or reference sample programs and/or inspection programs.

1.4 The Laboratory Manager shall establish and maintain the Quality Control System and all related forms and procedures.

1.5 Periodically, the manual will be reviewed and revised to incorporate the accumulated revisions and/or additions.

2. ORGANIZATION

2.1 Applied Construction Technologies, Inc. is an independent commercial laboratory providing the construction industry with geotechnical and materials engineering, inspection, and testing services.

The Company's facility is located at 210 Hayes Drive, Cleveland, Ohio 44131-1036. Services are rendered primarily in northeast Ohio but are not restricted to this geographical location.

Applied Construction Technologies, Inc. is incorporated in the State of Ohio. It is also registered as a professional corporation within the State of Ohio.

Applied Construction Technologies, Inc. is presently controlled by a Board of Directors representing both company management and independent shareholders of the corporation. Policies formulated at this level, as well as overall company operations, are administered by the President.

A management organizational chart is made a part of the Appendix to this manual to indicate current officers, directors, departmental supervisors, and the authoritative lines between them.

2.2 CLIENT SERVICES

Geotechnical Site Investigation
Foundation Engineering
Soils Laboratory
Specifications
Geo-Environmental Services
Failure Investigation
Pavement Design
Slpe Stability
Exploratory Borings

MATERIALS ENGINEERING

Mix Design (Concrete, Asphalt)
Asphalt Concrete Evaluation
Concrete, Brick & Block Testing
Aggregate Analysis
Mortar Testing
Roofing Evaluation
Soil Cement

INSPECTION

Earthwork Control
Structural Steel Inspection
Load Tests
Reinforcing Steel
Concrete Inspection
Batch Plant
Foundations

3. OPERATIONAL PROCEDURES

3.1 The general work flow at Applied Construction Technologies, Inc. is outlined in the Order Entry Flow Chart located in the Appendix to this manual. Portions of this flow chart are defined in the sections below for further clarification.

3.2 Work Order Preparation

All work to be performed by the laboratory, whether requiring specific analytical tests, inspection, or consultation, is noted on the "Work Order" form and/or "Laboratory Test Assignments" sheet by the party initiating the work order. This order is the client's purchase order, letter of transmittal or internally prepared work description. When feasible, the exact job requirements are reproduced in typewritten form on the work order. Too lengthy or detailed instructions are not copied but are referenced and attached thereto.

The work order number, composed of a six digit numerical code (four leading, decimal point, and two trailing), is contained in the upper right hand corner of each order and represents the primary internal reference to work in progress or work completed.

A properly prepared work order shall contain the following information:

1. Date
2. Client's Name and Address
3. Client Contact
4. Purchase Order and Shipper Number, if required
5. Referenced Test Requirements
6. Applicable Specification(s)
7. Report Distribution
8. Budget (cost estimate)
9. Billing information
10. Name of person initiating work order.

A log is maintained by the secretarial force which lists consecutively all prepared work orders with their identification numbers, client, project title and location.

3.5 Internal Document Control

Each Department Supervisor is responsible for assuring the availability of written procedures which adequately cover appropriate phases of testing, inspection, and analysis. In addition, the Supervisor is required to generate and update procedures to insure continuing compliance with governing specifications.

Internal written procedures shall include, at a minimum, a general outline of sequence of operations which will generate qualitative and/or quantitative results that satisfy purchase order requirements. These procedures may reference established and accepted specifications which describe preparation, performance, calibration, and reporting methods.

Each department maintains current revisions of government, military, prime contractor or independently produced specifications. When possible and economically feasible, continuing subscription service is maintained for acquiring latest revisions of pertinent specifications. Those specifications not acquired in this manner are ordered individually or in volumes, as necessary.

In each instance, the latest revision of the governing specification will be used unless the client specifically requests compliance with earlier revisions. Current specification indices will be used to establish the latest revision and the client will be notified of it's existence prior to testing.

3.6 Current Certifications, Applications, Qualifications and/or Approvals

1. American Council of Independent Laboratories, Inc.
2. Cement & Concrete REference Laboratory
3. Ohio Department of Transportation
4. City of Cleveland
5. Corps. of Engineers
6. State of Ohio Board of Registration for Professional Engineers and Surveyors
   a. Corporate Authorization to Practice Engineering
   b. Individual Registrations

4.0 PERSONNEL

4.1 Individual laboratory or department structure includes a Departmental Supervisor who, by education and/or practical experience, is qualified to direct, coordinate and supervise tests conducted and to assure utilization of proper procedures to achieve accurate and meaningful results. Direct accountability of the Department Supervisor concerning technical matters is to the President of the Company.

Within each Department, technical level personnel must account for their performance to the Department Supervisor. In most instances, conflicts arising from errors, omissions, or other inadequate performance by the technician are handled entirely at the departmental level.

4.2 One condition of hiring at Applied Construction Technologies, Inc. is that each new employee, regardless of technical level, undergo a month's probationary or training period. During these thirty days, a program of indoctrination or instruction is conducted by an immediate superior such that, when the time period elapses, a decision for continuing or terminating employment can be made. Results of this decision are communicated to the Board of Directors for it's review by means of an oral and/or written evaluation. When continued employment is the resulting decision, The Supervisor assumes responsibility for continued training and satisfactory job performance.

4.3 The Company strongly endorses certification of it's engineering technicians through the Institute For The Certification of Engineering Technicians. Requirements for the three grades of certification are dependent upon the number of years of experience under a professional engineer or equivalent.

4.4 Personnel files are maintained for all field and laboratory employees. Each file includes:

1. A record with date and results of each observation or examination of performance for each test method the staff member performs.

2. Certification of competence, if any, from recognized outside agencies.

3. Listing of training courses completed.

4.5 Training

All field and/or laboratory personnel will receive a copy of related A.S.T.M. procedures for his personal reference file when they are initially hired. It is each individual's responsibility to thoroughly familiarize himself/herself with these documents.

Training in these procedures will be conducted in-house and/or on the job. An initial review of the procedures will be conducted in-house by the Field Supervisor prior to assigning a "new hire" field inspector to a project. Hands-on field training will be conducted under the direct supervision of the Supervisor or a field technician who has satisfactorily maintained his proficiency rating in the procedure in question, as evidenced by personnel file proficiency records.

5.0 EQUIPMENT AND CALIBRATION

5.1 A system for control of accuracy and reliability of tools, gages, instruments and other measuring and testing apparatus forms the nucleus of Applied Construction Technologies, Inc.'s Quality Assurance System. The calibration system provides for the detection and prevention of equipment inaccuracy through comparison of measured properties against those obtained from higher standards of accuracy at regularly specified standards which have known relationship to National Standards where such Standards exist.

5.2 Records provide objective evidence that calibration schedules are complied with and that the accuracy of the equipment or standards is being maintained. Records for the inventory and calibration system of Applied Construction Technologies, Inc. is summarized below with more detailed information provided on the pages to follow.

5.2.1 Inventory

A tabulated inventory of test equipment, subject to calibration or operational check, is maintained in the Quality Control file. Essential elements of this listing are as follows:

1. Description of the test equipment.
2. Equipment identification number.
3. Name of manufacturer or source.
4. Model, style, or serial number or other identification.
5. Properties subject to calibration and/or verification.
6. Range of operation and range of calibration and/or verification.
7. Allowable error tolerances or maximum sensitivity.

8. Applied Construction Technologies, Inc.'s calibration/ verification procedure number.

9. Frequency of calibration or verification.

5.2.1.1 Inventory Numbers

Inventory numbers were developed to facilitate correlation between calibration records and the physical inventory. The first set of numbers contained in the equipment identification number identifies the type of equipment and the second set of numbers contains the unique item in that set.

The identification number may be found on an embossed plastic type or inscribed on the physical inventory. New items are added to the list consecutively according to the department and the equipment description.

5.2.1.2 Inventory List Location

Records for physical inventory are maintained in the Master File and the Unique File for each piece of equipment.

5.2.2 Procedures

The procedures used for the calibration, verification and maintenance is delineated for the various types of equipment in the Calibration Procedures Manual. Copies of each method is maintained at the departmental level for ready reference.

Information included with each procedure consists of the following as applicable:

1. Specifications, such as range of operation and range of calibration and/or verification, allowable error tolerances or maximum sensitivity. These may be listed in the manual or may be referenced.

2. Reference documents, such as ASTM Standards.

3. Equipment needed for calibration.

4. General instruction for pre-calibration inspection of the item.

5. Procedure to follow for the calibration and/or verification. This may be explicitly contained in the Procedures Manual or may be referenced so that it's location is clearly identified.

6. Reference Standard or Material.

7. Maintenance procedures.

5.2.2.1 Frequency

Determination of realistic calibration intervals is dependent upon the basis of the instrument or standard, it's purpose, and degree of usage. Intervals are used from existing specifications if available. Intervals shall be shortened as required to assure continued accuracy as evidenced by the results of preceding calibrations and may be lengthened only when the results of previous calibrations provide definite indications that such action will not adversely affect the accuracy of the system. Special calibration will be performed when accuracy of the equipment is suspect.

5.2.2.2 Refence Standards

Calibration of reference standards must be certified as being traceable to the National Bureau of Standards or derived from accepted values of natural physical constants.

Internal and External standards established for calibrating and measuring test equipment used in controlling product quality shall have the capabilities for accuracy, stability and range required for intended uses.

5.2.2.3 Tolerances

Qualifying calibration data shall fall within recognized tolerances established in the governing procedure.

5.2.2.4 Data Recording Sheets And Certificates

Each department maintains forms for routine calibrations and operational checks using proper standards, techniques, and data recording practices. All raw data and calculations are contained on these forms which are kept on file for each department.

Certificates of calibration/verification to reference standards are maintained on file when an outside source is used. Procedure used by the service is documented.

5.2.3 Labels

When size or function of the equipment is feasible, labels shall be attached to each piece of calibrated test equipment with the following information:

1. Inventory Number

2. Date of Calibration

3. Identification of certifying technicians

4. Due date for recalibration

Any equipment which has exceeded it's calibration interval, is out of tolerance, damaged, or in need of repair will be immediately removed from service until necessary repair or adjustments are made. Equipment consistently found to be out of calibration will be evaluated and repaired or replaced as necessary.

5.3 Calibration Recall System

5.3.1 General Comments

A calibration recall system, administered by the Quality Assurance Manager, assures the continued proper fuctioning of the calibration system.

At the beginning of each month, the system is searched so that proper communication regarding equipment renewal can be made with each Department. By the end of each month, new calibration data sheets must be submitted to the Quality Assurance Manager who, after review, will document receipt within the system. At the end of each month, the system is searched for delinquent calibrations so that appropriate steps can be taken to maintain compliance with the established schedule.

5.3.2 Procedure For Recall

Each item to be calibrated and/or verified for compliance to specifications is stored on a computer tape/disk with the date for calibration and/or verification. At the beginning of each month, a listing of equipment is printed out for calibration anddistributed to the respective Department Managers for processing.

5.4 Reference Programs

Annually, Applied Construction Technologies, Inc. will perform both a "within" and a "between" laboratory statistical evaluation of performance consistency conducted under the direction of the Laboratory Manager.

5.5 Internal Audit

5.5.1 General

Twice each calendar year, early in April and October, scheduled internal audits will be performed to assure compliance of the laboratory with all aspects of the Quality Assurance Program. Audit will be performed by the President of the Company. The established Calibration Recall System shall be made by the Quality Assurance Manager to the President who will select one or more staff members or senior technicians to perform the audit.

The auditor will conduct his/her survey as follows:

1. Two (2) inventoried items subject to calibration and one (1) item scheduled for operational check shall be selected at random from the master inventory file.

2. Written procedures for standardization of each item shall be made available.

3. Current calibration certificates or operational check lists shall be supplied.

The auditor shall independently review the above information for proper content and cross-referencing and shall answer each applicable question contained on a supplied "Audit Form" (a copy of which may be found in the Appendix to this Manual). Inspection of the physical inventory for identification, labeling, and condition shall be considered a pertinent aspect of the audit.

Procedures and calibration forms shall be returned to the files upon completion of the audit and marked "AUDITED" and dated in the column labeled "Date of Calibration". The Auditor shall initial these entries only when full system compliance is found. Deficiencies noted on completed "Audit Forms" shall be listed by the president in the Physical File for the equipment audited. At the end of April and October, a re-audit will be performed by the President. Correction of deficiencies will be indicated by initialling of "Audited" forms.

An unscheduled audit of the Quality Assurance System records shall be conducted by the President annually. Elements to be reviewed for currency and content are:

1. Equipment Inventory

2. Procedure File

3. Calibration Data

4. Scheduled Internal Audits

In subsequent discussions with the Department Managers, overall effectiveness of the program will be ascertained and such actions necessary to insure compliance formulated for timely implementation. A written record of unannounced audit results and proposed modifications or corrective actions shall be made part of the Audit File.

5.5.2 Instructions for Performance of Internal Audit

A. Select, at random, two (2) inventoried items subject to calibration and one (1) item scheduled for operational check listed in the Master Inventory File.

B. Search the Master File for calibration and operational check procedures.

C. Proceed to each department and request, from the Department Head or his assistant, the Department Quality Assurance Manual and location of equipment subject to audit.

D. Inspect and compare available documents and physical inventory for proper cross-referencing of inventory number, procedure number, date and frequency of re-verification.

E. Respond to questions 1.) through 8.).

F. Sign and date each Audit Form

G. Affix initials after "Audited" only when the results of the audit are deemed satisfactory (uninitialled inventory will be re-audited).

H. Present all audit materials and forms and one copy of the Internal Audit Form to the President.

5.6 External Audits

Applied Construction Technologies, Inc. maintains a continuing policy of cooperation with governmental, military, and industrial control inspectors. The Company realizes the advantages of maintaining approved equipment, test procedures, and quality control systems. To this end, the Company makes available it's records of calibration, operational checks, written procedures, records and test reportsl. Inspections are welcomed at the convenience of the agency and the Company. Any and reasonable demands for additions, modifications or revisions to the established Company's Quality Assurance System will be given careful consideration. Subsequent decisions will be made in the best interests of our clients and Applied Construction Technologies, Inc.

BRECHBUHLER SCALES, INC.
SALES - SERVICE - RENTALS - SYSTEMS
24 Hour Service — Periodic Inspection

SCALE INSPECTION REPORT

For: Cypplia Const Tech
210 Hayes dr Brooklyn Hts Ohio

P.O.# ..... Att: ..... Date 4-17-90

A 58561

☐ 1424 Scale St. S.W. CANTON, OHIO 44706 216-453-2424
☒ 520 Brookpark Rd. CLEVELAND, OHIO 44109 216-661-2660
☐ 4070 Perimeter Dr. COLUMBUS, OHIO 43228 614-274-9009
☐ 9914 Crescent Park Drive WEST CHESTER, OHIO 45069 513-777-5800
☐ 1060 National Parkway MANSFIELD, OHIO 44906 419-529-3006 or 529-5195
☐ 1001 Findlay Road LIMA, OHIO 45801 419-228-5021
☐ 1221 N. Meridian Rd. YOUNGSTOWN, OHIO 44509 216-792-7772
☐ 526 31st St. PARKERSBURG, WV 26101 304-428-1792
☐ 4200 First Ave., Suite 114 NITRO, WV 25143 304-755-0688

Test weights used are traceable to U.S. Government Standards

Company:

Accepted by: Leroy J. Tazer

Code:
G - Good
F - Fair
P - Poor
X - Condemned
A - Added Oil

Serviced by: Chad

Inspector, Weights & Measures: BSI 274

NO	LOCATION	MFG	CAPACITY	SERIAL NO	Cleaned & Tested	Lever Pivots & Brgs.	Platform	Pit - Or Scale Frame	Indicating Head	Dashpot	Printer	General Condition	Load Cell	Count	Weight Applied	Error Corrected	RECOMMENDATIONS
1	Shop	mettler	3600gr	3600	✓	-	G	G	G	-	-	G	G	-	200gr / 400gr / 1kg / 2kg / 50GR	-3gr	Applied wts as shown to Scale. Recalibrated Scale was 3gr light at 2kg. 1kg needed for Cal Scale ok.
2	"	OHAUS	2610g	N/A #1	✓	G	G	G	G	-	-	G	-	-	5G / 200gr / 400gr / 600gr / 2kg	0	No adj needed Scale within 1/10 of a gram at 2kg Scale ok.
3	"	OHAUS	311g	Suite C	✓	G	G	G	G	-	-	G	~~G~~	-	1G / 5G / 200G / 100G	-1	Rezeroed Scale app wt as shown scale. Good. No cal needed.
4	"	OHAUS	2610g	N/A	✓	G	G	G	G	-	-	G	-	-	5G / 200G / 400GR / 600G / 2kg	0	Applied wt as shown no Cal or adj needed.
5	Bldg 2	ES	100#	18782	✓	-	G	G	G	-	-	G	G	-	25# / 50# / 4kg / 5kg	±.05# / ±.005kg	Applied wt to Scale 25# + 50# ok ±.05. Applied 5kg ±.005 Scale ok.

BRECHBUHLER SCALES, INC.

SALES - SERVICE - RENTALS - SYSTEMS

24 Hour Service — Periodic Inspection

SCALE INSPECTION REPORT

For: Applied Const tech

210 Hayes Rd Brooklyn Hts Ohio 44

P.O.# ..... Att: ..... Date 4-17-90

A 58505

- ☐ 1424 Scale St. S.W. CANTON, OHIO 44708 216-453-2424
- ☑ 520 Brookpark Rd. CLEVELAND, OHIO 44109 216-661-2660
- ☐ 4070 Perimeter Dr. COLUMBUS, OHIO 43228 614-274-9009
- ☐ 9914 Crescent Park Drive WEST CHESTER, OHIO 45069 513-777-5800
- ☐ 1080 National Parkway MANSFIELD, OHIO 44906 419-529-3006 or 529-5195
- ☐ 1001 Findlay Road LIMA, OHIO 45801 419-226-5021
- ☐ 1221 N. Meridian Rd. YOUNGSTOWN, OHIO 44509 216-792-7772
- ☐ 526 31st St. PARKERSBURG, WV 26101 304-428-1792
- ☐ 4200 First Ave., Suite 114 NITRO, WV 25143 304-755-0686

Test weights used are traceable to U.S. Government Standards

Company:

Accepted by: Leroy J. [illegible]

Code:
G - Good
F - Fair
P - Poor
X - Condemned
A - Added Oil

Serviced by: Chuf

Inspector, BSI 274.
Weights & Measures

NO	LOCATION	MFG	CAPACITY	SERIAL NO	Cleaned & Tested	Lever Pivots & Brgs.	Platform	Pit - Or Scale Frame	Indicating Head	Dashpot	Printer	General Condition	Load Cell	Count	Weight Applied	Error Corrected	RECOMMENDATIONS
6	Bldg 2	OHAUS	20kg / 45#	N/A	✓										4kg	0	NO ADJUSTment
															5kg		needed applied wt
															[illegible]		as shown to Scale
															50G		Scale Good ✓
															100G		
															[illegible]		
															1000G		
															2kg		
															7kg		
															400g		
															200g		
															600g		

Calibration Services, Inc.
P.O. Box 425
Edinburg, PA 16116
412/654-3899 or 654-2533

Client	APPLIED CONSTRUCTION TECHNOLOGY, INC.	Report Date	11-02-90	File Number	CAPC1090
Address	210 Hayes Dr., Suite C	Calib. Date	10-31-90		
City/State	Cleveland, OH	Engineer	Paul J. Marano		
Zipcode	44131	Temperature	23 degrees C		
Location	210 Hayes Dr., Suite C	Device Type	proving ring	Ring Size:	10,000 lbs.
City/State	Cleveland, OH 44131	Manufacturer	Humboldt	Ring Serial #	204

CALIBRATION INSTRUMENTS USED: STRAINSENSE LOAD CELL SYSTEM S/N 830711

5 K. S/N 850416 Dead Wt. Mach. S/N M-4644;NIST LAB No.737.229759

50 K. S/N 870430 Dead Wt. Mach. S/N M-4644;NIST Lab No. 737.229759
and 50 K. S/N 881111 (T/C) Trans. Std. S/N 4858; NIST LAB No.732.07/243760

150 K. S/N 830711B Trans. Std. S/N 5779;NIST LAB No.732.07/241081
Trans. Std. S/N 4858;NIST LAB No. 732.07/243760
700 K. S/N 830711-C Trans. Std. S/N 5628;NIST LAB No. 732.07/241081;
Trans. Std. 4287;NIST LAB No. 732.07/243758; Trans Std S/N 5779;NIST LAB No. 732.07/241081

Calibrated according to ASTM E-74 specifications by Morehouse Instr. Co.
Calibrated: Dec. 13, 1989 Recalibration Due: December 1991

Certification Statement

This is to certify that the above described testing device has been calibrated by the acredited representative of this company in accordance with applicable ASTM Standard Procedures (E-4).

Signed Paul J. Marano
Paul J. Marano, CE

This Certificate is issued as a statement of the fact that on this date the above instrument(s) had an accuracy as indicated. It should not be construed or regarded as a Guarantee or Warranty of any kind (in favor of the client, the client's customers, or the public at large) that the instrument(s) will continue to retain the same percentage (%) of accuracy or efficiency as determined on the date when the calibration, and adjustments if required, was performed and reported by Calibration Services, Inc., since the calibrator has absolutely no control over the future operation, damage, maintenance, repairs, and overall condition of the instrument(s) and hereby expressly disclaims any and all liability for damage, or loss sustained by all parties arising or resulting from deterioration, obsolescence, malfunction, or substandard, performance of said instruments(s); which shall be deemed to be and which shall remain the sole responsibility of the machine's regular custodian, owner, and/or manufacturer.

Calibration Services. Inc.
P.O. Box 425
Edinburg, PA 16116
412/654-3899 or 654-2533

Client	Applied Construction Technology, Inc.	Report Date	11-02-90	File Number	BAPC1090
Address	210 Hayes Drive	Calib. Date	10-31-90		
City/State	Cleveland, OH	Engineer	Paul J. Marano		
Zipcode	44131	Temperature	23 degrees C		
Location	210 Hayes Drive Suite C	Device Type	proving ring	Ring Size:	1500 lbs.
City/State	Cleveland, OH 44131	Manufacturer	Soiltest, Inc.	Ring Serial #	21729

CALIBRATION INSTRUMENTS USED: STRAINSENSE LOAD CELL SYSTEM S/N 830711

5 K. S/N 850416 Dead Wt. Mach. S/N M-4644; NIST LAB No.737.229759

50 K. S/N 87043 Dead Wt. Mach. S/N M-4644; NIST LAB No. 737.229759
and 50 K. S/N 881111 (T/C) Trans. Std. S/N 4858; NIST LAB No. 732.07/243760

150 K. S/N 830711B Trans. Std. S/N 5779; NIST LAB No. 732.07/241081 &
Trans. Std. S/N 4858; NIST LAB No. 732.07/243760
700 K. S/N 830711-C Trans. Std. S/N 5628; NIST LAB No. 732.07/241081 &
Trans Std. 4287; NIST LAB No. 732.07/243758 & Trans Std. S/N 5779;NIST
LAB No. 732.07/241081

Calibrated according to ASTM E-74 specification by Morehouse Instr. Co.
Calibrated: December 13, 1989 Recalibration Due: December 1991

Certification Statement

This is to certify that the above described testing device has been calibrated by the acredited representative of this company in accordance with applicable ASTM Standard Procedures (E-4).

Signed Paul J. Marano
Paul J. Marano, CE

This Certificate is issued as a statement of the fact that on this date the above instrument(s) had an accuracy as indicated. It should not be construed or regarded as a Guarantee or Warranty of any kind (in favor of the client, the client's customers, or the public at large) that the instrument(s) will continue to retain the same percentage (%) of accuracy or efficiency as determined on the date when the calibration, and adjustments if required, was performed and reported by Calibration Services, Inc., since the calibrator has absolutely no control over the future operation, damage, maintenance, repairs, and overall condition of the instrument(s) and hereby expressly disclaims any and all liability for damage, or loss sustained by all parties arising or resulting from deterioration, obsolescence, malfunction, or substandard, performance of said instruments(s); which shall be deemed to be and which shall remain the sole responsibility of the machine's regular custodian, owner, and/or manufacturer.

APPENDIX

6.2 ORDER ENTRY FLOW CHART

1 CLIENT CONTACT:
- Telephone
- Walk-In
- Letter

2 ASCERTAIN SCOPE:
- Client
- Project
- Distribution
- Services Likely

3 ESTABLISH FILE:
- Assign Project File No.
- Make-Up File Folder

4 WORK REQUESTED

4.1 Geotechnical Services
- Geotechnical Engineer Assigned
- Obtain support services as required
- Evaluate
- Generate and distribute formal report
- Billing

4.2 Drilling
- In-House
- Contract

4.3 Laboratory
- Identify file
- Note services required as idendified on Laboratory Test Assignment Sheets
- Schedule work activity
- Assign laboratory technician
- Perform laboratory activity
- Obtain support services as required
- Store samples
- Generate and distribute formal report
- Billing

4.4 Field
- Identify file
- Determine services required
- Schedule work activity
- Assign field technician
- Perform field activity
  - Material laboratory
  - Specimen pick up coordination
- Generate and distribute formal report
- Billing

5.3 CONCRETE CYLINDER REPORT FLOW CHART

Field Inspector	Office	Materials Laboratory
1. Complete Field 3 - Part Form a. General field information b. Concrete class, yardage and mix proportions c. Field Test: Truck No. Cylinder No., field test results, specific location(s). Affix metal tag to each cylinder. d. Narrative of day's operations.		
2. Send field white copy to office	5. Type (1) and 6. Insert F.O., Bldg., Field, and File No. 9. Distribute and temp. file.	
3. Affix field pink copy to fabricated concrete cylinders enclosed in plastic bag for Materials Lab.		7. Assign Lab & Lab Report No. Date of Receipt 8. Determine 7-Day Strength Type of break
	10. Update for (7) and (8) 11. Distribute, return pink and temp. file.	
		12. Determine 28-Day Strength Type of break
	13. Update for (12) 14. Final file, return pink Master/Field White Distribute.	

6.4 SERVICES RENDERED BY EXTERNAL ORGANIZATIONS FOR CALIBRATION AND STANDARDIZATION

COMPANY	SERVICE FOR
Accurate Instrument Service Co 4228 W 130 St Cleveland, Ohio 44135	Dial Indicate
Densicon, Inc. 30 Sherman St. Elmwood, Conn. 06110	Windsor Probe
Troxler Electronic Lab Inc. P. O. Box 12057 Research Triangle Park, NC 27709	Troxler, Nuclear Gages
Zormco Electronics corp P. O. Box 4444 Cleveland, Ohio 44125	Thickness Gages
E. J. Deiber	Torque Wrench Multiplier
Calibration Services, Inc. P. O. Box 425 Edinburg, Pa. 18118	Compression Machine and Load Rings
Brechbuhler Scales, Inc. 520 Brookpark Road Cleveland, Ohio 44109	Balances and Scales

APPLIED CONSTRUCTION TECHNOLOGIES, INC.
210 HAYES DRIVE, #C
CLEVELAND, OHIO 44131-1036

6.5 INTERNAL AUDIT FORM

Department _____

INVENTORY NO.	DESCRIPTION	PROCEDURE NO.	FREQUENCY OF RE-VERIFICATION	CURRENT CALIBRATION DOCUMENT AVAILABLE?

Please answer the following questions:

1. Does the inventory number appear
   A. On the inventory List? _____
   B. On the equipment or storage container? _____
2. Does the procedure number appear on the inventory list? _____
3. Is the calibration current? _____
4. Is a dated and initialled label attached to the equipment or storage container? _____
5. Are operational checks performed at prescribed intervals? _____

6.5 INTERNAL AUDIT FORM

(Continued)

6. Do calibration forms show currency of calibration? _____

7. List noted discrepancies and make additional comments below:

_____

_____

_____

_____

_____

8. Is a re-audit necessary? _____

   If so, which inventory number(s)? _____

Name 1.) _____ Department 1.) _____

2.) _____ 2.) _____

6.6 SAMPLE RUNS

ENGINEERING • TESTING • INSPECTION

APPLIED CONSTRUCTION TECHNOLOGIES, INC.

[illegible] BROOKPARK ROAD • CLEVELAND [illegible] • [illegible]

ACT PROJECT NO. _____

WORK ORDER

CLIENT: _____

PROJECT: _____

LOCATION: _____

BILLING INFORMATION: ( ) _____

COPIES TO:

( ) _____ ( ) _____

( ) _____ ( ) _____

SITE CONTACT PERSON: _____ SITE PHONE _____

SCOPE OF WORK:

DIRECTIONS TO SITE:

SPECIAL INSTRUCTIONS:

Initiated by: _____ Date: _____

APPLIED CONSTRUCTION TECHNOLOGIES, INC.
1615 BROOKPARK ROAD
CLEVELAND, OHIO 44109 (216) 485-TEST
ENGINEERING • TESTING • INSPECTION

PROJECT: _____
SHEET NO: _____ OF _____
CLIENT: _____
DATE ENTERED: _____ COMPLETED: _____
PERSON IN CHARGE: _____

LABORATORY TEST ASSIGNMENTS

TYPE OF TEST	SAMPLE SOURCE/SAMPLE DEPTH
VISUAL CLASSIFICATION	
SIEVE ANALYSIS	
HYDROMETER ANALYSIS	
ATTERBERG LIMITS	
AASHTO CLASSIFICATION	
ODOT CLASSIFICATION	
MOISTURE CONTENT	
UNIT WEIGHT	
UNCONFINED COMPRESSION	
Shelby Tube	
Remolded Sample	
Rock Core	
Concrete Core	
MOISTURE DENSITY RELATIONSHIP	
Standard	
Modified	
PERMEABILITY	
Undisturbed Sample	
Compacted Sample	
SWELL TEST	
FHA (Swell Pressure)	
PENDOT (Amount Swell)	
Proctor Sample (Amount Swell)	
CALIFORNIA BEARING RATIO:	
Compactive Effort	
Surcharge	
Soaking Time	
OTHER TEST - SPECIFY	

REMARKS

6.7

Standard Test Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

$^{\epsilon 1}$ NOTE—Section 19 was added editorially in September 1990.

1. Scope

1.1 This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 µm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 µm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-µm), or No. 200 (75-µm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-µm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 µm.

2. Referenced Documents

2.1 *ASTM Standards:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

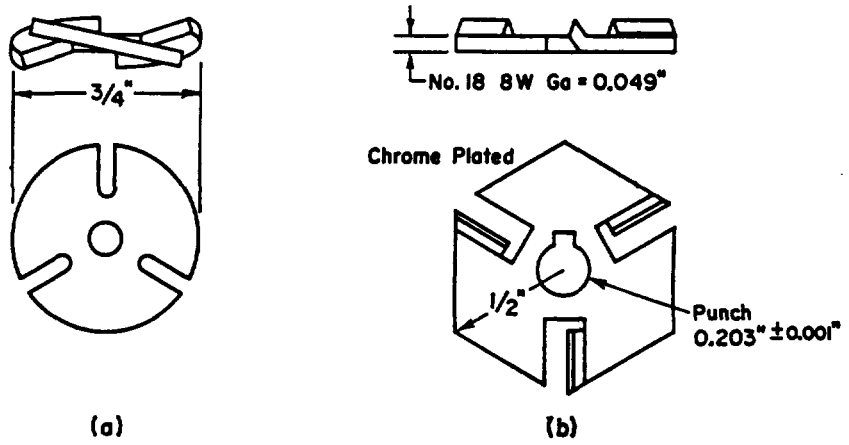
Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 - 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.



Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-µm)
1½-in. (37.5-mm)	No. 40 (425-µm)
1-in. (25.0-mm)	No. 60 (250-µm)
¾-in. (19.0-mm)	No. 140 (106-µm)
⅜-in. (9.5-mm)	No. 200 (75-µm)
No. 4 (4.75-mm)	

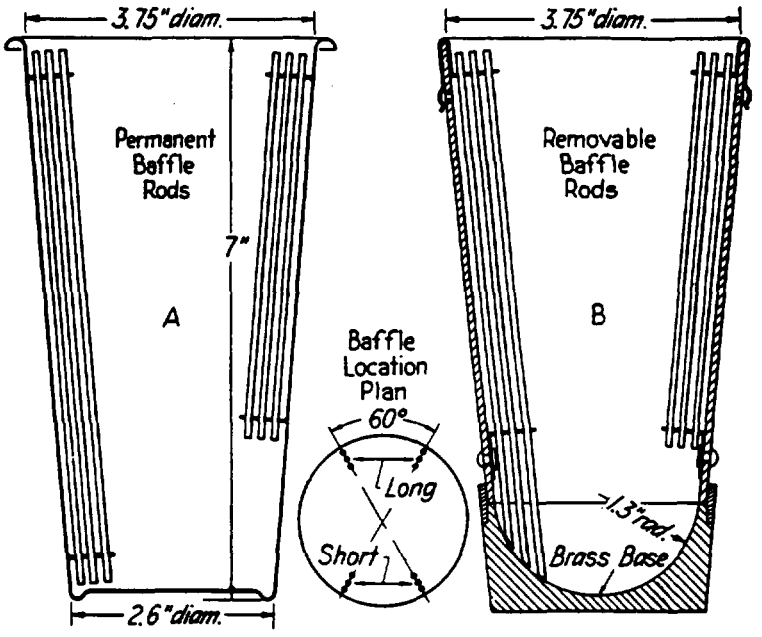
NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-µm)
¾-in. (19.0-mm)	No. 50 (300-µm)
⅜-in. (9.5-mm)	No. 100 (150-µm)
No. 4 (4.75-mm)	No. 200 (75-µm)
No. 8 (2.36-mm)	

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

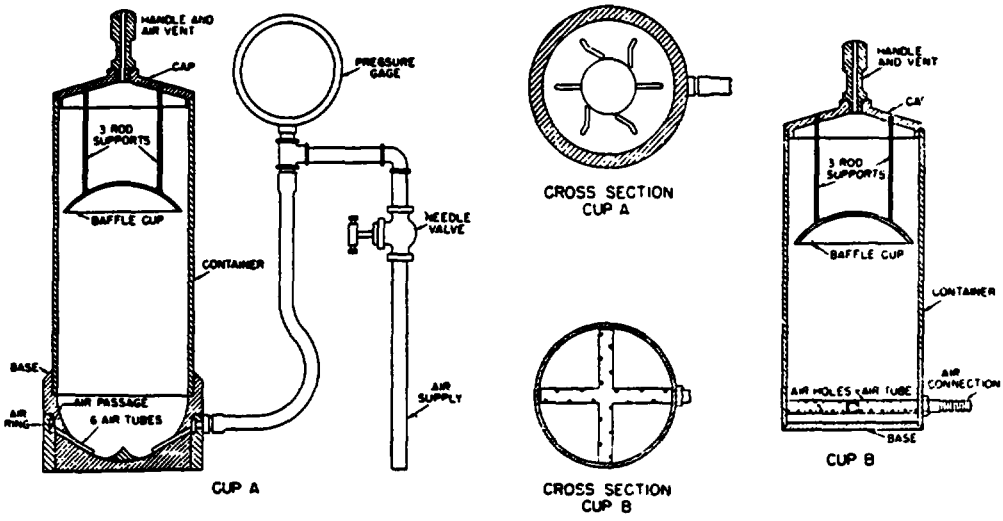


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

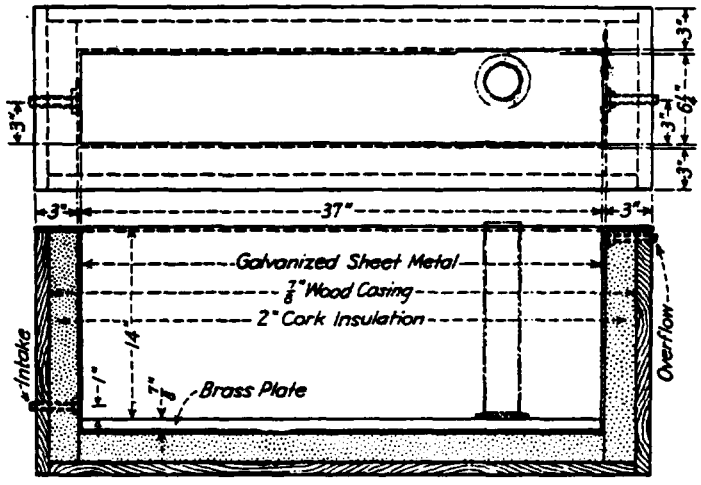
5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.



Metric Equivalents

in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cyclinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns.

Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

TABLE 1 Values of Correction Factor, a, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,

η = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)),

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of soil particles, and

G_l = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm^3, and

A = cross-sectional area of sedimentation cylinder, cm^2

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm^3

A = 27.8 cm^2

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(3) Silt size, 0.074 to 0.005 mm ..... %
(4) Clay size, smaller than 0.005 mm ..... %
  Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-µm)	.....
No. 200 (75-µm)	.....

HYDROMETER ANALYSIS

0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-µm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

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