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*New York State
Department of Environmental Conservation
Division of Hazardous Waste Remediation*

REMEDIAL INVESTIGATION AND FEASIBILITY STUDY WORK PLAN

**ROXY CLEANERS SITE
TOWN OF NORTH GREENBUSH
RENSSELAER COUNTY, NEW YORK
SITE I.D. NO. 4-42-024**

**VOLUME II
QUALITY ASSURANCE PROJECT PLAN**

OCTOBER 1991

Prepared By:

**M&E Metcalf & Eddy
of New York, Inc.**

303 South Broadway, Tarrytown, New York 10591

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TOWN OF NORTH GREENBUSH
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**SITE I.D. NO. 4-42-024
STANDBY CONTRACT WORK ASSIGNMENT NO. D002406-11.1**

**PREPARED FOR

NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION**

**BY

METCALF & EDDY OF NEW YORK, INC.
TARRYTOWN, NEW YORK**

OCTOBER 1991

ROXY CLEANERS SITE
NORTH GREENBUSH
RENSSELAER COUNTY, NEW YORK
SITE I.D. NO. 4-42-024

Date: 9/11/91

Date: 9/10/91

Date: _____

Date: _____

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1.0 PROJECT DESCRIPTION, ORGANIZATION AND OBJECTIVES

1.1 INTRODUCTION

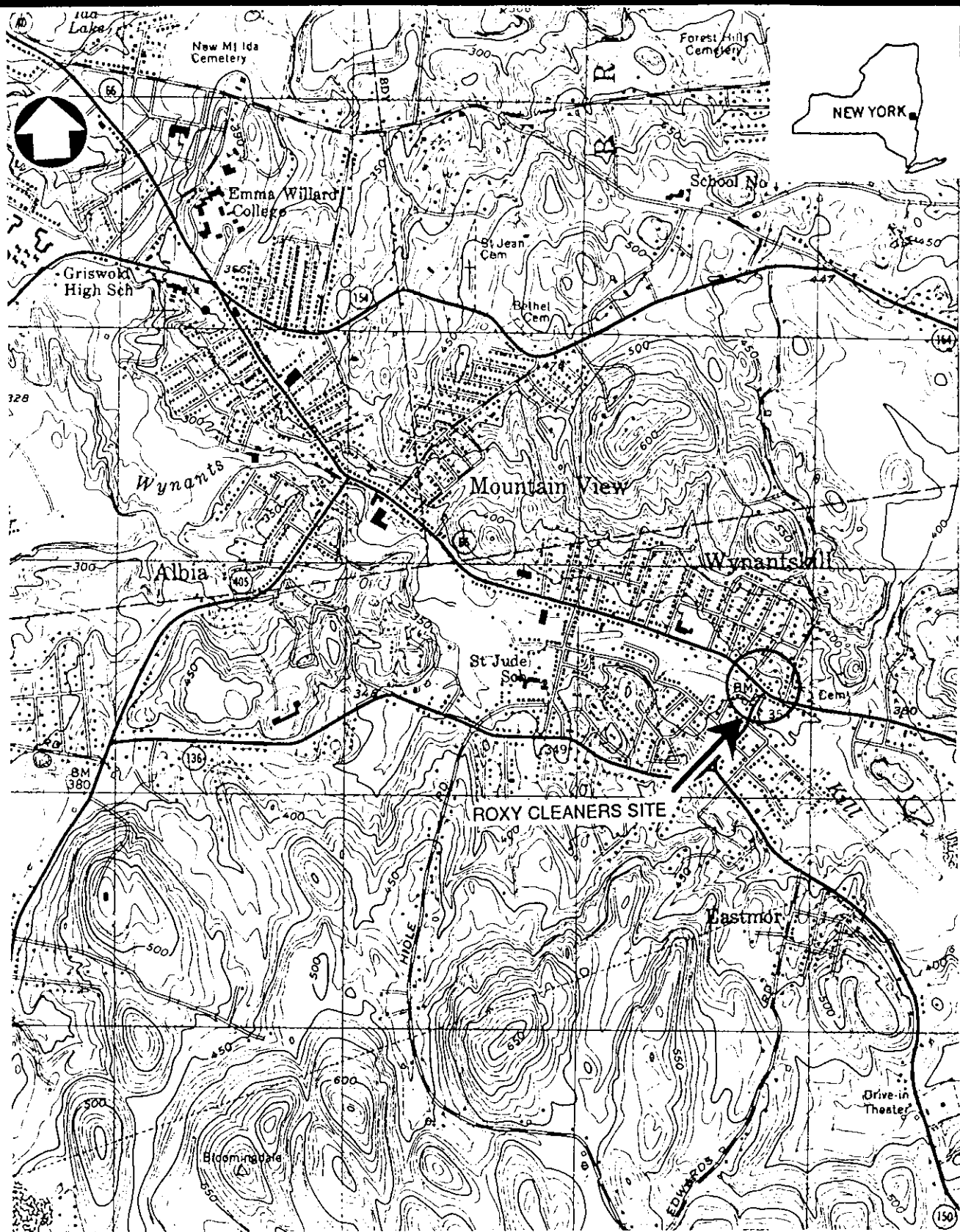
This Quality Assurance Project Plan (QAPP) presents a detailed description of the site-specific Quality Assurance and Quality Control (QA/QC) requirements for the Remedial Investigation/Feasibility Study (RI/FS) for the Roxy Cleaners Site in North Greenbush, New York. The scope of work is based on available data concerning site history, previous analytical testing, and geologic/hydrogeologic conditions. The purpose of the Roxy Cleaners Site RI/FS is to determine the nature and extent of the contamination at the site and adjacent areas, identify/confirm the source(s) of contamination, determine the risk to public health and the environment, and identify, evaluate and recommend a cost-effective, environmentally sound, long-term remedial action plan.

This QAPP shall be reviewed and approved by the NYSDEC and other agencies as appropriate to ensure agreement on the tasks and methodology to be undertaken prior to commencing the work. Also, the QAPP will serve as a guideline for field personnel.

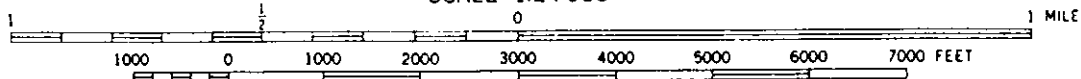
1.2 PROJECT DESCRIPTION

1.2.1 Site Description

The Roxy Cleaners Site is a Class 2 New York State Superfund Site located in the Hamlet of Wynantskill in the Town of North Greenbush, Rensselaer County, New York (see Figure 1-1). Roxy Cleaners is situated on Main Avenue, near the intersection of NYS Route 66 (Main Avenue) and Route 150 (West Sand Lake Road) approximately 250 feet north of the Wynants Kill, also known as Wynantskill Creek (see Figure 1-2).



SCALE 1:24 000



M&E Metcalf & Eddy
of New York, Inc.
A Metcalf & Eddy Company

ROXY CLEANERS SITE
RENSSELAER COUNTY, NEW YORK

FIGURE 1-1
SITE LOCATION MAP

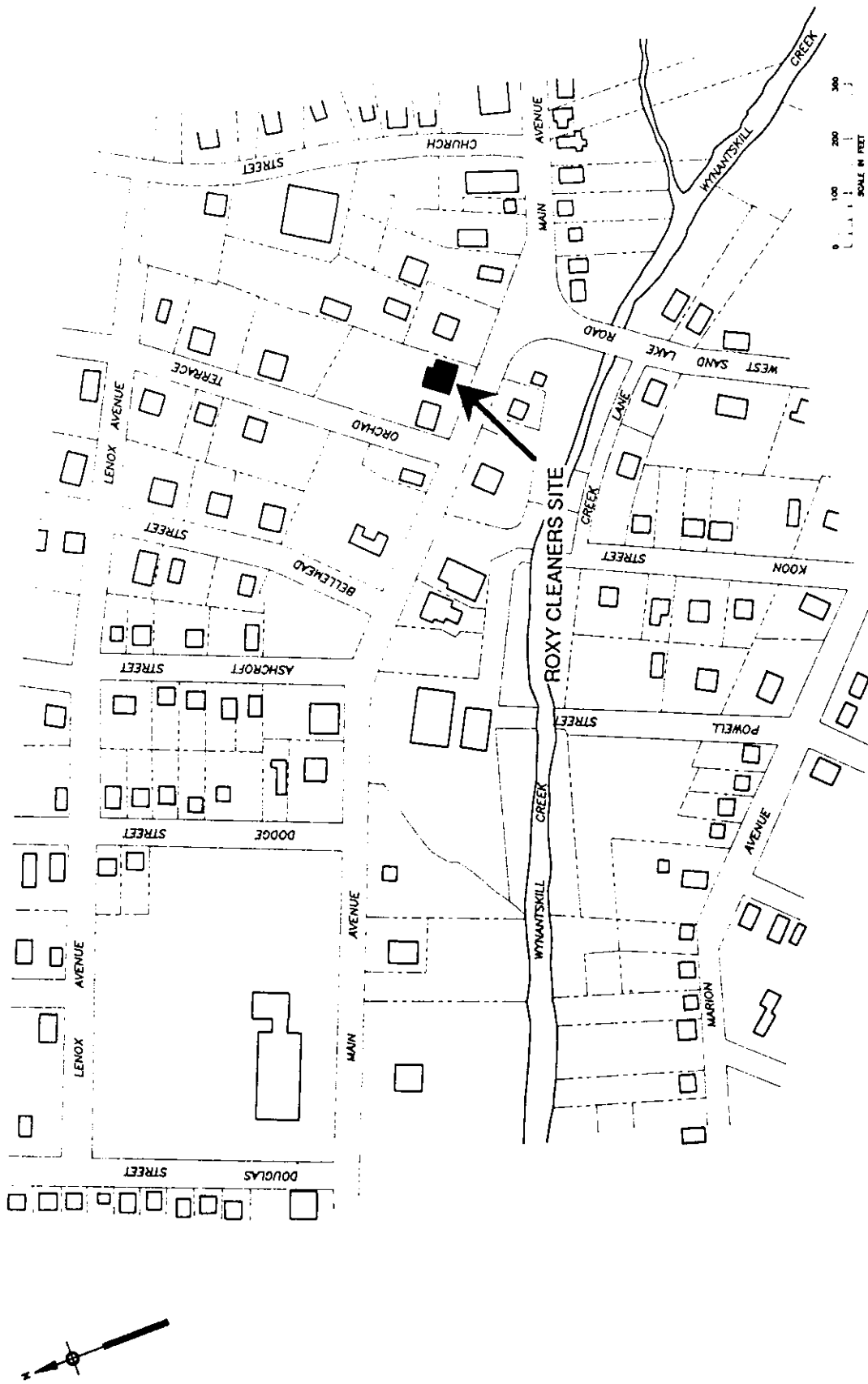


**FIGURE 1-2
SITE MAP**

ROXY CLEANERS SITE
RENSSELAER COUNTY, NEW YORK

The Roxy Cleaners Site is located in an area of light commercial and residential development. Directly west of the site is a vacant building (formerly Stewart's Ice Cream Shop) and to the east is an automotive shop (Jewett's Automotive Shop). Other auto repair shops and an abandoned gasoline station (formerly Stewarts) are located south of the site. Residential development is located directly north of the Roxy Cleaners Site. The site lies upgradient from residences and commercial establishments which obtain potable water from individual private wells.

1.2.2 Site History

Roxy Cleaners is a dry cleaning establishment that has been in operation for over 15 years, however; the actual cleaning of fabrics ceased several years ago. Since the cessation of their cleaning process, the facility has been used only as a distribution center while dry cleaning is performed at other locations.

Prior to the facility ceasing its dry cleaning operations, the solvent tetrachloroethene, also known as perchloroethylene (PCE), was used as part of the dry cleaning process. Due to improper disposal of this solvent, groundwater (and water supply including private wells at residences and commercial establishments) and on-site soils became contaminated with volatile and chlorinated organic compounds, including PCE and two of its breakdown compounds, trichloroethene (TCE) and cis-1,2-dichloroethene (DCE).

According to the owner of Roxy Cleaners, the only known incident which could have caused the groundwater contamination in the area was the spillage of a 55 gallon drum of PCE at the site in 1984. However, it appears that other unreported practices at the facility also contributed to the contamination. The origin of soil and groundwater contamination appears to be along the northern side of the Roxy Cleaners building, as a result of dry cleaning solvents being improperly disposed of to the ground surface in this area.

In May 1989, the NYSDEC Region 4 Spill Bureau was notified of a groundwater and water supply contamination concern when the owner of Roxy Cleaners

informed NYSDEC of PCE contamination in the private well utilized by both Roxy Cleaners and the adjacent Stewart's shop. NYSDEC obtained groundwater samples from the water supply well and it was found that the predominant contaminants detected in the samples were PCE (tetrachloroethene) and its degradation by-products, trichloroethene (TCE) and dichloroethene (DCE).

As a result of the preliminary findings, NYSDEC informed the New York State Department of Health (NYSDOH) of the groundwater/water supply contamination. The NYSDOH sampled several residential water supply wells in June and July 1989. The results of the sample analyses indicated that a number of water supply wells were contaminated with PCE and other chlorinated organic compounds.

Based on the analytical results of the samples, NYSDEC directed the site owner to employ the services of a consulting engineering firm in order to evaluate the nature of the contamination and to formulate possible remedial actions. Upon NYSDEC review of the plans prepared by the consultant selected by the owner, it was determined that the work plan was unacceptable since it did not include work to be performed off-site. Subsequently, in order to expedite the completion of the work, NYSDEC chose to undertake the work directly, through the involvement of Empire Soils Investigations, Inc. (Empire).

In June 1989, a site investigation was performed by Empire at the request and authorization of NYSDEC in the area centered around Roxy Cleaners. This investigation was undertaken by NYSDEC in response to complaints regarding contamination in the private water supply well utilized by Roxy Cleaners and the adjacent Stewart's shop, and due to contamination of other private water supply wells in the area by tetrachloroethene and other associated chlorinated organic compounds.

Residents have made numerous complaints regarding contamination of their private water supply wells. These complaints have been brought to the attention of NYSDEC, NYSDOH and the Rensselaer County Department of Health.

Several response actions and Interim Remedial Measures (IRMs) have been undertaken since the discovery of soil, groundwater and water supply contamination at and in the vicinity of the site.

Residents and owners of commercial establishments that have water supply wells affected by the contamination were initially instructed to use bottled water for cooking and consumption until activated carbon filters were installed. Afterward, the residents and business owners had activated carbon filters installed on their water supply systems to remove organic compounds.

NYSDEC authorized Metcalf & Eddy of New York, Inc. (M&E) to undertake an IRM for the improvement of existing activated carbon filter units and provision of new systems, where necessary. In addition, M&E is contracted to monitor and maintain the systems at 16 private residences/businesses, in the vicinity of the site. At present, NYSDEC has authorized M&E to perform another IRM for design and construction services necessary to remediate at least 300 cubic yards of contaminated soil on the Roxy Cleaners property.

As discussed previously, this document has been prepared for the Remedial Investigation and Feasibility Study (RI/FS) that is currently being undertaken by M&E and NYSDEC. The first phase of the remedial investigation will include the inspection of existing wells and installation of new monitoring wells, sampling of borehole soils, groundwater, surface water and sediment, and seeps (if present), a soil gas survey, site survey and mapping, and hydrogeological testing including packer and slug tests.

1.2.3 Available Analytical Data

Based on the results of previous sampling data, it appears that tetrachloroethene (PCE) is the primary contaminant of concern. In addition, two breakdown products of PCE, namely, cis-1,2-dichloroethene (DCE) and trichloroethene (TCE) are also chemicals of concern.

1.3 PROJECT ORGANIZATION AND RESPONSIBILITY

Figure 1-3 shows the overall project organization chart for the RI/FS. Figure 1-4 presents the Quality Assurance (QA) Organization Chart for this project and identifies the individuals responsible for each element of the overall program. Table 1-1 presents the staff assignments for this project.

The QA Data Coordinator is responsible for quality assurance and reports to the QA Technical Advisor who has Corporate QA responsibility. Mr. Michael Noonan is the QA Data Coordinator for this study.

Analytical results shall be reported directly to the QA Data Coordinator, who shall review and provide the data to the Project Manager. The Project Manager for this project is Mr. Errol Kitt.

Various professional staff members shall work on-site during the field sampling portion of this project. Personnel involved in site management are also shown in Figure 1-3.

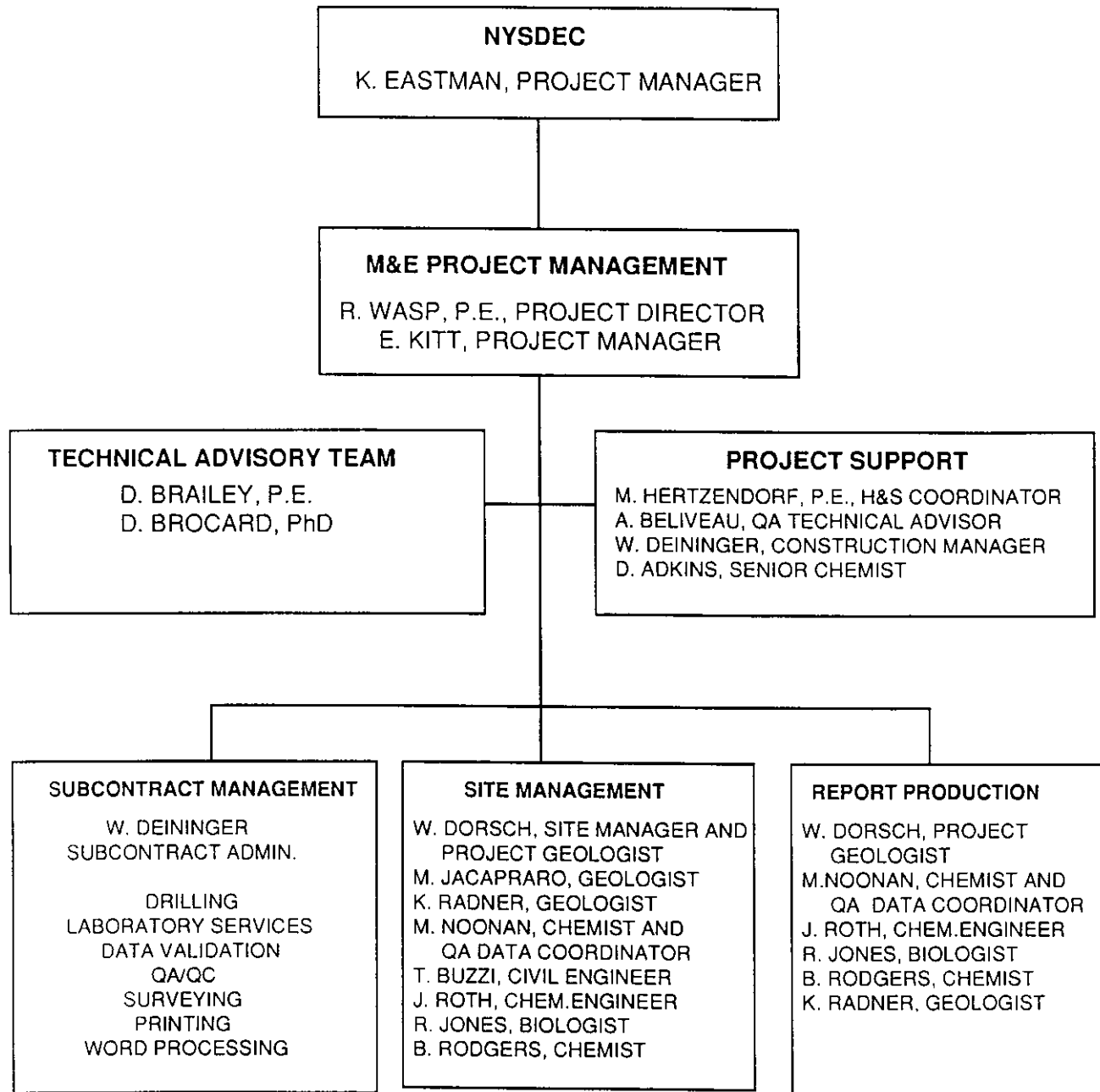
The subcontract laboratory, which shall provide analytical services during the sampling episode shall be determined.

The key individuals responsible for implementation of quality assurance procedures and their specific QA responsibilities are as follows:

QA Technical Advisor - Andrew Beliveau, Metcalf & Eddy

- Company QA technical advisory role
- Acts in conjunction with the Project Manager to develop site specific QAPP
- Oversees technical aspects of QAPP and approves QAPP
- Provides technical guidance to QA Data Coordinator
- Has authority to override decisions by Project Manager in areas where QA/QC elements may be compromised

ROXY CLEANERS SITE RI/FS
WA # D002406-11.1



ROXY CLEANERS SITE RI/FS
WA # D002406-11.1
QA ORGANIZATION CHART

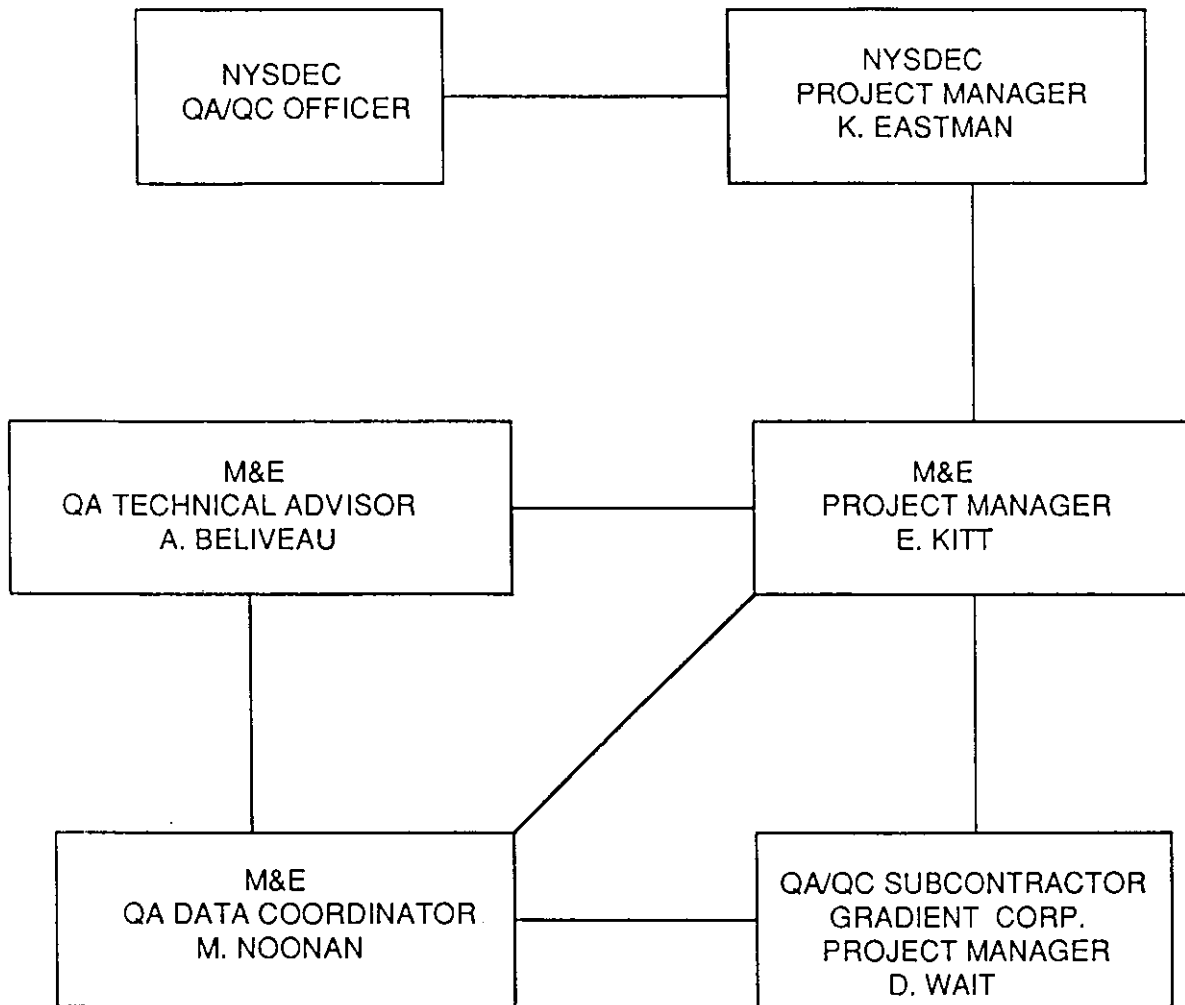


TABLE 1-1
STAFF ASSIGNMENTS

<u>STAFF</u>	<u>ASSIGNMENT</u>
Robert G. Wasp	Project Director
Donald M. Brailey Dominique N. Brocard	Staff Consultants (SC)
Martin S. Hertzendorf Andrew F. Beliveau	Technical Specialists (TSI)
Errol S. Kitt	Project Manager (PM)
William Deininger	Construction Manager (CM) and Subcontract Administrator (SA)
Andrew F. Beliveau	QA Technical Advisor
Michael Noonan	QA Data Coordinator
William Dorsch	Project Geologist (PG) and Site Manager (SM)
Karen Radner	Geologist (GE)
Michael Jacapraro	Geologist (GE)
Martin S. Hertzendorf	Health and Safety Coordinator
Thomas Buzzi	Civil Engineer (EN)
Joan Roth	Chemical Engineer (EN)
Dale Adkins	Senior Chemist
Michael Noonan	Chemist (CH)
Brad Rogers	Chemist (CH)
Rick Jones	Project Biologist (PB)
Valli Wasp	Word Processing Supervisor (WP)
Joseph Jelisavcic	Drafter (DR)

QA Data Coordinator - Michael Noonan, Chemist II, Metcalf & Eddy

- Prepares QAPP in conjunction with QA Technical Advisor and Project Manager
- Assists Project Manager in the development of the sampling and analytical portions of the QAPP
- Directs and tracks all laboratory measurement data
- Interfaces with laboratory subcontractor on daily matters
- Interfaces with laboratory and data validation subcontractors, makes requests and resolves problems
- Reviews and provides laboratory analytical data to M&E Project Manager
- Responsible for preparation of QA reports to management
- Interfaces with QA/QC subcontractor (Gradient Corporation) on daily matters
- Attends lab audit(s) with QA/QC subcontractor
- Interfaces with NYSDEC QA/QC representatives
- Assures that samples are collected in accordance with approved QA/QC procedures
- Assures that all documentation of field sampling activities is complete and accurate.
- Records and reports any problems or changes associated with field sampling activities
- Logs in data packages received from laboratory, checks data packages for completeness, transmits data packages to data validation subcontractor
- Logs in data validation submittals, checks for completeness, sends validation reports and data packages to data usability subcontractor

Project Manager - Errol Kitt, Metcalf & Eddy

- Reports to M&E Vice President, Hazardous Waste Group
- Reviews and approves field operating procedures

- Assures that approved procedures meet project objectives
- Coordinates field activities with QA Data Coordinator
- Responsible for implementation of recommendations made by QA Technical Advisor and QA Data Coordinator
- Management of subcontracts, QA/QC subcontractor (Gradient) and Data Validation subcontractor.

QA/QC Subcontractor - Gradient Corporation

- Performs laboratory audit
- Performs data usability and prepares report
- Performs field audits
- Reports to M&E QA Data Coordinator on technical matters
- Interfaces with NYSDEC QA/QC representatives on technical matters only
- Reports to M&E Project Manager on policy and contractual matters

Site Management - Metcalf & Eddy Personnel

- Reports to Project Manager, Metcalf & Eddy, Inc.
- Responsible for collection of representative samples
- Assures that samples are collected in accordance with approved QA/QC procedures
- Assures that all documentation of field sampling activities is complete and accurate
- Records and reports any problems and changes associated with field sampling activities

1.4 QUALITY ASSURANCE OBJECTIVES

Comprehensive Quality Assurance (QA) objectives for the Roxy Cleaners Site remedial investigation have been developed to provide guidelines for all field and laboratory procedures. These guidelines encompass all analysis performed

during the project. The intention of the sampling and analysis effort is to produce data of acceptable quality to identify and delineate the extent of the present site contamination and to allow for a comprehensive design of remedial alternatives.

The main QA objective for this work is that all measurements be representative of the actual present site conditions, and that all data resulting from sampling and analysis activities be comparable to previous analyses. The use of accepted, published, sampling and analysis methods, as well as the use of standardized units, shall aid in ensuring the comparability of the data.

The five major characteristics of data quality to be addressed by the laboratory during the analyses of environmental samples are defined below.

1.4.1 Accuracy

Accuracy is defined as the degree of agreement of measurement (or measurement average) with an accepted reference or true value. It is a measure of system bias and is usually expressed as a percentage of the true value.

Accuracy shall be determined in the laboratory through the use of site specific matrix spike and matrix spike duplicate (MS/MSD) analyses. The laboratory shall select one environmental sample in 20 to be analyzed for accuracy. The sample shall be split into three aliquots. One aliquot shall be analyzed routinely for the parameters of interest, while the remaining two aliquots shall be spiked with known quantities of particular target compounds prior to analysis. The percent recoveries of the target compounds shall be calculated and used as an indication of the accuracy of the analyses performed.

Sampling accuracy shall be maintained by the implementation and adherence to strict procedural protocols. Trip blanks and equipment blanks shall be collected and analyzed to ensure that all samples are representative of the particular site from which they are collected and to ensure that no cross-contamination occurred.

1.4.2 Precision

Precision is a measure of agreement among individual measurements of the same property under similar conditions. It is expressed in terms of relative percent difference (RPD) between replicates or in terms of the standard deviation when three or more replicate analyses are performed.

Precision shall be determined through the use of MS/MSD analyses as described in Section 1.4.1. The RPD between the two results shall be calculated as a measure of analytical precision.

Sampling precision shall be measured in the laboratory by the analysis of field replicates.

1.4.3 Completeness

Completeness is a measure of the accountability of each sample taken during a sampling episode. The ability to account for 100% of the samples taken through the entire sampling, transport and analysis phases is the goal of completeness.

1.4.4 Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a data population, process condition, a sampling point, or an environment. For this project, grab samples shall be taken, and such samples are, by definition, representative of only the conditions at the point in time collected, within sampling and analytical error.

1.4.5 Comparability

Comparability expresses the confidence with which one data can be compared to another. To achieve comparability in this project, the data generated shall

be reported using units of ug/l and mg/kg. By using consistent and approved sampling and analysis procedures, all data sets shall be comparable within a specific site and between sites at Roxy Cleaners to ensure that a consistent data base is used from which decisions concerning remedial action are made. To ensure data comparability, NYSDEC ASP standard reference materials shall be analyzed to establish that analytical procedures are generating valid data.

1.4.6 Procedures for Data Assessment

Data quality objectives for analyses conducted under the scope of this laboratory QA Program Plan are presented in Tables 1-2 through 1-4. The precision values presented therein represent variability for replicate measurements of the same parameter, and are expressed in terms of relative percent difference for duplicate measurements made on samples. Accuracy values include components of both random error (i.e., variability due to imprecision) and systematic error (i.e., bias), and thus reflect the total error for a given measurement, expressed as a percentage of the true value. The objectives listed in Table 1-2 through 1-4 are based primarily on performance data derived from method validation studies for analyses. These are not intended to represent validation criteria, per se; rather, they represent the performance capability of the methods.

An overview of the procedures to be used in assessing data quality include the following:

- Accuracy
 - Computing percent recoveries for spiked samples
 - Calculate the standard deviation in the overall average recovery value
 - Applying Chauvenet's criterion for detecting bad recovery data
 - Determining the range of uncertainty at a given level of confidence
 - Adjusting the laboratory data to correct any systematic errors (bias) that are discovered

TABLE 1-2

SUMMARY OF ESTIMATED LABORATORY PRECISION, ACCURACY AND COMPLETENESS OBJECTIVES
(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Metals				
Arsenic	NYSDEC-ASP	20%	75-125%	100%
Barium	NYSDEC-ASP	20%	75-125%	100%
Cadmium	NYSDEC-ASP	20%	75-125%	100%
Chromium	NYSDEC-ASP	20%	75-125%	100%
Copper	NYSDEC-ASP	20%	75-125%	100%
Iron	NYSDEC-ASP	20%	75-125%	100%
Lead	NYSDEC-ASP	20%	75-125%	100%
Mercury	NYSDEC-ASP	20%	75-125%	100%
Nickel	NYSDEC-ASP	20%	75-125%	100%
Selenium	NYSDEC-ASP	20%	75-125%	100%
Silver	NYSDEC-ASP	20%	75-125%	100%
Zinc	NYSDEC-ASP	20%	75-125%	100%
Antimony	NYSDEC-ASP	20%	75-125%	100%
Aluminum	NYSDEC-ASP	20%	75-125%	100%
Beryllium	NYSDEC-ASP	20%	75-125%	100%
Calcium	NYSDEC-ASP	20%	75-125%	100%
Cobalt	NYSDEC-ASP	20%	75-125%	100%
Manganese	NYSDEC-ASP	20%	75-125%	100%
Magnesium	NYSDEC-ASP	20%	75-125%	100%
Molybdenum	NYSDEC-ASP	20%	75-125%	100%
Potassium	NYSDEC-ASP	20%	75-125%	100%
Sodium	NYSDEC-ASP	20%	75-125%	100%
Thallium	NYSDEC-ASP	20%	75-125%	100%
Vanadium	NYSDEC-ASP	20%	75-125%	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Volatile Organics (VOC)				
Benzene	524.2	NDG	NDG	100%
Bromobenze	524.2	NDG	NDG	100%
Bromochloromethane	524.2	NDG	NDG	100%
Bromodichloromethane	524.2	NDG	NDG	100%
Bromoform	524.2	NDG	NDG	100%
Bromomethane	524.2	NDG	NDG	100%
n-Butylbenzene	524.2	NDG	NDG	100%
sec-Butylbenzene	524.2	NDG	NDG	100%
tert-Butylbenzene	524.2	NDG	NDG	100%
Carbon tetrachloride	524.2	NDG	NDG	100%
Chlorobenzene	524.2	NDG	NDG	100%
Chloroethane	524.2	NDG	NDG	100%
Chloroform	524.2	NDG	NDG	100%
Chloromethane	524.2	NDG	NDG	100%
2-Chlorotoluene	524.2	NDG	NDG	100%
4-Chlorotoluene	524.2	NDG	NDG	100%
Dibromochloromethane	524.2	NDG	NDG	100%
1,2-Dibromo-3-chloropropane	524.2	NDG	NDG	100%
1,2-Dibromoethane	524.2	NDG	NDG	100%
Dibromoethane	524.2	NDG	NDG	100%
1,2-Dichlorobenzene	524.2	NDG	NDG	100%
1,3-Dichlorobenzene	524.2	NDG	NDG	100%
1,4-Dichlorobenzene	524.2	NDG	NDG	100%
Dichlorodifluoromethane	524.2	NDG	NDG	100%
1,1-Dichloroethane	524.2	NDG	NDG	100%
1,2-Dichloroethane	524.2	NDG	NDG	100%
1,1-Dichloroethene	524.2	NDG	NDG	100%
cis-1,2-Dichloroethene	524.2	NDG	NDG	100%
trans-1,2-Dichloroethene	524.2	NDG	NDG	100%
1,2-Dichloropropane	524.2	NDG	NDG	100%
1,3-Dichloropropane	524.2	NDG	NDG	100%
2,2-Dichloropropane	524.2	NDG	NDG	100%
1,1-Dichloropropene	524.2	NDG	NDG	100%
cis-1,3-Dichloropropene	524.2	NDG	NDG	100%
trans-1,3-Dichloropropene	524.2	NDG	NDG	100%
Ethylbenzene	524.2	NDG	NDG	100%
Hexachlorobutadiene	524.2	NDG	NDG	100%
Isopropylbenzene	524.2	NDG	NDG	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Volatile Organics (VOC)				
4-Isopropyltoluene	524.2	NDG	NDG	100%
Methylene chloride	524.2	NDG	NDG	100%
Naphthalene	524.2	NDG	NDG	100%
n-Propylbenzene	524.2	NDG	NDG	100%
Styrene	524.2	NDG	NDG	100%
1,1,1,2-Tetrachloroethane	524.2	NDG	NDG	100%
1,1,2,2-Tetrachloroethane	524.2	NDG	NDG	100%
Tetrachloroethene	524.2	NDG	NDG	100%
Toluene	524.2	NDG	NDG	100%
1,2,3-Trichlorobenzene	524.2	NDG	NDG	100%
1,2,4-Trichlorobenzene	524.2	NDG	NDG	100%
1,1,1-Trichloroethane	524.2	NDG	NDG	100%
1,1,2-Trichloroethane	524.2	NDG	NDG	100%
Trichloroethene	524.2	NDG	NDG	100%
Trichlorofluoromethane	524.2	NDG	NDG	100%
1,2,3-Trichloropropane	524.2	NDG	NDG	100%
1,2,4-Trimethylbenzene	524.2	NDG	NDG	100%
1,3,5-Trimethylbenzene	524.2	NDG	NDG	100%
Vinyl chloride	524.2	NDG	NDG	100%
o-Xylene	524.2	NDG	NDG	100%
m-Xylene	524.2	NDG	NDG	100%
p-Xylene	524.2	NDG	NDG	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Volatile Organics (VOC)				
Acetone	NYSDEC-ASP	NDG	NDG	100%
Benzene	NYSDEC-ASP	11%	76-127%	100%
Bromodichloromethane	NYSDEC-ASP	NDG	NDG	100%
Bromoform	NYSDEC-ASP	NDG	NDG	100%
Bromomethane	NYSDEC-ASP	NDG	NDG	100%
2-Butanone (MEK)	NYSDEC-ASP	NDG	NDG	100%
Carbon disulfide	NYSDEC-ASP	NDG	NDG	100%
Carbon tetrachloride	NYSDEC-ASP	NDG	NDG	100%
Chlorobenzene	NYSDEC-ASP	13%	75-130%	100%
Chloroethane	NYSDEC-ASP	NDG	NDG	100%
2-Chloroethylvinyl ether	NYSDEC-ASP	NDG	NDG	100%
Chloroform	NYSDEC-ASP	NDG	NDG	100%
Chloromethane	NYSDEC-ASP	NDG	NDG	100%
Dibromochloromethane	NYSDEC-ASP	NDG	NDG	100%
1,1-Dichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,1-Dichloroethene	NYSDEC-ASP	14%	61-145%	100%
trans-1,2-Dichloroethene	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichloropropane	NYSDEC-ASP	NDG	NDG	100%
cis-1,3-Dichloropropene	NYSDEC-ASP	NDG	NDG	100%
trans-1,3-Dichloropropene	NYSDEC-ASP	NDG	NDG	100%
Ethylbenzene	NYSDEC-ASP	NDG	NDG	100%
2-Hexanone	NYSDEC-ASP	NDG	NDG	100%
Methylene chloride	NYSDEC-ASP	NDG	NDG	100%
4-Methyl-2-Pentanone (MIBK)	NYSDEC-ASP	NDG	NDG	100%
Styrene	NYSDEC-ASP	NDG	NDG	100%
1,1,2,2-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
Tetrachloroethene	NYSDEC-ASP	NDG	NDG	100%
Toluene	NYSDEC-ASP	13%	76-125%	100%
1,1,1-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,1,2-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
Trichloroethene	NYSDEC-ASP	14%	71-120%	100%
Vinyl acetate	NYSDEC-ASP	NDG	NDG	100%
Vinyl chloride	NYSDEC-ASP	NDG	NDG	100%
Xylenes	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. (1) Reference	Precision (2) (as RPD)	Accuracy (3) (Recovery)	Completeness
Semivolatile Organics (SVOC)				
Base/Neutral Extractables:				
Acenaphthene	NYSDEC-ASP	31%	46-118%	100%
Acenaphthylene	NYSDEC-ASP	NDG	NDG	100%
Anthracene	NYSDEC-ASP	NDG	NDG	100%
Benzo(a)anthracene	NYSDEC-ASP	NDG	NDG	100%
Benzo(b)fluoranthene	NYSDEC-ASP	NDG	NDG	100%
Benzo(k)fluoranthene	NYSDEC-ASP	NDG	NDG	100%
Benzo(ghi)perylene	NYSDEC-ASP	NDG	NDG	100%
Benzo(a)pyrene	NYSDEC-ASP	NDG	NDG	100%
Benzoic acid	NYSDEC-ASP	NDG	NDG	100%
Benzyl alcohol	NYSDEC-ASP	NDG	NDG	100%
Benzyl butyl phthalate	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroethoxy)methane	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroethyl)ether	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroisopropyl)ether	NYSDEC-ASP	NDG	NDG	100%
Bis(2-ethylhexyl)phthalate	NYSDEC-ASP	NDG	NDG	100%
4-Bromophenyl phenyl ether	NYSDEC-ASP	NDG	NDG	100%
4-Chloroaniline	NYSDEC-ASP	NDG	NDG	100%
2-Chloronaphthalene	NYSDEC-ASP	NDG	NDG	100%
4-Chlorophenyl phenyl ether	NYSDEC-ASP	NDG	NDG	100%
Chrysene	NYSDEC-ASP	NDG	NDG	100%
Dibenzo(a,h)anthracene	NYSDEC-ASP	NDG	NDG	100%
Dibenzofuran	NYSDEC-ASP	NDG	NDG	100%
Di-n-butyl phthalate	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichlorobenzene	NYSDEC-ASP	NDG	NDG	100%
1,3-Dichlorobenzene	NYSDEC-ASP	NDG	NDG	100%
1,4-Dichlorobenzene	NYSDEC-ASP	28%	36-97%	100%
3,3-Dichlorobenzidine	NYSDEC-ASP	NDG	NDG	100%
Diethyl phthalate	NYSDEC-ASP	NDG	NDG	100%
Dimethyl phthalate	NYSDEC-ASP	NDG	NDG	100%
2,4-Dinitrotoluene	NYSDEC-ASP	38%	24-96%	100%
2,6-Dinitrotoluene	NYSDEC-ASP	NDG	NDG	100%
Di-n-octylphthalate	NYSDEC-ASP	NDG	NDG	100%
Fluoranthene	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Fluorene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorobenzene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorobutadiene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorocyclopentadiene	NYSDEC-ASP	NDG	NDG	100%
Hexachloroethane	NYSDEC-ASP	NDG	NDG	100%
Indeno (1,2,3-cd)pyrene	NYSDEC-ASP	NDG	NDG	100%
Isophorone	NYSDEC-ASP	NDG	NDG	100%
2-Methylnaphthalene	NYSDEC-ASP	NDG	NDG	100%
Naphthalene	NYSDEC-ASP	NDG	NDG	100%
2-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
3-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
4-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
Nitrobenzene	NYSDEC-ASP	NDG	NDG	100%
N-Nitrosodi-n-propylamine	NYSDEC-ASP	38%	41-116%	100%
Phenanthrene	NYSDEC-ASP	NDG	NDG	100%
Pyrene	NYSDEC-ASP	31%	26-127%	100%
1,2,4-Trichlorobenzene	NYSDEC-ASP	28%	39-98%	100%
Acid Extractables:				
4-Chloro-3-methylphenol	NYSDEC-ASP	42%	23-97%	100%
2-Chlorophenol	NYSDEC-ASP	40%	27-123%	100%
2,4-Dichlorophenol	NYSDEC-ASP	NDG	NDG	100%
2,4-Dimethylphenol	NYSDEC-ASP	NDG	NDG	100%
2,4-Dinitrophenol	NYSDEC-ASP	NDG	NDG	100%
2-Methyl-4,6-dinitrophenol	NYSDEC-ASP	NDG	NDG	100%
2-Methylphenol	NYSDEC-ASP	NDG	NDG	100%
4-Methylphenol	NYSDEC-ASP	NDG	NDG	100%
2-Nitrophenol	NYSDEC-ASP	NDG	NDG	100%
4-Nitrophenol	NYSDEC-ASP	50%	10-80%	100%
Pentachlorophenol	NYSDEC-ASP	50%	9-103%	100%
Phenol	NYSDEC-ASP	42%	12-89%	100%
2,4,5-Trichlorophenol	NYSDEC-ASP	NDG	NDG	100%
2,4,6-Trichlorophenol	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-2 (Continued)

(WATER ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Pesticides and PCBs				
Aldrin	NYSDEC-ASP	22%	40-120%	100%
Alpha-BHC	NYSDEC-ASP	NDG	NDG	100%
Beta-BHC	NYSDEC-ASP	NDG	NDG	100%
Delta-BHC	NYSDEC-ASP	NDG	NDG	100%
Gamma-BHC (Lindane)	NYSDEC-ASP	NDG	56-123%	100%
Chlordane	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDD	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDE	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDT	NYSDEC-ASP	27%	38-127%	100%
Dieldrin	NYSDEC-ASP	18%	52-126%	100%
Endosulfan I	NYSDEC-ASP	NDG	NDG	100%
Endosulfan II	NYSDEC-ASP	NDG	NDG	100%
Endosulfan sulfate	NYSDEC-ASP	NDG	NDG	100%
Endrin	NYSDEC-ASP	21%	56-121%	100%
Endrin ketone	NYSDEC-ASP	NDG	NDG	100%
Heptachlor	NYSDEC-ASP	20%	40-131%	100%
Heptachlor epoxide	NYSDEC-ASP	NDG	NDG	100%
Methoxychlor	NYSDEC-ASP	NDG	NDG	100%
Toxaphene	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1016	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1221	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1232	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1242	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1248	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1254	NYSDEC-ASP	NDG	NDG	100%
Aroclor-1260	NYSDEC-ASP	NDG	NDG	100%

NDG No data generated for this analysis

- (1) NYSDEC Analytical Services Protocols, September 1989. Superfund Category.

Superfund CLP. - Volatile Organic NYSDEC, Method 89-1
 Superfund CLP. - Semi-Volatile Organic NYSDEC, Method 89-2
 Superfund CLP. - Pesticides/PCBs NYSDEC, Method 89-3
 Superfund CLP. - Inorganic NYSDEC
 Method 524.2 - Measurement of Purgeable Organic Compounds by Capillary Column GC/MS.

- (2) Precision - Relative Percent Difference (RPD) between duplicate matrix spike recoveries, or duplicate analyses, except where noted for organics
- (3) Accuracy - Expected recovery for QC check samples or as specified by the method, for matrix spike recoveries, except where noted for organics

TABLE 1-3

SUMMARY OF ESTIMATED LABORATORY PRECISION, ACCURACY, AND COMPLETENESS OBJECTIVES
(SOIL ANALYSES)

Parameter	Method No. (1) Reference	Precision (2) (as RPD)	Accuracy (3) (Recovery)	Completeness
Metals				
Arsenic	NYSDEC-ASP	20%	75-125%	100%
Barium	NYSDEC-ASP	20%	75-125%	100%
Cadmium	NYSDEC-ASP	20%	75-125%	100%
Chromium	NYSDEC-ASP	20%	75-125%	100%
Copper	NYSDEC-ASP	20%	75-125%	100%
Iron	NYSDEC-ASP	20%	75-125%	100%
Lead	NYSDEC-ASP	20%	75-125%	100%
Mercury	NYSDEC-ASP	20%	75-125%	100%
Nickel	NYSDEC-ASP	20%	75-125%	100%
Selenium	NYSDEC-ASP	20%	75-125%	100%
Silver	NYSDEC-ASP	20%	75-125%	100%
Zinc	NYSDEC-ASP	20%	75-125%	100%
Antimony	NYSDEC-ASP	NDG	NDG	NDG
Aluminum	NYSDEC-ASP	20%	75-125%	100%
Beryllium	NYSDEC-ASP	20%	75-125%	100%
Calcium	NYSDEC-ASP	20%	75-125%	100%
Cobalt	NYSDEC-ASP	20%	75-125%	100%
Manganese	NYSDEC-ASP	20%	75-125%	100%
Magnesium	NYSDEC-ASP	20%	75-125%	100%
Molybdenum	NYSDEC-ASP	20%	75-125%	100%
Potassium	NYSDEC-ASP	20%	75-125%	100%
Sodium	NYSDEC-ASP	20%	75-125%	100%
Thallium	NYSDEC-ASP	20%	75-125%	100%
Vanadium	NYSDEC-ASP	20%	75-125%	100%
Wet Chemistry				
Total Organic Carbon	USEPA- Region II	NDG	NDG	NDG

TABLE 1-3 (Continued)

(SOIL ANALYSES)

Parameter	Method No. (1) Reference	Precision (2) (as RPD)	Accuracy (3) (Recovery)	Completeness
Volatile Organics (VOC)				
Acetone	NYSDEC-ASP	NDG	NDG	100%
Benzene	NYSDEC-ASP	21%	66-142%	100%
Bromodichloromethane	NYSDEC-ASP	NDG	NDG	100%
Bromoform	NYSDEC-ASP	NDG	NDG	100%
Bromomethane	NYSDEC-ASP	NDG	NDG	100%
2-Butanone (MEK)	NYSDEC-ASP	NDG	NDG	100%
Carbon disulfide	NYSDEC-ASP	NDG	NDG	100%
Carbon tetrachloride	NYSDEC-ASP	NDG	NDG	100%
Chlorobenzene	NYSDEC-ASP	21%	60-133%	100%
Chloroethane	NYSDEC-ASP	NDG	NDG	100%
2-Chloroethylvinyl ether	NYSDEC-ASP	NDG	NDG	100%
Chloroform	NYSDEC-ASP	NDG	NDG	100%
Chloromethane	NYSDEC-ASP	NDG	NDG	100%
Dibromochloromethane	NYSDEC-ASP	NDG	NDG	100%
1,1-Dichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,1-Dichloroethene	NYSDEC-ASP	22%	59-172%	100%
trans-1,2-Dichloroethene	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichloropropane	NYSDEC-ASP	NDG	NDG	100%
cis-1,3-Dichloropropene	NYSDEC-ASP	NDG	NDG	100%
trans-1,3-Dichloropropene	NYSDEC-ASP	NDG	NDG	100%
Ethylbenzene	NYSDEC-ASP	NDG	NDG	100%
2-Hexanone	NYSDEC-ASP	NDG	NDG	100%
Methylene chloride	NYSDEC-ASP	NDG	NDG	100%
4-Methyl-2-Pentanone (MIBK)	NYSDEC-ASP	NDG	NDG	100%
Styrene	NYSDEC-ASP	NDG	NDG	100%
1,1,2,2-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
Tetrachloroethene	NYSDEC-ASP	NDG	NDG	100%
Toluene	NYSDEC-ASP	21%	59-139%	100%
1,1,1-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
1,1,2-Trichloroethane	NYSDEC-ASP	NDG	NDG	100%
Trichloroethene	NYSDEC-ASP	24%	62-137%	100%
Vinyl acetate	NYSDEC-ASP	NDG	NDG	100%
Vinyl chloride	NYSDEC-ASP	NDG	NDG	100%
Xylenes	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-3 (Continued)

(SOIL ANALYSES)

Parameter	Method No. (1) Reference	Precision (2) (as RPD)	Accuracy (3) (Recovery)	Completeness
Semivolatile Organics (SVOC)				
Base/Neutral Extractables:				
Acenaphthene	NYSDEC-ASP	19%	31-137%	100%
Acenaphthylene	NYSDEC-ASP	NDG	NDG	100%
Anthracene	NYSDEC-ASP	NDG	NDG	100%
Benzo(a)anthracene	NYSDEC-ASP	NDG	NDG	100%
Benzo(b)fluoranthene	NYSDEC-ASP	NDG	NDG	100%
Benzo(k)fluoranthene	NYSDEC-ASP	NDG	NDG	100%
Benzo(ghi)perylene	NYSDEC-ASP	NDG	NDG	100%
Benzo(a)pyrene	NYSDEC-ASP	NDG	NDG	100%
Benzoic acid	NYSDEC-ASP	NDG	NDG	100%
Benzyl alcohol	NYSDEC-ASP	NDG	NDG	100%
Benzyl butyl phthalate	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroethoxy)methane	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroethyl)ether	NYSDEC-ASP	NDG	NDG	100%
Bis(2-chloroisopropyl)ether	NYSDEC-ASP	NDG	NDG	100%
Bis(2-ethylhexyl)phthalate	NYSDEC-ASP	NDG	NDG	100%
4-Bromophenyl phenyl ether	NYSDEC-ASP	NDG	NDG	100%
4-Chloroaniline	NYSDEC-ASP	NDG	NDG	100%
2-Chloronaphthalene	NYSDEC-ASP	NDG	NDG	100%
4-Chlorophenyl phenyl ether	NYSDEC-ASP	NDG	NDG	100%
Chrysene	NYSDEC-ASP	NDG	NDG	100%
Dibenzo(a,h)anthracene	NYSDEC-ASP	NDG	NDG	100%
Dibenzofuran	NYSDEC-ASP	NDG	NDG	100%
Di-n-butyl phthalate	NYSDEC-ASP	NDG	NDG	100%
1,2-Dichlorobenzene	NYSDEC-ASP	NDG	NDG	100%
1,3-Dichlorobenzene	NYSDEC-ASP	NDG	NDG	100%
1,4-Dichlorobenzene	NYSDEC-ASP	27%	28-104%	100%
3,3'-Dichlorobenzidine	NYSDEC-ASP	NDG	NDG	100%
Dimethyl phthalate	NYSDEC-ASP	NDG	NDG	100%
Dimethyl phthalate	NYSDEC-ASP	NDG	NDG	100%
2,4-Dinitrotoluene	NYSDEC-ASP	47%	28-89%	100%
2,6-Dinitrotoluene	NYSDEC-ASP	NDG	NDG	100%
Di-n-octylphthalate	NYSDEC-ASP	NDG	NDG	100%
Fluoranthene	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-3 (Continued)

(SOIL ANALYSES)

Parameter	Method No. (1) Reference	Precision (2) (as RPD)	Accuracy (3) (Recovery)	Completeness
Fluorene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorobenzene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorobutadiene	NYSDEC-ASP	NDG	NDG	100%
Hexachlorocyclopentadiene	NYSDEC-ASP	NDG	NDG	100%
Hexachloroethane	NYSDEC-ASP	NDG	NDG	100%
Indeno (1,2,3-cd)pyrene	NYSDEC-ASP	NDG	NDG	100%
Isophorone	NYSDEC-ASP	NDG	NDG	100%
2-Methylnaphthalene	NYSDEC-ASP	NDG	NDG	100%
Naphthalene	NYSDEC-ASP	NDG	NDG	100%
2-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
3-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
4-Nitroaniline	NYSDEC-ASP	NDG	NDG	100%
Nitrobenzene	NYSDEC-ASP	NDG	NDG	100%
N-Nitrosodi-n-propylamine	NYSDEC-ASP	38%	41-126%	100%
Phenanthrene	NYSDEC-ASP	NDG	NDG	100%
Pyrene	NYSDEC-ASP	36%	35-142%	100%
1,2,4-Trichlorobenzene	NYSDEC-ASP	23%	38-107%	100%
Acid Extractables:				
4-Chloro-3-methylphenol	NYSDEC-ASP	33%	26-103%	100%
2-Chlorophenol	NYSDEC-ASP	50%	25-102%	100%
2,4-Dichlorophenol	NYSDEC-ASP	NDG	NDG	100%
2,4-Dimethylphenol	NYSDEC-ASP	NDG	NDG	100%
2,4-Dinitrophenol	NYSDEC-ASP	NDG	NDG	100%
2-Methyl-4,6-dinitrophenol	NYSDEC-ASP	NDG	NDG	100%
2-Methylphenol	NYSDEC-ASP	NDG	NDG	100%
4-Methylphenol	NYSDEC-ASP	NDG	NDG	100%
2-Nitrophenol	NYSDEC-ASP	NDG	NDG	100%
4-Nitrophenol	NYSDEC-ASP	50%	11-114%	100%
Pentachlorophenol	NYSDEC-ASP	47%	17-109%	100%
Phenol	NYSDEC-ASP	35%	26-90%	100%
2,4,5-Trichlorophenol	NYSDEC-ASP	NDG	NDG	100%
2,4,6-Trichlorophenol	NYSDEC-ASP	NDG	NDG	100%

TABLE 1-3 (Continued)

(SOIL ANALYSES)

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Pesticides/PCBs				
Aldrin	NYSDEC-ASP	43%	34-132%	100%
Alpha-BHC	NYSDEC-ASP	NDG	NDG	100%
Beta-BHC	NYSDEC-ASP	NDG	NDG	100%
Delta-BHC	NYSDEC-ASP	NDG	NDG	100%
Gamma-BHC (Lindane)	NYSDEC-ASP	50%	46-127%	100%
Chlordane	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDD	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDE	NYSDEC-ASP	NDG	NDG	100%
4,4'-DDT	NYSDEC-ASP	50%	23-134%	100%
Dieldrin	NYSDEC-ASP	38%	31-134%	100%
Endosulfan I	NYSDEC-ASP	NDG	NDG	100%
Endosulfan II	NYSDEC-ASP	NDG	NDG	100%
Endosulfan sulfate	NYSDEC-ASP	NDG	NDG	100%
Endrin	NYSDEC-ASP	45%	42-139%	100%
Endrin ketone	NYSDEC-ASP	NDG	NDG	100%
Heptachlor	NYSDEC-ASP	31%	35-130%	100%
Haptachlor epoxide	NYSDEC-ASP	NDG	NDG	100%
Methoxychlor	NYSDEC-ASP	NDG	NDG	100%
Toxaphene	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1016	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1221	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1232	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1242	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1248	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1254	NYSDEC-ASP	NDG	NDG	100%
Arochlor-1260	NYSDEC-ASP	NDG	NDG	100%

NDG No data generated for this analysis.

- (1) NYSDEC Analytical Services Protocols, September 1989, Superfund Category (and USEPA, where noted).

Superfund CLP. - Volatile Organic NYSDEC, Method 89-1

Superfund CLP. - Semi-Volatile Organic NYSDEC, Method 89-2

Superfund CLP. - Pesticides/PCBs NYSDEC, Method 89-3

Superfund CLP. - Inorganic NYSDEC

USEPA Region II - Lloyd Kahn Method for Determination of Total Organic Carbon in Sediment, July 1986.

- (2) Precision - Relative Percent Difference (RPD) between duplicate matrix spike recoveries, or duplicate analyses, except where noted for organics.
- (3) Accuracy - Expected recovery for QC check samples or as specified by the method, for matrix spike recoveries, except where noted for organics.

TABLE 1-4

SUMMARY OF ESTIMATED FIELD PRECISION, ACCURACY AND COMPLETENESS OBJECTIVES

Parameter	Method No. ⁽¹⁾ Reference	Precision ⁽²⁾ (as RPD)	Accuracy ⁽³⁾ (Recovery)	Completeness
Standing Water Levels	Stainless Steel Tape	± .01 ft	.005 ft	100%
Temperature	E170.1, Mercury Thermometer	± 0.1°C	.05°C	100%
Conductivity	E120.1, Electrometric	± 25 umho/cm ²	10 umho/cm ²	100%
pH	E150.1, Electrometric	± .1 pH units	.05 pH units	100%
Sieve Analysis	ASTM D-422	--	--	--

- (1) E100-500 METHODS
METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES, EPQA 600/4-79-020,
March 1983
- ASTM D-422-63 (Reapproved 1990), STANDARD TEST METHOD FOR PARTICLE-SIZE
ANALYSIS OF SOILS.
- NYSDEC ANALYTICAL SERVICES PROTOCOLS, September 1989.
Superfund Category.
- (2) Expressed as Relative Percent Difference of field duplicates
- (3) Expected based on equipment manufacturer specifications

- Precision
 - Examining split samples and pairs of samples for differences in inter- and intra-sample scatter
 - Determining if sampling error has occurred by comparing inter and intra-samples
 - Validating data on groups of samples, all of which should have the same composition, by examining the scatter in each group in comparison to the overall scatter (invalid data are discharged)
 - Computing an overall relative standard deviation that is applicable to all the field investigation data from a particular sampling campaign
- Completeness
 - Computing the fraction of QA test data that remains valid after discarding any invalid accuracy or precision data
 - The measure of completeness is the accountability of each sample taken during the episode. The ability to account for 100% of the samples taken through the entire sampling, transport, and analysis phases is the goal of completeness.
- Representativeness and Comparability
 - Determining whether these terms have meaning within the project framework
 - Identifying the appropriate statistical methods
 - Correctly applying the statistical methods and reporting the results

2.0 SAMPLING AND ANALYTICAL PROCEDURES

2.1 SAMPLING

Sampling will be performed during separate stages of the project. The first stage, the Phase I Remedial Investigation (Phase I RI), will involve the most concentrated sampling effort. The types of environmental samples to be collected include: soils, groundwater, surface water, and sediment.

Sampling procedures to be used for each of these samples types are detailed in Section 4.3 of the QAPP. All sampling methods described in this plan are standard Metcalf & Eddy procedures based on recognized USEPA and NYSDEC protocols when available.

Groundwater samples will be obtained from the sixteen (16) existing monitoring wells, and the eight (8) new monitoring wells to be installed during the Phase I RI. The sixteen (16) existing monitoring wells will be sampled on two separate occasions during the Phase I RI. The initial round of groundwater sampling (Round 1) will occur prior to the installation of the new monitoring wells in order to further define appropriate locations for these wells. The second round (Round 2) of groundwater sampling will include the existing 16 monitoring wells and the 8 new monitoring wells.

Three (3) surface water samples will be collected from the Wynants Kill (Wynantskill Creek). The objective of sampling the surface water is to determine whether groundwater contamination has entered the Wynants Kill and the impact on surface water quality.

Two (2) water samples will be collected from seeps (if present) without the use of any intermediate sampling instrument. Exact sample locations will be

determined in the field by the M&E site geologist with approval by the NYSDEC site representative.

The objective of sampling seeps in conjunction with groundwater, surface water and sediment sampling is to evaluate background and surface contamination concentrations. Data will be used to define source areas of contamination, the potential pathways of vertical and horizontal migration, and health risk evaluations to types and concentrations of contaminants present at the surface.

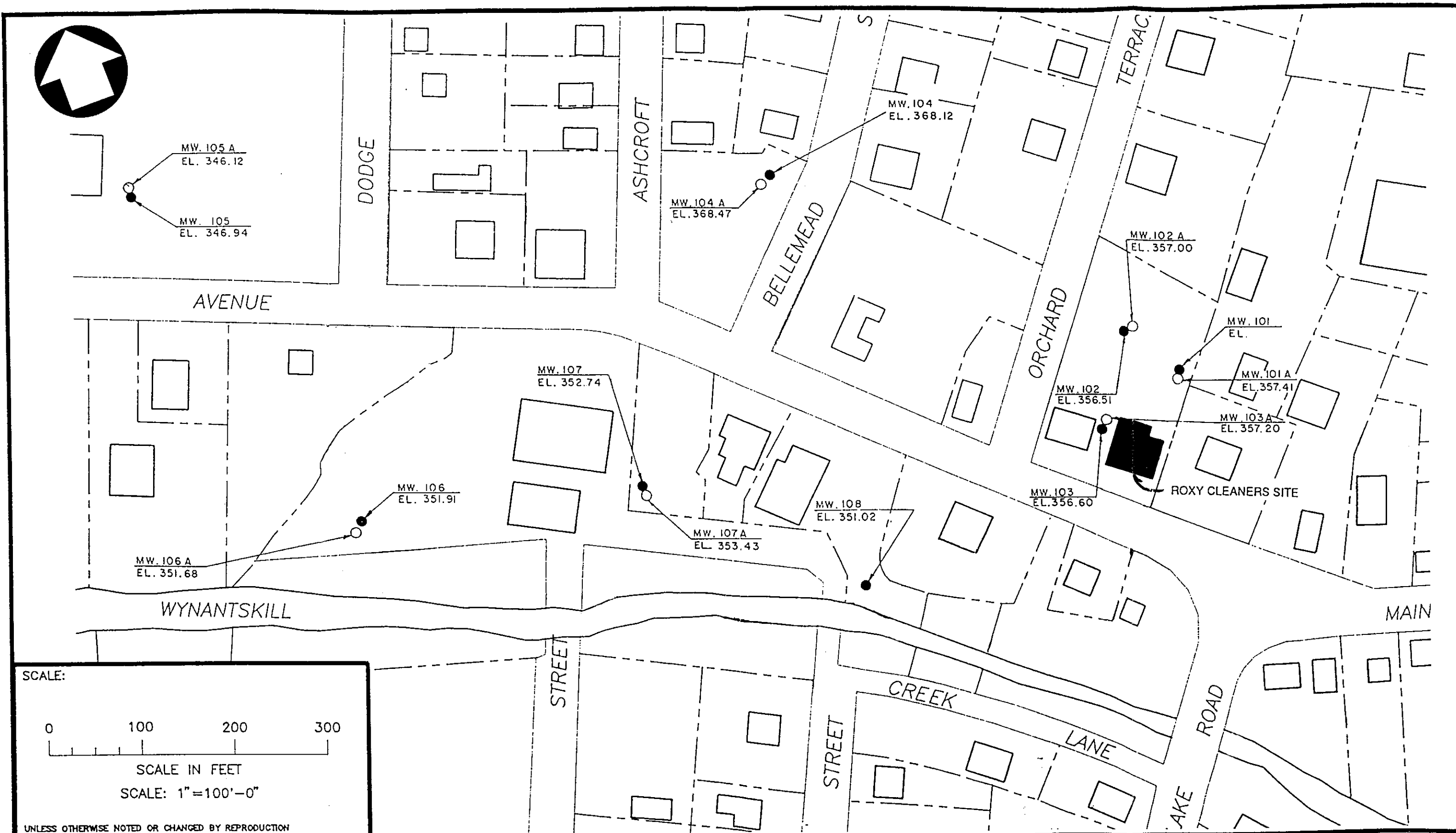
Sediment samples will be collected in conjunction with the surface water samples in order to help define the partitioning of the contaminants between soil and water. The samples will be taken at the same three (3) locations as the surface water samples. The samples will be collected directly below the sediment surface using a hand auger and stainless steel scoop.

During well drilling, three (3) borehole soil samples will be sampled for TCL Volatile Organics. Four (4) borehole soil samples will be analyzed for Total Organic Carbon (TOC). The borehole soil samples will be obtained only at the deep borehole locations of the new monitoring wells. The shallow borings shall be advanced without sampling unless requested by the NYSDEC geologist.

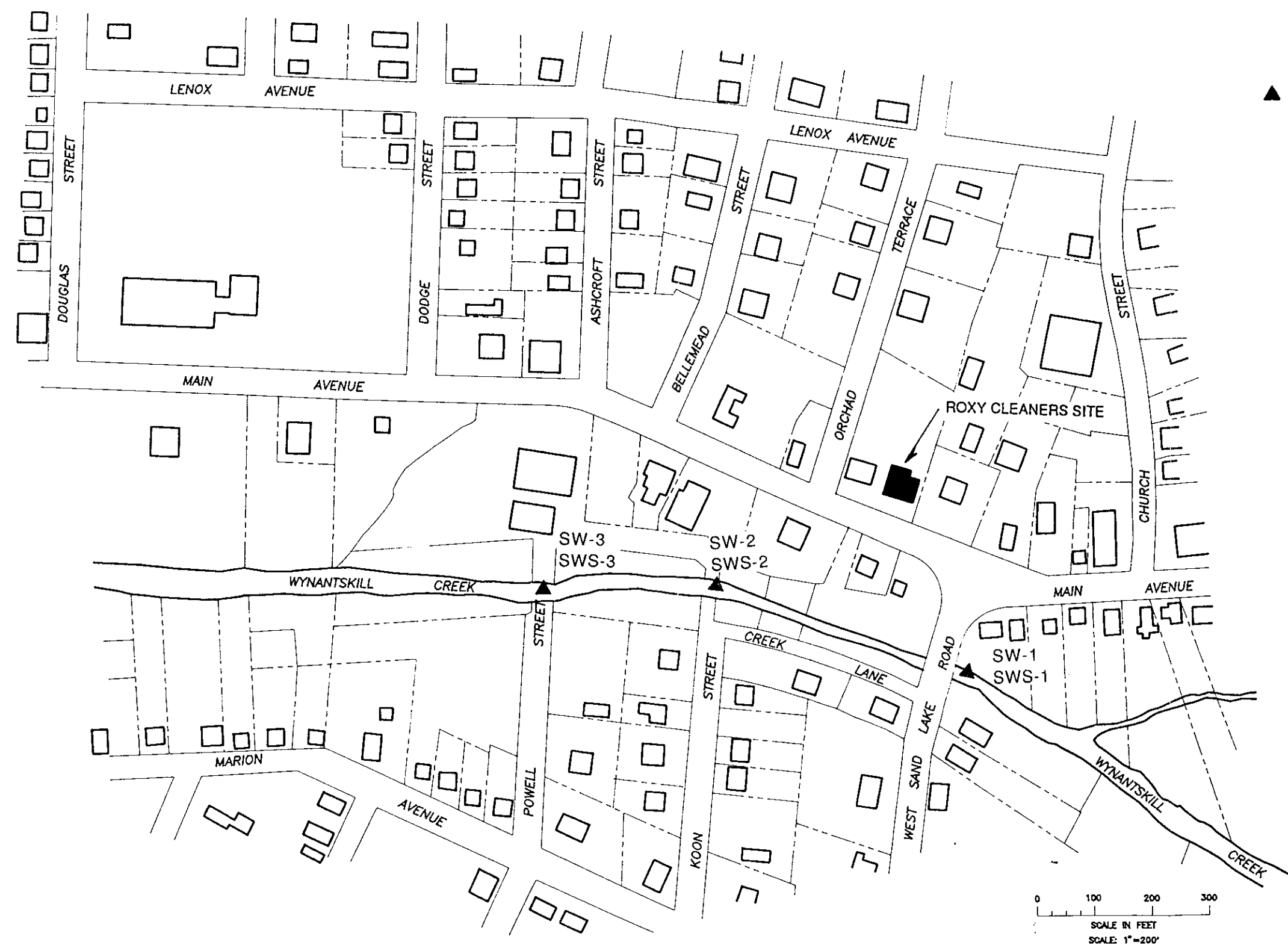
A soil gas survey will be performed at the Roxy Cleaners Site and surrounding study area. A maximum of 100 soil gas samples will be collected and analyzed, utilizing a Hewlett-Packard 5890 Series II Gas Chromatograph (CG) configured with an Electron Capture Detector (ECD) and a Photoionization Detector (PID).

2.1.1 Samples To Be Collected

Figures 2-1 through 2-3 show the sample collection locations for the project. Figure 2-2 shows the proposed locations of the new monitoring wells. The exact locations of these wells will be determined in the field by M&E site geologist with the approval by the NYSDEC site representative. Table 2-1 summarizes the types of samples that will be obtained along with a list of pertinent analysis/testing to be performed on each of these samples.







LEGEND

▲ -- Surface water (SW) and surface water sediment (SWS) sampling locations.

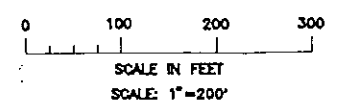


TABLE 2-1
SAMPLING SUMMARY

Activity	Number ⁽¹⁾ of Samples	Analysis
Groundwater From Existing Wells ⁽²⁾	16	TCL Volatile Organics
	6	TCL Semivolatile Organics
	6	TCL Metals
	6	TCL Pesticides/PCBs
Groundwater From Existing Wells ⁽³⁾	16	TCL Volatile Organics ⁽⁴⁾
Groundwater From New Wells	8	TCL Volatile Organics
Surface Water (Wynants Kill)	3	TCL Volatile Organics
Sediment (Wynants Kill)	3	TCL Volatile Organics
	3	Total Organic Carbon
Seep Water Samples	2	TCL Volatile Organics
Borehole Soils	3	TCL Volatile Organics
	4	Total Organic Carbon

(1) Number of samples listed does not include field duplicates and QC Samples (i.e., equipment and trip blanks, etc.)

(2) First round of sampling.

(3) Second round of sampling.

(4) Existing wells that showed non-detectable levels of contaminants in the first round of sampling may be analyzed by USEPA method 524.2 during the second round.

2.1.2 Sample Collection and Preservation Methods

Project-specific standard operating procedures for the collection of samples for both laboratory and field analyses are discussed in Section 4.0 of this QAPP. Sample preservation techniques are outlined in Table 4-3.

2.1.3 Sample Transport and Storage

Sample transport and storage will proceed following the guidelines described in detail in Section 3.1 of this QAPP.

2.2 ANALYTICAL PROCEDURES

2.2.1 Standard Analytical Methods

The standard analytical methods to be utilized for the analysis of samples collected at Roxy Cleaners Site are summarized in Table 2-2 and Table 2-3. Additional information on procedural techniques is included in the method references listed in the tables.

2.2.2 Project Specific Detection Limits

Method detection limits expected to be achieved during the analyses are shown in Table 2-4.

The ASP and SW846 provides the option to the analytical laboratory whether or not to employ analytical cleanups. Analytical cleanups are mandatory for soil samples or where matrix interferences are noted. It may be necessary to use both GPC and alumina cleanups for some samples. No sample will be diluted more than 1 to 5. The sample shall be either re-extracted, re-sonicated, re-steam distilled, etc., or be subjected to any one analytical cleanup noted in SW846 or a combination thereof. The analytical laboratory will expend such effort and discretion to demonstrate good laboratory practice and demonstrate an attempt to best achieve the method detection limit.

TABLE 2-2 ANALYTICAL PROCEDURES FOR AQUEOUS SAMPLES
(GROUNDWATER, SURFACE WATER AND SEEP)

Parameter	Method	Reference
LABORATORY ANALYSES		
TCL Metals	NYSDEC ASP/ Inorganic	Superfund NYSDEC ASP, Inorganics, NYSDEC Method
TCL Volatile Organics	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Volatile Organics, NYSDEC Method 89-1
TCL Semivolatile Organics	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Semivolatile Organics, NYSDEC Method 89-2
TCL Pesticides/PCBs	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Pest/PCBs, NYSDEC Method 89-3
Purgeable Volatile Organic Compounds	524.2/ USEPA	NYSDEC ASP, Volume 7, page D-XXVII-460

TABLE 2-3 ANALYTICAL PROCEDURES FOR SOIL/SEDIMENT SAMPLES
(BOREHOLE SOIL AND SURFACE WATER SEDIMENT)

Parameter	Method	Reference
LABORATORY ANALYSES		
TCL Metals	NYSDEC ASP/ Inorganic	Superfund NYSDEC ASP, Inorganics, NYSDEC Method
TCL Volatile Organics	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Volatile Organics, NYSDEC Method 89-1
TCL Semivolatile Organics	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Semivolatile Organics, NYSDEC Method 89-2
TCL Pesticides/PCBs	NYSDEC ASP/ Organic	Superfund NYSDEC ASP, Pest/PCBs, NYSDEC Method 89-3
Total Organic Carbon	USEPA	USEPA-Region II, Lloyd Kahn Method for Determination of Total Organic Carbon in Sediment, July 1986

TABLE 2-4 COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a)(c)(e)(f)
<u>Metals</u>				
Aluminum	NYSDEC ASP Inorganic	200	NYSDEC ASP Inorganic	20
Antimony	NYSDEC ASP Inorganic	60	NYSDEC ASP Inorganic	20
Arsenic	NYSDEC ASP Inorganic	10	NYSDEC ASP Inorganic	1000
Barium	NYSDEC ASP Inorganic	200	NYSDEC ASP Inorganic	20,000
Beryllium	NYSDEC ASP Inorganic	2	NYSDEC ASP Inorganic	0.2
Cadmium	NYSDEC ASP Inorganic	5	NYSDEC ASP Inorganic	500
Calcium	NYSDEC ASP Inorganic	5000	NYSDEC ASP Inorganic	500
Chromium	NYSDEC ASP Inorganic	10	NYSDEC ASP Inorganic	1000
Cobalt	NYSDEC ASP Inorganic	50	NYSDEC ASP Inorganic	4
Copper	NYSDEC ASP Inorganic	25	NYSDEC ASP Inorganic	2500
Iron	NYSDEC ASP Inorganic	100	NYSDEC ASP Inorganic	--
Lead	NYSDEC ASP Inorganic	5	NYSDEC ASP Inorganic	500

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a)(c)(e)(f)			
Magnesium	NYSDEC ASP Inorganic	5000	NYSDEC ASP Inorganic			500	
Manganese	NYSDEC ASP Inorganic	15	NYSDEC ASP Inorganic			1	
Mercury	NYSDEC ASP Inorganic	0.2	NYSDEC ASP Inorganic			20	
Nickel	NYSDEC ASP Inorganic	40	NYSDEC ASP Inorganic			4000	
Potassium	NYSDEC ASP Inorganic	5000	NYSDEC ASP Inorganic			500	
Selenium	NYSDEC ASP Inorganic	5	NYSDEC ASP Inorganic			500	
Silver	NYSDEC ASP Inorganic	10	NYSDEC ASP Inorganic			1000	
Sodium	NYSDEC ASP Inorganic	5000	NYSDEC ASP Inorganic			500	
Thallium	NYSDEC ASP Inorganic	10	NYSDEC ASP Inorganic			50	
Vanadium	NYSDEC ASP Inorganic	50	NYSDEC ASP Inorganic			4	
Zinc	NYSDEC ASP Inorganic	20	NYSDEC ASP Inorganic			2000	

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a)(c)(e)(f)
<u>Volatile Organics (g)</u>				
Acetone	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Benzene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Bromodichloromethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Bromoform	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Bromomethane	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
2-Butanone (MEK)	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Carbon disulfide	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Carbon tetrachloride	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Chlorobenzene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Chloroethane	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
2-Chloroethylvinyl ether	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Chloroform	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Chloromethane	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Dibromochloromethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,1-Dichloroethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,2-Dichloroethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a)(c)(e)(f)
1,1-Dichloroethene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
trans,1,2-Dichloro- ethene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,2-Dichloropropane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
cis-1,3-Dichloro- propene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
trans-1,3-Dichloro- propene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Ethylbenzene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
2-Hexanone	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Methylene chloride	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
4-Methyl-2-Pentanone (MIBK)	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Styrene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,1,2,2-Tetrachloro- ethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Tetrachloroethene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a) (c) (e) (f)
Toluene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,1,1-Trichloroethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
1,1,2-Trichloroethane	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Trichloroethene	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Vinyl acetate	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
Vinyl chloride	NYSDEC ASP Organic	10	NYSDEC ASP Organic	10
Xylenes	NYSDEC ASP Organic	5	NYSDEC ASP Organic	5
<u>Semivolatile Organics</u> ^(d)				
Base/Neutral Extractables:				
Acenaphthene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Acenaphthylene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Anthracene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzo(a)anthracene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (mg/kg) (a)(c)(e)(f)
Benzo(b)fluoranthene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzo(k)fluoranthene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzo(ghi)perylene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzo(a)pyrene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzoic Acid	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
Benzyl alcohol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Benzyl butyl phthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Bis(2-chloro- ethoxy)methane	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Bis(2-chloroethyl) ether	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Bis(2-chloro- isopropyl) ether	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Bis(2-ethylhexyl) phthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
4-Bromophenyl phenyl ether	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
4-Chloroaniline	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg) (a) (c) (e) (f)
2-Chloronaphthalene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
4-Chlorophenyl phenyl ether	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Chrysene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Dibenzo(a,h)anthracene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Dibenzofuran	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Di-n-butylphthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
1,2-Dichlorobenzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
1,3-Dichlorobenzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
1,4-Dichlorobenzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
3,3'-Dichlorobenzidine	NYSDEC ASP Organic	20	NYSDEC ASP Organic	660
Diethyl phthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Dimethyl phthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4-Dinitrotoluene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,6-Dinitrotoluene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Di-n-octylphthalate	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg)(a)(c)(e)(f)
Fluoranthene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Fluorene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Hexachlorobenzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Hexachlorobutadiene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Hexachloro- cyclopentadiene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Hexachloroethane	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Indeno(1,2,3-cd)pyrene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Isophorone	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2-Methylnaphthalene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Naphthalene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2-Nitroaniline	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
3-Nitroaniline	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
4-Nitroaniline	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
Nitrobenzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg) (a) (c) (e) (f)
N-Nitrosodi- n-propylamine	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Phenanthrene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
Pyrene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
1,2,4-Trichloro- benzene	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
<u>Semivolatile Organics</u> ^(d)				
Acid Extractables:				
4-Chloro-3-methyl- phenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2-Chlorophenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4-Dichlorophenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4-Dimethylphenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4-Dinitrophenol	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
2-Methyl-4,6-dinitro- phenol	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
2-Methylphenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
4-Methylphenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2-Nitrophenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg) (a) (c) (e) (f)
4-Nitrophenol	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
Pentachlorophenol	NYSDEC ASP Organic	50	NYSDEC ASP Organic	1600
Phenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4,5-Trichlorophenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
2,4,6-Trichlorophenol	NYSDEC ASP Organic	10	NYSDEC ASP Organic	330
<u>Pesticides/PCBs</u>				
Aldrin	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Alpha-BHC	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Beta-BHC	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Delta-BHC	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Gamma-BHC (Lindane)	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Chlordane	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0
4,4' -DDD	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
4,4' -DDE	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
4,4' -DDT	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Dieldrin	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg) (a) (c) (e) (f)
Endosulfan I	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Endosulfan II	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Endosulfan sulfate	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Endrin	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Endrin ketone	NYSDEC ASP Organic	0.10	NYSDEC ASP Organic	16.0
Heptachlor	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Heptachlor epoxide	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	8.0
Methoxychlor	NYSDEC ASP Organic	0.05	NYSDEC ASP Organic	80.0
Toxaphene	NYSDEC ASP Organic	1.00	NYSDEC ASP Organic	160.0
Arochlor-1016	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0
Arochlor-1221	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0
Arochlor-1232	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0
Arochlor-1242	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0
Arochlor-1248	NYSDEC ASP Organic	0.50	NYSDEC ASP Organic	80.0

TABLE 2-4 (Continued) COMPOUND SPECIFIC ANALYTICAL METHODS AND REQUIRED DETECTION LIMITS

Parameter	Method	Water (ug/l)	Method	Soil/Sediment (ug/kg) (a) (c) (e) (f)
Arochlor-1254	NYSDEC ASP Organic	1.0	NYSDEC ASP Organic	160.0
Arochlor-1260	NYSDEC ASP Organic	1.0	NYSDEC ASP Organic	160.0

- (a) Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the contract, will be higher.
- (b) The estimated instrumental detection limits as shown are given as a guide. The actual method detection limits are sample dependent and may vary as the sample matrix varies.
- (c) Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Volatile TCL Compounds are 125 times the individual Low Soil/Sediment CRQL.
- (d) Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for SemiVolatile TCL Compounds are 60 times the individual Low Soil/Sediment CRQL.
- (e) Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Pesticide/PCB TCL compounds are 15 times the individual Low Soil/Sediment CRQL.
- (f) Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.
- (g) Volatile organics analyzed by method no. 524.2 will have lower detection limits.

100-500 METHODS

METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES, EPA 600/4-79-020, March 1983.

1000-9000 METHODS

TEST METHODS FOR EVALUATING SOLID WASTE, SW-846, Third Edition, U.S. EPA, November 1986.

ASTM METHODS

ANNUAL BOOK OF ASTM STANDARDS, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat Materials, 1987.

NYSDEC Analytical and Services Protocols, September, 1989 "Superfund Category"

Superfund ASP - Volatile Organic NYSDEC Method 89-1.

Superfund ASP - Semi-volatile Organic NYSDEC Method 89-2.

Superfund ASP - Pesticides/PCBs NYSDEC Method 89-3.

Superfund ASP - Inorganics NYSDEC.

3.0 FIELD AND LABORATORY PROCEDURES

3.1 SAMPLE CUSTODY

An overriding consideration essential for the validation of environmental measurement data is the necessity to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of the sample traceability from collection to shipment, laboratory receipt, and laboratory custody (until proper sample disposal and the introduction of field investigation results as evidence in legal proceedings when pertinent) must be documented. A sample is considered to be in a person's custody if the sample is:

- In a person's actual possession
- In view after being in a person's possession
- Locked so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel.

The site chemist/engineer is responsible for overseeing and supervising the implementation of proper sample custody procedures in the field. He is also designated as the field sample custodian and is responsible for ensuring sample custody until the samples have been transferred to a courier or directly to the laboratory. The samples must be delivered to the laboratory within 24-48 hours of collection.

Once the samples have been received by the laboratory, a designated person is responsible for maintaining a file of all the original documents (e.g., sample labels, chain of custody forms, custody seals, traffic reports, special analytical services (SAS) request forms, etc.) pertinent to sample custody and sample analysis protocol.

3.1.1 Chain of Custody

The chain of custody procedures are initiated in the field following sample collection. The procedures consist of: 1) preparing and attaching a unique sample label to each sample collected, 2) completing the chain of custody form, and 3) preparing and packing the samples for shipment. These procedures are further described in the following sections.

Sample Labels

Field personnel are responsible for uniquely identifying, labeling and sealing all samples collected during a field investigation. All labeling must be done in indelible/waterproof ink. Any errors are crossed out with a single line, dated, and initialled. Each sample label securely affixed to the appropriate sample container must contain the following information (see Figure 3-1 for an example sample label):

- Client/site name
- Unique project-specific sample identification number (i.e., station number)
- Sample location/description number
- Type of analysis to be performed and the name of the laboratory to whom the samples are being sent
- Sample volume, container type, and the type of chemical preservation used
- Sampling date and time
- Initials of the person obtaining the sample.

Chain of Custody Record

A chain of custody form (see Figure 3-2) must be completed for each sample set collected at a sampling location. The form is maintained as a record of sample collection, transfer, shipment, and receipt by the laboratory. The forms must also contain pertinent information concerning sampling location,



Metcalf & Eddy

303 South Broadway
Tarrytown, NY 10591

Project Number _____
Unique Sample Number _____
Sample Description (such as borehole
& depth, or grid coordinates) _____
Sampling Date and Time _____
Method of Sample preservation/
conditioning, if any _____

METCALF & EDDY LABORATORY SAMPLES
CHAIN OF CUSTODY SEAL
DO NOT ACCEPT IF SEAL IS BROKEN
CALL 914/631-7273

date, and times; signatures of the sampling team members; types of samples collected along with a unique sample identification number; the number of samples collected and shipped for analysis in each lot; the project name and number; and the name of the laboratory to which the samples are being sent.

Transfer of Custody

Samples shall be accompanied by an approved and completed chain of custody form during each step of custody, transfer, and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them shall sign, date, and record the time on the chain of custody form. In the case of sample shipment by an overnight courier, a properly prepared airbill shall serve as an extension of the chain of custody form while the samples are in transit.

Laboratory Sample Custody Procedures

Upon sample receipt, the laboratory representative responsible for accepting incoming sample shipment must compare the samples received against the list on the chain of custody record and examine all the samples to determine if the proper temperature was maintained during shipment. If the samples were damaged during transfer, the remaining samples must be carefully examined to determine whether they were affected. All samples must be carefully examined to determine whether they were affected. Any samples so affected must also be considered damaged and the pertinent information noted on the chain of custody record (specifying which samples were damaged and that the samples were removed from the sampling program). Field personnel are immediately notified of any sample damage so that resampling can take place or the testing program can be changed.

The laboratory representative must also: (1) verify that sample holding times have not been exceeded, (2) sign and date the chain of custody record, (3) list the received samples in the laboratory sample master log-in book which contains the following information:

- Project identification number
- Sample numbers
- Type of samples
- Date picked by transfer personnel
- Date received in laboratory

(4) notify the Laboratory Manager of sample arrival and alert him of any analyses requiring immediate attention due to short holding time, and (5) store the samples according to the requirements of the analytical protocols.

The chain of custody records are maintained and filed in laboratory's sampling log-in area and Laboratory Information Management System (LIMS) according to contract name and project number, becoming part of the permanent project documentation. All analysts are required to sign for receipt and return of samples obtained for analysis. See Figure 3-3 for an example of a sample tracking sheet for Laboratory "XYZ".

Laboratory Sample Tracking Procedures

The laboratory uses a computerized Laboratory Information Management System (LIMS) to electronically track samples. XYZ procedures for sample tracking are described in detail in their QAPP on sample tracking procedures.

Laboratory Storage of Samples

Samples shall be maintained in storage in one of the locked storage refrigerators prior to sample preparation and analysis. The operating procedures for sample storage are described in detail in the XYZ Quality Assurance Plan contained in Appendix A. A summary of this procedure is included in the following paragraphs.

Storage refrigerators are maintained at $4^{\circ}\pm 2^{\circ}\text{C}$. The temperature is monitored and recorded daily in a bound logbook by the sample custodian. During working hours, if equipment failure (compressor failure, door left open, etc.) results in the temperature of the storage refrigerator exceeding the upper or lower

XYZ SAMPLE TRACKING SHEET

XYZ SAMPLE ID #	CLIENT ID	ANALYSIS REQUIRED	TEST CODE	FRIG	SAMPLES/ BOTTLE	COND

control limits, an audible alarm will sound and the samples will be moved to suitably controlled storage until the problem has been corrected.

Refrigerator storage is designed to segregate samples to prevent cross-contamination and to prevent sample mix-up. This includes storage of volatiles samples separate from semivolatiles and inorganics samples. Within the refrigerators, samples are stored according to CLP protocol by batch number for easy retrieval. Analysts request samples for analysis from the sample custodian and both sign the chain-of-custody acknowledging transfer of custody to the analyst.

3.1.2 Sample Packaging and Shipping

Following sample collection, all samples will be brought to an on-site location for batching and paperwork checks. At this central location, like sample types are matched (i.e., solids, liquids, etc.) with similar sample types from all sample locations. Labels and log information are checked to be sure there is no error in sample identification. The samples are packaged to prevent breakage and/or leakage, and the shipping containers are labeled in accordance with the DOT regulations for transport.

As soon as field personnel are ready to transport samples from the field to the laboratory, the laboratory shall be notified by telephone of the shipment along with the estimated time of arrival. All samples shall be shipped directly to the laboratory via overnight carrier. The field team shall determine whether it is best to directly transport the packages to the shipping office or to arrange for on-site pick-up. For each sample shipment to a specific subcontracting laboratory, an overnight airbill must be properly completed. An example of a properly prepared airbill used when shipping non-hazardous samples to the laboratory is shown in Figure 3-4.

In order to ensure safe, secure delivery of all collected samples to XYZ, the following packaging and shipping procedures have been prepared for this project. All procedures presented below are written to comply with applicable DOT regulations for transportation by surface and/or air.

Conductivity and pH meter readings shall be taken for each liquid samples. The collected data shall be recorded in the field logbook along with the documentation of the shipping procedures utilized. Any pertinent information with regard to particular sample hazards (i.e., any dangers anticipated upon opening of the sample bottle) is included on the chain of custody form.

Unless field collected information indicates otherwise, all environmental samples collected will be treated as non-hazardous aqueous liquids and non-hazardous soil.

Non-Hazardous Packaging and Shipping

Because of the expected non-hazardous nature of the collected samples, packaging and shipping criteria have been designed only to maintain chain of custody protocol, as well as to prevent breakage of the sample containers. The packaging and shipping procedures will be as follows:

- Place a layer of cushioning material (e.g., vermiculite) in the bottom of the watertight insulated metal or equivalent strength plastic shipping containers.
- Wrap the properly labeled and secured glass sample bottles and purgeable vials with plastic bubble wrap. Place the wrapped containers into watertight zip lock bags and seal the bags closed.
- Place sample bottles (top side up) into the shipping container arranging the bottles so that the glass bottles are surrounded by plastic bottles.
- Using the necessary packing material, pack the sample bottles to ensure that they do not shift during transport.
- Fill any void spaces of the shipping container, around and on top of the sample bottles, with ice cubes or chips sealed in plastic bags or with blue ice.
- Seal the appropriate chain of custody form(s) in a zip-lock plastic bag, and tape it securely to the inside of the shipping container lid.
- Close and lock/latch the shipping container. Seal the space between the container body and lid with waterproof tape. (If the shipping container used is a picnic cooler, tape the drain plug

closed to prevent any leakage of water as the ice packs melt during transport.)

- Apply several wraps of chain of custody tape around the shipping containers perpendicular to the seal to ensure that the lid remains closed if the latch is accidentally released or damaged during shipment. Do not obscure any stickers or labels on the shipping container with the chain of custody tape.
- Place a completed overnight carrier airbill (see Figure 3-4 for an example of a properly completed airbill) on the lid of the shipping container. Include the name, address, and telephone number of the receiving laboratory and the return address and telephone number of the shipper on the airbill.
- Place a "This End Up" label on the lid and on all four sides of the shipping container.
- Each shipping container must not weigh more than 150 pounds if it is to be shipped overnight by Federal Express.

3.1.3 Documentation

A simple program designed to ensure that field performed analyses yield valid, useful data is summarized in Table 3-1. In all cases, the Field Task Manager will maintain a concise, detailed field logbook containing accounts of all field activities and actions taken, as well as documentation of observations made.

All sampling procedures, instrument calibration, and information pertinent to sampling conditions, progress, and any field data collection must be documented following a prescribed set of guidelines. The documentation serves as a permanent and traceable record of all activities related to the Roxy Cleaners Site remedial investigation. The record must be legible and accessible to allow ease in verifying sampling activities and addressing future questions which may arise concerning such issues as sample integrity, sample traceability, etc.

TABLE 3-1 FIELD DOCUMENTATION OBJECTIVES FOR
VALID DATA COLLECTION

Objective	Action	Responsible Person(s)
Verify sample and location information conforms to conditions and requirements specified	Review labeled samples and in-process samples using daily sample inventory	Field sampling team
Verify incoming field data and sample completeness	Maintain daily count of incomplete items	Field sampling team
Verify completeness of field log books	Review daily	Field team leader
Review field calibration criteria and record test calibration acceptance	Perform as necessary	Field team leader
Ensure all data forms are properly completed	Review and check off during each sample collection	Field team leader
Verify all field generated QC samples were collected as required	Review requirements and confirm sample collection	Field team leader

Sample Designation/Identification

All samples will be identified by a unique sample identification number which includes the project number hyphenated with a consecutive numbering system specific to the samples collected for the project (see the sample number as listed on the Chain of Custody Form in Figure 3-2). This identification number is used to ensure sample traceability. Also, within a specific project, all samples shall be identified using a numbering system cross-referenced to descriptive sample information. The cross-index is to be designed and maintained by the field team leader. An example sample identifier used by the field team is shown in Figure 3-2 (Chain of Custody Form) as the sample location, MW-107(1), which corresponds to groundwater monitoring well number 107 (first round of sampling).

Both the unique sample identification number (sample number) and project sample identification number (sample location) will be recorded on the chain of custody forms accompanying each sample to the subcontract laboratory for sample analysis.

Daily Log

The field team leader has the responsibility to maintain the daily field documents pertaining to sample identification and control. Special emphasis is placed on logbook completeness and accuracy. Field logbooks, field data forms, and chain of custody forms must contain entries made with indelible ink and must be dated, signed, and contain statements that are legible, accurate, and an inclusive documentation of project activities. At a minimum, entries into the logbook shall include weather conditions, date and time of each sample collection, and references to any difficulties encountered and how such difficulties were resolved. Because the logbook, field data forms, and chain of custody forms provide the basis for reports written later, they must contain accurate facts and observations. Language must be objective, factual, and free of personal interpretations or other terminology that might prove inappropriate.

Corrections to Documentation

All documentation must be recorded in permanent ink. Corrections to errors in documentation or recorded calculations will be made by first striking out the error with a single line so as not to obliterate the original entry. Then the replacement entry or value shall be inserted where appropriate. The person originating the change will initial each separate change. All revisions, deletions, and changes must be made in indelible ink.

Photographs

The field team leader will document, through the use of color photography, various on-site activities and sampling specifics as deemed necessary under the Statement of Work. Examples of items that may require such photographic documentation include:

- General site topography
- Well drilling locations
- Exact sample locations, (i.e., point sampled in streams, ponds, test-pits, etc.)
- Contents of split-spoons or corings if recovered material seems unusual
- Well development water after development is complete (more particularly described in well installation plan)

Records

Field Logbooks

A field logbook shall be maintained by each field team member for this hazardous waste field investigation project. The logbook will be comprised of a bound book with consecutively numbered pages; no pages must be skipped when filling in the logbooks. The integrity of field documentation is further ensured by the use of field logbooks containing paper treated to repel the

rain or any other aqueous splashings experienced during field documentation. Should more than one field logbook be required, they will be numbered sequentially.

The front of each field logbook shall contain the following information:

- Project name and number
- Name of the contract under which the investigation is being conducted
- Date(s) of use

The field logbook will contain a diary of all pertinent project activities. Standard information recorded in the field logbook includes: general observations made in the field, identification and calibration of instruments used, and field data. See Figure 3-5 for an example of typical information that may be included at the beginning of each field logbook. Figures 3-6, 3-7, and 3-8 serve as guidelines for field logbook data entries for various sample collection procedures.

The field logbook will contain the following information:

- Date and time of personnel entries on-site, weather conditions, temperature, etc.
- List of start-stop times of all subcontractors hired for activities such as drilling, steam cleaning, well development, etc.
- List of the personnel present on-site during each sampling day including all M&E personnel, subcontractors, and visitors
- List of the equipment decontaminated along with a reference to the procedures used
- Description of the sampling locations in reference to permanent landmarks
- List of any changes from standard operating procedures, decisions made in the field, and other pertinent information
- QA/QC samples associated with the samples collected

FIELD LOGBOOK INITIAL FIELD INFORMATION

Instrument Calibration

Include a brief description of the calibration procedures used for each instrument utilized in the field and the designated pages on which daily calibration entries are made. The field instruments may include:

- HNu photoionization detector
- pH meter
- Conductivity meter
- MX 241 Combustible gas indicator

Decontamination Procedures

Include a brief description of the decontamination procedures used to clean all sampling equipment that may come in direct contact with the sample and designated pages on which daily decontamination procedure entries are made. The equipment may include:

- Pump
- Bailer
- Filtration apparatus
- Split spoon sampler
- Hand trowel
- Measurement tape (used to measure static water level)

Quality Control Sample Preparation

Include a brief description of all QC samples prepared in the field for both aqueous and soil/sediment sampling and the designated pages on which daily QC samples preparation entries are made. The QC samples may include:

- Trip blank
- Ambient conditions blank
- Equipment blank
- Field duplicates

Field Water

Include a brief description of the field water sources for tap, deionized, and organic-free deionized water. Outline the daily water usages, the applications of when each water type is used, and the designated pages on which daily use entries are made.

FIELD LOGBOOK GROUNDWATER MONITORING WELL WATER SAMPLING DATA

Date _____ Location _____

Samplers, Pumps, Bailers, Used _____

Description of sampling location _____

Weather _____

Water parameters/measurements:

pH _____

Conductivity _____

Temperature _____

Depth of well _____

Static groundwater level collection _____

Well water volume _____

Time of bailing: start _____ finish _____

Final groundwater water level _____

Air monitoring (list equipment used as well as the measured reading):

In well _____

In personnel breathing area _____

Sample description _____

Field preservation _____

Decontamination (page number reference):

Bailer _____

Measurement tape _____

Filtration apparatus _____

Pump _____

Sampler _____

Signature (of field team personnel making data entry) _____

FIELD LOGBOOK SOIL/SEDIMENT SAMPLING DATA

Date _____ Location _____

Samplers Used _____

Drawing of sampling location (including location description as well as the presence of debris, surface sheens, recent excavations, vegetation, etc.)

Weather _____

Soil/sediment sampling parameters:

Description of sample _____

Time of sample collection _____

Depth of water (for sediment sampling) _____

Decontamination (page number reference) _____

Spoons or spatulas _____

Trowel _____

Hand corer _____

Hand auger _____

Bowls _____

Split spoons _____

Photograph frame numbers _____

Signature (of field team personnel making data entry) _____

FIELD LOGBOOK SURFACE WATER AND LEACHATE WATER SAMPLING DATA

Date _____ Location _____

Samplers Used _____

Drawing of sampling location (including location description as well as the presence of debris, surface sheens, current, vegetation, etc.)

Weather _____

Water sampling parameters:

pH _____

Conductivity _____

Temperature _____

Depth of water body _____

Time of sample collection _____

Final groundwater water level _____

Field preservation _____

Decontamination (page number reference):

Filtration apparatus _____

Sampler _____

Photograph frame numbers _____

Signature (of field team personnel making data entry) _____

- Equipment and/or instrument identification numbers (if available) for those used
- Sample preservation techniques performed
- Air monitoring information gathered (e.g., HNu, radiation meter readings, etc.)
- Level of personnel protection mandated (e.g., Level B, C, D) and record of pertinent time intervals spent by each field team member at each level (e.g., time spent in Level C developing a well, time spent in Level C sampling soil from a backhoe, etc.)
- Other logs/paperwork used to document activities
- Instrument calibration information including the instruments calibrated during the day and the individual who performed the calibration (Note: Instrument calibration information should be documented in the field logbook as well as on the instrument calibration log kept with each instrument and serving to document instrument response over time.)
- List of the samples collected by media (i.e., soil, water, etc.)
- Comments relative to any problem areas that occurred during the day's activities, their final resolution, and any anticipated impact on the outcome of the field investigation

The last item to be entered at the bottom of each page of the field logbook shall be the signature of the person responsible for completing the data entry.

Project Logbook

A unique project-specific logbook shall be maintained for the entire hazardous waste field investigation. The logbook shall be comprised of a bound book with consecutively numbered pages. Should more than one project logbook be required, they will be numbered sequentially.

The primary purpose of this logbook is to contain all the data specific to a particular field activity in one central location. In general, the project logbook shall contain essentially the same information as the field logbook. However, the project logbook need not list all the actual field data and

instrument calibration information generated during a sampling episode. References to other documents that contain specific field activity descriptions, outlines of any administrative occurrences that have affected the field work for any given sampling activity descriptions, outlines of any administrative occurrences that have affected the field work for any given sampling activity, as well as a summary of the field activities, must be documented in the project logbook and kept up-to-date on a daily basis.

Field Equipment Logbook

A unique field equipment logbook is required for each piece of field equipment (i.e., the HNu photoionization detector, pH meter, conductivity meter, etc.). This logbook is used for the documentation of the proper use, maintenance, and calibration of the field equipment as well as for any information concerning the conformance and decontamination status of each piece of equipment.

The field equipment logbook is maintained and kept up-to-date following every use, maintenance, inspection, repair, and/or calibration of the equipment. Entries into the logbook should contain the following information:

- Signature of the person making the entry
- Date of entry
- Status of equipment in terms of its operational and decontamination standing
- Reference to the procedures used for calibration or maintenance as well as the procedural results and/or description
- Name of person(s) using the equipment and a brief description of the nature of the work
- Calibration log forms used to record all on-site calibrations

The field equipment logbook is comprised of a 3-ring notebook. Every page in the logbook must be signed by the field equipment maintenance person responsible for keeping each piece of equipment in proper repair and

X

maintained for field use as a reflection of their review and approval of each entry.

Field Data Forms

Along with the completion of data entry in each of the above-mentioned logbooks, field data forms should also be completed and filed in a 3-ring notebook maintained at the sampling site for all field activities.

These forms include the following:

- Site Health and Safety Log - to maintain accurate health and safety records for each field team member
- Geologic Boring Log - to document soil boring operations
- Rock Coring Log
- Monitoring Well Installation Form
- Monitoring Well Development Form
- Monitoring Well Sampling Worksheet

Chain of Custody Record

Possession of samples shall be traceable from the time a sample is collected until it is used as evidence in legal proceedings, if applicable. To adequately track sample possession, a documented chain of custody must be maintained. See Section 3.1.1 for more detailed guidelines concerning sample custody.

3.2 CALIBRATION PROCEDURES AND FREQUENCIES

3.2.1 Laboratory Instrumentation

This section describes calibration procedures and policies pertinent to the analysis of samples collected at the site under this investigation. All

instruments shall be calibrated according to the guidelines specified in the NYSDEC Analytical Services Protocol (ASP).

Instruments and equipment used at XYZ Laboratory are controlled by a formal calibration program as outlined in Tables 3-2 and 3-3. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration.

Before any instrument is used as a measurement device, the instrumental response to known reference materials must be determined. The manner in which various instruments are calibrated is dependent on the particular type of instrument and its intended use. All sample measurements are made within the calibrated range of the instrument. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook.

Instrument calibration typically consists of two types: initial calibration and continuing calibration. Initial calibration procedures establish the calibration range of the instrument and determine instrument response over that range. Typically, three to five analyte concentrations are used to establish instrument response over a concentration range. The instrument response over the range is generally absorbance, peak height, etc., which can be expressed as a linear model with a correlation coefficient (e.g., for Atomic Absorption, Inductively Coupled Plasma, UV-Visible-Infrared Spectrophotometry, Ion Chromatography) or as a response factor or amount vs. response plot (e.g., for Gas Chromatography, Gas Chromatography/Mass Spectrometry, High Performance Liquid Chromatography).

Continuing calibration usually includes measurement of the instrument response to fewer calibration standards and requires instrument response to compare with certain limits (e.g., $\pm 10\%$) of the initial measured instrument response. Continuing calibration may be used within an analytical sequence to verify stable calibration throughout the sequence, and/or to demonstrate that

TABLE 3-2 CALIBRATION FREQUENCY AND MECHANISM FOR
MAJOR INSTRUMENTS AT XYZ LABORATORY

Instrument	Frequency of Calibration	Standard	Indicator Parameters
GC/MS	Daily (or every 12 hrs)	Standard soln. of analytes to be measured	Response- Sensitivity
GC (Hall, PID, EC, NPD, FID, FPD)	Daily (or more frequently as required)	Standard soln. of analytes to be measured	Retention Time Response- Sensitivity
AA	Daily (or more frequently as required)	Standard soln. of analytes to be measured	Response Linear Range
ICP	Daily (or more frequently as required)	Standard soln. of analytes to be measured	Response Linear Range
Ion Chromatograph	Daily	Standard soln. of analytes to be measured	Retention Time Response
Spectro- photometers	Daily	Standard soln. of analytes to be measured	Response Linear Range
Technicon Auto Analyzer	Daily	Standard soln. of analytes to be measured	Response Linear Range
PH Meter	Daily	Standard buffer solutions	Response and Linearity
Conductivity Meter	Daily	Standard soln. of analytes to be measured	Response
Analytical Balance	Daily, when used	Class S wts. (NBS cert)	Accuracy
Ovens	Quarterly	Thermometer (NBS-cert)	Accuracy

TABLE 3-3 GC/MS CALIBRATION CHECK COMPOUNDS (CCC)
AND SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCC)

Volatiles:	Semivolatiles:
# chloromethane	* phenol
* vinyl chloride	* 1,4-dichlorobenzene
* 1,1-dichloroethene	# N-nitroso-di-N-propylamine
# 1,1-dichloroethane	* 2-nitrophenol
* chloroform	* 2,4-dichlorophenol
* 1,2-dichloropropane	* hexachlorobutadiene
# bromoform	* 4-chloro-3-methylphenol
# 1,1,2,2-tetrachloroethane	# hexachlorocyclopentadiene
* toluene	* 2,4,6-trichlorophenol
# chlorobenzene	* acenaphthene
* ethylbenzene	# 2,4-dinitrophenol
	# 4-nitrophenol
	* N-nitrosodiphenylamine
	* pentachlorophenol
	* fluoranthene
	* benzo-a-pyrene

* = calibration check compounds

= system performance check compounds

instrument response did not drift during a period of non-use of the instrument.

Routine analytical services are performed using standard EPA/ASP-approved methodology. In some cases, modification of standard methods may be necessary to provide accurate analyses of particularly complex matrices. When modifications to standard analytical methods are performed, the specific alterations, as well as the reason for the change, will be reported with the results of analysis.

The laboratory's reported method detection limits (MDLs) are based on program requirements, sample matrix, and in-house instrument capabilities. These MDLs may be higher than published method detection limits. However, published MDLs are generally determined using clean matrices which are free of interferences, such as deionized water, and which are analyzed under optimal laboratory conditions. For actual sample analysis, these MDL's may not be routinely achievable. Procedures in place to demonstrate that reported detection limits are obtainable, are described in the ensuing subsections of this section.

Individual sample detection limits may vary from the laboratory's routinely reported detected limits. This may be due to dilution requirements, variability in sample weight or volume used to perform the analysis, dry weight adjustment for solid samples, the presence of analytical background contaminants, or other sample-related or analysis-related conditions.

Balances

Laboratory balances are calibrated and serviced annually by a factory representative. In addition, the analyst must check the balance before each use with two masses: one in the gram range and one in the milligram range. A record of calibrations and daily checks is kept in the balance log.

The Class P weights used by the analysts for daily balance checks are calibrated annually against a set of Class S certified weights.

Thermometers

Oven and refrigerator thermometers are calibrated annually against a National Bureau of Standards (NBS) certified thermometer in the range of interest. Annual calibrations are recorded in a calibration notebook. Daily readings are recorded with the respective oven or refrigerator.

3.2.2 Field Test Equipment

Before any test equipment is used in the field, proper calibration must be ensured. Specific calibration procedures for various instruments are described in detail in the following sections, as well as the instrument operations manuals contained in Appendices B and C.

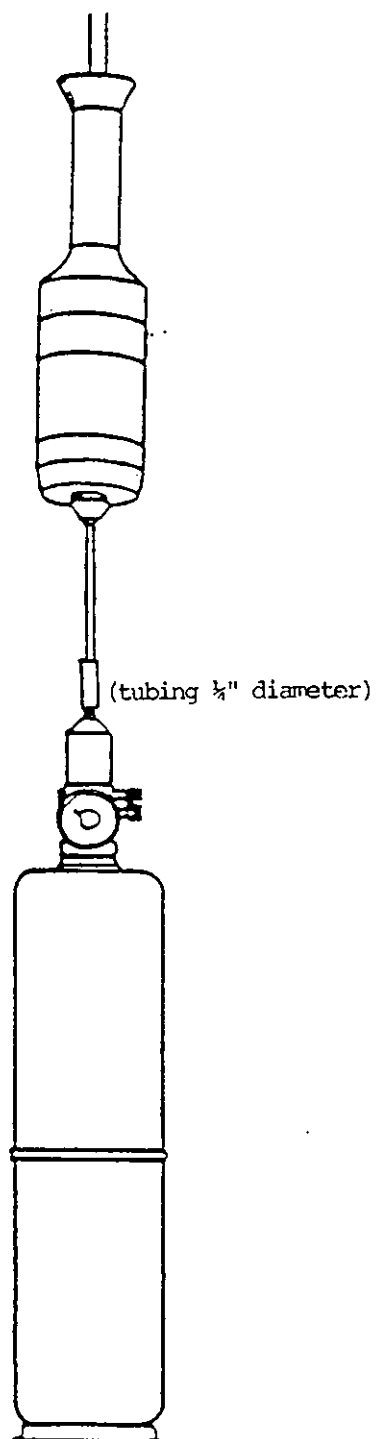
Photoionization Detector

The photoionization detector (PID) shall be used in the field to monitor volatile organic compound (VOC) vapors and to screen for the magnitude of VOC contamination in soils. The PID shall be used to detect the vertical extent of contamination and also to determine if cuttings from the wells require containment as a hazardous material and whether or not the sample should be collected for laboratory analysis for VOCs.

When calibrating the PID, the gas standards used for calibration should be at ambient temperature and pressure, and at the proper flow rate. The frequency of calibration is dictated by the frequency of use and the toxicity of the species measured. After the analyzer has been serviced or repaired, it should be calibrated to verify proper operation and performance. It is recommended that calibration be checked frequently at first (daily or every other day) and then regularly as the confidence in the instrument increases.

An accurate and reliable method of checking the calibration is to use an analyzed gas cylinder in a test set-up as shown in Figure 3-9 and described in the paragraphs to follow. Additional material on calibration is given in the instrument operations manual.

PID CALIBRATION TEST SET-UP



Calibration of the PID by the Analyzed Gas Cylinder Method

The analyzed gas cylinder is a compressed gas cylinder containing the species of interest at known concentrations in an air matrix at or near the concentrations expected in the field. If the species of interest are unstable in air, another gaseous matrix shall be used. If the expected concentrations are wholly unknown, then a concentration should be chosen that causes a scale displacement of 50 to 80% on the X10 range. Calibration on the X10 range provides accurate values on the X1 range as well.

A two-standard calibration is preferred when using the 0 to 2000 ppm range: one at 70 to 85% of the linear range and the other at 25 to 35% of the linear range. With a linear range of approximately 600 ppm for most compounds, these points would lie between 420 to 510 ppm and 150 to 210 ppm, respectively.

The calibration gas must be stable within the cylinder during the period of use. If the calibration is required in the field, a small cylinder is recommended. The cylinder material must be compatible with the calibration gas. The cylinder must be equipped with a regulator which fits properly and is also compatible with the calibration gas. The operator should contact the supplier in the event of uncertainties. Extreme care must be taken in the handling of gas cylinders; the contents are under high pressure and, in some cases, the contents may be hazardous. Many gas suppliers provide data sheets for the mixtures upon request.

One method of sampling the calibration gas is illustrated in Figure 3-9. The cylinder is connected to one leg of a tee, a flow meter to the opposite leg, and the probe to the third leg. The flow meter does not require a valve (If there is a valve, it must be left wide open.) and is used only to indicate any excess gas flow. All connections as well as the main cylinder valve must be leak-tested to prevent toxic or hazardous materials from leaking into the work area. The flow from the regulator is adjusted such that only a small amount of excess flow is registered at the flow meter. This adjustment insures that the calibration gas flowing to the PID is at atmospheric pressure and is close to the ambient temperature.

A gas calibration cylinder should not be used below a pressure of 200 to 300 psi, because the loss of pressure could cause the concentration to vary. The cylinder should not be used past the recommended shelf-life of its contents.

The Probe

The PID probe must be set-up properly prior to the overall instrument calibration. The required steps to follow in this procedure are outlined below:

1. Identify the probe by the lamp label. The energy of the lamp is etched into the glass envelope.
2. Connect the probe to the readout assembly, making sure the red interlock switch is depressed by the ring on the connector.
3. Set the SPAN potentiometer to the proper value for the probe being calibrated (refer to the calibration memo accompanying the probe).
4. Check the Ionization Potential (IP) of the calibration gas to be used and verify that the IP of the calibration gas is at or below the IP of the lamp.
5. Proceed with the calibration as described below.

Calibration Method

The HNu will be prepared for use each day according to the following procedure:

1. WARM-UP: 5-10 minutes.
2. BATTERY CHECK: Turn the function switch to the battery check position. The needle should reach within or above the green area on the scaleplate. If the needle is in the lower portion of the battery arc, the instrument must be recharged prior to making any measurements. If the red LED comes on, the battery must be recharged.
3. CHECK UV LIGHT SOURCE: Turn the function switch to the "on" position. In this position, the UV light source should be on. Check this by looking quickly into the end of the probe to see the purple glow of the lamp.

4. ZERO INSTRUMENT: Turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. (Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotation yields a d# as necessary.)

0-2000 Range: The instrument is first calibrated with the higher concentration standard using the span potentiometer, then the instrument is calibrated at the lower concentration using the zero potentiometer. These steps should be repeated several times to ensure that a good calibration is obtained. If the analyzer is to be used subsequently on the 0-20 or 0-200 range, it must be recalibrated as described above.

6. RECHARGING INSTRUMENT: The PID must be recharged overnight after each day's use. To charge the battery, place the mini phone plug into the jack on the left side of the bezel and plug the charger into a 120 VAC source. (NOTE: When disconnecting the charger, remove it from the 120 VAC source before removing the mini phone plug.) The battery completely recharges in 14 hours. To ensure that the charger is functioning properly, turn the function switch to the battery check position, place the phone plug into jack, and plug the charger into an AC outlet. The needle reading should go upscale if the charger is working properly and is correctly inserted into the jack.

Calibration Check

A short-cut method may be used to quickly check the calibration of the PID in the field. Immediately after a calibration has been completed, a reading is taken on an isobutylene standard. The reading obtained is used as a benchmark throughout the day. Readings of the isobutylene standard may be taken and adjustments to the PID may be made as required. This is an indirect method of calibration, one that maintains the calibration on the isobutylene standard to indicate the fidelity of the calibration using the original gas mixture.

pH METER

A field pH meter shall be used throughout this investigation in order to determine the acidity of various water samples prior to, during, and upon final sample collection. Calibration of the pH meter shall proceed as described below.

All pH buffer solutions used to calibrate field pH meters are typically purchased from Fisher Scientific Company. These buffer solutions, manufactured in Fairlawn, New Jersey, are traceable to the National Bureau of Standards.

Two-Buffer Calibration

This procedure is recommended for precise measurement and is as follows:

1. Choose two buffer solutions which bracket the expected sample pH. The first should be pH 7 and the second appropriate for the expected sample pH (pH 4 or pH 10).
2. The buffer solutions should be at room temperature (if the samples are at varying temperatures, the appropriate temperature compensation, as described in the instrument instruction manual, is recommended).
3. Rinse the electrode with distilled water and blot dry.
4. Place the electrode in the pH 7 buffer solution. Wait for a stable display. Set the meter to the pH value of the buffer at its measured temperature. (A table of pH values at various temperatures is supplied with the buffer solution.)
5. Rinse the electrode with distilled water and blot dry.
6. Place the electrode in the second buffer solution. When the display reading is stable, set the meter to the actual pH value of the buffer as described in the meter instruction manual.
7. Rinse electrode with distilled water and blot dry.
8. Place the electrode in the sample solution. When display reading is stabilized, record the displayed pH.

Conductivity Meter

A field conductivity meter shall be used throughout this investigation in order to determine the relative conductivity of various water samples prior to, during, and upon final sample collection. Initial calibration checks are performed on the conductivity meter at M&E prior to use at the project site. Continuing calibration is performed in the field at regular intervals during the course of the investigation. (Typically, calibration is checked at each test site or every 4 hours, whichever is more frequent.) Final calibration checks shall be performed at M&E following use at the project site. If the initial and final calibration checks differ by more than 15 to 20 percent, reanalysis of the field samples is required.

All standard conductivity solutions used to calibrate field conductivity meters shall be traceable to the National Bureau of Standards.

Initial (and Final) Calibration

A full range calibration check is performed on the conductivity meter prior to and following each field investigation. Typically, four standard conductivity solutions ranging from 10 to 10,000 mmhos/cm, one for each instrumental analytical range, are analyzed. One standard, generally the 10 mmhos/cm concentration, is analyzed and the meter is adjusted to read 10 mmhos/cm on the lowest range setting. Then 100, 1,000, and 10,000 mmhos/cm standards are analyzed on their appropriate range settings and the results are reported. The instrument is presumed to be operating correctly if the initial and final calibration difference is less than 20 percent. (Note: Care must be taken to rinse the probe with deionized water and plot it dry between analyses.)

Continuing Calibration

Continuing calibration is performed in the field at a frequency depending on the sample concentration range. Typically, a sample is analyzed to determine the appropriate instrumental range setting. Then, while at this setting, the appropriate calibration standard is analyzed, the instrumental range is

fine-tuned, and the sample is reanalyzed. (Note: Care must be taken to rinse the probe with deionized water and blot it dry between analyses.)

3.2.3 Standards and Reagents

Primary sources of standard reference materials used for calibration, calibration checks, and accuracy control are the USEPA and National Bureau of Standards repositories. Reliable commercial manufacturers represent a secondary source. Certain projects, especially those involving pesticide registration, may necessitate the use of reference standards supplied by the client.

New standards are routinely validated against known standards that are traceable to EPA or NBS reference materials if possible.

Reagents used in the preparation of matrix spike, surrogate standard, and internal standard spiking solutions for ASP work are validated using standards obtained directly from EPA or traceable to EPA. Quality Control Check Samples from the EPA EMSL Quality Assurance Branch in Cincinnati are routinely requested, received, and analyzed by the XYZ Laboratory.

Standards are periodically analyzed for concentration changes and visually inspected for signs of deterioration such as color change and precipitate formation. A Standards Preparation Logbook, which contains all pertinent information regarding the source and preparation of each analytical standard, is maintained by XYZ laboratory.

3.2.4 Soil Gas Survey Equipment Calibration

Analytical Methods

Because of anticipated interference from hydrocarbon contamination originating offsite, two detectors will be used for the analyses. A Hewlett-Packard 5890 Series II Gas Chromatograph (GC) will be configured with an Electron Capture Detector (ECD) and a Photoionization Detector (PID). It is anticipated that

the GC will be equipped with an injection tee (see Supelco catalog part #2-3664 M), allowing one injection which will split into two capillary columns. However, if this is not possible, one injection will be made into each column using two syringes of equal volume. A 30 meter (0.52 millimeter inside diameter) VOCOL column with a 3.0 micron film thickness will be installed to the ECD (Supelco catalog #2-5320). A 30 meter SPB-5 column (0.53 millimeter inside diameter, 1.50 micron film thickness) will be installed to the PID. Two HP3396A integrators will be used to record detector response. Helium will be the carrier gas with Nitrogen added as a makeup gas for the ECD. It is anticipated that the GC will be operated in the splitless mode. Flow rates, temperature ramps, and column head pressures will be adjusted to maximize chromatographic separation. Once the operating conditions are established, all subsequent analyses will proceed according to the set parameters. All operating conditions, including temperature programming information, gas flow rates, column head pressures, attenuation settings, and PID voltage settings, will be recorded in water proof ink in a sequentially bound laboratory notebook. Additional information, including instrument serial numbers and types, will also be recorded in the notebook.

Initial Calibrations

The analyte of interest is perchloroethylene (PCE). Additionally, benzene, toluene, ethylbenzene and total xylenes (BTEX) are possible contaminants that may be detected from an offsite gasoline leak. Initial calibrations will include the following:

Analytes	Concentrations	Vendor/Source
Benzene	9.44 ppm	Scotty
	0.941 ppm	Scotty
Ethylbenzene	9.860 ppm	Scotty
	0.901 ppm	Scotty
M-xylene	9.440 ppm	Scotty
	0.942 ppm	Scotty
O-xylene	9.790 ppm	Scotty
	0.962 ppm	Scotty
P-xylene	9.980 ppm	Scotty
	0.957 ppm	Scotty
Perchloroethylene	1.000 ppm	NIST
	0.100 ppm	NYSDEC

The initial calibration will include a three point calibration using two gas standards in nitrogen and zero air. Two curves will be generated from the BTEX standard and PCE standard. The linear regression coefficient will be greater than 0.995 using area or height versus concentrations of analyte. Using the line generated from the initial calibration ($y=a+bx$), sample concentrations will be determined from the area or height response. Dilution factors will be used, if appropriate, for the final report.

In the event that a sample falls outside the high point on the calibration curve (1 ppm for PCE and 10 ppm for BTEX), the location will be resampled and a smaller volume injected into the instrument for analysis.

Continuing Calibrations

At the beginning of each day, or every twelve hours, a continuing calibration sample will be injected for BTEX and PCE in concentrations of approximately 1 ppm and 0.100 ppm, respectively. The measured concentration, as determined

from the initial calibration curve, will not vary more than plus or minus twenty percent. In the event the twenty percent criterion is exceeded, a new initial calibration curve must be generated and the continuing calibration reanalyzed.

Quality Control Samples

Ambient air samples will be obtained by loading ten microliters of air in an area next to the first probe location of each day into a Hamilton gas tight syringe. Ten microliters of zero air will be injected at the beginning of each day and every twenty samples thereafter as a laboratory blank. An equipment blank will be obtained by drawing ten microliters of ambient air through the sampling train and analyzing it as a regular sample.

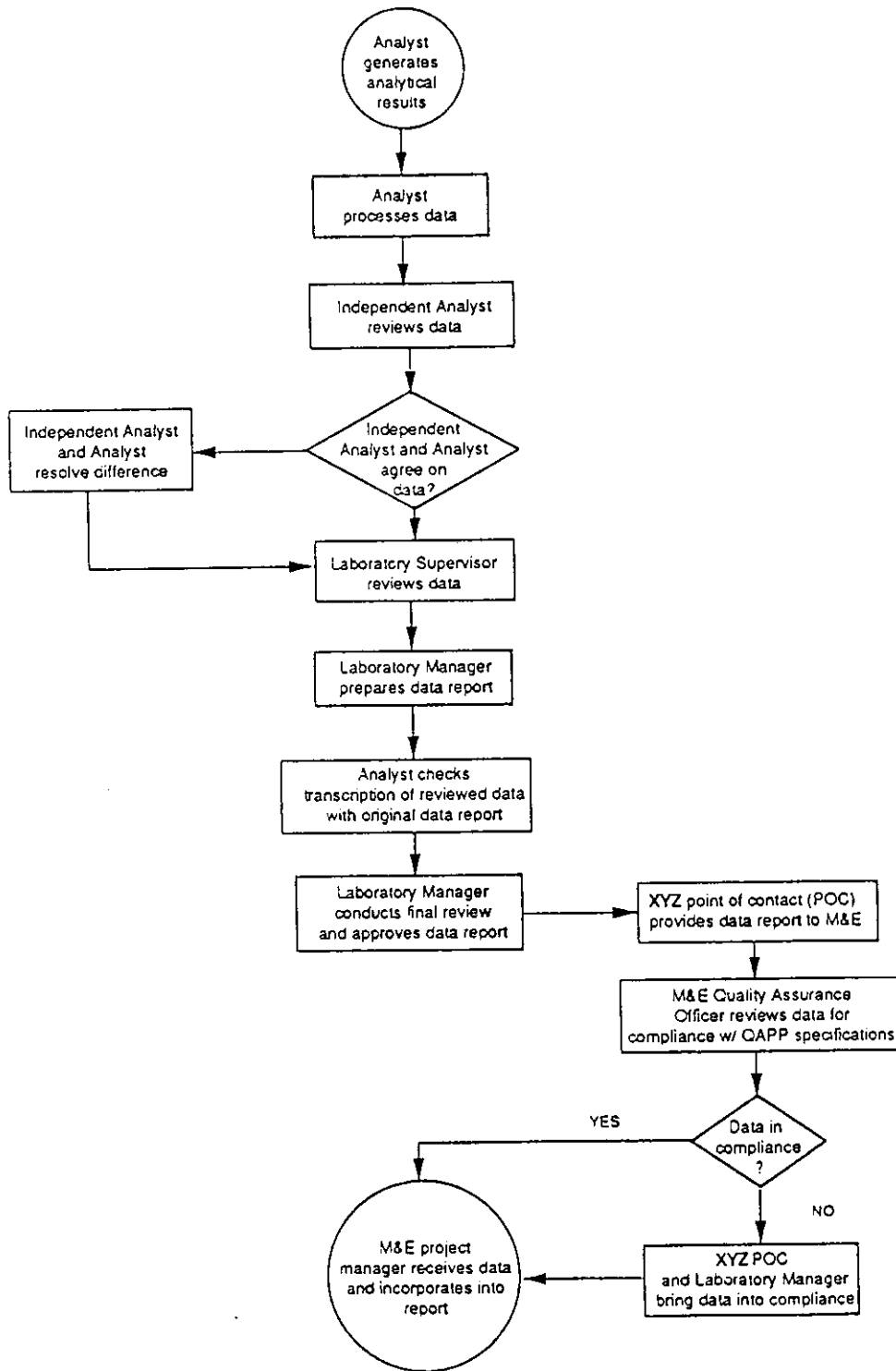
3.3 DATA COLLECTION, REDUCTION, VALIDATION, AND REPORTING

3.3.1 XYZ Procedures

This section describes data reduction, validation, and reporting procedures which shall be used at XYZ, the subcontract laboratory participating in this project. A flow chart outlining the steps which shall occur during the data review process is shown in Figure 3-10. Primary responsibility for implementation of these procedures within the laboratory resides with the Laboratory Manager. The Laboratory Manager conducts the final review and approves of all data reports before transferring the information to the Point of Contact (POC) Officer within XYZ. The POC Officer in turn, communicates with the M&E Project Manager and QA Project Officer in an effort to review the laboratory data for compliance with QAPP specifications. Final responsibility for validation and reporting of data resides with the M&E QA Project Officer.

Data processing begins with the collection of data and continues through the reduction and validation processes to the final data reporting procedure. Discussion of this process is outlined in the following sections.

DATA REVIEW PROCESS FLOWCHART



Data Collection

At XYZ, all analytical data are recorded in bound laboratory notebooks issued by the QA coordinator. Data recorded shall at a minimum contain: a unique sample identification number, a project sample identity, the analytical method used, the analyst's signature and data of analysis, reagent concentrations, instrument settings (where applicable), raw data, and any comments or descriptions pertinent to the analysis. Sample preparation forms shown in Figures 3-11 through 3-17 shall be included either in the case narrative or with the data package.

The laboratory analysts must sign and date all notebook entries on a daily basis. The notebooks shall be available for periodic review by the appropriate laboratory section manager.

Copies of all instrument outputs (chromatograms, strip charts, etc.) and computer-generated data shall be maintained in a project-specific file and be easily available for review.

Computerized internal checks may be used to avoid errors in the laboratory data collection process and to ensure analytical accuracy. These checks may include instrumental tuning and performance evaluation; initial and continuing calibration verification; analysis of matrix spikes and matrix spike duplicates, surrogate spike recoveries, duplicate samples, and field, method, and equipment blanks; and verification of analysis performance within the allotted sample holding time.

Data Reduction

Data reduction includes the process of validation, reduction, and verification. Data reduction may also include statistical and mathematical analysis of the raw data in order to reduce the size or dimensionality of the data set.

SAMPLE IDENTIFICATION AND ANALYTICAL REQUIREMENT SUMMARY

[illegible]

*Check Appropriate Boxes

*CLP, Non-CLP

*HSL, Priority Pollutant

B/N-A ANALYSES

[illegible]

ORGANIC ANALYSES

[illegible]

VOA
ANALYSES[illegible]

SAMPLE PREPARATION AND ANALYSIS SUMMARY
PESTICIDE/PCB
ANALYSES

[illegible]

INORGANIC ANALYSES

[illegible]

INORGANIC ANALYSES

[illegible]

Initial Data Reduction

Initial data reduction is performed by the individual analysts and consists of calculating concentrations in samples from the raw data obtained from the analytical instruments. The complexity of the data reduction depends on the specific analytical method and the number of discrete operations (i.e., extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured.

Initial Data Validation

Validation is achieved by ensuring that the internal checks in the data collection system are being addressed properly. Chromatographic determinations shall be screened manually according to ASP guidelines. Actual in-depth validation shall be performed using "Region II Laboratory Data Validation, Functional Guidelines for Evaluating Organic and Inorganic Analyses." The reviewer shall make professional judgment and express concerns and comments on the validity of this data package.

The initial data validation shall be performed by the XYZ Quality Assurance Manager or his designee. The information generated during the validation shall be used as an internal quality assurance check only and shall not be included in the deliverables to be sent to the client unless specifically requested.

Data Verification

The verification process addresses the chain-of-custody protocol of each sample (as described in NEIC Policies and Procedures EPA-330/9-78-001-R, revised February 1983) which sets guidelines for sample traceability from field sampling through data collection and validation. This protocol verifies that the data reported for each sample corresponds to the appropriate site sampled. A software package may be used to retrieve appropriate information from the computer mainframe and produce a concise and accurate flow chart of

sample trafficking information. The use of such a computer package eases the task of manual review and time constraints of informational access.

Data Review/Validation

Data validation is an essential element of the QA evaluation component. Validation is the process of data review and subsequent acceptance or rejection based on established criteria. System reviews are performed at all levels. The individual analyst constantly reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews are further verified by an independent analyst or unit leader prior to data submission to the Section Managers or the Analytical Project Manager. The independent analyst shall be given the entire data package including all the raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters (e.g., response factor), etc. generated during sample analysis. The reviewing analyst (checker) shall validate the data by checking the appropriateness of the equations used, the correctness of numerical input, the numerical correctness of all calculations (by reperforming approximately 20% of all numerical computations), as well as the correct interpretation of strip charts, etc.

Raw data that is automatically acquired and processed does not require any validation at this point beyond that previously discussed.

The data package is then submitted to the appropriate Laboratory Section Manager and/or the Analytical Project Manager for subsequent review for consistency and reasonableness with other generated data. The reviewing manager shall also determine if the project requirements have been satisfied. Selected hard-copy output of data (chromatograms, spectra, etc.) are reviewed to ensure that results have been interpreted correctly. Unusual or unexpected results are reviewed, and a resolution is made as to whether the analysis should be repeated. In addition, the Analytical Project Manager or Section Manager recalculates selected results to verify the calculation procedure. If, during the checking process, errors are determined, the checking process shall be expanded to include the entire data set.

The Quality Assurance Officer independently conducts a complete review of selected projects to determine if laboratory and client quality assurance/quality control requirements have been met. Discrepancies shall be reported to the appropriate Section Manager and/or Analytical Project Manager for resolution.

The final routine review is performed by the Laboratory Manager prior to reporting the results to the client. Non-routine audits are performed by regulatory agencies and client representatives. The level of detail and the areas of concern during these reviews are dependent on the specific program requirements.

The following criteria are employed by XYZ in the evaluation of data:

- Accuracy requirements
- Precision requirements
- Detection limit requirements
- Completeness
- Representativeness
- Correctness (of manual and computer calculations)
- Contractual requirements
- Documentation requirements

As in the case of ASP procedures, data acceptance limits may be defined within the method. The same windows are used for similar types of analyses if the sample matrix permits. As a tracking mechanism of data acceptability, quality control charts may be plotted for specific parameters determined in the identical, homogeneous matrices. Control limits for methods development and research data may be statistically determined as analytical results are generated.

Validation includes data review at both the technical and editorial levels. Technical review evaluates the application of analytical protocols and resultant effects on the data generated. Editorial review assesses the content, lucidity, conciseness, and completeness of the data report.

Data Reporting

Initial Laboratory Data Report

Reports shall contain final results (uncorrected for blanks and recoveries), methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported must be consistent with the limits of uncertainty inherent in the analytical method; most analytical results are reported to no more than two (2) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight per unit volume (e.g., micrograms per liter). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per gram).

Report Review

Review of data reports is required to verify that information reported by XYZ corresponds with processed analytical results. Review is only required of the data as it is presented for issuance. Preparation of the report is the responsibility of the Laboratory Manager or his designated representative.

After the draft data report is prepared, the reported results should be checked against the reviewed processed data so that transcription errors do not occur. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.

The draft data report should be reviewed to ensure that the items cited for data presentation in Sections 3.3.1 and 3.3.2 are complete and correct.

If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout. If

X

computer output is used directly as the data report without further transcription, only the input requires review.

After the reviewer has checked the data report, the report shall be given to the Laboratory Manager or his designated representative for final review. This step is not intended to verify any reported data; it serves as a final check to determine that the report is complete and meets project requirements. The data report is approved for issue by the Laboratory Manager.

Final Laboratory Data Report

The final presentation shall be checked in accordance with data verification requirements specific for this project and approved by the Laboratory Manager. Data shall be presented in a tabular format whenever possible and shall be formatted as a standard lab report. An example is shown in the XYZ Quality Assurance Plan as included in Appendix A.

At a minimum, data presentation shall include:

- Sample identification number used by XYZ and/or the sample identification provided to the laboratory, if different than identification used in the laboratory
- Chemical parameters analyzed, reported values, and units of measurement
- Detection limit of the analytical procedure if the reported value is less than the detection limit
- Data for a chemical parameter reported with consistent significant figures for all samples
- Results of Quality Control sample analysis if appropriate
- Achieved accuracy and precision of data
- Footnotes referenced to specific data if required to explain reported values

If explanatory text is not issued with the analytical results, a letter of transmittal shall be prepared and shall include:

- Name(s) of person(s) receiving the data
- Name of person transmitting the data
- Documentation of whether the chain of custody was incorrect or not provided; whether any samples were damaged in shipment; whether sample containers were inappropriate for analysis; if samples were not preserved; and whether the quantity of sample provided was adequate for proper analysis
- Brief discussion of samples analyzed and the analytical program
- Discussion of any apparent data anomalies
- Discussion of any analytical difficulties and follow-up actions taken to remedy such difficulties

3.3.2 Metcalf & Eddy Validation Procedures

The validation of all analytical data for drinking water samples, generated by XYZ Laboratories will be subcontracted by ABC, Inc. ABC, Inc. will use the most recent NYSDEC ASP data validation protocols (1989 or current issue).

3.4 INTERNAL QUALITY CONTROL CHECKS

All analyses performed in support of this investigation shall use standardized laboratory procedures. The QC program developed by M&E shall use both known and unknown (or "blind") QC samples, calibration check samples, method blanks, field blanks, trip blanks, and replicate aliquot samples (duplicates). The various types of both field/laboratory-generated QC samples are described below. The type and frequency of analysis for each QC sample is shown in Table 3-4. The laboratory control limits for matrix spikes, etc. are listed in Tables 3-5 and 3-6.

Type	Purpose	Frequency	Criteria	Corrective Action
Trip Blank	Verifies no contamination during sample transport/storage	1 per batch of VOC shipped from field	No compound of interest shipped from field 5 times MDL ⁽¹⁾	Qualify data or resample
Method Blank	Verifies clean reagents, instrument systems and lab environment	1 per 12-hour analysis day per batch	No compound of interest > 5 times MDL	Reanalyze; if second blank exceed criteria, clean and recalibrate system; document corrective action
Equipment Blank	Verifies effective decontamination procedures used in field	1 per day per sampling team	BDL ⁽²⁾	Qualify data or resample
Laboratory Control Samples (for AA, ICP)	Verifies analyst proficiency with method and instrumentation	> 1 per sample batch	< 20% RSD ⁽³⁾ + 15% recovery	Check system; require additional operator training
System Performance Check (for GC/MS compounds)	Checks performance of analytical system	When sample matrix interference is suspected	≥ 0.05 response	Check system; recalibrate; reanalyze
Laboratory Replicates (including matrix spike duplicates)	Precision check on analytical method	Per batch of 20 samples as supplied from field	RPD ⁽⁴⁾ as outlined in Tables 1.4.1a and 1.4.1b	Compare with field duplicates; check for matrix interference
Calibration Check Sample	Verifies calibration curve	≥ 1 per analysis day	± 20% initial calibration	Recalibrate; check system
Matrix Spikes and Duplicates (MS/MSD)	Checks recovery from real matrix	Performed at 5% frequency of samples	Specified in analytical methods (see Table 1.10.3)	Qualify data or recalibrate, reanalyze, and document corrective action
Surrogate Standards	Determine recoveries, control limits matrix effects	All GC/MS samples and all GC samples	As specified in analytical methods, SH846 3rd edition (see Table 1.10.2)	Reanalyze samples; Qualify or reject data
Field Duplicate	Submit "blind" to lab to determine sample variability	5% of samples	± 10% RPD	Compare to lab replicates; check systems for possible matrix interferences or improper sample collection procedure

⁽¹⁾ MDL - Method Detection Limit
⁽²⁾ BDL - Below Detectable Levels
⁽³⁾ RSD - Relative Standard Deviation of at least 5 Replicates
⁽⁴⁾ RPD - Relative Percent Difference of Duplicate Measurements
 , No data generated for Ag and Sb analyses

QC SAMPLE TYPES, CRITERIA AND CORRECTIVE ACTION

ROXY CLEANERS SITE
RENSSELAER COUNTY, NEW YORK

TABLE 3-5 QA OBJECTIVES FOR ACCURACY FOR ORGANIC SURROGATE ANALYSIS

Fraction Soil/Sediment	Surrogate Compound	Percent Recovery	
		Low/Medium Water	Low/Medium
VOA	Toluene-d8	88-100	81-177
VOA	4-Bromofluorobenzene	86-115	74-121
VOA	1,2-Dichloroethane-d4	76-114	70-121
BNA	Nitrobenzene-d5	35-114	23-120
BNA	2-Fluorobiphenyl	43-116	30-115
BNA	p-Terphenyl-d14	33-114	118-137
BNA	Phenol-d5	10-94	24-113
BNA	2-Fluorophenol	21-100	25-121
BNA	2,4,6-Tribromophenol	10-123	19-122
PEST/PCBs	Dibutylchloroendate	24-154	20-150

TABLE 3-6 QA OBJECTIVES FOR ACCURACY AND PRECISION
FOR MATRIX AND MATRIX SPIKE DUPLICATES

Fraction	Matrix Spike Compound	% Recovery Limits		% RPD Limits	
		Water	Soil/Sed	Water	Soil/Sed
VOA	1,1-Dichloroethene	61-145	59-172	14	22
VOA	Trichloroethene	71-120	62-137	14	24
VOA	Chlorobenzene	75-130	60-133	13	21
VOA	Toluene	76-125	59-139	13	21
VOA	Benzene	76-127	66-142	11	21
BN	1,2,4-Trichlorobenzene	39-98	38-107	28	22
BN	Acenaphthene	46-118	31-137	31	19
BN	2,4-Dinitrotoluene	24-96	28-89	38	47
BN	Pyrene	26-127	35-142	31	36
BN	N-nitroso-di-N-propylamine	41-116	41-126	38	38
ACID	Pentachlorophenol	9-103	17-109	50	47
ACID	Phenol	12-89	26-90	42	35
ACID	2-Chloropheno	127-123	25-102	40	50
ACID	4 Chloro-3-methyl-phenol	23-97	26-103	42	33
ACID	4-Nitrophenol	10-80	11-114	50	50
PEST/PCBs	Gamma-BHC (Lindane)	56-123	46-127	15	50
PEST/PCBs	Neptachlor	40-31	35-130	20	31
PEST/PCBs	Aldrin	40-120	34-132	22	43
PEST/PCBs	Dieldrin	52-126	31-134	18	38
PEST/PCBs	Endrin	56-121	42-139	21	45
PEST/PCBs	4,4' -DDT	38-127	23-134	27	50

% RPD = Relative Percent Difference

This list includes those compounds most commonly used for QA/QC accuracy and precision control in the groups of analytes shown based on current U.S. EPA Contract Laboratory Program (CLP) requirements. (USEPA SOW 10/86 as revised through 8/87). Stated control limits will be updated to the current CLP protocol, as required.

3.4.1 Field Generated QC Samples

Trip Blank

One (1) trip blank shall be submitted to the laboratory for every batch of VOC samples (both soils and water) collected. The results of this blank will serve as a baseline measurement of any contamination that the samples may be exposed to during transport. A trip blank is comprised of a sample bottle filled with ASTM Type-II reagent water, transported to the sample collection site, handled like a sample, and returned to the laboratory for analysis (trip blanks are not to be opened in the field). The trip blank for soils is Type-II reagent water, just as is the case for water samples.

Equipment Blank

Five (5) percent of all samples collected that are submitted to the laboratory for analysis shall be equipment blanks. The analysis of these blanks serves to verify the cleanliness of the sampling equipment. An equipment blank is taken by rinsing the field equipment with ASTM Type-II reagent water, transferring the water to a sample bottle, and sending the sample for analysis.

Field Duplicates

Five (5) percent of all the samples shall be collected in duplicate and submitted to the laboratory for analysis. Duplicates are two samples collected independently at a sampling location during a single episode of sampling. Duplicates provide statistical information about sample variability. Field duplicates shall be indistinguishable from other analytical samples so that personnel performing the analyses are not able to determine which samples are duplicates.

Documentation and Review of Quality Control Activities

Field quality control sample custody shall be documented from the time of sample origin throughout transfer of the sample to the laboratory. Documentation of field quality control sample custody provides evidence that the samples have reached the laboratory without alteration.

Documentation of sample collection, shipment, laboratory receipt, and laboratory custody must be maintained in order to accomplish this.

Documentation is maintained through a chain of custody record which contains a list of all samples and the individuals responsible for the sample collection, shipment, and receipt.

In addition to initiating the chain of custody form, field personnel are responsible for uniquely identifying, labeling, and tagging field quality control samples prior to shipment. Every field quality control sample chain of custody form must contain the following information:

- Project number
- Unique sample number
- Sample description (i.e., equipment blank, trip blank)
- Date and time of sample origin
- Name of person obtaining the sample
- Method of sample preservation

Chain of custody records shall be initiated for every field quality control sample by the person collecting the sample. All chain of custody form entries shall be made with indelible ink.

Field quality control samples shall be packed and shipped along with their corresponding environmental samples in iced and sealed coolers with the chain of custody form taped to the inside lid. Upon receipt of the samples, the laboratory must sign and date the chain of custody form to verify receipt of the samples. The laboratory must also note any physical alternatives to samples (i.e., chain of custody seal broken, sample bottle broken, etc.).

3.4.2 Laboratory Generated QC Samples

The XYZ Laboratory shall comply with the QC sample requirements of each analytical method specified. The QC sample types generally required by the analytical methods are described below.

Laboratory Control Standard

One (1) laboratory control standard (LCS) shall be analyzed for all parameters for each batch of samples tested for metals and inorganics. The LCS is routinely used to establish that an instrument or procedure is precise and accurate. The analytical results of the LCS must be recorded in the instrument logbook and on the control chart; results must be within the acceptable control limits. A LCS solution is prepared by adding known quantities of an EMSL-Cincinnati Standard, a NBS Standard Reference Material, or a reference-traceable stock material to deionized water or the solvent of interest. A LCS solution is typically carried through the entire sample preparation and analysis procedure.

Calibration Check Sample

One (1) calibration check sample (CCS) shall be analyzed for every 10 samples analyzed sequentially for all project-specific parameters. A CCS is chosen as one of the mid-range working calibration standards that is reanalyzed periodically throughout the sample analysis to verify that the original calibration is still valid.

Method/Reagent Blank

One (1) method/reagent blank shall be analyzed with each sample batch tested. A method blank is comprised of laboratory-pure, analyte-free water carried through the entire sample preparation and analysis procedure. Analysis of the method blank provides a check of the background contamination due to sample preparation procedures.

Laboratory Replicates

One sample per batch of twenty (20) samples analyzed for project required parameters shall be analyzed in replicate. A replicate sample is produced by dividing a single collected sample into two equal parts for the purpose of determining analytical precision.

Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates shall be performed every 20 samples, or once for every group of less than 20 samples. Matrix spikes and matrix spike duplicates shall be performed for each group of samples having a similar concentration range and matrix composition. The analyte spike shall be added prior to digestion/distillation of the sample. If the spike recovery is not within the acceptable criteria limits specific to this project, the data of those samples associated with that spiked sample must be handled appropriately.

Surrogate Samples

All collected samples requiring organic analysis by GC/MS or GC shall be spiked with an appropriate set of surrogate standards prior to sample preparation. The surrogate standards shall encompass the full range of types of organics to be analyzed in the sample and shall also serve as checks on any matrix interference exhibited by the samples. If the percent recoveries of the surrogates are outside the acceptable project-specific criteria limits, the associated samples may be reanalyzed if the problem appears to be due to lab error. An independent analyst or unit leader shall determine whether to reanalyze the sample or qualify the data.

Documentation and Review of Quality Control Activities

The analyses of quality control samples are entered chronologically onto quality control charts specifically maintained for each analytical procedure. These control charts are labeled with upper and lower warning and control limits, the analysis which is being charted and the value (e.g., % recovery, % RPD, etc.) which is being monitored. Control charts are updated monthly and are used to demonstrate method performance and help identify system errors.

The laboratory documents its Quality Control activities by means of discrete sections of their report that include:*****

- A case narrative describing any problems encountered with method blanks, matrix spike, and matrix spike duplicates, surrogate recoveries, initial calibration, and continuing calibration
- Compilation of method blanks data
- Compilation of matrix spike and matrix spike duplicate
- Surrogate recoveries data
- Initial calibration and continuing calibration information

3.4.3 Documentation of Laboratory Tracking Procedure

The tracking system employed at XYZ Laboratory utilizes a computerized Laboratory Information Management System (LIMS). The system may be accessed to ascertain and document the status of any sample in process or upon analysis completion. The LIMS system may additionally be accessed by the client if the availability of this option is chosen at the beginning of the contract.

3.5 PERFORMANCE AND SYSTEMS AUDITS

3.5.1 Metcalf & Eddy Auditing Practices

The M&E QA project officer or agent will perform audits of the subcontractor laboratory. He will also be responsible for running field audits.

* All QA activities are documented in the laboratory but are only delivered upon request of the contracting agency. Delivery of the entire QA/QC package must be contracted prior to sample analysis.

3.5.2 XYZ Laboratory Auditing Practices, Performance Audits and Frequency

As a participant in several certification programs and various contracts, the XYZ laboratory is frequently subjected to rigorous performance evaluations and on-site inspections by the EPA, other regulatory agencies and commercial clients. The XYZ Quality Assurance staff performs routine internal audits of the laboratory as well. The audits ensure that all laboratory systems including sample control, analytical procedures, data generation, and documentation meet contractual requirements and comply with good laboratory practice standards.

Systems Audits

The NYSDEC Contract Laboratory Program provides for complete, on-site, systems audits of contract laboratories by a team of auditors on a regular basis. XYZ will have already undergone several such EPA and NYSDEC inspections. (To be checked when Laboratory is selected.)

A list of recent systems audits of the XYZ Laboratory will be provided in their Quality Assurance Plan (see Appendix A). In addition, the XYZ laboratories are audited routinely by QA staff members in order to detect any sample flow, analytical, or documentation problems and to ensure adherence to the good laboratory practices as described in XYZ laboratory operating manuals. The items covered in an internal systems audit at XYZ are outlined on a general basis in Figure 3-18.

Performance Audits (Check after Laboratory is selected)

The XYZ laboratories are also subjected to frequent performance evaluations. Recent XYZ performance evaluations will be provided in their Quality Assurance Plan (see Appendix A).

The NYSDEC Contract Laboratory Program requires successful performance of pre-award performance evaluation samples prior to acceptance into the program. Once established in the program, a lab must continue to demonstrate

LABORATORY AUDIT OUTLINE

Laboratory Audit - General Considerations

- Sample Flow Through Lab
- Chain of Custody
 - Usual
 - Strict
- Sample Storage
 - Controlled Access
 - Proximity to Chemical Storage
 - Physical Conditions, e.g., Temperature
 - Holding Times
- Sample Work-up and/or Analysis
 - SOPs
 - Logbooks
 - Standards Preparation
 - Instruments - Sample Analysis
 - Calibration/Tune
 - Standards Analyses
 - Check Samples
 - Balance
 - Temperature
 - Notebooks
 - Dates
 - Signature
 - Filled Pages
 - Initialed Errors with Single-line Crossouts
 - Units Recorded
 - QC Samples
 - Blanks
 - Spikes
 - Duplicates
 - Surrogates
 - Control Charts
- Data File Storage
 - Hard Copies
 - Other Media - Magnetic Tape, Disk
- Laboratory Safety
- Organization, Order
- Sampling
 - Container Preparation
 - Preservative(s)
 - Techniques
- QA Access - Corrective Action Forms

performance capabilities by successfully analyzing blind samples sent by NYSDEC each quarter.

Performance is monitored internally on a daily basis at XYZ through the use of surrogate standards and matrix control samples. Check samples obtained from NYSDEC and from independent commercial sources are employed routinely in each of the XYZ laboratories and ensure continuing high level performance.

3.6 PREVENTIVE MAINTENANCE

3.6.1 Laboratory Equipment

Preventive maintenance is a routine practice at XYZ for each analytical instrument. Scheduled preventive maintenance minimizes instrument downtime and consequent interruption of analysis. The laboratory personnel at XYZ are familiar with the maintenance requirements of the instruments they operate. This familiarity is based on conventional education, specialized courses, and hands-on experience. Designated staff members are trained at manufacturer facilities in the routine maintenance procedures for major analytical instrumentation. (An example logbook page for GC/MS routine maintenance is presented as Figure 3-19.)

In the case of a downed instrument, the problem is diagnosed as quickly as possible. If necessary, replacement parts are ordered and repairs performed by skilled in-house personnel. Key instrumentation is maintained under service contract. As a third option, a service call is placed with the manufacturer.

Instrument Maintenance Logbooks

Each analytical instrument is assigned an instrument logbook in which is recorded all maintenance activities, problems, and repairs. The information entered in the instrument logbook includes:

- Date of service
- Person performing service
- Type of service performed and reason for service
- Replacement parts installed (if appropriate)
- Miscellaneous information

If service is performed by the manufacturer, a copy of the service record is taped into the page facing the notebook page where the above information is entered. An example logbook page for GC/MS non-routine instrument maintenance is presented in Figure 3-20.

Instrument Calibration and Maintenance

The routine calibration procedures used for analytical instrumentation are described in Section 3.2.1 and shown in Table 3-2. Preventative maintenance and calibration by manufacturer service representatives are provided on a routine basis. The maintenance procedures and frequencies for major analytical instrumentation are given in Table 3-7.

XYZ Laboratory service agreements provide for preventative maintenance, emergency service, and emergency shipping of spare parts. The service contracts for the GC, GC/MS, AA, and ICP require emergency on-site response within 48 to 72 hours. (Typically, service representatives are on site within 24 hours of a service call.) The service contracts also provide for 24-hour delivery of critical spare parts in response to a service request.

Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. This inventory should emphasize those parts (and supplies) which:

- Are subject to frequent failure
- Have limited useful lifetimes
- Cannot be obtained in a timely fashion should failure occur

GC/MS NON-ROUTINE INSTRUMENT MAINTENANCE

Instrument: _____	PROBLEM SITE:
Date Reported: _____ By: _____	_____ GC
Date Resolved: _____ By: _____	_____ Gas Line
Comments on Repair: _____	_____ Dectector
_____	_____ Other (DESCRIBE)
_____	_____
_____	_____

DESCRIPTION OF PROBLEM:

RESOLUTION:

Instrument	Items Checked/Serviced	Frequency	Documentation	Reference
LABORATORY EQUIPMENT				
Atomic Absorption Spectrophotometer	3-point calibration performed Clean burner head Clean nebulizer Tune optics Check lamps Check electronics Check pump Replace tubing	When serviced by repairman Each shift used Each shift used Each shift used As required As required As required	Log Book	Manufacturer's Manual
Analytical Balance	Internal weight train, gears, electronics	Annual service	Log Book, Service Sticker	Manufacturer's Manual
Inductively Coupled Plasma Spectrophotometer	Sample introduction system Clean optics Check pumps Check electronics	Each analysis day As required As required As required		Manufacturer's Manual
pH Meter	Electronics checked Electrolyte changed	Each analysis day Checked weekly, changed when low	Log Book	Manufacturer's Manual
GC/MS	Replace column Clean source Change glass wool plug Clean insert Replace septa Gases (purity checks) Flow controller check	As required As required As required As required Each analysis day New cylinders Semi-annually	Log Book	Manufacturer's Manual
GC	Replace column Gases (purity checks) Flow controller check Replace Septa	As required New cylinders Semi-annually Each analysis day	Log Book	Manufacturer's Manual
FIELD EQUIPMENT				
Stainless Steel Tape	Replace if damaged or bent	As necessary		-
Thermometer	Check calibration against NBS thermometer	Yearly	Log Book	-
pH Meter	Electronics checked Electrolyte changed	Each analysis day Checked weekly, changed when low	Log Book	Manufacturer's Manual
Conductivity Meter	Check response Check probe Check batteries	With each use Daily Daily	Log Book	Manufacturer's Manual
Photoionization Detector	Clean UV lamp and ion chamber Replace lamp	As required As necessary	Log Book	Manufacturer's Manual

3.6.2 Field Equipment

Preventive maintenance of field equipment is also required in order to ensure the collection of valid field measurements. All necessary maintenance procedures are fully documented in the project-specific field log book. The type and frequency of such preventive maintenance is outlined in Table 3-7.

3.7 PROCEDURES FOR ASSESSING PRECISION, ACCURACY AND COMPLETENESS

Following are the procedures recommended for evaluating the precision and accuracy of all environmental measurement data generated by this project. The protocol used for QC requirements is in accordance with specific analytical procedures.

3.7.1 Review of QC Sample Data

When the analysis of a sample set is completed, the results are reviewed and evaluated to assess the validity of the data set. The review is based on the criteria in Table 3-4, and is outlined as follows:

- **Reagent Blank Evaluation** - The reagent and/or method blank results are evaluated for high readings characteristics of background contamination. If high blank values are observed, laboratory glassware and reagents are checked for contamination and the analysis is halted until the system is brought under control. A high background value is defined as a value sufficiently large enough as to affect the sample value, and if not corrected may result in an increase in the sample value greater than or equal to the smallest significant digit known to be true.
- **Trip Blank Evaluation** - Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis must be reviewed. If both the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of the contamination is probably within the laboratory.

- **Matrix Spike Evaluation** - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value (the percent recovery) may be plotted on a control chart for the parameter determined to show method performance.
- **Calibration Standard Evaluation** - The calibration curve is evaluated to determine linearity through its full range, and to verify that sample values are within the range defined by the low and high standards. If the curve does not meet method calibration criteria, corrective action is taken. Only the analysis of matrix samples (blanks, spiked blanks) trigger corrective action.
- **Replicate Sample Evaluation** - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). If the results of replicate analyses (RPD's) are outside of the recommended range, XYZ shall reanalyze samples.
- **Reference Standard Evaluation** - Standard Reference Material analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide corrective action. All sample analyses should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed.
- **Check Standard Evaluation** - The results of check standard analyses are compared with the true values and the percent recovery of the check standard is calculated. If correction is required, the check standard should be reanalyzed to demonstrate that the corrective action has been successful.
- **Surrogate Standard Evaluation** - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Average recovery values for surrogates in each GC/MS fraction will be plotted on control charts.

3.7.2 Evaluation of Data Using Control Charts

XYZ shall apply precision and accuracy criteria to each parameter that is analyzed. When analysis of a sample set is completed, the quality control

data are reviewed and evaluated through the use of control charts to validate the data set.

Control charts shall be established for all major analytical parameters. A minimum of ten measurements of precision and accuracy are required before control limits can be established. Once established, control limits are updated (by the Quality Control Coordinator) as additional precision and accuracy data become available.

3.7.3 Evaluation of Analytical Precision

For replicate results D_1 and D_2 , the RPD is calculated from:

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

When the RPD is obtained for at least ten replicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^n m_i}{n}$$

and

$$S_m = \frac{[\sum_{i=1}^n (m - \bar{m})^2]^{1/2}}{n - 1}$$

where

m = the RPD of a replicate pair,

$\bar{m}$ = the average of the Relative Percent Difference determinations,

S_m = the standard deviation of the data set of RPD Determinations, and

n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

$$\text{Upper Control Limit} = m + 3 S_m$$

$$\text{Lower Control Limit} = m - 3 S_m$$

$$\text{Upper Warning Limit} = m + 2 S_m$$

$$\text{Lower Warning Limit} = m - 2 S_m$$

A control chart is established by plotting the RPD of each replicate on a graph generated as follows:

- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Charts. These control charts are used to show method performance and to document that the default control limits used by the laboratory for method control are met on a routine basis.
- If the RPD plots are outside the Control Limits for control spikes (blanks spikes of LCS's), the source of error is determined and corrective action is implemented.

3.7.4 Evaluation of Analytical Accuracy

To determine the accuracy of an analytical method and/or the laboratory analyst, a periodic program of sample spiking is conducted. The results of sample spiking will be used to calculate the quality control parameter for accuracy evaluation, the percent recovery (%R).

The %R is defined as 100 times the observed concentration, minus the sample concentration, divided by the true concentration of the spike.

$$\%R = \frac{O_i - O_s}{T_i} \times 100\%$$

where

$\%R$ = the percent recovery
 O_i = the observed spiked sample concentration,
 O_s = the sample concentration, and
 T_i = the true concentration of the spike

The true Concentration is calculated from:

$$T_i = \frac{\text{Spike Concentration [c] (mg/L) x Volume of Spike (in mL)}}{\text{Volume of Sample (in ML) + Volume of Spike [in mL]}}$$

When the Percent Recovery is obtained for at least ten spiked samples, the mean percent recovery and the standard deviation are calculated using the formulae:

$$\% \bar{R} = \frac{\sum_{i=1}^n \%R_i}{n}$$

and

$$S_R = \frac{\left\{ \sum_{i=1}^n (\%R_i - \% \bar{R})^2 \right\}^{1/2}}{n - 1}$$

where

$\% \bar{R}$ = the mean percent recovery
 $\%R_i$ = the percent recovery of a single spiked sample
 n = the number of results, and
 S_R = the standard deviation of the data set of percent recovery determinations.

The Warning Control Limits are then calculated from the following equations:

$$\text{Upper Control Limit} = \bar{R} + 3 S_R$$

$$\text{Lower Control Limit} = \bar{R} - 3 S_R$$

$$\text{Upper Warning Limit} = \bar{R} + 2 S_R$$

$$\text{Lower Warning Limit} = \bar{R} - 2 S_R$$

A control chart is generated by plotting the percent recovery data on a graph as follows:

- The average of the percent recovery determinations for the original data set is established as the midpoint on the Y axis above the mean of the percent recovery of the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the percent recovery determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines.
- If the percent recovery is plotted outside the Control Limits for reference controls (blank spikes and duplicates, LCS and duplicates, the source of error is determined and corrective action is implemented. Once the error source has been resolved, the data set shall be reanalyzed on a case by case basis.
- On a periodic basis, the Warning and Control Limits are recalculated for the entire data set and the Control Chart for the corresponding parameter is updated.

All control charts are maintained by the Quality Assurance Coordinator, as well as distributed to appropriate management.

3.7.5 Evaluation of Completeness

Completeness is calculated as the percentage of total samples accounted for compared to the total samples that were planned to be collected to achieve particular project requirements. Data points may not be valid if samples

exceeded holding times, if quality control sample criteria were not met and reanalysis of samples was not possible, or if sample containers were broken or otherwise destroyed.

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

$$\% \text{ completeness} = \frac{\text{Total samples accounted}}{\text{Total samples planned}} \times 100$$

The overall completeness objective for this project is 100%

3.8 CORRECTIVE ACTION PROCEDURES

Corrective action procedures for this program normally are initiated by the analytical laboratory personnel (and their supervisors) directly involved with the analysis of the samples and the implementing of the procedures presented in this QAPP; corrective action may also be a result of validation checks. Quality control records for daily instrument calibration, replicate analyses, and surrogate analysis are utilized to indicate the necessity for corrective action. Control records shall be established for each procedure indicating upper and lower limit ranges. At the point when the control records indicate a determination is outside the warning ranges, investigation as to the cause shall be initiated.

The laboratory analyst shall verify that all quality control procedures are followed and that the results of the analysis of quality control samples are within the allowable acceptance criteria. This requires that the analyst assess the correctness of all of the following items as appropriate:

- sample preparation procedure
- initial calibration
- calibration verification
- method blank result
- duplicate analysis
- laboratory control standard
- fortified sample result

If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. The analyst notifies the appropriate supervisor and QA Manager of the problem and, if possible, identifies potential causes and corrective action. In turn, the Lab QA Manager shall notify the prime contractor if the quality of data deliverables has been compromised or if the laboratory has determined that the problem is field related.

The nature of the corrective action obviously depends on the nature of the problem. For example, if a continuing calibration verification is determined to be out of control, the corrective action may require recalibration of the analytical system and reanalysis of all the samples analyzed since the last acceptable continuing calibration standard was run.

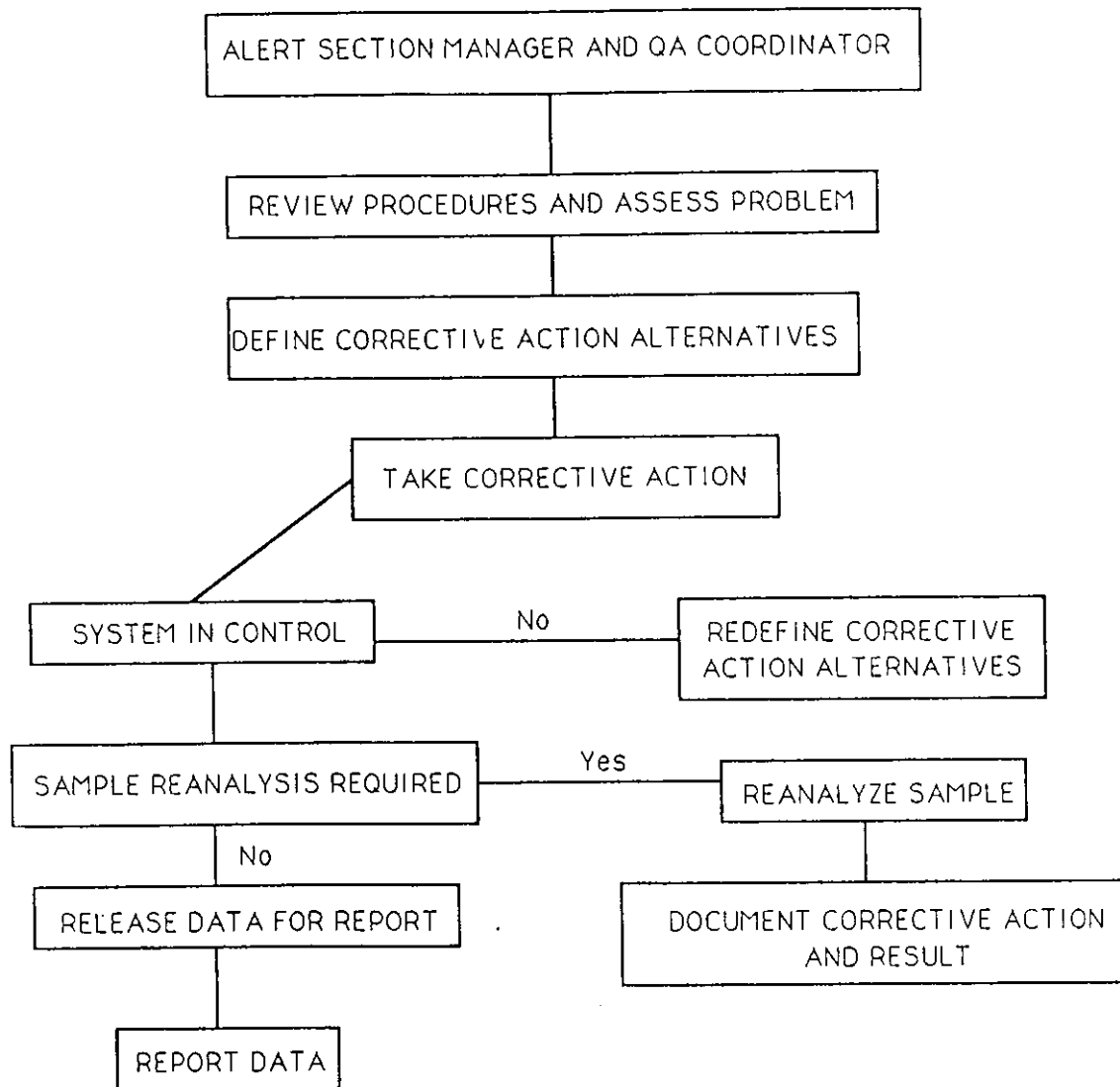
When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control", the analyst documents the problem and the corrective action. Copies of the form summarizing these actions are provided to the Laboratory Section Manager and QA Coordinator.

Data generated concurrently with an out-of-control system shall be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data is reported and the deficiency is noted in the case narrative. Where sample results are impaired, the Laboratory Project Manager is notified and appropriate corrective action (e.g., reanalysis) is taken.

The critical path for assessing the correctness and acceptability of analytical data is shown in Figure 3-21.

The corrective action relative to the control records is related more to precision than to accuracy. These records give clues as to when some factor, generally of a procedural nature, is causing the results to drift or when an unexpected difference beyond the control limits occurs. Data within the upper and lower control limits of the control records are well within the precision, accuracy, and completeness criteria specified for this investigation.

CRITICAL PATH FOR CORRECTIVE ACTION IN THE LABORATORY



The routine analytical corrective action procedures within the laboratory are documented and may result in the reanalysis of samples or recalibration of analytical instrumentation. Routine corrective action shall take place as necessary and does not require the approval of the M&E Quality Assurance Manager. However, should significant events occur such as sample breakage or loss, exceeding sample holding times, extensive instrumentation downtime, or changes or additions to sample clean-up for removal of interferences, the laboratory shall report these events to M&E immediately.

Corrective actions are also initiated as a result of other project QA activities including performance audits, systems audits, and laboratory comparison studies. Problems signaling significant and systematic deficiencies are addressed with a Recommendation for Corrective Action (RCA) form (see Figure 3-22) which is issued only by the M&E Quality Assurance Manager or his designee. Each RCA shall address a specific problem or deficiency encountered during laboratory analysis.

Each Recommendation for Corrective Action requires a correction action resolution to be made by the Project Manager and the QA Project Manager. A QA data files is used to track RCAs. On a monthly basis a summary of the unresolved RCAs is prepared by the M&E QA Manager and submitted to project management or to the subcontractor management. These reports list all RCAs that have been issued to the work areas for which each manager is responsible as well as the current status of each RCA. Each RCA requires a listing of the corrective response and verification by the M&E QA Manager that the corrective action has been implemented before the status is changed on the monthly report. If there is no response to an RCA within 30 days, or if the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved. The corrective action scheme is shown in the form of a flow chart as Figure 3-23.

As stated previously, corrective actions start in the laboratory when the project is in the Environmental Data Collection Activities or EDCA mode. Corrective actions may also be a result of problems in the field, transportation problems, or the results of human error. Early detection of a

RECOMMENDATION FOR CORRECTIVE ACTION

A. Initial Information

RCA NO.:	DATE:	URGENCY LEVEL <input style="width: 40px; height: 20px;" type="checkbox"/> 1. Potential for major data loss or invalidation. 2. Potential for failure to achieve data quality objectives. 3. Suggested improvement.
ORIGINATOR:	APPROVED BY:	
ORGANIZATION/INDIVIDUAL RESPONSIBLE FOR ACTION:		

B. Problem Identification

SITE/LAB:	SYSTEM:	DATE PROBLEM IDENTIFIED:
DESCRIPTION OF PROBLEM:		

C. Recommended Corrective Action

DESCRIPTION:	IMPLEMENT BY:

D. Problem Resolution

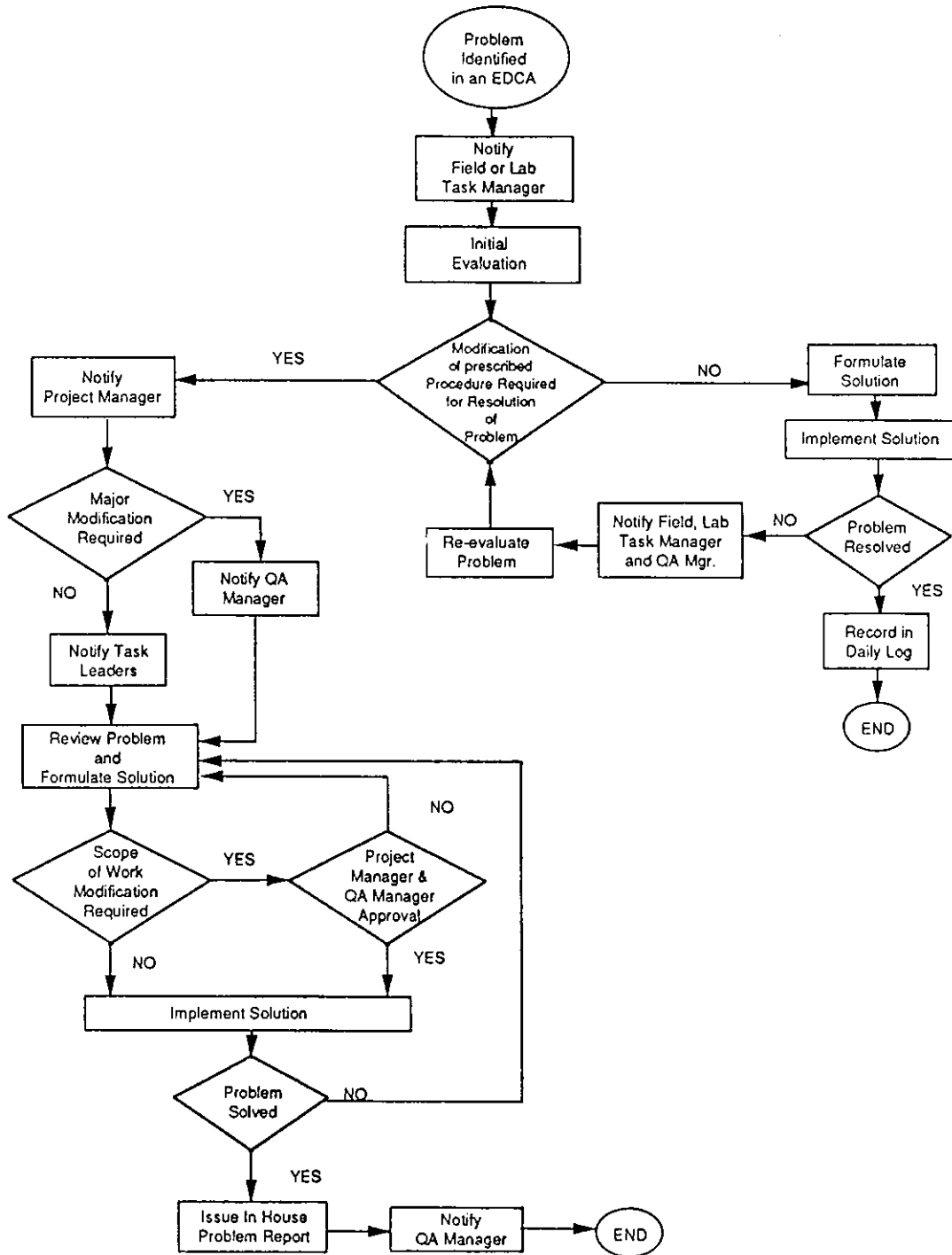
PLANNED CORRECTIVE ACTION:	PROPOSED BY:	DATE PROPOSED:	SCHEDULE IMPLEMENTATION:
IMPLEMENTED CORRECTIVE ACTION:			DATE IMPLEMENTED:

E. QA Verification

VERIFIED BY:	DATE:	COMMENTS:

White: Return for Implemented Corrective Action Yellow: Return for Planned Corrective Action Pink: File Copy Gold: Originator's Copy
 Information Copies Distribution: _____ _____ _____ _____ _____ _____

CORRECTIVE ACTION FLOW CHART



problem and punctual corrective action is the key for prevention of poor quality data. Early discovery of a problem usually takes place at a sub-project or task level. In these cases, the task manager must recognize problems and action them as soon as possible.

3.9 QUALITY ASSURANCE REPORTS

3.9.1 Metcalf & Eddy, Inc.

At monthly intervals (when data is available) for the duration of the field sampling and chemical analysis activities of this project, the M&E Project Engineer shall prepare a summary report on the performance of the measurement systems and the corresponding data quality. These reports shall address, at a minimum, the following:

- Results of performance audits of all field sampling and laboratory analysis activities performed during the subject reporting period
- Results of system audits
- Assessment of measurement data accuracy, precision, completeness, and comparability including review of all XYZ Laboratory measurement data

Data assessment shall, in more detail, address the following issues to ensure proper data reduction, validation, and reporting:

- Accuracy
 - Compute percent recoveries for spiked samples
 - Calculate the standard deviation in the overall average recovery value
 - Apply Chauvenet's criterion for detecting poor recovery data
 - Determine the range of uncertainty at a given level of confidence
 - Adjust the laboratory data to correct any systematic errors (bias) that are discovered

- Precision
 - Examine split samples and pairs of samples for differences in inter- and intra-sample scatter
 - Determine if sampling error has occurred by comparing inter- and intra-sample scatter
 - Validate data for groups of samples having similar compositions, by examining the scatter in each group in comparison to the overall scatter (invalid data are discarded)
 - Compute an overall relative standard deviation that is applicable to all the field investigation data from a particular sampling campaign
- Completeness
 - Compute the fraction of QA test data that remains valid after discarding any invalid accuracy or precision data
- Representativeness and Comparability
 - Determine whether these terms have meaning within the project framework
 - Identify the appropriate statistical methods
 - Correctly apply the statistical methods and report the results

Information provided in the periodic QA reports shall be summarized for inclusion in the final project report.

3.9.2 XYZ Laboratory

Performance and Reporting

A case narrative outlining nonconformance pertaining to project samples shall accompany all sample results.

Nonconformance/Corrective Action Resolution from System Audits

The Laboratory Manager and Quality Assurance Manager shall seek to directly resolve all differences concerning cited nonconformances, requests for corrective action, or the completion of corrective action. If the differences cannot be resolved, higher levels of management shall be the arbitrator.

Management Review of the Quality Assurance Program

The review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. The Laboratory Manager must present recommended changes to the divisional Quality Assurance Director as they arise.

The divisional QA Director in turn must provide quarterly reports to the corporate QA Manager. These reports summarize QA activities for the reporting period including: results of performance audits (external and internal), results of system audits (external and internal), summaries of corrective action used to remedy any situations which have caused QA data to be outside the prescribed criteria requirements, and recommendation for the revision of laboratory procedures to improve the analytical systems. The Project Manager is notified immediately of all laboratory QA situations requiring immediate corrective action.

During system audits, the Quality Assurance Program is discussed. The corresponding audit report shall document all system revision recommendations made by either the Laboratory Manager, the M&E Project QA Officer, or the audit team.

4.0 QUALITY ASSURANCE/QUALITY CONTROL - FIELD SAMPLING

4.1 SAMPLING LOCATION AND FREQUENCY

This section outlines the location, procedures and frequency of samples that will be taken for analytical testing. The field sampling program includes the following:

- Groundwater sampling
 - 16 existing monitoring wells
 - eight new monitoring wells
- Surface water sampling (Wynants Kill)
- Sediment sampling (Wynants Kill)
- Borehole soil sampling
- Soil gas sampling

4.1.1 Monitoring Wells

There are 16 existing groundwater monitoring wells (eight well pairs) in the vicinity of Roxy Cleaners. During the first round of sampling, all 16 existing wells will be sampled for TCL Volatile Organics. One upgradient well pair and two downgradient, highly contaminated well pairs will also be sampled for TCL Semi-volatile Organics, TCL Pesticides/PCBs and TCL Metals. After the eight new wells are installed, the second round of groundwater sampling will be performed. During the second round of sampling, both the existing 16 wells and eight new wells will be sampled for volatile organics only, unless the results of the first round of sampling indicate the presence of other contaminants. The samples from wells which showed non-detectable levels of contaminants during the first round of sampling will be analyzed using EPA Method 524.2. The use of EPA Method 524.2 makes a lower detection

limit possible. Samples that showed detectable levels of contaminants during the first round of sampling will be analyzed for TCL volatiles.

4.1.2 Surface Water Samples

One upstream and two downstream surface water locations will be sampled along the Wynants Kill and analyzed for TCL Volatile Organics only. The objective of the surface water sampling is to determine whether groundwater contamination has reached the Kill and if surface water quality has been impacted.

4.1.3 Sediment Samples

Three sediment samples will be collected from the Wynants Kill in conjunction with the surface water samples. All three sediment samples will be analyzed for TCL Volatile Organics and Total Organic Carbon (TOC). The samples will be collected directly below the sediment surface using a hand auger and stainless steel scoop. The results of the TOC analyses will be used in calculating sediment criteria for the Wynants Kill.

4.1.4 Seep Water Samples

Two water samples will be collected from seeps (if present) without the use of any intermediate sampling instrument. The two seep water samples will be analyzed for TCL Volatile Organics. The seep sample locations will be determined in the field by the M&E site geologist with approval by the NYSDEC site representative.

The objective of sampling seeps in conjunction with groundwater and sediment sampling is to evaluate background and surface contamination concentrations. Data will be used to define source areas of contamination, the potential pathways of vertical and horizontal migration, types and concentrations of contaminants present at the surface, and health risk evaluations.

4.1.5 Borehole Soil Samples

A total of four borehole soil samples will be analyzed for Total Organic Carbon (TOC). In addition, three borehole soil samples which exhibit high readings on the flame ionization detector (FID) or photoionization detector (PID) will be submitted for TCL Volatile Organics analysis. These boreholes will be used for the installation of the new groundwater monitoring wells. The results of the TOC analyses will be used in calculating the soil cleanup criteria.

4.1.6 Soil Gas Survey

A maximum of 100 soil gas probes will be driven into the ground surrounding the Roxy Cleaners Site to determine the presence of organic vapors and evaluate the concentrations of volatile organic compounds in the vadose zone. The soil gas survey locations cover a developed area containing stores, homes and roads. Therefore, if an obstruction is blocking a location, then that location may be moved several feet. Soil probes will be driven at approximately 50 foot intervals. Thus, there will be approximately one soil probe per 2,500 square feet of surface area. The soil gas will be analyzed using a Hewlett-Packard 5890 Series II Gas Chromatograph (GC) configured with an Electron Capture Detector (ECD) and a Photoionization Detector (PID) (refer to Section 3.2.4 for calibration of these instruments and Section 4.4.1 for their decontamination).

4.1.7 Quality Assurance Parameters

Table 4-1 summarizes the field QA/QC samples in addition to the environmental samples to be collected at the Roxy Site Cleaners Site and surrounding study area.

4.2 SAMPLE DESIGNATION

The establishment of a standard sample designation/labeling protocol is essential to ensure adequate quality assurance/quality control (QA/QC) with

WELL PURGING WORKSHEET

Job Name _____ Job No. _____ Samplers _____

Well ID _____ Date Sampled _____ Time: Start _____ End _____

Casing Diameter _____ inches + 12 = _____ (d) ft. Well secured upon arrival? Y/N

Depth of well from T.O.C. _____ ft. Standing water (gal.) = _____

Depth of water from T.O.C. _____ ft. x _____ well volumes

Feet of standing water _____ (h) ft. = _____ gallons to purge

$$\begin{aligned} \text{Standing Water} &= \pi [(d)^2 + 4] (h) \\ \text{Volume} &= 3.14 [(d)^2 + 4] (h) \times 7.48 \text{ gal/ft}^3 = \text{gals.} \end{aligned}$$

PID Readings (ppm)

Breathing _____

Well _____

Purging method _____ Purge: Time Start _____ End _____

pH Conductivity Temperature, (C)

1 well volume = _____ gal. _____

2 well volume = _____ gal. _____

3 well volume = _____ gal. _____

Final volume = _____ gal. _____

Sample Collection: Time Start _____ End _____ Bailer ID# _____

Sample Characteristics (Circle all applicable)

Describe odor: none sulfide fishy musty petroleum _____

Describe color: colorless black brown orange red _____

Describe appearance: turbid silty sand clay floaters sheen
clear multiphased foaming slimy algae _____

Organic Layer? _____ Length? _____ Samples preserved? _____

Comments _____

TABLE 4-1

QUALITY ASSURANCE PARAMETERS
ENVIRONMENTAL SAMPLING AND TESTING

Parameters	Matrix	Total Samples	Field Duplicates	Trip Blanks	Equipment Blanks	MS/MSD/ DS
TCL Volatile Organics	Existing Groundwater Monitoring Wells (First Round Sampling)	16	1	8	1	1
TCL Semi- Volatile Organics	Existing Groundwater Monitoring Wells (First Round Sampling)	6	1	--	1	1
TCL Pesticides/PCBs	Existing Groundwater Monitoring Wells (First Round Sampling)	6	1	--	1	1
TCL Metals	Existing Groundwater Monitoring Wells (First Round Sampling)	6	1	--	1	1
TCL Volatile Organics ⁽¹⁾	Existing Groundwater Monitoring Wells (Second Round Sampling)	16	1	8	1	1
TCL Volatile Organics	New Groundwater Monitoring Wells (Second Round Sampling)	8	1	4	--	1
Total Organic Carbon	Borehole Soils	4	1	--	1	1
TCL Volatile Organics	Borehole Soils	3	1	3	1	1
TCL Volatile Organics	Surface Water	3	1	1	--	1
TCL Volatile Organics	Sediment	3	1	--	1	1
Total Organic Carbon	Sediment	3	1	--	1	1
Total Volatile Organics	Seep Water	2	1	1	--	--

* MS/MSD/DS - Matrix Spike/Matrix Spike Duplicate or Duplicate Spike

⁽¹⁾ Monitoring wells that show non-detectable levels of contaminants in the first round of sampling may be analyzed by Method 524.2 in the second round.

respect to the traceability of samples and their associated analytical data. Proper labeling allows for the tracking of samples beginning from the time of sample collection, through analysis, and following project completion should future data correlation be deemed necessary. The proper labeling of samples is also critical in ensuring that samples are analyzed within the required sample holding times as specified by the appropriate analytical methods. Sample tracking is initiated in the field with the assignment of an identification number to each sample as it is collected. This identification number consists of a unique project-specific sample identification number coupled with a sample location/description number. The project-specific identification number (i.e., station number) is composed of a project number hyphenated with a consecutive numbering system representative of the order in which the samples are collected. For example,

Station number: 005089-093

is the designated project-specific identification number representative of the 93rd sample collected from project 005089. The sample location number is composed of an initial or initials typifying the sample location (e.g., MW107 for existing monitoring well number 107, SW for surface water samples, SWS for surface water sediment samples, etc.). Also, existing monitoring wells will be sampled during two rounds of sampling. Therefore, the monitoring well location number will be hyphenated with the number of the sampling round. For example,

Location number: MW107-1

is representative of a sample from monitoring well number 107 during the first round of sampling.

The identification number shall be recorded on the sample label and chain of custody forms (refer to Figures 3-1 and 3-2). The number shall also be recorded in the field logbook along with any additional comments relative to the sample description and collection methodology.

4.3 SAMPLING PROCEDURES

The following sections outline the equipment and sampling procedures that will be used for the collection of sediment samples, soil gas samples and groundwater samples at the Roxy Cleaners Site and surrounding study area. All samples shall be delivered to the analytical laboratory within 48 hours of collection. Decontamination procedures for the sampling equipment is outlined in Section 4.4.

4.3.1 Sediment Sampling

Equipment

The following equipment may be used during sediment sampling:

- Stainless steel trowel, scoop, or hand core sampler with stainless steel liner tubes
- Stainless steel spoons
- Sample containers
- Stainless steel bowls

Sampling Procedures

To prevent cross-contamination of samples, sample collection will start at the most downstream location and continue upstream. Samples shall be collected while standing at the downstream side of each sample collection point according to the following procedures:

1. Record the physical characteristics of the surface water and sediment such as color, odor, and texture.
2. Make a sketch of the surface water boundaries and sediment sampling locations.
3. Photograph the sampling locations and conditions.
4. Trowel or scoop: Wearing a new pair of PVC gloves, collect equal portions of sediment at a minimum of three points in the cross-section of the sampling area, and transfer the samples into a stainless steel bowl.

- X
5. For samples being collected for volatile organics analysis, minimize any disturbance or mixing of the sediment. Invert a 40 ml VOA vial and insert it directly into the sample sediment. Twist the sample into the sediment and fill the container as completely as possible to minimize the air space.

After volatile organics have been collected, the remaining samples may be mixed or composited. Fill the containers at least 3/4 full for all other analyses.

6. Immediately label and tag (as required); refrigerate/ice; and log the samples into the bound field logbook and complete the sample chain of custody form.

Documentation

In addition to the observations noted previously, the following information shall be documented and reported in the field logbook when sampling surface soils:

- Description of the sample
- Approximate depth of the sample collection
- Information on whether the sample is a grab or composited sample
- Method of composting
- Whether the sample was sent to be analyzed or not, and why

Refer to Figure 3-5 for a general outline of a typical data entry page in the field logbook.

4.3.2 Surface Water Sampling

Equipment

The following equipment may be used during surface water sampling:

- Sample containers
- Required sample preservatives (if sample containers do not contain preservatives)
- Disposable polystyrene cups for collection of field monitoring aliquot

- Turbidity meter
- pH meter
- Conductivity meter
- Thermometer
- Standard pH buffer solutions of pH 4.0, 7.0 and 10.0
- Single-use KCl conductivity calibration standard solutions

Sampling Procedures

To prevent cross-contamination of samples, sample collection will start at the most downstream location and continue upstream. Samples shall be collected while standing at the downstream side of each sample collection point according to the following procedures:

1. Estimate the depth of water. (If a pole is to be used, wait to perform this task until after actual sampling to avoid water turbulence.)
2. Record the weather conditions and other on-site particulars.
3. Record the physical characteristics of the water body such as odor, color, temperature, pH, conductivity, presence of any dead vegetation and surface sheens, etc.
4. Make a sketch of the surface water boundaries and sampling locations.
5. Photograph the sampling locations and conditions.
6. Collect a sample by immersing the sample container in the water; rinse the container in the water to be collected and pour it out. Do not collect samples at the surface; instead, the sample container should be inverted, lowered to the approximate sampling depth, and held at about a 45-degree angle. If the surface water is a flowing stream, river, or brook, hold the sample container at about a 45-degree angle with the mouth of the bottle facing upstream.

Samples collected for volatile organic analysis must contain no air bubbles in the VOA vial after it has been capped; ensure this by turning the vial upside down and tapping it lightly. Fill all other sample containers to the shoulder.

7. Collect a final sample aliquot in a disposable container and immediately measure and record the pH, temperature, and conductivity of that sample.

8. Immediately label and tag (as required); preserve if necessary; refrigerate/ice; and log the samples into a bound field logbook and complete the chain of custody form.
9. Calculate an estimate of the surface water flow (if any) from a measurement of the linear cross-section flow velocity (using a bobber and a stop watch) and an estimate of the flow volume's cross-sectional area.

Documentation

In addition to the sampling observations listed previously, the following observations should be documented in the field logbook when sampling surface water:

- Time of sample collected
- Description of water body, color, odor, appearance
- Depth of water
- Depth of sample collection for each specimen collected
- Description of sampling location (e.g., left bank, eddy, middle, etc.)

Refer to Figure 3-5 for a general outline of a typical data entry page in the field logbook.

4.3.3 Groundwater Sampling

Equipment

- Stainless steel measurement tape weighted and used to measure the water level
- Bladder, Fultz, centrifugal, or positive displacement hand pump to purge the well (The entire pump and discharge lines should be constructed of non-contaminating materials.)
- Decontaminated, disposable High Density Polyethylene (HDPE) bailers for sample collection (Clear teflon bailers can be used for floating product estimations.)

- Teflon-coated stainless steel cable to be used as leader line for bailers (It must be decontaminated between uses.)
- Sample containers
- Required sample preservatives (if sample containers do not already contain preservatives)
- Disposable polystyrene cups for collection of field monitoring aliquots
- pH meter
- Thermometer, non-mercury type
- Conductivity meter
- Standard pH buffer solutions of pH 4.0, 7.0, and 10.0 units
- Single-use KCl conductivity calibration standard solutions
- Photoionization detector (PID) and/or flame ionization detector (FID)

Sampling Procedures

Samples shall be collected beginning with the well located in the area suspected of least contamination (upgradient) and proceeding to the areas suspected of highest levels of contamination. Other factors, such as well recovery time, may also influence the order of sample collection. The following procedure shall be used:

1. Unlock the protective casing on the well.
2. Sample the air in the well head for organic vapors using a PID or FID, and record the measurements.
3. Decontaminate the measuring tape as per this site-specific QAPP. Using the measuring tape, measure and record the well's static groundwater level and the depth to the bottom of the well. Record the measurement from the water surface to the top of the well casing to the nearest 0.01 foot.
4. From the well diameter and the measured depth of the standing water, calculate (or use a well volume table to determine) the volume of the standing water in the well. Note and record the volume.

5. (Optional) If a floating layer is suspected to be present on top of the well water, gently lower a dedicated, clear teflon bailer to below the water surface and withdraw. Using the PID or FID, measure the headspace of the sample in the bailer and record the reading. Examine the surface of the water in the bailer for the presence of floating materials. If possible, measure and record the thickness of this layer.
6. Wearing a new pair of PVC gloves, begin purging the well with a purging pump or bailer. The actual number of well volumes to be removed during a purging may vary depending on the site characteristics, well design, and the chemicals or parameters for which analysis shall be performed. At a minimum, three well volumes should be purged from each well, unless the well is pumped to dryness. The purged volumes are estimated by discharging the purge water into a container of known volume. During the purging of each well volume, measure and record the water's temperature, conductivity, and pH as it is removed from the well at the end of each well volume. Discontinue purging when three well volumes have been removed provided the pH and conductivity readings have stabilized. If the measurements have not stabilized (i.e., any given pH or conductivity reading demonstrates more than a 10 percent difference from the previous reading), further purging may be necessary. A minimum of three readings is required.

If the well goes to dryness during the purging process, several options are available. The options are based on the time required for the water-level recovery, the type of analysis to be performed, and the type of purging and sampling equipment being used. The option to be used for each investigation should be approved on a site-specific basis prior to sampling. The options may include the following:

- For water-level recoveries of 0.5 to 1 hour, purge three well volumes. Sampling shall be performed within three hours of the final purge.
- Whenever full well recovery exceeds 3 hours, samples should be collected in order of their volatility, as soon as sufficient water volume is available for a sample for each parameter or compatible set of parameters. Parameters that are not pH-sensitive or subject to loss through volatilization should be collected last.
- The discharged water collected from each well purge is disposed of as described in the site-specific waste disposal agreement made with the state authority.

7. Using a decontaminated disposable HDPE bailer, withdraw two full bailers of groundwater from the well. Dispose of these first two bailers of sample along with the discharged well purge water as described in the site-specific waste disposal agreement with the controlling agency. (Personnel handling the bailer must wear new PVC gloves.)
8. Using the same bailer and leader line, collect groundwater samples from the well. Record the time at the beginning and end of the bailing. (During sample collection, the leader line and bailer should not touch the ground or any objects except for the well casing.)
9. Immediately, wearing new PVC gloves, transfer the groundwater sample directly from the bailer to the appropriate sample containers.

All sample containers should be filled to the shoulder, approximately 3/4's full, except for those samples requiring volatile organic analyses. Samples collected for volatile organics analysis must be collected first. For the collection of volatile organic samples in VOA vials, be sure that there are no air bubbles in the vial after it has been capped; ensure this by turning the vial upside down and tapping it lightly.

10. Collect a final sample aliquot in a disposable container and immediately measure and record the pH, temperature, and conductivity of that sample.
11. Immediately label and tag (as required); preserve if necessary; refrigerate/ice; and log the samples into a bound field logbook and complete the chain of custody form.
12. Remeasure and record the standing water level in the well after sampling.
13. Replace the protective cap on the well and lock.

Documentation

In addition to the sampling observations listed previously, the following observations should be documented and reported in the field logbook when sampling monitoring wells: well purging data (see Figure 4-1) sample characteristics, and a note as to whether the well was secured upon arrival. Refer to Figure 3-6 for a general outline of a typical data entry page in the field logbook for the actual collection of samples.

4.3.4 Borehole Soil Sampling

A truck-mounted hollow-stem auger drill rig will be used during drilling of the boreholes, and a split-barrel sampler will be utilized to obtain the borehole soil samples. Continuous split spoon samples will be collected from the deep boring in each well pair. The shallow boring shall be advanced without sampling unless requested by the NYSDEC geologist. Upon extraction of the sampler from the boring at each sample interval, the sampler will be placed on a dry, clean surface. Prior to the disassembly of the sampler, the tip of an organic vapor analyzer will immediately be inserted into the open end of the sampler and the reading recorded. After opening, the vapor analyzer probe will be passed over the surface of the sample and the highest reading recorded. The highest measurement obtained will then be recorded on the boring log.

Once the sampler is disassembled, soil samples will be simultaneously visually inspected and logged, and removed from the sampler and placed into several sample containers for possible shipment to the certified project laboratory.

A field decision will be made on which samples (maximum of three samples) are to be selected for quantitative analysis for TCL Volatile Organic Compounds at a certified laboratory. In general, samples from each boring representing the highest level of contamination as indicated by a screening method should be analyzed in the laboratory.

In addition, four borehole soil samples will be obtained and analyzed for Total Organic Carbon (TOC). Again, a field decision will be made on which samples are to be selected for analysis at the certified laboratory.

All soil samples designated for lab analysis will be sealed, labelled, and kept cool and shipped within 48 hours to the laboratory. Chain-of-custody forms will be completed by M&E field personnel and shipped to the laboratory with the samples.

4.3.5 Soil Gas Sampling Methods

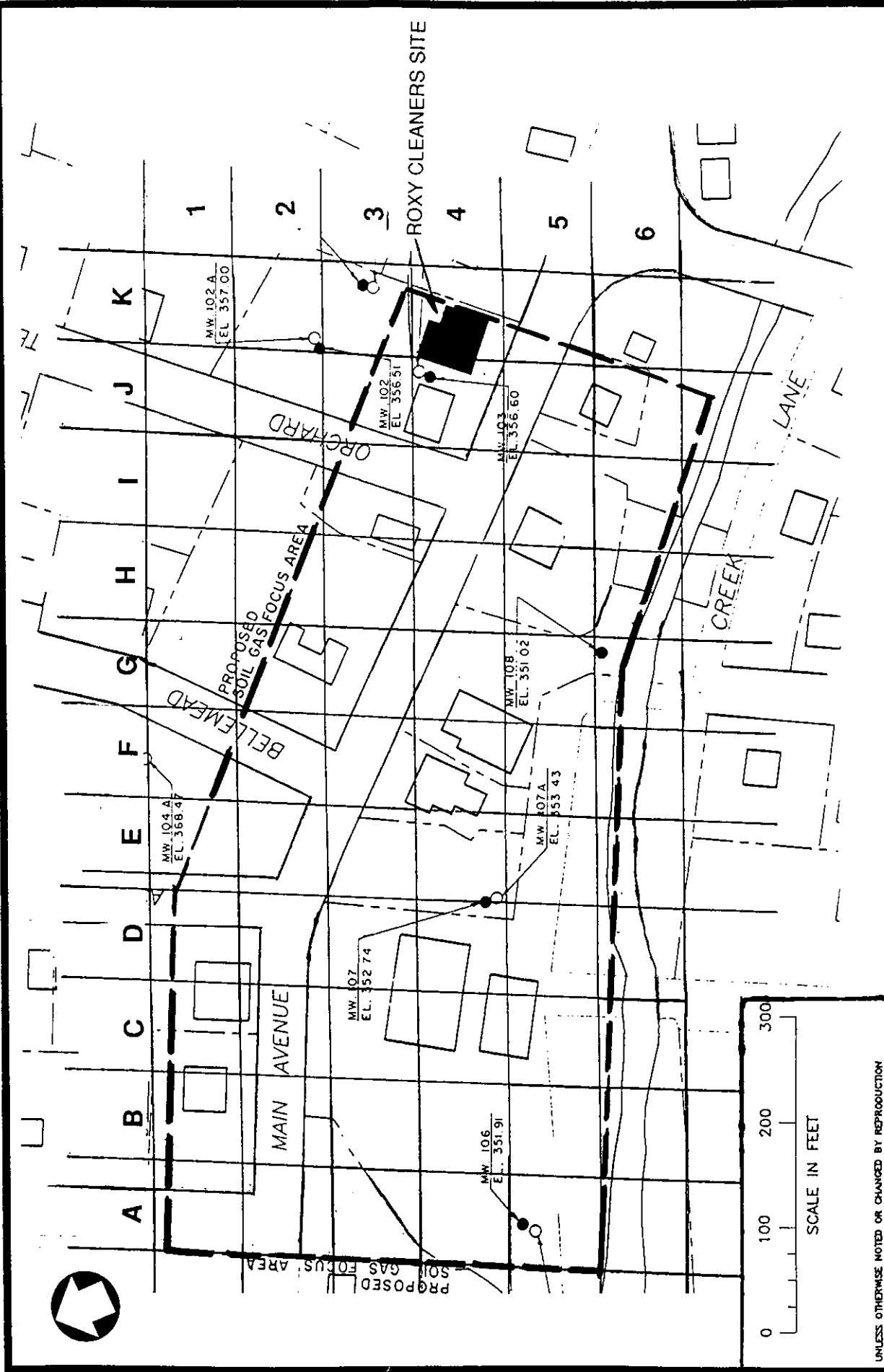
A soil gas survey will be performed as part of the Phase I RI at the Roxy Cleaners Site and surrounding study area. Soil gas surveys are semi-quantitative in nature and are only useful for comparing relative concentrations of a putative contaminant within a specific area. Accordingly, for this study, a comparison of relative instrument responses to background conditions over an array of test locations will be used to preliminarily delineate the areas exhibiting elevated concentrations of tetrachloroethene (perchloroethylene or PCE) within the study area boundaries.

Sampling Methodology

Beginning at the site and using it as a focal point, a sampling grid will be constructed in quadrants across the study area with sampling points marked at approximately 50 foot intervals with either orange tipped wooden stakes or paint marks on asphalt surfaces. All of these points (maximum of 100 sample probe locations) will be labelled and located on a base map for future reference. Each sampling point on the grid will cover approximately 2,500 square feet. Based on previous data and consultation with NYSDEC, the grid will be biased downgradient from the site (see Figure 4-2).

Soil gas samples will continue to be collected in all directions outward from the grid until two consecutive probe points indicate PCE below the detection limit of the method (a line of two probe points with non-detections).

Sampling Train. The sampling train will consist of six foot black steel rods (probes) with carbon steel points. The soil gas sample will be drawn from a septa sealed tee fitting attached to the top of the probe. The syringe needles will draw directly from the top of the probe with zero dead volume. Gas samples will only contact metal surfaces and are never in contact with potentially sorbing materials (see Figure 4-3). Attached to the tee above the sampling point will be tygon tubing leading to a photoionization detector, HNu Model HW101, with a positive displacement pump.



UNLESS OTHERWISE NOTED OR CHANGED BY REPRODUCTION

M&E Metcalf & Eddy
of New York, Inc.
 A Metcalf & Eddy Company

ROXY CLEANERS SITE
 RENSSELAER COUNTY, NEW YORK

SOIL GAS SURVEY SAMPLING GRID

FIGURE 4-2

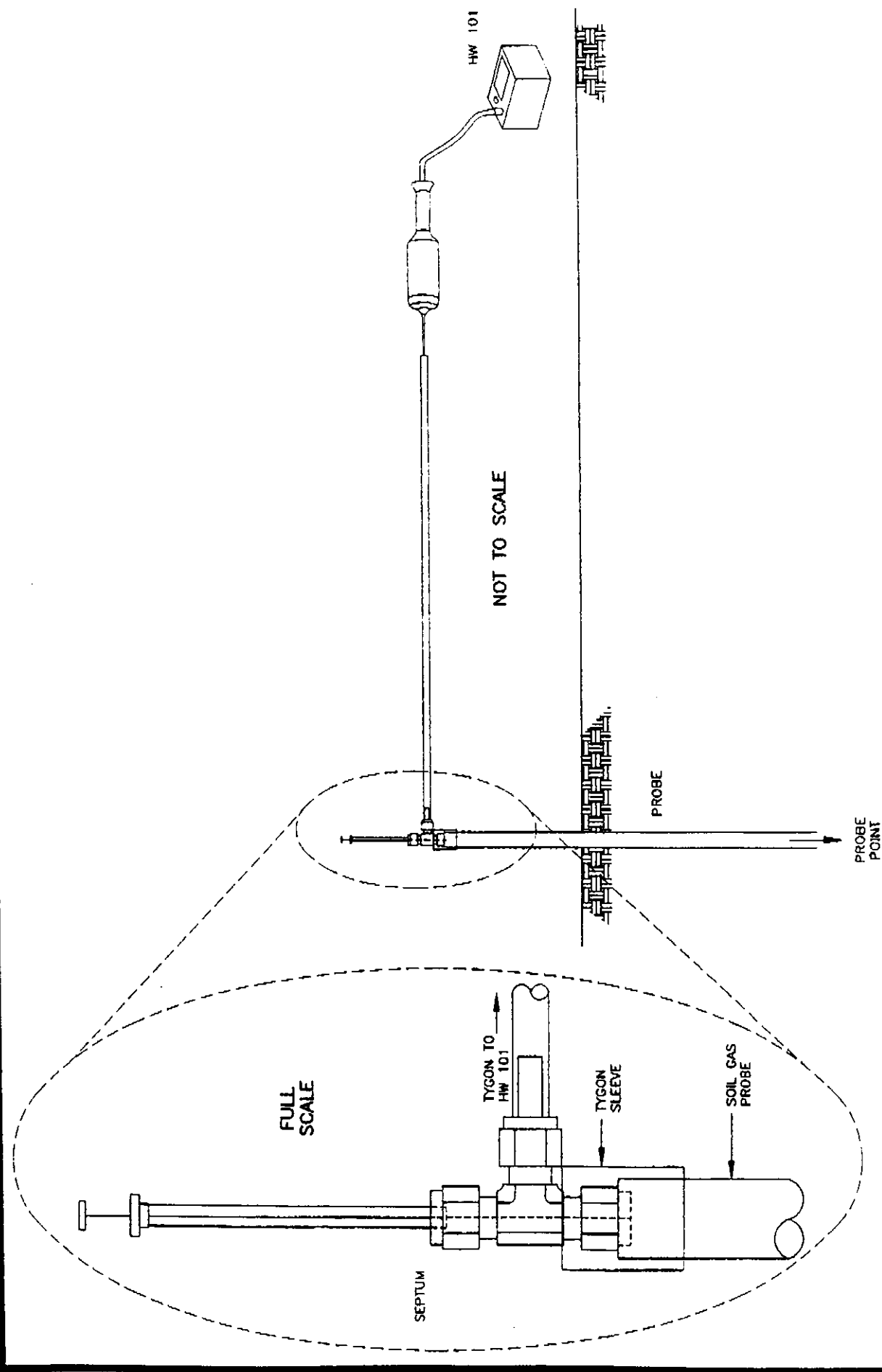


FIGURE 4-3	SOIL GAS SAMPLING TRAIN
ROXY CLEANERS SITE RENSSELAER COUNTY, NEW YORK	M&E Metcalf & Eddy of New York, Inc. A Metcalf & Eddy Company

Probe Installation. The sampling probe with a carbon steel point will be driven into the ground with a slide hammer to a depth of approximately four to five feet. The steel point will be left behind as the probe is pulled back approximately six to eight inches, exposing the formation to pumping. If the formation is unproductive, as determined by the work load on the pump, the probe will be withdrawn until a productive formation is found or a depth of two feet below grade, whichever occurs first. Bentonite/clay will be used to ensure an adequate seal at the ground surface. If a sampling point is abandoned, another area within that sampling grid area will be selected for another attempt.

Probe Purging. The soil gas probe will be purged one to three times the sample train volume unless determined otherwise in the field depending upon the volume determined experimentally at a probe location as calculated by the following equation:

$$V = D(1/2)r^2L (28.3 \text{ liters/cubic foot})$$

V = volume of gas at STP in liters
r = radius of inside diameter of sampling train in feet
L = length of sampling train
D = pi

The pump flow rate will be determined by using a Gillibrator bubble flow meter. The flow rate will be the mean average of five measurements.

Gas Sampling Procedures. The purge volume before sampling will be determined experimentally. A probe will be installed at or near the site and the instrument reading from the PID closely monitored. The purge volume with the highest PID reading will be used for all other points in the survey.

Samples will be obtained at the determined purge volume by drawing ten microliters into each of the two Hamilton gas tight syringes. After removing the syringes, septums will be placed over the needles to prevent accidental

evacuation. A label will be fixed to the syringes with the sample number corresponding to the grid point. The grid point and sample time will be recorded in a bound sequentially numbered field logbook with water proof ink. The syringes will be transported to the on-site analytical area in a horizontal position within a cushion metal tray.

Analytical Methods

Because of anticipated interference from hydrocarbon contamination originating off-site, two detectors will be used for the analyses. A Hewlett-Packard 5890 Series II Gas Chromatograph (GC) will be configured with an Electron Capture Detector (ECD) and a Photoionization Detector (PID). It is anticipated that the GC will be equipped with an injection tee (see Supelco catalog part #2-3664 M), allowing one injection (using one syringe) which will split into two capillary columns. However, if the injection tee cannot be added, one injection will be made into each capillary column using two syringes of equal sample volume. A 30 meter (0.52 millimeter inside diameter) VOCOL column with a 3.0 micron film thickness will be installed to the ECD (Supelco catalog #2-5320). A 30 meter SPB-5 column (0.53 millimeter inside diameter, 1.50 micron film thickness) will be installed to the PID. Two HP3396A integrators will be used to record detector response. Helium will be the carrier gas with Nitrogen added as a makeup gas for the ECD. It is anticipated that the GC will be operated in the splitless mode. Flow rates, temperature ramps, and column head pressures will be adjusted to maximize chromatographic separation. Once the operating conditions are established, all subsequent analyses will proceed according to the set parameters. All operating conditions, including temperature programming information, gas flow rates, column head pressures, attenuation settings, and PID voltage settings, will be recorded in water proof ink in a sequentially bound laboratory notebook. Additional information, including instrument serial numbers and types, will also be recorded in the notebook.

Sample Handling and Injection. When each sample arrives in the laboratory area the sample number from the tag and the arrival time will be entered in waterproof ink in a sequentially numbered bound laboratory notebook. The injection number, as it corresponds to the chromatogram number from the integrators, will also be entered. The septums will be removed from the needles just before injection and all ten microliters from each syringe loaded onto the GC.

As stated previously, if a sample concentration falls outside of the high point (10 ppm for BTEX and 1 ppm for PCE) on the calibration curve, then the sample area will be resampled and a smaller volume of sample will be injected for the next analysis.

Records. The laboratory notebook will include all information necessary to trace analytical work, including sample calculations, injection order, sample numbers, instrument parameters, and any and all deviations or variances from the standard protocols. Observations or extraordinary circumstances will also be noted. The analyst's name will appear at the top of each page, the words "continued to page" and "continued from page" will be added along with the appropriate page number. A table of contents will be constructed at the beginning of the notebook. Chromatograms will remain in a continuous roll throughout the day and the sample ID added. If a roll of paper is torn or a new roll is used, an explanation will be included at the beginning. The integrator sample number, time, and date will be checked each morning and verified by entry into the laboratory notebook. Once the study is completed, copies of all chromatograms and laboratory notebooks will be provided to NYSDEC. Inspection of the data will be available at all reasonable times during the study if such inspection does not interrupt or interfere with ongoing work.

4.3.6 Quality Control Samples

During each sampling episode, a number of quality control (QC) samples must be collected and submitted for laboratory analysis. The number and frequency of the QC sample collection is determined by the individual project requirements and is outlined in Table 4-1 which is contained in Section 4.1 of this QAPP.

A list of the types of QC samples that will be collected, along with a brief description of each sample type is outlined in the following sections.

Trip Blanks

Trip blanks are collected for chemical analysis of volatile organics. The analytical results serve as a baseline measurement of volatile organics contamination that samples may be exposed to during transport and laboratory storage prior to analysis.

Trip blanks originate in the laboratory. They are comprised of organic-free ASTM Type II reagent water which is placed in sample containers by the subcontracting laboratory, transported to the sample collection site, handled along with the samples, and returned to the laboratory along with samples of water and/or soil collected for volatile organics analysis. The trip blank containers are not to be opened in the field.

Typically, one trip blank is included in each shipping container of up to 20 samples collected for volatile organics analysis, is stored in the laboratory with the samples, and is analyzed by the laboratory (for volatile organics only).

Equipment Blanks

Equipment blanks (sometimes called field blanks) are collected for each piece of sampling equipment used in the collection of samples when equipment other

than the sample bottle itself is required. The analysis of these blanks serves to verify the cleanliness of the sampling equipment.

Equipment blanks originate in the laboratory. They are comprised of organic-free ASTM Type II reagent water which is placed in sample containers by the subcontracting laboratory, transported to the sample collection site, opened, poured into the sampling device following equipment decontamination procedures (or pumped through it as in the case of sampling pumps), transferred back to the sample bottle, and returned to the laboratory for analysis. The equipment blanks are analyzed for the same parameters as the associated samples.

Typically, 5 percent of all samples collected for laboratory analysis are equipment blanks.

Field Duplicates

Field duplicates are defined as two samples collected independently of each other at a sampling location during a single episode of sampling. Analysis of these duplicates provides statistical information relating to sample variability and serves as a check on the precision of any sample collection method as it pertains to the sampled area.

Typically, 5 percent of all samples are collected in duplicate and submitted for laboratory analysis. Field duplicates shall be labeled in such a manner so that persons performing laboratory analyses are not able to distinguish duplicates from other collected samples.

4.3.7 Sieve Analysis

During the construction of the two new monitoring well clusters on the south side of the Wynants Kill, eight borehole soil samples will be obtained from distinct geological formations for grain-size/sieve analysis. The sieve analyses will be performed by the drilling subcontractor in accordance with ASTM D 422-63 (see Appendix D).

4.4 DECONTAMINATION PROCEDURES

Decontamination is the process of removing contaminants that have accumulated on excavation and drilling equipment and sampling apparatus. Proper decontamination is essential in minimizing the transfer of harmful materials into clean areas, in the prevention of cross-contamination between samples due to the use of improperly decontaminated field/sampling equipment, and in protecting workers from hazardous substances.

4.4.1 Equipment

All equipment involved in site investigation activities shall be decontaminated prior to leaving the site. The general outline for such decontamination procedures is included in the following sections.

Non-Sampling Field Equipment

Non-sampling field equipment is any equipment that may potentially contact a sample area. All equipment and power tools used as non-sampling equipment (i.e., drilling equipment, well casings and screens, backhoes, dredges, augers, etc.) shall be decontaminated before and following usage and prior to removal from the site. Large and heavy drilling equipment will be steam cleaned. Light or small equipment such as hand tools shall be rinsed with tap water, scrubbed with a water/mild soap solution, rinsed again with tap water, rinsed with deionized water and pesticide-grade methanol and hexane, and air dried. All equipment decontamination will be performed at a decontamination station specified in the site-specific Health and Safety Plan (HASP).

Sampling Equipment

Prior to field use, the cleaning procedures outlined in this section shall be used by M&E personnel for the cleaning of all sampling equipment that will come in direct contact with the sample being collected. A sufficient amount of clean equipment and sample containers should, whenever possible, be transported to the project site to minimize the need for excessive cleaning of

equipment in the field. Since off-site decontamination is not always possible, field cleaning procedures are outlined in the following sections. Table 4-2 can be used as a guide for typical items used during field decontamination.

Decontamination Procedures For Soil Gas Survey Equipment

All disposable probes will be steam cleaned and rinsed with DI Water prior to use. Care should be taken to ensure that the probe is dry prior to use since moisture in the probe may cause sample interference and monitoring instrument damage.

The equipment blank check consists of zeroing the monitoring instrument to ambient air, attaching the instrument to the probe, and drawing ambient air through the probe. A positive response on the instrument indicates that further decontamination and equipment blank procedures should be repeated until no response deviation is noted on the monitoring instrument when the sample probe is on-line.

Cleaning Material

The materials used throughout the cleaning procedures may be dangerous if improperly handled. Caution must be exercised by all personnel, and all applicable safety procedures must be followed. At a minimum, the following precautions shall be taken in the field during these cleaning operations:

- Safety glasses with splash shields or goggles, chemical resistant gloves, and appropriate protective clothing must be worn during all cleaning operations.
- No eating, smoking, drinking, chewing, or any other hand-to-mouth contact (including application of cosmetics) is permitted during cleaning operations.

TABLE 4-2
TYPICAL MATERIALS REQUIRED FOR DECONTAMINATION EQUIPMENT

Item	Usage Rate	Unit
Aluminum Foil	1/10 samples	100 sq ft
Bottle Brushes	2/crew	
Equipment Decontamination Basin	2/site	
Equipment Decontamination Brush	2/site	
2.5 - 5 Gallon Carboy, Glass (DI Water)	1/20 samples	2.5 - 5 gal
2.5 - 5 Gallon Carboy (Tap Water)	2/day	each
Non-Phosphate Laboratory Detergent, Liquid	1/crew/week	2 1/2 gal
Non-Phosphate Laboratory Detergent, Solid	1/crew/week	4 lbs
Nitric Acid (Reagent Grade)	1/25 aqueous samples	1 pint
Personnel Decontamination Basin (for hands and face)	3/site	
Personnel Decontamination Brush	3/site	
Plastic Sheeting (250 sq ft/roll)	1/crew/week	1 roll
Polyethylene Wash Bottles	5/crew	1 pint
Tap Water	1/20 samples	5 gallons
Teflon Wash Bottles	6/crew	1 pint
Trash Bags (20/box)	1/crew	box
Trash Barrels	3/site	
Type IV Quality Deionized Water	1/20 samples	5 gallons

Project-specific guidelines should be used when choosing proper cleaning materials. The laboratory detergent solutions, and rinse waters used to clean equipment shall not be reused. The cleaning materials referred to throughout the Sampling Procedures are defined in the following paragraphs.

Phosphate-Free Laboratory Detergent: A standard brand of phosphate-free detergent such as Sparkleen^R, Liquinox^R, or Alconox^R is used as the standard laboratory detergent for decontaminating field equipment. The use of any other detergent must be justified and documented in the field logbooks.

Nitric Acid Solution: When trace metal analysis is to be performed on a sample, a nitric acid solution is used for decontamination of sampling equipment involved in collection of this sample. The standard solution is made as a 1:10 dilution of ACS reagent-grade nitric acid in deionized water. Other types of acids (e.g., hydrochloric, sulfuric, etc.) may be used as specified in other project-specific Quality Assurance Plans. If nitrates are to be analyzed, nitric acid should not be used for decontamination purposes.

Tap Water: Tap water from any municipal potable water supply system may be used for initial rinsing of field equipment. An untreated or non-potable water supply is not an acceptable substitute for tap water.

Deionized Water: Deionized water shall be used for the final water rinsing of all field equipment. An ASTM Type II reagent water is used for this purpose; ASTM Type II water has a maximum conductivity of 1.0 umhos/cm and a maximum residue concentration of 0.1 ppm. Other grades of reagent water may be used as required by the site-specific project plans. (Distilled water purchased from local supply stores or supermarkets is not Type II reagent water.) The field team shall retain the analytical data or manufacturer's certification which verifies the quality of the reagent water used by the field team.

Organic-Free Water: Organic-free water is defined as tap water that has been both deionized and treated with activated carbon. Organic-free water should contain no detectable amounts of pesticides, herbicides, and extractable

organic compounds, and less than 20 ug/l of commonly used laboratory solvents. All other compounds should be below the detection limits as measured by a low level GC/MS analysis (typically 0.5 ug/l).

Brushes: The use of brushes with wire-wrapped bristle should be avoided due to contamination from the wire. Totally plastic brushes should be used, if possible. However, plastic brushes with wooden handles are acceptable.

Cleaning Procedures

Guidelines for routine decontamination of equipment used in the collection of samples at hazardous waste sites are outlined in the following sections.

(Note: Rinse the sampling equipment thoroughly with tap water in the field as soon as possible after use to aid in the ease of future decontamination).

Paint or coatings (e.g., rust) must be removed from any part of the equipment that may contact the sample.

Teflon, Polyethylene, and/or Glass Equipment:

1. Wash the equipment thoroughly with phosphate-free laboratory detergent and tap water. Use a brush to remove any particulate matter or surface film.
2. Rinse the equipment thoroughly with tap water.
3. If the sampling equipment is being used to collect samples for trace metals analysis, rinse the equipment with a nitric acid solution.
4. Rinse the equipment thoroughly with deionized water.
5. Wrap the equipment completely with aluminum foil to prevent contamination during storage and/or transport to the field.

Stainless Steel Equipment:

1. Wash the equipment thoroughly with phosphate-free laboratory detergent and tap water or by steam cleaning. Use a brush to remove any particulate matter or surface film.

2. Rinse the equipment thoroughly with tap water.
3. Rinse the equipment thoroughly with deionized water and allow it to air dry.
4. Wrap the equipment completely with aluminum foil to prevent contamination during storage and/or transport to the field.

Electronic Well Sounding Tapes and/or Measuring Tapes Used to Measure Groundwater Levels:

1. Wash the electronic well sounding tapes and/or measuring tapes with phosphate-free laboratory detergent and tap water.
2. Rinse the equipment thoroughly with tap water.
3. Rinse the equipment thoroughly with deionized water.
4. Place the equipment in a polyethylene bag or wrap it with aluminum foil to prevent contamination during storage and/or transport to the field.

Ice Chests and Shipping Containers:

All ice chests and reusable shipping containers shall be washed on-site (interior and exterior) with phosphate-free laboratory detergent, rinsed with tap water, and air dried before storage. In the event that an ice chest or shipping container becomes severely contaminated with waste or other toxic material, it shall be cleaned as thoroughly as possible and disposed of properly.

Vehicles:

All vehicles used by M&E that enter contaminated areas shall be steamed cleaned or washed under a high pressure water spray at the site vehicle decontamination station (as specified in the site-specific Health & Safety Plan) at the conclusion of each field trip. If a steam cleaner is not available, then a high pressure sprayer may be used. This routine maintenance minimizes any chance of contamination of equipment, samples, or other sites due to the contamination of vehicles. A thorough interior and exterior

cleaning is mandatory at the conclusion of all field investigations. At a minimum, this decontamination procedure will consist of washing the exterior of the vehicle with a water/mild soap solution, rinsing it with tap water, and vacuuming the interior. It is the responsibility of the field team leader to ensure this procedure is followed.

4.4.2 Quality Control Procedures

Equipment that has been used to collect samples shall be decontaminated before it is returned from the field. At a minimum, this decontamination procedure will consist of washing the equipment with a phosphate-free laboratory detergent solution and rinsing it with tap water.

All equipment that is decontaminated using the procedures outlined previously shall be labeled or marked with the decontamination date and decontaminators signature prior to being stored. If there is any procedural deviation from the cleaning procedures outlined in this section, this fact should be noted in the appropriate field logbooks.

Field or sampling equipment that require repair will be identified with a tag. Any problems encountered with the equipment during use, as well as suggestions/recommendations as to the needed repairs shall be noted on this tag. Field equipment or reusable sample containers requiring cleaning and/or repairs will not be stored with decontaminated equipment, sample tubing, or sample containers. The effectiveness of the decontamination procedures used to clean all equipment that comes in direct contact with the samples being collected is monitored by rinsing the sampling equipment with organic-free water and collecting the rinsate in a sample container and submitting this for analysis. This sample is designated as an equipment blank. The frequency of such sample collection is specified in this QAPP. Typically, at least one such quality control sample is collected per day of equipment use and decontamination per sample medium.

4.4.3 Disposal of Decontamination Solutions and Other Sampling Derived Wastes

Solid Waste

Air monitoring scans are generally performed on all solid wastes using a photoionization detector (PID), an organic vapor analyzer, or other appropriate detection equipment. If the scans indicate that the level of contamination in the solid waste (containing such materials as disposable coveralls, gloves, duct tape, broken glassware, and general trash) could be hazardous, the wastes are packed in DOT approved drums and a composite of the drums analyzed. If the waste is determined to be hazardous, then it is disposed of according to applicable State and Federal Laws as specified by the site-specific waste disposal agreement between the contractor and the State of New York.

Liquid Waste

The liquid waste generated on-site during decontamination procedures shall be disposed of by allowing it to drain back on-site. However, if the decontamination liquids contain solvents or acids, the wastewater must be drummed or contained in a storage tank for off-site disposal and/or on-site treatment by the treatment system owned by NYSDEC, as described in the site-specific waste disposal agreement.

4.4.4 Documentation

All decontamination procedures performed during the course of a field investigation must be documented in the site-specific field logbook. Any deviations from the standard decontamination protocols must be noted.

4.5 SAMPLE HANDLING FOR ANALYSTS

4.5.1 Sample Preservation

All samples shall be preserved immediately following collection. Table 4-3 summarizes the sampling parameters, containers, and preservation requirements for the aqueous (groundwater, surface water and seep water) and soil/sediment samples to be collected at the Roxy Cleaners Site and surrounding study area.

4.5.2 Sample Custody

An overriding consideration essential for the validation of environmental measurement data is the necessity to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of the sample traceability from collection to shipment, laboratory receipt, and laboratory custody (until proper sample disposal and the introduction of field investigation results as evidence in legal proceedings when pertinent) must be documented. A sample is considered to be in a person's custody if the sample is:

- In a person's actual possession
- In view after being in a person's possession
- Locked so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel.

The site chemist/ environmental engineer is responsible for overseeing and supervising the implementation of proper sample custody procedures in the field. He is also designated as the field sample custodian and is responsible for ensuring sample custody until the samples have been transferred to a courier or directly to the laboratory.

Once the samples have been received by the laboratory, a designated person is responsible for maintaining a file of all the original documents (e.g., sample

TABLE 4-3

SAMPLING PARAMETERS, CONTAINERS AND PRESERVATION
REQUIREMENTS FOR SAMPLES COLLECTED AT THE ROXY CLEANERS SITE

Parameter	Container ⁽¹⁾⁽⁶⁾	Preservative ⁽²⁾	Holding Time ⁽³⁾
[AQUEOUS SAMPLES]			
Volatile Organics	2x40 ML G Septa-vial teflon-lined cap	Cool to 4°C	7 days VTSR ⁽⁴⁾
Semi-Volatile Organics	2 1 liter, G teflon-lined cap	Cool to 4°C	5 days until extraction, 40 days after VTSR
Pesticides/PCBs	1 liter, G teflon-lined cap	Cool to 4°C	5 days until extraction, 40 days after VTSR
Metals	1 liter G or P	Cool to 4°C, HNO ₃ <2pH	6 months ⁽⁵⁾
[SOIL AND SEDIMENT SAMPLES]			
Volatile Organics	4 ounce wide mouth, G	Cool to 4°C	7 days
Semi-Volatile Organics	16 ounce wide mouth, G	Cool to 4°C	5 days until extraction, 40 days after VTSR
Metals	P,G 1 liter	Cool to 4°C	6 months ⁽⁵⁾
Pesticides/PCBs	8 ounce wide mouth, G	Cool to 4°C	5 days until extraction, 40 days after VTSR
Total Organic Carbon	4 ounce wide mouth, P or G	Cool to 4°C	26 days

Notes:

- (1) Some aqueous samples require acidification and cooling for sample preservation; soil samples require cooling only.
- (2) For liquid samples only.
- (3) Liquid samples collected for metals analysis do not require refrigeration, however, samples are generally refrigerated along with the rest of the samples collected for ease in transport and shipping.
- (4) VTSR - Validated Time of Sample Receipt
- (5) The holding time for mercury analysis is 26 days from VTSR
- (6) Sample bottles must be of traceable quality (i.e., ICHM-300, Eagle Pitcher or equivalent).

labels, chain of custody forms, custody seals, traffic reports, routine analytical services (RAS) request forms, etc.) pertinent to sample custody and sample analysis protocol.

Chain of Custody

The chain of custody procedures are initiated in the field following sample collection. The procedures consist of: 1) preparing and attaching a unique sample label to each sample collected, 2) completing the chain of custody form, and 3) preparing and packing the samples for shipment. These procedures are further described in the following sections.

Sample Labels

Field personnel are responsible for uniquely identifying and labeling all samples collected during a field investigation. All labeling must be done in indelible/waterproof ink. Any errors are crossed out with a single line, dated, and initialled. Each sample label securely affixed to the appropriate sample container must contain the following information (refer to Figure 3-1 for an example sample label):

- Client/site name
- Unique project-specific sample identification number (i.e., station number)
- Sample location/description number
- Type of analysis to be performed and the name of the laboratory to whom the samples are being sent
- Sample volume, container type, and the type of chemical preservation used
- Sampling date and time
- Initials of the person obtaining the sample.

Chain of Custody Record

A chain of custody form (refer to Figure 3-2) must be completed for each sample set collected at a sampling location. The form is maintained as a record of sample collection, transfer, shipment, and receipt by the laboratory. The forms must also contain pertinent information concerning sampling locations, dates, and times; signatures of the sampling team members; types of samples collected along with a unique sample identification number; the number of samples collected and shipped for analysis in each lot; the project name and number; and the name of the laboratory to which the samples are being sent.

Transfer of Custody

Samples shall be accompanied by an approved and completed chain of custody form during each step of custody, transfer, and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them will sign, date, and record the time on the chain of custody form. In the case of sample shipment by an overnight courier, a properly prepared air-bill shall serve as an extension of the chain of custody form while the samples are in transit.

Sample Packaging and Shipping

Following sample collection, all samples shall be brought to an on-site location for batching and paperwork checks. At this central location, like sample types are matched (i.e., solids, liquids, etc.) with similar sample types from all sample locations. Labels, tags (as required), and log information are checked to be sure there is no error in sample identification. The samples are packaged to prevent breakage and/or leakage, and the shipping containers are labeled in accordance with the DOT regulations for transport.

As soon as field personnel are ready to transport samples from the field to the laboratory, the laboratory shall be notified by telephone of the shipment along with the estimated time of arrival. All samples will be shipped

X

directly to the laboratory via overnight carrier (or will be picked-up by the laboratory courier and transported back to the lab that same day/night). The field team shall determine whether it is best to directly transport the packages to the shipping office or to arrange for on-site pick-up. For each sample shipment to a specific subcontracting laboratory, an overnight air-bill must be properly completed. An example of a properly prepared air-bill used when shipping non-hazardous samples to the laboratory is shown in Figure 3-4. Unless field collected information indicates otherwise, all environmental samples collected shall be treated as non-hazardous aqueous liquids and non-hazardous soil/sediment.

Non-Hazardous Packaging and Shipping

Because of the expected non-hazardous nature of the collected samples, packaging and shipping criteria have been designed only to maintain chain of custody protocol, as well as to prevent breakage of the sample containers. The packaging and shipping procedures shall be as follows:

- Place a layer of cushioning material (e.g., vermiculite) in the bottom of the watertight insulated metal or equivalent strength plastic shipping containers.
- Wrap the properly labeled and secured glass sample bottles and purgeable vials with plastic bubble wrap. Place the wrapped containers into watertight zip lock bags and seal the bags closed.
- Place sample bottles (top side up) into the shipping container arranging the bottles so that the glass bottles are surrounded by plastic bottles.
- Using the necessary packing material, pack the sample bottles to ensure that they do not shift during transport.
- Fill any void spaces of the shipping container, around and on top of the sample bottles, with ice cubes or chips sealed in plastic bags or with blue ice.
- Seal the appropriate chain of custody form(s) in a zip-lock plastic bag, and tape it securely to the inside of the shipping container lid.

- Close and lock/latch the shipping container. Seal the space between the container body and lid with waterproof tape. (If the shipping container used is a picnic cooler, tape the drain plug closed to prevent any leakage of water as the ice packs melt during transport.)
- Apply several wraps of chain of custody tape around the shipping containers perpendicular to the seal to ensure that the lid remains closed if the latch is accidentally released or damaged during shipment. Do not obscure any stickers or labels on the shipping container with the chain of custody tape.
- Place a completed overnight carrier air-bill (refer to Figure 3-4 for an example of a properly completed Federal Express air-bill) on the lid of the shipping container. Include the name, address, and telephone number of the receiving laboratory and the return address and telephone number of the shipper on the air-bill.
- Place a "This End Up" label on the lid and on all four sides of the shipping container.
- Each shipping container must not weigh more than 150 pounds if it is to be shipped overnight by Federal Express.

Hazardous Packaging and Shipping

The packaging and shipping of known hazardous wastes must be in accordance with Department of Transportation regulations described in 49 CFR Parts 172 & 173. The packaging procedures (in addition to those listed above) must also include:

- Proper identification and classification of the hazardous materials
- Proper description and shipping name of the materials
- Usage of DOT approved shipping containers
- Proper marking of the shipping container to include:
 - Commodity description and DOT labeling
 - "This end up" labels for shipping containers of liquid samples
 - Name and address of shipper
 - Characteristics of hazardous materials such as corrosivity, ignitability, or flammability

- Sample container labeling consistent with shipping papers
- Inclusion of proper shipping papers listing the name and classification of the hazardous material and assurance that the labeling is consistent with the shipping papers. An example of a completed Federal Express air-bill for dangerous goods is shown in Figure 4-4.

4.5.3 Documentation

All sampling procedures, instrument calibration, and information pertinent to sampling conditions, progress, and field data collection must be documented following a prescribed set of guidelines. The documentation serves as a permanent and traceable record of all activities related to a specific field investigation project. The record must be legible and accessible to allow ease in verifying sampling activities and addressing future questions which may arise concerning such issues as sample integrity, sample traceability, etc.

All documentation must be recorded in permanent ink. Any errors must be crossed out with a single line, dated, and initialed; the use of white-out is not permissible.

The following serves as a guideline for the types of documentation that are required for site investigation at the Roxy Cleaners Site.

Field Logbook

A field logbook shall be maintained by each field team member for this hazardous waste field investigation project. The logbook shall be comprised of a bound book with consecutively numbered pages; no pages must be skipped when filling in the logbooks. The integrity of field documentation is further ensured by the use of field logbooks containing paper treated to repel the rain or any other aqueous splashings experienced during field documentation. Should more than one field logbook be required, they shall be numbered sequentially.

EXAMPLE FEDERAL EXPRESS DANGEROUS ARTICLES AIRBILL

[illegible]

The front of each field logbook shall contain the following information:

- Project name and number
- Name of the contract under which the investigation is being conducted
- Date(s) of use

The field logbook shall contain a diary of all pertinent project activities. Standard information recorded in the field logbook includes: general observations made in the field, identification and calibration of instruments used, and field data (refer to Figure 3-5 for an example of typical information that may be included at the beginning of each field logbook). Figures 3-6, 3-7 and 3-8 serve as guidelines for field logbook data entries for various sample collection procedures. In addition, Figure 4-5 depicts a field logbook data entry format for soil gas testing data.

Daily documentation in field logbooks shall include at a minimum:

- Date and time of personnel entries on-site, weather conditions, temperature
- List of start/stop times of all subcontractors hired for activities such as drilling, steam cleaning, well development
- List of the personnel present on-site during each sampling day to include all M&E personnel, subcontractors, and visitors
- List of the equipment decontaminated along with a reference to the procedures used
- Description of the sampling locations in reference to permanent landmarks
- List of any changes from standard operating procedures, decisions made in the field, and other pertinent information
- QA/QC samples associated with the samples collected
- Equipment and/or instrument identification numbers (if available) for those used
- Sample preservation techniques performed

Date: _____

[illegible]

- Air monitoring information gathered (e.g., HNu, radiation meter readings)
- Level of personnel protection mandated (e.g., Level B, C, D) and record of pertinent time intervals spent by each field team member at each level (e.g., time spent in Level C developing a well, time spent in Level C sampling soil from a backhoe, etc.)
- Other logs/paperwork used to document activities
- Instrument calibration information including the instruments calibrated during the day and the individual who performed the calibration (Note: Instrument calibration information should be documented in the field logbook as well as on the instrument calibration log kept with each instrument and serving to document instrument response over time.)
- List of the samples collected by media (i.e., soil, water, etc.)
- Comments relative to any problem areas that occurred during the day's activities, their final resolution, and any anticipated impact on the outcome of the field investigation

The last item to be entered at the bottom of each page of the field logbook shall be the signature of the person responsible for completing the data entry.

Project Daily Logbook

A unique project-specific logbook shall be maintained daily for each hazardous waste field investigation project. The logbook shall be comprised of a bound book with consecutively numbered pages. Should more than one project logbook be required, they shall be numbered sequentially.

The primary purpose of this logbook is to contain all the data specific to a particular project in one central location. In general, the daily logbook shall contain similar information as the field logbook. However, the daily logbook need not list all the actual field data and instrument calibration information generated during a sampling episode. References to other documents that contain specific field activity descriptions, outlines of any

administrative occurrences that have affected the field work for any given sampling activity, as well as a daily summary of the field activities, must be documented in the project daily logbook and kept up-to-date.

Field Equipment Logbook

A unique field equipment logbook is required for each piece of field equipment (i.e., the HNu photoionization detector, pH meter, conductivity meter, etc.). This logbook is used for the documentation of the proper use, maintenance, and calibration of the field equipment as well as for any information concerning the conformance and decontamination status of each piece of equipment.

The field equipment logbook is maintained and kept up-to-date following every use, maintenance, inspection, repair, and/or calibration of the equipment. Entries into the logbook should contain the following information:

- Signature of the person making the entry
- Date of entry
- Status of equipment in terms of its operational and decontamination standing
- Reference to the procedures used for calibration or maintenance as well as the procedural results and/or description
- Name of person(s) using the equipment and a brief description of the nature of the work
- Calibration log forms used to record all on-site calibrations

The field equipment logbook is comprised of a 3-ring notebook. Every page in the logbook must be signed by the field equipment maintenance person responsible for keeping each piece of equipment in proper repair and maintained for field use as a reflection of their review and approval of each entry.

Field Data Forms

Along with the completion of data entry in each of the above-mentioned logbooks, field data forms should also be completed and filed in a 3-ring notebook maintained at the sampling site for all field activities.

These forms include the following:

- Site Health and Safety Log - to maintain accurate health and safety records for each field team member
- Geologic Boring Log - to document soil boring operations
- Rock Coring Log
- Monitoring Well Installation Form
- Monitoring Well Development Form
- Monitoring Well Sampling Work Sheet

Chain of Custody

Possession of samples shall be traceable from the time a sample is collected until it is used as evidence in legal proceedings, if applicable. To adequately track sample possession, a documented chain of custody must be maintained (refer to Section 4.5.2 for more detailed guidelines concerning sample custody).

Materials Certification

Documentation (or copies) concerning the quality of all materials used on-site shall be retained on-site for the duration of the site investigation. The following list of materials certification documentation serves as an example of the minimum that shall be maintained:

- Analysis of all decontamination rinse water (i.e., both tap water and ASTM Type II water)

- Sieve analysis of filter pack material
- Manufacturer and lot number for calibration standards
- Material certificates for all construction materials
- Certificates of analysis for materials used in decontamination
- Certificates of cleaning or decontamination furnished by the equipment manufacturer (The certificate shall detail the cleaning procedure.)

Material blanks are samples of the construction materials used in sampling equipment, such as monitoring wells and lysimeters, that are archived for future analysis. Material blanks are required for all the applicable construction materials used during the site investigation. The types of material that shall be sampled are pipe, well screen, filter pack material, drilling additives, bentonite, and cement.

Dry measure materials such as cement, bentonite, and silica sand shall be sampled and labeled in a similar fashion as described for the environmental and field QC samples. Approximately 2 pounds of dry material is required per sample. Liquid materials such as drilling additives, paints, and lubricants require at least 1 liter of sample. Rigid materials such as riser pipe and well screen require at least 2 feet of material per sample.

The archived samples shall be stored in clean containers and protected from exposure to the elements for the duration of the site investigation. The NYSDEC project manager for the Roxy Cleaners Site shall determine the length of time material blanks are to be stored following completion of the site investigation.

Variances

A variance is a deviation from project requirements. All variances from procedural and planning/design documents and other project requirements shall be documented on the Field Change Request form. The NYSDEC project manager

for the Roxy Cleaners Site shall approve field changes that have a major impact on cost, schedule, and/or technical performance prior to incorporation.

Field changes and deviations from project planning documents shall be reviewed and approved by the Project Manager or Project Quality Assurance Officer. All deviations from procedural and planning documents shall be recorded in the site logbook. Project reports shall detail all field changes and deviations.

4.6 REFERENCES

The following references were cited in Section 4.2 of this document:

Burris, D., "Examination of Soil Vapor Sampling Techniques," Sound National Outdoor Action Conference on Aquifer Restoration, Groundwater Monitoring, and Geophysical Methods, Las Vegas, Nevada, May 23-26, 1988.

Spittler, T.M., R. Siscanaw, H.W. Lataille, and P.A. Parks, "Correlation Between Field GC Measurement of Volatile Organic Compounds and Laboratory Confirmation of Collected Field Samples Using GC/MS," APCA Specialty Conference on Measurement and Monitoring of Non-Criteria (Toxic) Contaminants in Air, Chicago, Illinois, 1983.

U.S. Environmental Protection Agency, "Soil Gas Sensing for Detection and Mapping of Volatile Organics," EPA-600/8-87-036, 1987.

The following references were cited in Section 4.3 of this document:

Driscoll, F.G., "Groundwater and Wells," Johnson Division, St. Paul, Minnesota, 1986.

Gilham, R.W., Robin, M.J.L., Barker, J.F., Cherry, J.A., "Groundwater Monitoring and Sample Bias," Environmental Affairs Department, American Petroleum Institute, 1983.

Morrison, R.D., "Ground Water Monitoring Technology," TIMCO Mfg., Inc., Prairie du Sac, Wisconsin, 1983.

Roy, S.P., Milner, P., D'Amore, D., Drake, D., White, E.C., Groundwater Monitoring Handbook, Commonwealth of Massachusetts DEQE, publication #13,994-152-80-4-85-CR, April, 1985.

U.S. Department of Interior, National Handbook of Recommended Methods for Water-Data Acquisition, U.S. Geological Survey, Reston, VA, Vol. 2, 1987.

U.S. Environmental Protection Agency, "A Compendium of Superfund Field Operations Methods," EPA-540/P-87/001, December 1987.

U.S. Environmental Protection Agency, "Manual of Groundwater Quality Sampling Procedures," EPA-600/2-81-160, 1981.

U.S. Environmental Protection Agency, "RCRA Groundwater Monitoring Technical Enforcement Document, U.S. Government Printing Office: 1987 0-172-437, 1986.

U.S. Environmental Protection Agency, "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA-600/2-80-018, 1980.

U.S. Environmental Protection Agency, "Soil Sampling Quality Assurance User's Guide," EPA-600/4-84-043, 1984.

U.S. Environmental Protection Agency, "Test Methods for Evaluating Solid Wastes, 3rd Edition," SW-846, November 1986.

EPA Technical Monographs:

- No. 15, Purposes and Objectives of Sampling
- No. 16, Water Sampling Methods
- No. 17, Soil and Sediment Sampling Methods
- No. 18, Sampling and Biological Specimens
- No. 19, Methods of Collecting Concentrated (Hazardous) Samples
- No. 20, Container Opening Techniques
- No. 21, Geohydrological Techniques
- No. 22, Sample Handling, Packaging, and Shipping Procedures

The following references were cited in Section 4.4 of this document:

U.S. Environmental Protection Agency, "A Compendium of Superfund Field Operations Methods," EPA-540/P-87/001, December 1987.

U.S. Environmental Protection Agency, "Manual of Groundwater Quality Sampling Procedures," EPA-600/2-81-160, 1981.

EPA Technical Monographs:

- No. 19, Methods of Collecting Concentrated (Hazardous) Samples

APPENDIX A

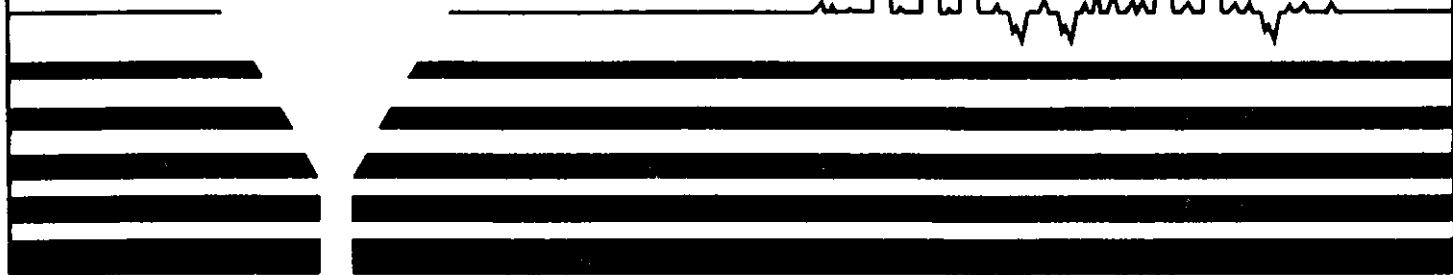
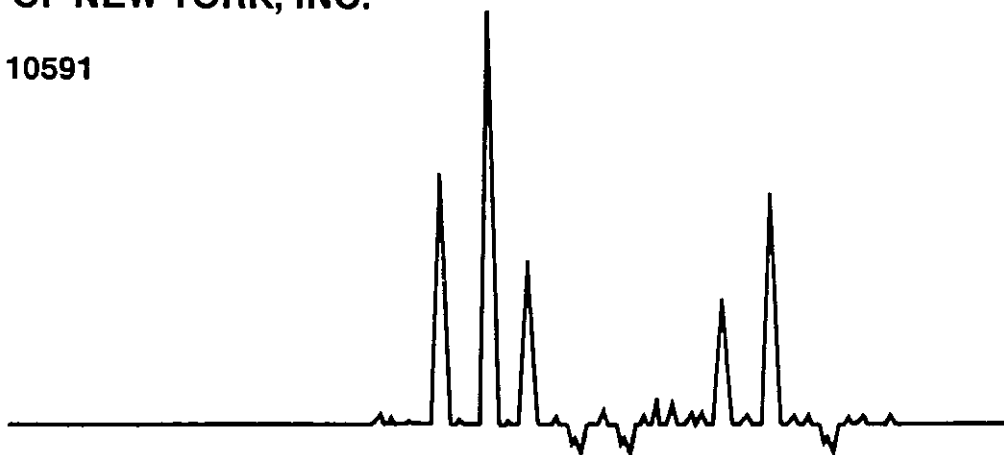
227125-36

**LABORATORY SERVICES
ROXY CLEANERS SITE**

SUBMITTED TO:

METCALF & EDDY OF NEW YORK, INC.
303 South Broadway
Tarrytown, New York 10591

AUGUST 1991



H2M LABS, INC.

Environmental Testing Laboratories
575 Broad Hollow Road, Melville, New York 11747

1. Sample Preservation and Receiving



H2M LABS, INC.

1. SAMPLE PRESERVATION AND RECEIVING

1.1 SAMPLE CONTAINERS

Sample containers will be provided by H2M. The wide scope of this project necessarily mandates that several types of sampling containers may be used. Materials must be selected that would not result in interference with the analysis. Each sample container must have a label which contains all the information necessary to identify the sample. The amount of information on the label may vary depending on the source and other factors, but, in general, should include:

- number of bottles per analysis
- collector's name
- sample identification
- sample location
- date and time of collection
- water temperature
- depth of sample
- tide stage
- atmospheric conditions

For CLP projects, bottles will be supplied by I-Chem Research, Hayward, California. These will be prepared using CLP-approved cleaning protocols, quality control analyzed and certified by the vendor. (See Figure 4.3 and 4.4 for an example of the certification of these containers.) The bottles used from I-Chem are verified as non-contaminated by checking a random sampling of bottles (5 percent) and filling with distilled water and analyzing for the parameters that would be analyzed from that bottle. This record is kept on file in the Quality Control department. Any positive readings for any analytes are flagged and the supervisor and QA Manager are notified. No bottles from the effected lot are used until the source of contamination is determined and remedied.

1.2 SAMPLE PRESERVATION AND SHIPMENT

Sample preservation is dependent upon which analyses are to be performed. A summary of sample container types and preservation methods is presented in Table 1A. The majority of samples will need to be packed in ice and shipped to the laboratory within 24 hours of collection. Samples requiring only trace metals analyses would be the only exception. Table 1B reference the sample container code with a figure of the bottle type to be used.

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1.3 SAMPLE RECEIVING

Upon arrival at H2M, samples are to be inspected for integrity. That is, they are examined for breakage, leakage, air bubbles (for purgeables) and proper labeling.

Personnel are in the laboratory during the following hours:

Mondays to Fridays; 7:00 a.m. to 10:00 p.m.
Saturdays and Sundays; 9:00 a.m. to 3:00 p.m.

We would request, however, if deliveries must be made later than 5:00 p.m. on weekdays, or anytime on weekends, that we be contacted in advance so that arrangements can be made with our staff to ensure proper receipt of samples.

1.4 LABORATORY WATER PREPARATION

All laboratory water for reagents, trip and field blanks, and general laboratory water is prepared daily using the ultra pure still in the GC laboratory. The Wet Chem and metals labs use water from the Belco all glass stills.

The GC lab and GC/MS lab use the water from the ultra pure system as the source for the method blanks each day. No volatile organics or semi-volatile organics greater than 1 ug/l can be detected in this water. The conductivity of this water is checked daily. Monthly, the conductivity is determined by comparison to another conductivity meter. These values are recorded in a log book.

1.5 FIELD AND TRIP BLANK SAMPLE PREPARATION

Laboratory distilled water, which is certified as pure, is used for all field and trip blanks. This water is verified as pure by analysis prior to filling the trip and field blank bottles by analysis on the day the bottles are to be filled. These samples are analyzed for volatile organics, base-neutral-acid extractables and pesticide/PCB's. A record of this is on file in the QC department. No organic analytes can be detected in these samples above 1 ug/l.

Preservatives are added to the sample containers prior to shipment.

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1.6 NOTES TO TABLES OF REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

- 1) Polyethylene (P) or Glass (G).
- 2) Sample preservation should be performed immediately upon sample collection.
- 3) When any sample is to be shipped by common carrier or sent through via US mail, it must comply with the Department of Transportation's Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements, the Office of Hazardous Materials, Materials Transportation Bureau, and Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCL) in water solutions at concentrations of 0.04 percent by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15 percent by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35 percent by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080 percent by weight or less (pH about 12.30 or less).
- 4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5) Should only be used in the presence of residual chlorine.
- 6) Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- 7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- 8) Guidance applies to samples to be analyzed by GC, LC or GC/MS for specific compounds.

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- 9) Sample receiving no pH adjustment must be analyzed within seven days of sampling.
- 10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
- 11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 40C, reducing residual chlorine with 0.008 percent sodium thiosulfate, storing in the dark and adjusting the pH to 6-9; samples preserved in this manner may be held seven days before extraction and 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote five (Re: the requirement for thiosulfate reduction of residual chlorine), and footnotes 12 and 13 (Re: the analysis of benzidine).
- 12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 + 0.2 to prevent rearrangement to benzidine).
- 13) Extracts may be stored up to seven days before analysis of storage is conducted under an inert (oxidant-free) atmosphere.
- 14) For the analysis of dipheylnitrosamine, add 0.008 percent Na₂A₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- 15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008 percent Na₂S₂O₃.

TABLE 1A

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, HOLDING TIMES

<u>Parameter Name</u>	<u>Container(1)</u>	<u>Preservation (2)(3)</u>	<u>Maximum Holding Time(4)</u>
CLP Purgeable halocarbons	40ml vial G. Teflon lined septum	Cool 4°C.	Water 7 days Soil 7 days
CLP Purgeable aromatics	40ml vial G. Teflon lined septum	Cool 4°C.	Water 7 days Soil 7 days
Acrolein acrylonitrile	1 liter G. G. Teflon lined cap	Cool 4°C.	7 days
Phenols (11)	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5)	5 days until extraction: analysis 40 days after extraction (13)
Benzidines (11)	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5)	5 days until extraction: analysis 40 days after extraction
Phthalate esters	1 liter G. Teflon lined cap	Cool 4°C.	5 days until extraction: analysis 40 days after extraction
Nitro-samines(11)(14)	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ store in dark	
CLP PCB's (11)	1 liter G. Teflon lined cap	Cool 4°C. store	5 days until extraction: analysis 40 days extraction
Nitroaromatic and Isophorones	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5) store in dark	Water 5 days/Soil 10 days until extraction: analysis 40 days after extraction
Polynuclear aromatic hydrocarbons(11)	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5) store in dark	Water 5 days/Soil 10 days until extraction: analysis 40 days after extraction
Haloethers (11)	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5) store in dark	Water 5 days/Soil 10 days until extraction: analysis 40 days after extraction
Chlorinated hydrocarbons	1 liter G. Teflon lined cap	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5) store in dark	Water 5 days/Soil 10 days until extraction: analysis 40 days after extraction
CLP Pesticides	1 liter G. Teflon lined cap	Cool 4°C. pH 5-9 (15)	Water 5 days/Soil 10 days until extraction: analysis 40 days after extraction
Purgeable aromatics method 602	40ml vial G. Teflon lined septum	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ to pH 2 (9) with ascorbic acid	7 days

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TABLE 2.1A (cont'd)

<u>Parameter Name</u>	<u>Container(1)</u>	<u>Preservation (2)(3)</u>	<u>Maximum Holding Time(4)</u>
Purgeable aromatics method 601	40ml vial G. Teflon lined septum	Cool 4°C. 0.008% Na ₂ S ₂ O ₃	7 days
CLP Acid extractables	1 liter G. Teflon lined	Store at 4°C.	5 days for extraction: analysis 40 days after extraction
CLP Base/neutral extractables	1 liter G. Teflon lined	Store at 4°C.	5 days for extraction: analysis 40 days after extraction
Phosphorus, total	P.G	Store at 4°C. H ₂ SO ₄ to pH <2	26 days
Residue, total	P.G	Store at 4°C.	5 days
Residue, filterable	P.G	Store at 4°C.	5 days
Residue, non-filterable (TSS)	P.G.	Store at 4°C.	5 days
Residue, settleable	P.G.	Store at 4°C.	24 hours
Residue, volatile	P.G.	Store at 4°C.	5 days
Silica	P	Store at 4°C.	26 days
Specific conductance	P.G	Store at 4°C.	26 days (field test)
Sulfate	P.G	Store at 4°C.	26 days
Sulfide	P.G	Store at 4°C. zinc acetate plus sodium hydroxide to pH >9	5 days
Sulfite	P.G	None required	Analyze immediately
Sufactants	P.G	Store at 4°C.	24 hours
Temperature	P.G	None required	Analyze immediately (field test)
Turbidity	P.G	Store at 4°C.	48 hours
Coliform, fecal and total	P.G	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5)	6 hours
Fecal streptococci	P.G	Cool 4°C. 0.008% Na ₂ S ₂ O ₃ (5)	6 hours

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TABLE 1B

SAMPLE CONTAINER CODES

Code	Type	Analysis Code
<u>INORGANIC</u>		
A	Unpreserved plastic	acidity & CO ₂ , volatile acids alkalinity, BOD 5, bromide chloride, Cl ₂ demand, chloride res, color, conductivity, MBAS, fluoride, NO ₃ , NO ₂ , odor, pH, ortho phosphate, total phosphate, solids, sulfate, sulfite, turbidity, hex, chrom.
B	(HNO ₃) plastic	metals
C	(H ₂ SO ₄) 1 qt. glass	COD, phenois, TKN, ammonia
D	(H ₂ SO ₄) small glass	TOC, POC
E	(Na SO ₂) small glass	TOX, POX
F	unpres. 40 ml vials	sulfide
G	125 ml ster. glass	total coliform, fecal coliform
H	1 qt. glass plastic (NAOH)	cyanide
<u>ORGANIC</u>		
I	1 liter glass w/Teflon lined caps	PCB's organo-chloride pest., haloethers, phenois, chlorinated hydrocarbons, herb, triazines, acid ext., base neutral ext.
J	40 ml glass vials w/Teflon lined septa	purgeable halocarbons, trihalomethanes, purgeable aromatics, priority pollutant volatiles
K	unpreserved glass 1 qt.	soil analysis, ignitability, reactivity corrosivity

TABLE 1C

BOTTLE WASHING METHODS

Bottle Type

A & H	1) Rinse 3 times with hot tap water 2) Rinse 3 times with distilled water 3) Air dry and cap
B	1) Wash containers and caps with hot water and non-phosphate detergent 2) Rinse 3 times with hot tap water 3) Rinse with 1:1 nitric acid 4) Rinse 3 times with distilled water 5) Air dry and cap
C	1) Wash containers and teflon lined caps with hot water and non-phosphate detergent 2) Rinse 3 times with hot tap water 3) Rinse with chromic acid 4) Rinse 3 times with hot tap water 5) Rinse 3 times with distilled water 6) Air dry and cap
D & E	1) Wash containers and teflon lined caps with hot water and non-phosphate detergent 2) Rinse 3 times with hot tap water 3) Rinse with chromic acid 4) Rinse 3 times with hot tap water 5) Rinse 3 times with distilled water 6) Air dry and cap
F & J	1) Wash vials, caps and septa in hot water and non-phosphate detergent 2) Rinse 3 times with hot water 3) Rinse 3 times with distilled water 4) Oven dry for 1 hour at 110°C 5) Cap vials while still hot
G	1) Wash with hot water and non-phosphate detergent 2) Rinse 8 times with cold tap water 3) Rinse 3 times with distilled water 4) Air dry 5) Add 3-4 drops of 10% Sodium thiosulfite and loosely cap 6) Autoclave for 30 minutes at 121°C 7) Remove from autoclave and tighten caps

2. Progression, COC, Sample Storage

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2. PROGRESSION, COC, SAMPLE STORAGE

H2M has a standard operating procedure for documenting the receipt, tracking and compilation of sample data. The chain of custody (COC) procedure begins with the preparation of the bottles. The cooler is sealed with chain custody tape and the COC form is completed with the number of bottles prepared and the name of the preparer. The bottles are then sent to or picked up by the client. After receipt of the samples at the laboratory, they are routed through a sequence of steps from preparation to analysis until the final report is issued to the customers.

A flow diagram summarizing all steps involved with the sample progress is presented in Figure 5.1. The diagram includes the tracking of a sample progress as well as analysis and reporting.

2.1 STANDARD PROCEDURES FOR SAMPLE CUSTODY

If samples collected require special security and documentation of custody a special procedure is in place for sample tracking as described below.

A. PREPARATION OF SAMPLE BOTTLES AND COOLERS

Coolers	a. Cooler(s) to be used for sample containers are scrubbed clean with a non-contaminating detergent. b. Cooler(s) are then flushed with organic-free distilled water and dried.
Glass and Plastic Sampling Containers:	a. All containers used are brand new. They are purchased specially cleaned and certified from I-Chem in Hayward, California. These containers are used only once for the particular job intended. In addition, all cases of containers are supplied with lot numbers for easy tracking if necessary. (See section 4.1 for I-Chem information.) b. Containers are assembled in conjunction with the number of sample locations (including: MS/MSD, trip blank(s) and field blank(s)) and the proper preservative, if any, for the particular

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analysis required: 50 percent nitric acid-Metals; 100 percent sulfuric acid-Phenol, TKN, Nitrogen Series, Oil/Grease etc.; Sodium Hydroxide-Cyanide.

- c. Each container is affixed with an official label (including a lot number). The label is then earmarked accordingly: HNO₃; H₂SO₄; or NaOH.

Packaging of Glass and Plastic Sampling Containers:

- a. All glass containers are individually fitted with resealable bubble-pak bags.
- b. Both glass and plastic containers are placed upright into coolers. (Bottles will be separated into individual location kits at the clients request.)
- c. 40 ml. vials are always packaged in bubble-pak bags, placed into small cardboard boxes and placed within the cooler or, because of their light weight and small size, laid on top of the larger sample containers.
- d. Minimum breakage is achieved following this technique.

External COC and Custody Seals:

- a. External chain of custodies (with H2M Labs logo, address and phone number) are prepared. All samples for NJDEP Task IV (CLP) word will be prepared with the NJDEP COC forms: DEP Form 060 (COC record with shuttle or DEP Form) and DEP Form 009 (COC without shuttle. See forms at end of section).
 - Container type (with or without preservative).
 - Number of containers for each sample location, including MS/MSD, trip blank and field blank.
 - Any distinctive notification.
 - Sample Custodian's signature.
 - Receiver's signature.

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- Date and time of relinquishment.
- COC is sealed into a zip-loc bag and placed into cooler.
- Samples preservative will be listed.

Custody seals for return trip are also included in plastic bag. Seal (1) per cooler is affixed.

- b. Custody seals are signed, dated and affixed to each cooler (signature on custody seal (1) must be positioned directly over groove betwixt the lid and basin parts of the cooler) just prior to customers receipt.
- c. Custody seals are reinforced onto cooler with fiber tape if required. For a soil sample designated as a MS/MSD sample, no additional sample is necessary for soil.

B. DELIVERY OF COOLERS TO CLIENT

The bottles are either sent to the client (Federal Express) or picked up by the client. The client fills the bottles, (all sample bottles must be sealed tightly in the field to prevent any leakage of samples or contamination) completes the COC of custody form, and completes the sample request form, fills coolers, seals cooler with custody tape and either delivers cooler to the laboratory or ships it to lab.

C. DELIVERY OF COOLER TO H2M SAMPLE CUSTODIAN

Sample custodian examines cooler. If any hazard is suspected, cooler is moved to special hood before opening. Hazard suspect would include any previous knowledge from client or broken glass detected.

D. OPENING OF COOLER

External COC form is removed and time and date of arrival is entered (for NJ Forms 009 or 060 used).

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- Shipment is checked for completeness. Samples are examined for damages.
- All sample bottles are checked that they are properly sealed.
- Shortages and damages are noted on the external COC.
- Sample request form is compared to external COC form. For NJDEP, sample request form DEP 059 will be utilized.
- A copy of external COC is given to sampling personnel. The original is filed in the client file with the sample custodian.
- Client is notified immediately of any damage or shortage of samples.

E. CONTINUING CHAIN-OF-CUSTODY

The custodian assigns H2M Lab numbers to the samples. Custodian transfers samples to departmental custodians. An internal chain-of-custody form is completed with client project number, date of receipt, and listing of samples by customer numbers and H2M Lab identification numbers. The sample custodian and departmental custodian sign for transfer with date and time. The department custodian places samples in secured areas for storage.

The departmental custodian relinquishes samples to the technicians for sample preparation and/or analysis. The analysts sign for the samples and extracts/digestives each time the samples exchange hands. Upon completion of analysis, samples are returned to the custodian.

F. LOG BOOKS FOR PREPARATION AND ANALYSIS

Separate log books are on file in the laboratory departments for recording all aspects of sample preparation and analysis, with signatures of the analysts and dates steps were performed.

G. INCLUSIONS IN REPORT SENT TO CLIENT

Copies of sample request forms, external chain-of-custody forms and internal custody are included in report. Copies of any work sheets/analysts log book pages submitted.

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2.2 STANDARD OPERATING PROCEDURE FOR HAZARDOUS WASTE GENERATION, STORAGE & DISPOSAL

H2M Labs, Inc. is classified as a small quantity generator of hazardous waste by the New York State Department of Environmental Conservation. H2M Labs has been assigned EPA ID number NYD982283053. The current rate of hazardous waste generation is approximately 550 kilograms per month. The operations involving hazardous waste generation, storage and disposal are highly regulated. The intent of this Standard Operating Procedure (SOP) is to ensure that hazardous waste management at H2M Labs remains in compliance with Federal and State regulations at all times. In addition, this SOP is intended to promote economical waste disposal practices, reduce potential for waste spills, and promote employee health and safety.

Waste Storage Facility:

H2M Labs waste storage facility has been designed and constructed in accordance with Article 12 of the Suffolk County Sanitary Code. This room includes secondary containment for fifteen 55 gallon drums, explosion proof lighting/HVAC systems and a fire suppression system. The storage facility is located adjacent to H2M Labs eastern lobby. Access to the waste storage facility is restricted and is controlled by the receiving department.

Waste Generation:

Inherent in the nature of H2M Labs operations is the utilization of various chemicals to conduct a wide range of analytical procedures on samples of varying composition. Once these chemicals are used in an analysis and are no longer useful to the laboratory, the chemicals along with the sample analyzed become waste. If the sample or chemical waste contains sufficient material that is regulated by State or Federal hazardous waste regulations, 6NYCRR Part 371 and/or 40 CFR 260, respectively, they are considered hazardous waste. Samples that are not hazardous or contaminated are disposed of through conventional means.

All hazardous wastes are accumulated for removal and disposal by an outside contractor permitted to transport, treat,

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store, and dispose of hazardous waste by NYSDEC and/or USEPA.

UNDER NO CIRCUMSTANCES ARE ANY HAZARDOUS WASTES DISCHARGED INTO ANY SINK OR DRAIN.

H2M Labs generates a variety of hazardous wastes. The major quantity of wastes generated are segregated into 55 gallon drums as follows: waste acids, waste methylene chloride/chloroform, waste ether, and waste granular activated carbon. The remaining hazardous wastes that are generated in small quantities primarily consist of contaminated samples, prepared samples, and expired or off spec. analytical standards.

Hazardous Waste Accumulation and Storage:

Both bulk and small quantity hazardous wastes are initially accumulated in the section of the laboratory where they are generated. Bulk wastes are initially stored in containers ranging from one liter to five gallons in size. Upon accumulation of a maximum of five gallons of bulk hazardous waste, the waste is transferred to a designated 55 gallon drum in the hazardous waste storage facility by the department head or an other authorized person appointed and trained to transfer the waste.

In order to gain access to the waste storage facility, a list of types and quantities of wastes to be transferred is first submitted to the receiving department. The receiving department maintains the lists of wastes transferred into the storage room.

2.3 HAZARDOUS WASTE DISPOSAL

All hazardous wastes are accumulated and stored for disposal by fully licensed transporter and treatment, storage and disposal facility (TSD). During transfer of wastes from the storage room by the disposal contractor, spill controlled equipment is maintained at hand to respond to potential spills. All wastes are manifested in accordance with federal and state regulations. Final disposal on all wastes is conducted through physical treatment and/or incineration.

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2.4 STORAGE

The samples are stored in the following areas.

CLP VOA:	Samples are stored in the GC/MS Laboratory in a locked refrigerator at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$ and protected from light from receipt until analysis.
CLP BNA and Pest/PCB:	Samples are stored in the Special Process Laboratory in a locked refrigerator at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$ and are protected from light from receipt until extraction and analysis. After analysis, extracts and unused samples are protected from light and stored at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$. The sample extracts are stored in the locked refrigerator between the GC/MS and Special Process Laboratory.
CLP Metals:	Samples are stored in a locked cabinet in the Metals Laboratory.
CLP Cyanide:	Samples are stored in a locked refrigerator at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in the Wet Chemistry storage area.
Final Storage:	Approximately 60 days after the report is submitted to the client, samples are moved to final storage. The CLP BNA and Pest/PCB extracts are stored in a locked refrigerator in the GC Lab. All other sample bottles are stored in a secured room in the laboratory. For NJDEP, 30 days prior to holding the samples for one year, a letter will be written to NJDEP requesting permission to dispose of the samples. No sample is to be disposed without written permission from the client.

2.5 SECURITY

The entire building is equipped with security system monitored by a private alarm company which notifies the local police in the event of an emergency. The laboratory area is divided into separate zones. The access doors to these areas are wired with sensors so that the zones can be operated individually. The GC/MS area is independently locked. Keys to the instrument room are in the possession of the senior analysts and management personnel.

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TABLE 2A

NUMBER AND TYPE OF WATER BOTTLES FOR A CLP PROJECT

<u>Analysis</u>	<u>No. of Bottles</u>	<u>Type of Bottle</u>	<u>Preservation</u>
CLP VOA	2	40 ml glass vials w/Teflon lined septa	Cool 4°C
CLP BNA CLP Pest/PCB	3	1 liter glass w/Teflon lined septa	Cool 4°C
CLP Metals	1	1 liter plastic	HNO ₃ added
CLP Cyanide	2	1 liter glass w/teflon lined cap	H ₂ SO ₄ preservative

NUMBER AND TYPE OF WATER BOTTLES FOR SAMPLE DESIGNATED AS MS/MSD

<u>Analysis</u>	<u>No. of Bottles</u>	<u>Type of Bottle</u>	<u>Preservation</u>
CLP VOA	4	40 ml glass vials w/Teflon lined septa	Cool 4°C
CLP BNA CLP Pest/PCB	7	1 liter glass w/Teflon lined cap	Cool 4°C
CLP Metals	2	1 liter plastic	HNO ₃ added
CLP Cyanide	4	1 liter glass w/teflon lined cap	H ₂ SO ₄ added

NUMBER AND TYPE OF SOIL BOTTLES PER SAMPLE FOR CLP PROJECT

<u>Analysis</u>	<u>No. of Bottles</u>	<u>Type of Bottle</u>	<u>Preservation</u>
CLP VOA CLP BNA CLP Pest/PCB CLP Metals CLP Cyanide	4	8 oz. amber glass jar w/teflon lined cap	Cool 4°C

For sample designated as the MS/MSD no additional sample is necessary for soil.

3. NYSDEC ASP Method 89-1 Volatile Organics

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3. NYSDEC ASP METHOD 89-1 VOLATILE ORGANICS

3.1 SUMMARY OF PROCEDURES

The protocol for volatile analysis in all types of solid waste matrices, soils and ground water that is employed in our laboratory is the NYSDEC CLP. The procedure includes purging with inert gas to release the compounds from the matrix, concentrating them on a trap and transfer onto the chromatographic system. The compounds are separated on a packed or capillary column in the GC and analyzed on a quadropole MS.

Three instruments are available, each comprising a "purge and trap" unit, an autosampler and a GC/MS instrument for analysis. The list of analytes and the quantitation limits for NYSDEC is listed in the table following this section.

3.2 HOLDING TIMES

All samples for the NYSDEC protocol must be analyzed within seven days regardless of matrix.

3.3 GC/MS CALIBRATION PROCEDURES

This section outlines the minimum operations necessary to satisfy analytical requirements associated with the determination of the organic target compound list analyses in water and soil/sediment samples. The following operations will be performed routinely: Documentation of Mass calibration and abundance pattern; Documentation of GC/MS RF stability of response factors against internal standards; Internal standard response and retention time monitoring.

3.4 TUNING GC/MS MASS CALIBRATION

Prior to initiating data collection, it is necessary to establish that the instrument meets the standard mass spectral abundance criteria. This is accomplished through the analysis of p-bromofluorobenzene (p-BFB) for volatile compounds. The ion abundance criteria for the calibration compounds should be met before any samples, blanks, or standards, can be analyzed. p-BFB must be analyzed every 12 hours. The criteria in the table below must be met.

BFB - Each GC/MS system used for the analysis of the volatile TCL compounds must be tuned to meet the abundance

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criteria which should be demonstrated every 12 hours. Results of the p-BFB analyses have to be filed by the analyst. The results have to be tabulated in the appropriate forms for documentation, apart from supplying the bar graph plots and mass abundance listing.

p-BFB Key ions and ion abundance criteria (FOR NYSDEC)

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	15.0 to 40.0% of the base peak
75	30.0 to 60.0% of the base peak
95	Base peak, 100% relative abundance
96	5.0 to 9.0% of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50.0% of the base peak
175	5.0 to 9.0% of mass 174
176	> 95.0% but <101.0% of mass 174
177	5.0 to 9.0% of mass 176

Whenever the laboratory takes corrective action which may change or affect the tuning criteria for p-BFB (e.g. ion source cleaning or repair, etc.), the unit must be verified prior to initiating sample analysis. p-BFB criteria should be met before any samples, sample extracts, blanks or standards are analyzed. Any samples analyzed when tuning criteria have not been met may require re-analysis.

3.5 INITIAL CALIBRATION

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must be initially calibrated at a minimum (five) concentrations to determine the response curve for all targeted compounds. Average response factors are computed for the linear range. For GC/MS analysis, typical linear ranges are 20 ug/L to 200 ug/L for volatiles.

It is important that the initial calibration is performed with the same instrument parameters as the samples are analyzed. This applies to settings for the GC and MS as well as purge and trap parameters (e.g. for heated purge and trap and different purge volumes individual initial calibration curves or average RRF's have to be prepared).

NYSDEC CLP criteria specify the concentrations levels for initial calibration and also the specific internal standard to be used on a compound-by-compound basis. The relative response factor (RRF) for each compound is calculated at each concentration level using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \quad (2.1)$$



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where: A_x = area of the characteristic ion
for the compound measured
 A_{is} = area of the characteristic ion
for the specific internal
standards
 C_{is} = concentration of the internal
standard (ng/ul)
 C_x = concentration of compound to be
measured (ng/l)

For NYSDEC, the concentration of the initial calibration is 20, 50, 100, 150 and 200 ug/L.

The internal standards are the same for all protocols; bromochloromethane, 1,4-difluorobenzene and chlorobenzene-d5 at a concentration of 50 ug/L each. Surrogate standards are added to NYSDEC samples; these include: 1,2-dichloroethane-d4, bromofluorobenzene and toluene-d8 at a concentration of 50 ug/L (see Table 3C and Table 3D for ions.)

3.6 CALIBRATION CHECK COMPOUNDS

Using the RRF from the initial calibration, the percent relative standard deviations (%RSD) are calculated for certain calibration check compounds, and six volatile compounds and as classified in the CLP, the following equation is used:

$$\%RSD = \frac{SD}{X} (100)$$

where: RSD = Relative Standard Deviation
SD = Standard Deviation of initial
five RF's (per compound)
X = mean of initial three or five
RF's (per compound)

The percent RSD for each individual calibration check compound must be less than 30 percent. The criteria must be met for the initial calibration to be valid.

NOTE: For NYSDEC work all compounds must show <35 percent for RSD for the calibration to be valid.

3.7 SYSTEM PERFORMANCE CHECK

A system performance check must be performed to ensure minimum average RRF's are met before the calibration curve is used. The CLP classifies certain compounds System Performance

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Check Compounds (SPCC's) for which a minimum relative response factor must be obtained.

For Volatiles, the five SPCC's 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 RF's 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.

Note: For calculating the RF's of the xylenes, since o-xylene is the single eluting isomer on the capillary column, its area and concentration are used to derive the RF.

For 1,2-dichloroethene total, the cis and trans stereoisomers separate on the capillary column. Total the area and concentrations from both the cis and trans for the RF calculation.

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 percent, the initial calibration is assumed to be valid. If the criteria are not met (>30 percent difference) for any one calibration check compound, corrective action must be taken. Problems similar to those listed under SPCC should affect the criterion. If no source of the problem can be determined after corrective action has been taken, a new initial five-point calibration must be generated. The criterion must be met before sample analysis begins.

NOTE: NYSDEC CLP protocol requires all compounds to show 35 percent RPD for the volatile scan.

3.8 CONTINUING CALIBRATION

A check of the initial calibration must be made every 12 hours. The concentration for each volatile compound in the continuing calibration standard(s) is 50 ug/L. All SPCC and CCC criteris, as mentioned above, must be met for the continuing calibration to be valid. All %D's must be less than 35 percent.

Internal standard area responses and retention times (RT) must be evaluated. The RT for any internal standard must not deviate by more than 30 seconds in any blank or sample as compared with the 12 hour check standard. The extracted ion

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current profile of the internal standard must be within (-50 percent to +100 percent).

3.9 SUMMARY OF SAMPLE ANALYSIS

For Water samples: Five ml of sample is purged at ambient temperature. The purgeables are transferred from the aqueous to the vapor phase. The vapor phase is trapped onto a sorbent. The sorbent is heated and backflushed with inert gas onto a GC column. The MS is used as the detector.

For Low Level Soils: Five grams of soil is purged with reagent water at an elevated temperature. (The same as above)

For Medium Level Soils: A measured amount of soil is extracted with methanol. A portion of the methanol extract is diluted to five mls with reagent water.

Water Samples. Water samples must warm to ambient temperature.

- . GC/MS operating conditions are listed in Table 3.1C.
- . Five ml of sample is loaded with syringes.
- . Add the surrogate standards (NYSDEC) and the internal standards to have a concentration of 50 ug/L of each.
- . The syringe is loaded onto the dedicated samplers of the autosampler.
- . The sample is then purged, trapped, desorbed and analyzed (see Table 3A)

Check analysis for saturation (above 200 ug/L). If saturation exists analyze a blank after the sample. The sample must be reanalyzed at a lesser amount to have the analyte within the linear range.

Soil Samples:. Fill a leur lock syringe with five ml of reagent water and add 10ul of the surrogate standards and 10ul of internal standard.

- . Weigh out the five grams of sample (note actual weight to the nearest 0.1 gm).

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- . Add the spiked reagent water to the purge device.
- . Weigh another five grams of sample and determine the percent moisture.
- . Heat the sample to 40°Celsius±/degrees Celsius and purge for 11 minutes.
- . Proceed with analyses.

Medium Level
(Ambient
Temp)

- . Weigh 4gm into a tared 15/ml vial. Determine percent moisture.
- . Add 9 ml of methanol to the vial. Add 1.0 ml of the surrogate (aka system monitoring compounds) to the vial.
- . Cap and shake for two minutes.
- . Transfer 1ml of extract into a 40ml vial.
- . Add 1ml of reagent methanol to the vial (Reagent Blank).
- . Estimate concentration range from low level analysis and make appropriate dilution.
- . Fill five ml of reagent water into a luer lock syringe. Add 10 ul of internal standard and the volume of the methanol extract.
- . Add to purging chamber.
- . Proceed with analysis.

3.10 QUALITATIVE ANALYSIS OF TARGETED COMPOUNDS

Compound identification is based on the computer algorithms for matches of the sample spectra with the library spectra developed in-house from standard injection. ("Reverse library search")

All ions in the standard mass spectrum of a relative intensity greater than 10 percent of the most abundant ion must be present. The relative intensities of the ions must agree within +/- 20 percent. The ions greater than 10 percent in the sample but not in the standard must be accounted for.

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As an aid in identification of compounds the relative retention time of the sample peak is compared with that in the standard analysis. The relative retention times should not deviate by more than six (6) percent.

3.11 COMPUTER IDENTIFICATION OF UNKNOWN COMPOUNDS ("TENTATIVELY IDENTIFIED COMPOUNDS")

Non-targeted compounds can be tentatively identified by matching their spectra to spectra in the NBS library by computer search ("Forward Search"). The algorithms for the match are based on the USEPA criteria:

The major ions in the reference spectra, greater than 10 percent of the most abundant ion, must be present. The relative intensities must agree. The molecular ions present in the reference spectra must be in the sample spectra.

The ions present in the sample spectra, but not in the reference spectra, are reviewed for contamination or coelution. Ions contributed by background and unresolved compounds can be subtracted by the computer software.

Ions present in the reference spectra greater than 10 percent must be found in the sample spectra.

If the above criteria are not met, the mass spectral interpretation analyst will report the compound as an "unknown". If more information about the "type" of the unknown is available, (e.g. aromatic hydrocarbon) this information will be reported.

3.12 QUANTITATIVE ANALYSIS

If the computer search establishes a positive, the concentration of the analyte is computed by the software program with the relative response factors of the calibration, established by internal standard method.

The factors applied are generally derived from the initial five point calibration, which is verified with a daily calibration check. The data system is capable of computing the reportable sample concentration, if the appropriate parameters are entered according to the following computation:

Water:

$$\text{Conc. (ug/L)} = \frac{(Ax) (Is)}{(Ais) (RRF) (Vo)}$$

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$$\begin{array}{l} \text{Soil/Sediment:} \\ \text{Conc. (ug/kg)} = \frac{(Ax) (Is)}{(Ais) (RRF) (Ws) (D)} \end{array}$$

where: Ax = area of sample ion.
Ais = are of internal standard ion.
Is = ng of internal standard.
Vo = volume of water purged (ml).
Vi = volume of extract injected (ul)
Vt = total volume of extract (ul)
WS = of sample weight (g)
RRF = relative response factor
D = $\frac{100 - \% \text{ moisture}}{100}$

If targeted analytes are detected, but are below the CRQL, these analytes are reported but flagged with the qualifier "J", if reporting by CLP.

For TIC's the concentrations are estimated, using the area counts from the total ion chromatograms for both the analyte and the internal standard. A relative response factor of one (1) is assumed.

3.13 METHOD BLANK

A method blank is 5ml of distilled and carbon-filtered water that is analyzed prior to samples to ensure the analytical system is free of interferences. For GC/MS volatiles a method blank is analyzed for each 12 hours of analysis. The interferences seen in the method blank must be less than the contract required detection limits except for methylene chloride, acetone, toluene and 2-butanone. These compounds must be less than five times the contract required detection limit. If the method blank exceeds the above criteria the source of the contamination must be found and the problem eliminated prior to continuing with analysis. All samples analyzed with a blank that does not meet the above criteria must be reanalyzed. Method blanks must be subjected to the same analytical conditions as the samples (e.g. medium level method blanks, methanol should be added). Ottawa sand is used for low level soils.

3.14 SURROGATE SPIKE ANALYSIS

Surrogate standards are added to all standards, blanks and samples. Their recoveries are calculated. This recovery data is then compared with the allowable for the particular matrix in question (water or soil). The following are the acceptable criteria:

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Surrogate Recovery Limits

<u>Surrogate Compound</u>	<u>Water</u>	<u>Low/Medium Soil</u>
toluene-d8	88-110	81-117
p-BFB	86-115	74-121
1,2 dichloroethane-d4	76-114	60-121

For method blank analysis all surrogate recoveries must meet the above mentioned limits. The reason for the surrogate recoveries exceeding the criteria must be uncovered prior to the analysis of any sample.

For volatile organics, all sample surrogate recoveries must meet the acceptance criteria. If it does not the cause of this must be determined. If it can not be determined, the sample must be reanalyzed.

3.15 MATRIX SPIKE/MATRIX DUPLICATE SAMPLE

A group of representative compounds has been chosen from the volatile organic analytes and is used to monitor the overall recovery of the samples. The sample is spiked with these compounds and then another aliquot of the same sample is spiked again. Both accuracy and precision can be monitored with this information. Matrix Spike/Matrix Duplicate Sample (MS/MSD) samples are analyzed with every 20 samples in a case, or with each case received or similar matrix, or they can be analyzed every 14 days, whichever is more frequent.

The following matrix spiking compounds are used as the spiking solution for volatile organics: chlorobenzene; toluene; benzene; 1,1-dichloroethene; trichloroethene. The percent recovery of each individual spike is calculated and compared to the replicate and the RPD is then calculated. These limits are for advisory purposes only.

Matrix Spike Recovery Limits

<u>Matrix Spike Compound</u>	<u>Water</u>	<u>RPD</u>	<u>Soil/Sediment</u>	<u>RPD</u>
1,1-dichloroethene	61-145	14	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

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3.16 FILING AND STORAGE OF DATA

All p-BFB, standards, blanks and samples are filed in chronological order in the VOA run book. Also, all raw data is archived onto magnetic tapes shortly after data acquisition is complete. Magnetic tapes are kept for a period of three years.

3.17 DOCUMENTATION

Chain of Custody	The volatile COC is kept in the GC/MS laboratory. Groups of samples are signed for by the department custodian and transferred to the custody of the analyst.
Instrument Maintenance Log	The instrument log(s) for the Volatile instruments is kept near the instrument at all times so that any work done on the instrument can be immediately recorded. This is a record of all maintenance done on the instrument: liner change, column changes, problems, solutions, calibrations, PM visits, source cleaning, etc.
MDL's (Method Detection Limits)	MDL's are a series of seven standards that have gone through every step of the method. When the results of the seven standard runs are obtained, calculations are made that will determine the method detection limit. This is performed once every six months and is documented in a binder kept in the GC/MS lab.
X & R Charts	These are graphical representations of the precision and accuracy of our MS/MSD recoveries. Recoveries and relative percent differences are tabulated and graphed, then stored in the volatile MS/MSD QC binder. Raw data for the MS/MSD runs is also stored there.

X

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Surrogates Surrogate recoveries are tabulated in the volatile surrogate QC binder. Recoveries that are outside QC limits are flagged with an asterisk.

Column Log Whenever a new column is installed in the instrument, it is noted in the instrument maintenance log and any paperwork that accompanies the column is stored in the column performance summaries binder. These are labelled with the date the column was received, the date it was installed, and the serial number of the column.

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TABLE 3A

INSTRUMENT PARAMETERS
FOR TCL VOLATILE ORGANICS BY GC/MS

PURGE PROCESS (TEKMAR LSC-2000)

Sample Size:	5ml
Purge Time:	11 minutes & 1 minute
Purge Flow:	40ml/minute
Purge Temp:	ambient for waters and medium level soils
Desorption flow:	15ml/minute
Desorption time:	4.0 +/- 0.1 minute
Desorption Temp:	180 degrees Celsius

GC CONDITIONS (PERKIN-ELMER SIGMA 3)

Column:	30ml long x 0.53 ID DB 624 fused silica wide bore 3 is full thickness
Flow rate:	15ml/minute
Initial Temp/Hold:	10 degrees Celsius
Ramp Rate Final Temp:	160 degrees Celsius
Carrier Final Hold:	Helium

MS CONDITIONS (HEWLETT PACKARD 5890)

Separator Temp:	225 degrees Celsius
Manifold Temp:	250 degrees Celsius
Scanning Parameters:	
masses:	35 to 300
scan time:	0.5 seconds
number of scans:	1590
threshold:	50
minimum peak area:	5000

TABLE 3B

SECTION 1
SUPERFUND-CLP ORGANICS

SUPERFUND TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS*

VOLATILES	CASE NUMBER	QUANTITATION LIMITS**	
		LOW WATER (µG/L)	LOW SOIL/SEDIMENT 1 ^A (µG/KG)
Chloromethane	74-87-3	10	10
Bromomethane	74-83-9	10	10
Vinyl Chloride	75-01-4	10	10
Chloroethane	75-00-3	10	10
Methylene Chloride	75-09-2	5	5
Acetone	67-64-1	10	10
Carbon Disulfide	75-15-0	5	5
1,1-Dichloroethylene	75-35-4	5	5
1,1-Dichloroethane	75-35-3	5	5
1,2-Dichloroethylene (total)	540-59-0	5	5
Chloroform	67-66-3	5	5
1,2-Dichloroethane	107-06-2	5	5
2-Butanone	78-93-3	10	10
1,1,1-Trichloroethane	71-55-6	5	5
Carbon tetrachloride	56-23-5	5	5
Vinyl acetate	108-05-4	10	10
Bromodichloromethane	75-27-4	5	5
1,2-Dichloropropane	78-87-5	5	5
cis-1,2-Dichloropropene	10061-01-5	5	5
Trichloroethene	79-01-6	5	5
Dichlorodifluoromethane	124-48-1	5	5
1,1,2-Trichloroethane	79-00-5	5	5
Benzene	71-43-2	5	5
trans-1,2-Dichloropropene	10061-02-6	5	5
Bromoform	75-25-2	5	5
4-Methyl-2-pentanone	108-10-1	10	10
2-Hexanone	591-78-6	10	10
Tetrachloroethene	127-18-4	5	5
Toluene	108-88-3	5	5
1,1,2,2-Tetrachloroethene	79-34-5	5	5
Chlorobenzene	108-90-7	5	5
Ethyl Benzene	100-41-4	5	5
Styrene	100-42-5	5	5
Total Xylenes	1330-20-7	5	5

* Specific quantitation limits are highly matrix dependent. The limits herein are provided for guidance and may not always be achievable.

** Quantitation Limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the protocol, will be higher.

a Medium Soil/Sediment CRQL are 125 times the individual Low Soil Sediment CRQL.

TABLE 3C

CHARACTERISTIC IONS FOR SURROGATE AND INTERNAL STANDARDS
FOR VOLATILE ORGANIC COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
<i>SURROGATE STANDARDS</i>		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d ₄	65	102
Toluene-d ₄	98	70, 100
<i>INTERNAL STANDARDS</i>		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d ₄	117	82-119

TABLE 3D

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

ANALYTE	PRIMARY ION*	SECONDARY ION(S)
Chloromethane	50	52
Bromomethane	94	96
Vinyl Chloride	62	64
Chloroethane	64	66
Methylene Chloride	84	49,51,86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61,98
1,1-Dichloroethane	63	65,83,85,98,100
1,2-Dibromoethane	96	61,98
Chloroform	83	85
1,2-Dichloroethane	62	64,100,98
2-Butanone	72	57
1,1,1-Trichloroethane	97	99,117,119
Cardontetrachloride	117	119,121
Vinyl acetate	43	86
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,2-Dichloropropane	63	65,114
trans-1,2-Dichloropropane	75	77
Trichloroethene	130	95,97,132
Dibromochloromethane	129	208,206
1,1,2-Trichloroethane	97	83,85,99,132,134
Benzene	78	--
cis-1,2-Dichloropropene	75	77
Bromoform	173	171,175,250,252,254,256
2-Hexanone	43	58,57,100
4-Methyl-2-pentanone	43	58,100
Tetrachloroethene	164	129,131,166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78,103
Total Zylenes	106	91

* The primary ion should be used unless interferences are present, in which case a secondary ion may be used.

4. NYSDEC ASP Method 89-2 Base Neutral Acid

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4. NYSDEC ASP METHOD 89-2 BASE NEUTRAL ACID

4.2 SUMMARY OF PROCEDURES

The protocol for semivolatile analysis of all types of solid waste matrices, soils and ground water that is employed in our laboratory is documented in detail in the CLP NYSDEC ASP 9/89.

Compounds that can be analyzed with this procedure comprise most neutral, acidic and basic organic compounds that are soluble in methylene chloride and capable of being eluted as sharp peaks from a GC fused silica capillary column coated with a slightly polar silicone. Unknown, or non-targeted compounds, can be subjected to a computer library search on the National Institute of Standards and Technology library for tentative identification. A listing of the analytes reported and the quantification limits are found in Table 4A for NYSDEC.

The basic steps for analysis of semivolatiles can be summarized as follows: the contaminants are removed from the matrix with the appropriate sample preparation procedure. The purified and concentrated solvent extract is then analyzed on a HP-MSD with capillary column. Data reduction for identification and quantification is accomplished with the Aquarius software or the Terwilliger software.

4.2 HOLDING TIMES

Samples have to be extracted within five (5) days and analyzed within 40 days for all work submitted to the DEC. All analyses are to be performed within 40 days.

4.3 SAMPLE PREPARATIONS

Separatory funnel extractions (NYSDEC only) or auto extractors are used for removing contaminants from water. Extraction of soil samples is performed with a sonic disruptor.

Soil extracts, as well as water extracts containing higher molecular weight organics, are subject to gel permeation chromatography (GPC) cleanup, followed by a analysis of semivolatile organics by GC/MS. The use of GPC cleanup for soil and sediment samples is mandatory for all regulatory agencies. A flow chart listing the preparation for waters is listed in figure 1. Surrogate and matrix spike standard analytes and concentrations are listed in table 4B.

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Liquid/ liquid extraction with separatory funnel (DEC only)

1. Bring water sample to room temperature and measure sample volume (regularly one liter) and pour into a separatory funnel.
2. Record all data in extraction book.
3. Adjust pH to >12 for extraction of base/neutral (B/N) compounds.
4. Add surrogate standards for base/neutral and acid extractables.
5. Extract three times with 60ml methylene chloride (MC) and combine portions.
6. Dry extract by passing through purified Na_2SO_4 .
7. Concentrate extract in Kuderna Danish glassware to a volume under 1ml.
8. Adjust pH of water sample to <2 for extraction of acid compounds.
9. Repeat steps five to seven.
10. Bring extract to final volume of 1ml.
11. Combine both extracts for base/neutral and acid extractables
12. Spike with 10ul of the internal standard mix solution before injection.

Extraction of Liquid Samples with continuous extractor (NJDEP and EPA)

1. Assemble extractor; add boiling chips to round bottom flasks.
2. Add 1 liter of sample to the continuous extractor.
3. Add 0.5 ml of surrogate standard and mix.
4. Check pH and adjust to 2.0 with 1:1 H_2SO_4 .
5. Add 500 ml methylene chloride to the distilling flask.

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6. Add reagent water.
7. Extract for 18 hours.
8. Cool flask.
9. Concentrate extracts in Kuderna Danish concentrator followed by nitrogen "blow-down" concentration or with Turbo vap.
10. Combine and transfer concentrate to graduated vial and adjust volume to 1ml.

Extraction of Liquid Samples with Continuous Extractor (DEC Prep)

1. Assemble extractor; add boiling chips to round bottom flasks.
2. Adjust pH to <12 for extraction of B/N compounds.
3. Measure sample volume (250 to 1000ml) and pour into extraction chamber.
4. Add all surrogate standards for B/N/A extraction.
5. Slowly add enough methylene chloride (MC) to extraction chamber so that ca. 75ml reach the round bottom flask through the overflow and MC/Water interface is at bottom of wide part of extraction chamber.
6. Set temperature on heating mantle to achieve slow refluxing.
7. Have MC drip through sample for at least 16 hours.
8. Drain bottom layer of MC from extraction flask chamber and add to round bottom flask.
9. Filter extract through cleaned Na_2SO_4 in filter tube.
10. Adjust pH of water sample to pH <2 for acid extractables.

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11. Repeat steps five to nine.
12. Concentrate extracts in Kuderna Danish concentrator followed by nitrogen "blow-down" concentration
13. Combine and transfer concentrate to graduated vial and adjust volume to 1ml.

Low Level Extraction of Soils/ Sediments with Sonic Disruptor (For DEC)

1. Mix sample with spatula.
2. Prepare a 1:1 water slurry with 50g of sample and measure the pH with a glass electrode after thorough mixing for one hour.
3. Determine percent moisture with routine procedure for percent solid analysis.
4. Weigh 30g of sample in a 400ml beaker; mix with 60g anhydrous sodium sulfate.
5. Add 100ml of 1:1 methylene chloride/acetone solvent mix and 1ml each of the base/neutral and acid extractables surrogate standard solution; for splits for the pesticides/PCB scan, also add 200ul of the DBC surrogate solution; all spikes are doubled if GPC is intended.
6. Optimize the voltage for the ultrasonic disruptor according to the manufacturer manual. The probe used is a Tekmar sonic disruptor (% Dutycycle 50; output control 10).
7. Place sample on jack in sonicator sound box. Raise jack until extender tip of the 3/4-inch horn is inserted about a half inch under the surface of the solvent.
8. Sonicate for three minutes at the 50% interval setting.

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9. Decant and/or centrifuge the slurry in two 50ml centrifuge tubes.
10. Repeat extraction procedure with two more 100ml portions of the solvent mix.
11. Combine extracts and take 5ml aliquot for prescreening; blow down the prescreening aliquot to a volume of 1ml with nitrogen; analyze on D5-5 column on FID/GC; recombine unused prescreening concentrate with sample extract.
12. Place extract into 500ml Kuderna Danish bulb with 10ml graduated concentrator tube; add 1-2 Carborundum boiling chips.
13. Concentrate rapidly in 60-80°C water bath, refluxing with Snyder column.
14. When liquid level reaches 1ml in receiving vial, remove from water bath and let cool for 10 minutes.
15. Rinse walls of Snyder column, Kuderna Danish bulb and ground glass connections; collect in receiving vial.
16. For splitting of extract for BN/A and pesticides/PCB analyses, or if GPC cleanup is performed, bring volume to 10ml; use vortexer to achieve homogeneous concentration; split 1:20 for the two analytical scans, or remove 5ml for GPC cleanup.
17. If no splitting or GPC is needed, concentrate extract further in Kuderna Danish receiving vial.
18. For final concentration, add new boiling chips and reflux with small Snyder column, or use blowdown technique with nitrogen.

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19. If volume of ca. 1/2ml is reached, cool down assembly and bring volume to 1ml with rinsings from Snyder column and tube walls.

20. Transfer extract to storage or autosampler vial and store at 4 C.

Gel Permeation Chroma- tography Cleanup (For DEC)

To remove interferences by high molecular weight organic substances, size exclusion chromatography is performed on low level soil extracts. The systems used were constructed in-house and permit simultaneous cleanup of four samples. They basically consist of a 5ml sampling loop with a filter at the injection port, a vent valve, a 650mm chromatographic column with Bio Beads SX-3, and a collection valve.

Calibration of each column system is performed with corn oil, checked gravimetrically, and a mix of the semivolatiles pentachlorophenol and bis(2-ethyl-hexyl)-phthalate. Concentration of the semivolatiles in each collected fraction is checked by GC/MS. The fractional distribution is verified with a calibration standard at an interval of approximately twenty (20) runs. Recovery of both compounds has to be above or equal to 85 percent.

Low Level Extraction of Soils/ Sediments with Sonic Disruptor (For DEC)

1. Mix sample with spatula.
2. Prepare a 1:1 water slurry with 50g of sample and measure the pH with a glass electrode after thorough mixing for one hour.
3. Determine percent moisture with routine procedure for percent solid analysis.
4. Weigh 30g of sample in a 400ml beaker; mix with 60g anhydrous sodium sulfate.
5. Add 100ml of 1:1 methylene chloride/acetone solvent mix and 0.5ml each of the base/neutral and acid extractables surrogate standard solution.

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6. Optimize the voltage for the ultrasonic disruptor according to the manufacturer manual. The probe used is a Tekmar sonic disruptor (% Dutycycle 50; output control 10).
7. Place sample on jack in sonicator sound box. Raise jack until extender tip of the 3/4-inch horn is inserted about a half inch under the surface of the solvent.
8. Sonicate for three minutes at the 50% interval setting.
9. Decant and/or centrifuge the slurry in two 50ml centrifuge tubes.
10. Repeat extraction procedure with two more 100ml portions of the solvent mix.
11. Combine extracts and take 5ml aliquot for prescreening; blow down the prescreening aliquot to a volume of 1ml with nitrogen; analyze on D5-5 column on FID/GC; recombine unused prescreening concentrate with sample extract.
12. Place extract into 500ml Kuderna Danish bulb with 10ml graduated concentrator tube; add 1-2 Carborundum boiling chips.
13. Concentrate rapidly in 60-80°C water bath, refluxing with Snyder column.
14. When liquid level reaches 1ml in receiving vial, remove from water bath and let cool for 10 minutes.
15. Rinse walls of Snyder column, Kudernal Danish bulb and ground glass connections; collect in receiving vial.
16. The sample is diluted to 10 ml with methylene chloride.
17. The sample is then cleaned up by the use of GPC.

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18. Bring the volume to 0.5 ml by concentration.

Gel Permeation Chromatography Cleanup

To remove interferences by high molecular weight organic substances, size exclusion chromatography is performed on low level soil extracts. The systems used were constructed in-house and permit simultaneous cleanup of four samples. They consist of a 5 ml sampling loop with a filter at the injection port, a vent valve, a 650 mm chromatographic column with Bio Beads SX-3 and a collection valve.

Packing the column

Soak 70g Bi-bead SX-3 in 400 ml beaker, with methylene chloride overnight. Place beads in column and flush at 5ml/min solvent with the flow direction going from bottom to top. After approximately one (1) hour adjust pressure to seven to 10 psi and pump for four hours. Maintain column pressure.

Calibration of the column

Load 3ml of corn oil into loop. Inject corn oil and collect 10ml fractions for 36 minutes. Inject the phthalate-phenol solution and collect 15ml fractions for 60 minutes.

Determine the corn oil elution pattern by evaporation of each fraction to dryness, followed by a gravimetric determination of the residue. Analyze the phthalate-phenol fraction by GC/MS and plot the amount recovered versus eluant volume. Choose a "dump time" which allows >85% removal of the corn oil and 85% recovery of the bis(2-ethylhexyl)phthalate. Choose the "collect time" to extend at least 10 minutes after elution of pentachlorophenol to recover later dichlorobenzenes. Wash the column for at least 15 minutes between samples. For EPA and NJDEP the phthalate; methoxychlor and perylene are added and resolutions must be greater than 85%.

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Sample Cleanup

Five (5) ml of extract is loaded into the sampling loop of the GPC system, passing it through a double filter mounted in a filter holder with luer adapter. Process the sample using the dup., collect and wash parameters determined from the aluminum foil. With every 23 samples, run a phthalatephenol calibration. Recovery must be >85%. Analyze by GC/MS - submit a copy with package, show percent recovery. Run a GPC blank methylene chloride after a particularly dirty sample. For NJDEP and EPA a weekly calibration of GPC column is required.

Final Concentration

After GPC the methylene chloride fraction collected is concentrated in a K-D concentrator to approximately 1 ml. If a split is taken with the semivolatile analysis, bring the volume to 10 ml and split 1/20, i.e. 0.5 ml for pesticides analysis. The pesticides extract is exchanged to hexane and brought to a volume of 0.5 ml hexane solution. For the last phase of concentration use nitrogen blowdown in a water bath at 35°C. (Do not go to dryness!). For NJDEP and EPA the methylene chloride fraction is concentrated to 0.5 ml.

4.4 GC/MS CALIBRATION PROCEDURES

This section outlines the minimum operations necessary to satisfy analytical requirements associated with the determination of organic extractables in water and soil/sediment samples. The following operations will be performed routinely in the laboratory: documentation of mass calibration and abundance pattern; documentation of GC/MS stability or response factors against internal standards; internal standard response and retention time monitoring

DFTPP KEY ions and ion abundance criteria for NYSDEC

<u>MASS</u>	<u>ION ABUNDANCE CRITERIA</u>
51	30.0-60.0% of mass 198
68	Less than 2.0% of mass 69
69	mass 69 relative abundance
70	Less than 2.0% of mass 69
127	40.0 -60.0% of mass 198

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197	Less than 1.0% of mass 198
198	Base peak, 100% relative abundance.
199	5.0-9.0% of mass 198
275	10.0-30.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17.0 -23.0% of mass 442

4.5 TUNING GC/MS MASS CALIBRATION

Prior to initiating data collection, it is necessary to establish that the instrument meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) for base/neutral and acid (BNA) compounds. The ion abundance criteria for each calibration compound should be met before any samples, blanks, or standards, can be analyzed. DFTPP must be analyzed every 12 hours.

DFTPP - Each GC/MS system used for the analysis for semivolatiles compounds must be tuned to meet the abundance criteria as listed in the above criteria table for a 50 nanogram (ng) injection of DFTPP. DFTPP may be analyzed separately or as part of the calibration standard, and the criteria must be demonstrated daily. Documentation of the calibration must be provided in the form of a bar graph plot and as a mass listing.

4.6 INITIAL CALIBRATION FOR CLP

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the response curve for all targeted compounds. Average response factors are computed for the linear range. For GC/MS analysis, typical linear ranges are 10 to 400ng for base neutrals and 10 to 1000ng for phenols.

It is important that the initial calibration is performed with the same instrument parameters as the samples are analyzed. This applies to settings for the GC and MS.

Internal standards are added to all calibration standards. The base peak ion from the specific internal standards is used as the primary ion for quantification unless interferences mandate that the secondary ion is used. (See table 7 for the internal standard and its referenced analytes) The primary and secondary ions are listed in tables 8 and 9 for the internal standards and targeted analytes respectively.

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Surrogate standards are added prior to extraction to monitor recoveries throughout the preparation. The surrogates are phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 for all agencies. The NJDEP and EPA and the additional surrogates 2-chlorophenol-d4 and 1,2-dichlorobenzene-d4 are also monitored.

The CLP Method criteria indicate the concentration range levels for initial calibration and also the specific internal standards to be used on a compound-by-compound basis.

The concentration range required for DEC samples is 20ng, 50ng, 80ng, 120ng, 160ng. The relative response factor (RRF) for each compound is calculated at each concentration level using the following equation:

$$RRF = \frac{A^X}{A^{is}} \times \frac{C^{is}}{C^X}$$

where: A^X = area of the characteristic ion for compound measured.
 A^{is} = area of the characteristic ion for the specific internal standards.
 C^{is} = concentration of internal standard (ng/ul).
 C^X = concentration of compound to be measured (ng/ul).

The % RSD should be calculated for each compound percent. The %RSD should be less than 30% for each compound for DEC.

$$RSD = 100 \frac{[SD]}{RRF}$$

4.7 CALIBRATION CHECK COMPOUNDS (DEC ONLY)

Using the RRF from the initial calibration, the percent relative standard deviations (%RSD) are calculated for certain calibration check compounds. Thirteen semivolatile compounds are classified in the protocol method 8270. The following equation is used:

$$\%RSD = 100 \frac{SD}{RF}$$

where: RSD = Relative Standard Deviation
SD = Standard Deviation of initial five RF's (per compound)

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The percent RSD for each individual calibration check compound must be less than 30 percent. The criterion must be met for the initial calibration to be valid. A listing of the calibration check compounds can be found in the Calibration Check Compounds table following.

4.8 SYSTEM PERFORMANCE CHECK (DEC ONLY)

A system performance check must be performed to ensure minimum average RRF's are met before the calibration curve is used. The method classifies certain compounds as the (SPCC) system performance check compounds. The CCC's and SPCC's for the DEC are listed in the table below.

CALIBRATION CHECK COMPOUNDS

Base Neutral/Fraction:	Acenaphthene 1,4-Dichlorobenzene Hexachlorobutadiene N-Nitroso-di-n-phenylamine Di-n-octylphthlate Fluoranthene Benzo (a) pyrene
Acid Fraction:	4-Chloro-3-Methylphenol 2,4-Dichlorophenol 2-Nitrophenol Phenol Pentachlorophenol 2,4,6-Trichlorophenol
System Performance Check Compounds:	N-Nitroso-di-n-propylamine Hexachlorocyclopentadiene 2,4-Dinitro-phenol 4-Nitrophenol

For semi-volatiles, the System Performance Check Compounds (SPCC's) are: N-Nitroso-Di-n-Propylamine; Hexachlorocyclopentadiene; 2,4-Dinitrophenol; 4-Nitrophenol.

These compounds typically have very low RF's (0.1 - 0.2) and tend to decrease in response as the chromatographic system or the standard material begins to deteriorate. These compounds are usually the first to show poor performance. Therefore, they must meet the minimum average RRF of 0.05 when the system is calibrated.

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The initial calibration is valid only after both the %RSD for CCC compounds and the minimum RF for SPCC have been met. Only after both these criteria are met can sample analysis begin.

4.9 CONTINUING CALIBRATION (GC/MS)

The instrument must be tuned with 50 ng of DFTPP. A calibration standard containing all targeted compounds, as well as all required surrogates, should be analyzed every 12 hours. The RF's from the 12 hour standard are compared with the initial calibration and must be within 30% for DEC. The criteria as established in the criteria table (see section 4.4 GC/MS Calibration Procedures) must be met for EPA and NJDEP.

The concentration for each semivolatile compound in the continuing calibration standard(s) is 50 total nanograms. Nine compounds; Benzoic Acid, 2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol and pentachlorophenol, will be analyzed at 80 total nanograms of each compound injected.

As in the initial calibration for DEC, the minimum RRF's of 0.05 must be met for the SPCC's. After the system performance, check is made, Calibration Check Compounds are used to check the validity of the initial calibration. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{\text{RRF}^i - \text{RRF}^C}{\text{RRF}^i} \times 100$$

where: RRF^i = average relative response factor from initial calibration.
 RF^C = relative response factor for continuous calibration.

If the percent difference for any compound is greater than 30 the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 30 percent, the initial calibration is assumed to be valid. If the criteria are not met (> 30 percent difference) for any one calibration check compound, corrective action must be taken. Problems similar to those listed under SPCC should affect the criterion. If no source of the problem can be determined after corrective action has been taken, a new initial five-point calibration must be generated. The criterion must be met before sample analysis begins.

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Up to 4 semivolatile compounds can fail to meet the criteria but the RRF's must be above 0.010 and the %D must be less than or equal to 40% for the continuing calibration to be valid.

Internal standard responses and retention times in all samples are monitored. The retention time for any internal standard must not change by more than 30 seconds from the most recent standard. The EICP area for any internal standard must be within (-50% to +100%). Re-analysis is required if the above criteria are not met.

4.10 QUALITITATIVE ANALYSIS OF TARGETED COMPOUNDS

Compound identification is based on the computer algorithms for matches of the sample spectra with the library spectra developed in-house from standard injections.

All ions in the standard mass spectrum of a relative intensity greater than 10% of the most abundant ion must be present. The relative intensities of the ions must agree within +/- 20%. The ions greater than 10% in the sample but not in the standard must be accounted for.

Another means of identifying compounds is by comparison of the relative retention time of the sample peak with that in the standard analysis. The relative retention times should not deviate by more than six (6) percent.

4.11 COMPUTER IDENTIFICATION OF UNKNOWN COMPOUNDS ("TENTATIVELY IDENTIFIED COMPOUNDS")

Non-targeted compounds can be tentatively identified by matching their spectra to spectra in the NBS library by computer search. The algorithms for the match are based on the EPA criteria:

The major ions in the reference spectra, greater than 10% of the most abundant ion, must be present. The relative intensities must agree. The molecular ions present in the reference spectra must be in the sample spectra.

The ions present in the sample spectra, but not in the reference spectra, are reviewed for contamination or coelution. Ions contributed by background and unresolved compounds can be subtracted by the computer software.

Up to 20 non surrogate semivolatile organics are identified and reported on form I's. Substances with

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responses less than 10% of the closest internal standard are not reported.

Ions present in the reference spectra that are not found in the sample spectra, are reviewed by the analyst for subtraction from sample spectra, because of background or coelution.

If the above criteria are not met, the mass spectral interpretation analyst will report the compound as an "unknown". If more information about the "type" of the unknown is available, (e.g., aromatic hydrocarbon) this information will be reported.

4.12 QUANTITATIVE ANALYSIS

If the computer search establishes a positive, the concentration of the analyte is computed by the software program with the relative response factors of the calibration, established by internal standard method.

The factors applied are generally derived from the initial five point calibration, which is verified with a daily calibration check. "Daily" response factors from the "continuous calibration" are used. The data system is capable of computing the reportable sample concentration, if the appropriate parameters are entered according to the following computation:

$$\text{Water: Conc. (ug/l)} = \frac{(A_x)}{(A_{is})} \frac{(I_s)}{(RRF)} \frac{(V_t)}{(V_o)} \frac{Df}{(V_i)}$$

Soil/

$$\text{Sediment: Conc. (ug/kg)} = \frac{(A_x)}{(A_{is})} \frac{(I_s)}{(RRF)} \frac{(V_t)}{(V_i)} \frac{Df}{(W^s)} \frac{(2.0)}{(D)}$$

where: Ax = area of sample
Ais = area of internal standard ion
Is = ng of internal standard
Vo = volume of water extracted (ml)
Vi = volume of extract injected (ul)
Vt = total volume of extract (ul)
D = 100% moisture/100
Ws = of sample weight (g)
RRF = relative response factor
Df = dilution factor 2.0 factor for
EPA and NJDEP only

For TIC's, the concentrations are estimated, using the area counts from the total ion chromatograms for both the

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analyte and the internal standard. A relative response factor of one (1) is assumed.

4.13 SURROGATE STANDARD RECOVERY

The percent recovery of the surrogate standards added to all samples, blanks and standards are calculated. The recovery is compared with the limits in table 12.

If one surrogate is outside the limits in either the base/neutral or acid extractable group, the sample does not require re-extraction.

If two base/neutral or two acid surrogates are out of limits or if recovery of any one base/neutral or acid surrogate reanalysis is required. If the reanalysis does not resolve the recovery problem re-extraction is required.

If the re-extract is also out, both sets of data are submitted. If re-extraction is within recovery limits submit only the re-extracted sample.

If any surrogate in the blank is out, all samples associated with the blank (extracted in the same batch) must be re-extracted and reanalyzed.

4.14 MATRIX SPIKE/MATRIX SPIKE DUPLICATE/ MATRIX SPIKE BLANK

A matrix spike/matrix spike duplicate/matrix spike blank (for DEC) are to be analyzed with each case or each group of samples of a similar concentration or every 14 days. The spike compounds and the recovery limits are listed in table 3.2.3A and Table 3.2.3B. The recovery is calculated by the following formula:

$$\text{Matrix Spike Recovery} = \frac{\text{SSR} - \text{SR} (100)}{\text{SA}}$$

Where: SSR = Spike sample result
SR = Sample result
SA = Spike added

The relative percent difference is calculated by the following formula:

$$\text{RPD} = \frac{1\text{MSR} - \text{MSRD1}}{(1/2) (\text{MSR} + \text{MSDR})}$$

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Where: RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

The limits are advisory, no corrective action is required if these criteria are not met.

For DEC a matrix spike blank is required. The matrix spike solution added to the MS/MSD is spiked into lab distilled water and the recovery is monitored. The recovery should be within 75-125 percent.

4.15 METHOD BLANK

Method blanks are prepared for each day the samples are extracted and for each procedure used in sample preparation. The method blank must not contain any targeted analytes above the CRDL except for the phthalate esters which may be up to five times the CRDL. If the method blank does not meet this criteria all samples associated with it must be reextracted and reanalyzed. As mentioned under surrogate recoveries all surrogate recoveries must be within for the method blank of re-extraction and re-analysis is required. For soils/sediment samples Ottawa sand is used as a blank for DEC and sodium sulfate is used for EPA and NJDEP.

4.16 INTERNAL STANDARDS

All samples, standards and blanks are spiked prior to analyses with a minimum of three internal standards. The internal standards in use at H2M are: 1,4-dichlorobenzene-d4, naphthalene-D8, acenaphthene-D-10, phenanthrene-D-10, chrysene-D-12 and perylene-D-12.

4.17 ACCURACY

USEPA EMSL'S are analyzed on a quarterly basis. The percent recovery is calculated and the data is plotted on a X chart. Standards from other sources are also analyzed and the data plotted.

4.18 FILING AND STORAGE OF DATA

All DFTPP, standards, blanks and samples are filed in chronological order in the BNA run book. Also, all raw data is archived onto magnetic tapes shortly after data acquisition is complete. Magnetic tapes are kept for a period of six years.

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4.19 DOCUMENTATION

Chain of Custody	The semi-volatiles COC is kept in the special process department. Groups of samples are signed for when completed extracts are transferred to the custody of the analyst.
Instrument Maintenance Log	The instrument log for the BNA instrument (HP#2) is kept near the instrument at all times so that any work done on the instrument can be immediately recorded. This is a record of all maintenance done on the instrument: liner change, column changes, problems, solutions, calibrations, PM visits, source cleaning, etc.
MDL's (Method Detection Limits)	MDL's are a series of seven standards that have gone through every step of the method from extraction to analysis. When the results of the seven standard runs are obtained, calculations are made that will determine the method detection limit. This is performed once every six months and is documented in a binder kept in the GC/MS lab.
X & R Charts	These are graphical representations of the precision and accuracy of our MS/MSD recoveries. Recoveries and relative percent differences are tabulated and graphed, then stored in the BNA MS/MSD QC binder. Raw data for the MS/MSD runs is also stored there.
Surrogates	Surrogate recoveries are tabulated in the semivolatile surrogate QC binder. Recoveries that are outside QC limits are flagged with an asterisk.
Column Log	Whenever a new column is installed in the instrument, it is noted in the instrument column log and any paperwork that accompanies the column is stored in the column performance summaries binder. These are labelled with the date the column was received, the date it was installed, and the serial number of the column.

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

Semivolatiles	CAS Number	Quantitation Limits**	
		Low Water $\mu\text{g/L}$	Low Soil/Sediment ^b $\mu\text{g/Kg}$
35. Phenol	108-95-2	10	330
36. bis(2-Chloroethyl) ether	111-44-4	10	330
37. 2-Chlorophenol	95-57-8	10	330
38. 1,3-Dichlorobenzene	541-73-1	10	330
39. 1,4-Dichlorobenzene	106-46-7	10	330
40. Benzyl alcohol	100-51-6	10	330
41. 1,2-Dichlorobenzene	95-50-1	10	330
42. 2-Methylphenol	95-48-7	10	330
43. 2,2'-oxybis(1-Chloro- propane	108-60-1	10	330
44. 4-Methylphenol	106-44-5	10	330
45. N-Nitroso-di-n-propylamine	621-64-7	10	330
46. Hexachloroethane	67-72-1	10	330
47. Nitrobenzene	98-95-3	10	330
48. Isophorone	78-59-1	10	330
49. 2-Nitrophenol	88-75-5	10	330
50. 2,4-Dimethylphenol	105-67-9	10	330
51. Benzoic acid	65-85-0	50	1600
52. bis(2-Chloroethoxy) methane	111-91-1	10	330
53. 2,4-Dichlorophenol	120-83-2	10	330
54. 1,2,4-Trichlorobenzene	120-82-1	10	330
55. Naphthalene	91-20-3	10	330
56. 4-Chloroaniline	106-47-8	10	330
57. Hexachlorobutadiene	87-68-3	10	330
58. 4-Chloro-3-methylphenol (p-chloro-m-cresol)	59-50-7	10	330
59. 2-Methylnaphthalene	91-57-6	10	330
60. Hexachlorocyclopentadiene	77-47-4	10	330
61. 2,4,6-Trichlorophenol	88-06-2	10	330
62. 2,4,5-Trichlorophenol	95-95-4	50	1600
63. 2-Chloronaphthalene	91-58-7	10	330

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

Semivolatiles (continued)	CAS Number	Quantitation Limits**	
		Low Water $\mu\text{g/L}$	Low Soil/Sediment ^b $\mu\text{g/Kg}$
64. 2-Nitroaniline	88-74-4	50	1600
65. Dimethyl phthalate	131-11-3	10	330
66. Acenaphthylene	208-96-8	10	330
67. 2,6-Dinitrotoluene	606-20-2	10	330
68. 3-Nitroaniline	99-09-2	50	1600
69. Acenaphthene	83-32-9	10	330
70. 2,4-Dinitrophenol	51-28-5	50	1600
71. 4-Nitrophenol	100-02-7	50	1600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. Diethylphthalate	84-66-2	10	330
75. 4-Chlorophenyl phenyl ether	7005-72-3	10	330
76. Fluorene	86-73-7	10	330
77. 4-Nitroaniline	100-01-6	50	1600
78. 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
79. N-nitrosodiphenylamine	86-30-6	10	330
80. 4-Bromophenyl phenyl ether	101-55-3	10	330
81. Hexachlorobenzene	118-74-1	10	330
82. Pentachlorophenol	87-86-5	50	1600
83. Phenanthrene	85-01-8	10	330
84. Anthracene	120-12-7	10	330
85. Di-n-butyl phthalate	84-74-2	10	330
86. Fluoranthene	206-44-0	10	330
87. Pyrene	129-00-0	10	330
88. Butyl benzyl phthalate	85-68-7	10	330
89. 3,3'-Dichlorobenzidine	91-94-1	20	660
90. Benz(a)anthracene	56-55-3	10	330
91. Chrysene	218-01-9	10	330
92. bis(2-Ethylhexyl)phthalate	117-81-7	10	330
93. Di-n-octyl phthalate	117-84-0	10	330
94. Benzo(b)fluoranthene	205-99-2	10	330

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

Semivolatiles (continued)	CAS Number	Quantitation Limits**	
		Low Water $\mu\text{g/L}$	Low Soil/Sediment ^b $\mu\text{g/Kg}$
95. Benzo(k)fluoranthene	207-08-9	10	330
96. Benzo(a)pyrene	50-32-8	10	330
97. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
98. Dibenzo(a,h)anthracene	53-70-3	10	330
99. Benzo(g,h,i)perylene	191-24-2	10	330

^b Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile TCL Compounds are 60 times the individual Low Soil/Sediment CRDL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

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TABLE 4B

SURROGATE STANDARDS

<u>Compound</u>	<u>Conc. for NYSDEC</u>
Phenol-5	200 ug/ml
2,4,6-tribromophenol	200 ug/ml
2-fluorophenol	200 ug/ml
Nitrobenzene-d5	100 ug/ml
Terphenyl-d14	100 ug/ml
2-fluorobiphenyl	100 ug/ml

MATRIX SPIKE COMPOUNDS

<u>Compound</u>	<u>Conc. for NYSDEC</u>
1,2,4-trichlorobenzene	100 ug/ml
Acenaphthene	100 ug/ml
2,4-dinitrotoluene	100 ug/ml
Pyrene	100 ug/ml
N-nitroso-di-n-propylamine	100 ug/ml
1,4-dichlorobenzene	100 ug/ml
Pentachlorophenol	200 ug/ml
Phenol	200 ug/ml
2-Chlorophenol	200 ug/ml
4-chloro-3-methylphenol	200 ug/ml
4-Nitrophenol	200 ug/ml

TABLE 4C

MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE

<u>Compound</u>	<u>% Recovery Water</u>	<u>RPD Water</u>	<u>% Recovery Soil</u>	<u>RPD Soil</u>
Phenol	12 - 89	42	26 - 90	35
2-chlorophenol	27 - 123	40	25 - 102	50
1,4-dichlorobenzene	36 - 97	28	28 - 104	27
N-Nitroso-di-n-propylamine	41 - 116	38	41 - 126	38
1,2,4-trichlorobenzene	39 - 98	28	38 - 107	23
4-chloro-3-methylphenol	23 - 97	42	26 - 103	33
Acenaphthene	46 - 118	31	31 - 137	19
2,4-dinitrotoluene	24 - 96	38	28 - 89	47
Pentachlorophenol	9 - 103	50	17 - 109	47
Pyrene	26 - 127	31	35 - 142	36

TABLE 4D

SURROGATE SPIKE PERCENT RECOVERY

<u>Compound</u>	Q.C. LIMITS	
	<u>Water</u>	<u>Soil</u>
nitrobenzene-d ₅	35 - 114	23 - 120
2-fluorobiphenyl	43 - 116	30 - 115
terphenyl-d ₁₄	33 - 141	18 - 137
phenol-d ₆	10 - 94	24 - 113
2-fluorophenol	21 - 100	25 - 121
2,4,6-tribromophenol	10 - 123	19 - 122

5. NYSDEC ASP Method 89-3 Pesticide/PCB

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5. NYSDEC ASP METHOD 89-3 PESTICIDE/PCB

Our procedure conforms with the ASP CLP requirements as set forth in Ref. #1a. This protocol has to be followed for CLP samples from DEC or other projects that require CLP deliverables that are submitted to NYSDEC.

5.1 SUMMARY OF PROCEDURE

The method details sample preparation for water and low and medium level soil.

Various extraction and concentration procedures are presented. They are followed by cleanup procedures utilized to remove interferences from the extracts.

Analysis is performed by GC/ECD on a widebore capillary column. Positives have to be confirmed on a secondary widebore capillary column. A specific sequence of sample and standard injections for 72 hours is followed to permit continuous calibration and system performance checks.

5.2 SAMPLE STORAGE AND HOLDING TIMES

Samples have to be stored in locked refrigerators at 4°C from receipt until extraction. For CLP protocol, water and soil sample extraction must be completed within five (5) days of sample receipt in the laboratory. Analysis must be completed within forty days of receipt for all samples.

5.3 SAMPLE PREPARATION

Preparation of Water Samples: One liter of water sample is extracted with methylene chloride using a separatory funnel or continuous extractor. If an emulsion is formed, the continuous extractor has to be used. The extract is dried, exchanged to hexane and adjusted to 10ml.

Separatory - Measure the sample of appr. 1l in
Funnel graduated cylinder and record the volume;
Extraction:

- Add 1ml surrogate standard solution, containing 20 ug/ml dibutylchloredate;
- Adjust the pH between 5-9 (with either 10N NaOH or 1:1 sulfuric acid);

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- For samples selected for MS/MSD also add 1ml of matrix spike solution to each one liter of sample.

Matrix Spike Solution

Lindane	2.0 ug/ml
Heptachlor	2.0 ug/ml
Aldrin	2.0 ug/ml
Dieldrin	5.0 ug/ml
Endrin	5.0 ug/ml
4,4'-DDT	5.0 ug/ml

- Add 60ml methylene chloride and extract by shaking for two minutes, venting periodically.
- Allow solvent layer to separate from water layer and drain methylene chloride.
- Repeat extraction two more times for a total of three extractions and combine extracts.

Sample Extraction with Continuous Liquid/Liquid Extractor	- Fill 200ml methylene chloride into extractor vessel.
	- Pour measured quantity of sample of appr. 1l on top of the methylene chloride and record the volume of sample.

(Note: If the interface between solvent and the sample does not show a clear separation, raise the level of the interface by adding less sample, or removing a measured quantity again by syphoning with a pipette. This will prevent floating material or emulsion to enter the overflow to the flask.)

- Add 1 ml surrogate standad solution and for matrix spike samples add 1 ml matrix spike solution.

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- Adjust pH between 5 and 9 with 10 N NaOH or 1:1 H₂SO₄
- Raise the temperature of the waterbath to 80°C or adjust the heating mantel to achieve a slow boil of the MC.
- Reflux the MC for 18 hours.
- Transfer the MC extract to concentrator for concentration.

K-D Concent- ration

- Pour combined extracts through funnel with 2" anhydrous granular sodium sulfate, baked for 1/2 hour in kiln at 400°C. Collect in KD concentrator or Zymark concentrator tube.
- Rinse with 20 to 30 ml of methylene chloride.
- Add clean boiling chips to evaporative flask and attach a three ball Snyder column.
- Pre-wet the Snyder column by adding 1 ml methylene chloride to the top.
- Place the KD on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in hot water and the lower rounded surface of the flask is in hot vapor.
- Concentrate to 1ml and allow to drain and cool for at least 10 minutes.

Solvent Exchange

- Add 50 ml hexane and new boiling chip.
- Pre-wet column by adding 1ml of hexane.

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- Concentrate again to 1ml. The concentration time should be approximately five to ten minutes.
- When the volume of the concentrate reaches about 1ml, remove the KD. Allow to drain and cool for at least 10 minutes.
- Remove the Snyder column and rinse with 1 to 2ml hexane. If sulfur crystals are present, follow optional sulfur clean-up.

Nitrogen Blow-Down Concent- ration

- Place the concentration tube in a warm water bath (35°C) and evaporate to 0.5ml using nitrogen. DO NOT BLOW DOWN TO DRYNESS!
- Dilute with 1/2 ml acetone and proceed with alumina column clean-up.

Concent- ration with the Zymark Turbo Vap Concent- rator

- Concentration and solvent exchange with nitrogen blow down in automated with the Turbo Vap. A photosensor stops the nitrogen flow, when the solvent level reaches 0.5 ml concentration to dryness is optional.

Procedure

- Set temperature at 25°C; Set N₂ pressure at 7 PSI; Select end volume of 0.5 ml.; Select number of times of solvent exchange with hexane of 2 times.
- Load 200 ml tubes with sample extracts and close cover
- When ready-light flashes, the endpoint of 0.5 ml is reached. The wall of the tube is rinsed by adding several ml of MC.

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- The concentrator is closed and the samples concentrated again until the endpoint of 0.5 ml is reached.
- With pasteur pipet transfer concentrate to autosampler vial or small storage vial. Bring up to required volume with solvent rinsings of the 200 ml concentrator tube. (Note: The rinsing process is very critical to obtain adequate recovery. Great care has to be taken to rinse the walls of the lower cone very thoroughly.

Extract Cleanup

- Water extracts only have to be cleaned up by alumina, whereas the other procedures are used as required. The procedures are described under the soil sample preparation protocol.

Sample Preparation for Soil/Sediment: The sample must be characterized as to concentration level. Samples of greater than 1000 ug/kg of pesticides/PCBs are considered "medium level", samples of a lower concentration are "low level". Different preparation protocols are followed for "medium" and "low" samples.

Prescreening on FID provides information about levels of organics in the sample but does not identify the targeted compounds and therefore cannot be relied upon for level determination of pesticides and PCBs. The decision, which protocol to use, is mostly based on prior knowledge of the sample.

The receiving department alerts the Special Process Lab of the possibility that the sample might contain more than 1000 ug/kg. The medium level procedure is then followed and this is regarded as a "screening procedure", i.e., if the extract shows lower levels of the targeted organics, the extraction has to be repeated with the low level protocol.

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- Sample Preparation for Low Level Soil: Extraction with Ultrasonic Disruptor (Sonication):
- Decant and discard any water layer on sediment. Mix samples thoroughly and discard any sticks, leaves and rocks.
 - Transfer 50g of soil to 100ml beaker, add 50 ml water and stir for one hour. Determine pH of sample with glass electrode while stirring. Note the pH. Discard this sample aliquot. (This step is performed in the Inorganic Department).
 - Weigh 5-10g of sediment into a tared crucible. Dry overnight at 105°C and determine the percent moisture. (This is also performed in the Inorganic Department).
 - Weigh 30g of sample to nearest 0.1g into 250 ml beaker, add 60g anhydrous powdered sodium sulfate. Mix well. Add 100ml 1:1 methylene chloride/ acetone to the sample.
 - Spike sample with 100 ul of surrogate solution containing 20 ug ml DBC in methanol. Double the spike volume, if GPC is intended.
 - For MS/MSD analysis weigh out two 30g portions. Add 400 ul of matrix spike and 100 ul of DBC surrogate solution to each portion. Double the spike quantities if GPC cleanup is intended.
 - Place beakers on rack in sonicator box. (Two beakers can be processed simultaneously using both horns). Position bottom of tip of the 3/4" disruptor horn about 1/2" below the surface of the solvent, but above the sediment layer. Sonicate for three minutes at 50% pulsing, with output control set at ten.

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- Decant and filter extract through filter tube with sodium sulfate. If phases of slurry don't separate, centrifuge slurry in 50ml centrifuge tubes at setting 3 for 2 minutes and decant extraction solvent.
- Repeat the extraction two more times with two additions of 100ml portions of 1:1 methylene chloride/acetone. Decant the extraction solvent after each sonication.
- Follow water protocol for KD concentration or concentration with the Turbo-Vap. The solvent has to also be exchanged to hexane.
- Bring the final volume to 10 ml for split with BNA or GPC cleanup. If no GPC or split is needed, bring volume to 0.5 ml for alumina cleanup.

Sample Preparation
for Medium
Level:

- Transfer 50g for soil to 100ml beaker. Add 50ml water and stir one hour, determine pH with glass electrode, report pH. Discard this part of sample. Ph analysis is executed in the wet chem department.
- Add one gram sample to 20ml vial; record weight; cap to prevent cross-contamination; Add two grams anhydrous powdered sodium sulfate (from kiln) to sample and mix; Add 50ul surrogate spiking solution of 20 ug/ml methanol; Add 1ml matrix spiking solution to each sample chosen for MS/MSD.

Matrix Spike Solution

Lindane	2.0 ug/ml
Heptachlor	2.0 ug/ml
Aldrin	2.0 ug/ml
Dieldrin	5.0 ug/ml
Endrin	5.0 ug/ml
4,4'-/DDT	5.0 ug/ml

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- Add 10ml hexane or 9ml of hexane for MS/MSD samples
- Sonicate in sonicator box with microtip of ultrasonic disruptor at 50% setting and output ten. Use divider between beakers to prevent cross contamination, if more than one sample is placed in box.
- Loosely pack pasteur pipettes with 2-3 cm glass wool plugs. Filter hexane layer through glass wool and collect at least 1ml.
- Transfer 2ml of the extract to K-D tube and concentrate to 1ml hexane extract with nitrogen.
- Follow procedure for low level soil for alumina cleanup and sulfur removal.

Extract Cleanup:

- Most soils contain contaminants that either interfere with the analysis or will cause problems for the analytical system. These contaminants have to be removed by appropriate cleanup procedures. Employed are size exclusion in gel permeation chromatography, (GPC) polarographic column chromatography, in alumina cleanup and TBA reaction for sulfur removal. GPC and alumina are mandatory in the DEC ASP. Also, sulfur cleanup has to be performed if residual sulfur is present after GPC cleanup, to avoid reanalysis.

Gel Permeation Chromatography (GPC) Cleanup:

Packing of the GPC column

- Soak 70g Bio Bead SX-3 in 400ml beaker with MC overnight. Tightly cap with alumina foil and leave hood running.
- Before filling column, make sure that top and bottom pistons fit tightly. If they slip too easily, replace the o-rings for tighter fit, otherwise gaps will occur in column later.

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- With bottom (inlet) seals in place and inlet valve open, place funnel on top and fill beads into column, draining MC into beaker under inlet. Add MC to slurry as needed keeping slurry "wet" and easily pourable. It is important to fill continuously to achieve a uniform packing without gaps for efficient chromatographic separation.
- Press down the piston of the outlet (top) seal, until the screen is touching the top of the packing
- Attach top, set valves to "run" position,
- Turn on rabbit pump and operate the manual "fast" button until MC reaches column.
- Pump MC through column at pump position at 5ml/min for several hours until all air bubbles are removed.

Calibration of the GPC column

- Prepare GPC calibration solution of 1000 ug/ml each of pentachlorophenol, bis(2-ethylhexyl)phthalate, and 1,4-dichlorobenzene (optional) in methylene chloride.
- Dissolve sulfur in warm cornoil to achieve concentration of 8 mg in 2500 mg. Dilute cornoil sulfur mix with MC to prepare solution of 2500 mg/8 ml.
- Attach teflon tube of GPC column outlet to inlet of analytical flow cell of 254 nm UV detector.
- Fill reference cell with MC.

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- Turn on UV detector and warm up for 1 hour.
- Pump MC through GPC column at 5 ml/min. (Measure for 10 min with graduated cylinder)
- Set recorder speed to 1 cm/2 min.
- "Zero" signal.
- Set Hamilton valve of GPC system to "load" position.
- Pull up 4 ml cornoil solution and 1 ml GPC calibration solution in syringe.
- Inject through filter into sampling loop.
- Switch valves to "run" position and start recorder.
- Continue pumping MC through system until baseline is reached after peak of last component (PCP or DCB, if contained in mix).
- Determine duration of fractions to be discarded and collected: *Fraction I* ("Dump" cycle): Contains cornoil and will be discarded; *Fraction II* ("Collect" cycle): Must contain at least 95% of BEHP and less than 15% of the cornoil. Select time in favor of BEHP to guarantee adequate recovery, even if a retention time shift occurs. Select collection time to continue at least 1 min after PCP is eluted, to include other compounds that have slightly later elution times; *Fraction III* ("Dump" cycle): Consists of a rinse with MC for 10 min, to clean out system between injections.

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- If a problem arises with insufficient resolution between peaks remedial action must be taken by cleaning the sytem or repacking the column.

Sample
Cleanup on
GPC Column:

- Determine organic content of sample. Due to the loading capacity of the column of ca 300 mg organics, the sample extract cannot contain more than 600 mg, if one half of the sample is to be subjected to cleanup. For samples with high organic content (e.g. sludges of ca 10%) several aliquots have to be cleaned up and combined after cleanup.
- Fill MC into reservoir.
- Insert glass fiber prefilter and 0.45 u filter in filter holder.
- Set Hamilton valve to "load" position and rinse loading port and sampling loop with appr. 10 ml MC.
- Load half of the concentrated sample extract (5ml) into glass syringe and inject the sampling loop, slowly passing it through filter holder not to rupture filter. (Extracts with high particle content may have to be filtered prior to mounting in order to prevent clogging).
- Set all valves to "run" position. Start pump using 5ml/min setting.
- Collect "dump" volume, as determined during calibration, in measuring cylinder and discard.

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- Collect sample fraction in measuring cylinder, until the "collect" volume is reached that was determined during calibration. This fraction is saved for analysis.
- Run GPC column for 10 min longer to remove residual contaminations. Discard this fraction.
- If sample is not split concentrate the collected sample fraction with N_2 , and solvent-exchange to 0.5 ml of hexane for alumina cleanup.
- If a BNA split is taken, bring volume to 5 ml and remove 0.5 ml for pesticides alumina cleanup. This constitutes 1/10 split instead of 1/20. (This makes up for the GPC aliquot of one half, and is performed instead of concentrating the final volume 0.5 ml). The aliquot of 0.5 ml has to be solvent exchanged to 0.5 ml of hexane for alumina cleanup.

Calibration Check of GPC Column

- With every 23 samples run, a GPC calibration check has to be performed.
- Mount 5ml GPC-CC solution.
- Collect the same "fraction II" as determined for collection of targeted compounds for samples.
- Analyze both, a 1/100 dilution of the GPC-CC and the unconcentrated fraction II on GC/MS.
- Calculate recovery of check compounds. The mandatory compounds must have at least 85% recovery.

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- File data.
- If recovery is too low, recalibrate GPC column with UV procedure.

For CLP packages the data for the calibration check, including the calibration standard for the GC/MS, must be submitted for the GPC column, on which the sample was cleaned. Alternatively the UV trace can be submitted for a GPC calibration.

Sulfur Cleanup:	As preventive measure to avoid reanalysis, all soil extracts are subject to sulfur removal.

- Concentrate hexane extract to 1ml.
- Transfer 1ml to a 12ml glass vial with Teflon lined screw cap.
- Rinse concentrator tube with 1ml hexane and add to bottle.
- Prepare TBA - sulfite reagent: Dissolve 3.39 g tetrabutylammonium hydrogen sulfate in 100 ml water. Extract with hexane to purify and add 25 g sodium sulfite. (Discard hexane)
- Add 1ml TBA-sulfite reagent and shake for one minute. If sample is colorless or color unchanged, and if clear crystals are present, enough sodium sulfite is present.
- If the precipitated sodium sulfite disappears, add more crystalline sodium sulfate in approximate 100mg portions until a solid residue remains after repeated shaking.

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- Add 5ml distilled water and shake for one minute.
- Allow to stand 5-10 minutes.
- Transfer the hexane (top) layer to a crimp-top autosampler vial, seal and store for analysis

Alumina Cleanup:

This cleanup procedure removes polar interferences as well as the polar BNA surrogates that are added, if BNA compounds are extracted together with the pesticides/PCBs and the extracts are split. To remove the surrogate compounds without losing pesticides, activity III alumina has to be prepared, and the batch of alumina has to be tested by the "equivalency check", before the material can be used.

Preparation- Prepare activity III by adding 7% (v/w)
of Alumina: reagent water to Super I neutral alumina.

- Tumble for a minimum of 2 hours or preferably overnight. (There should be no lumps present).
- Store in a tightly sealed glass container.
- A 25-cycle soxhlet extraction of the alumina with methylene chloride is required, if a solvent blank passed through alumina indicates any interferences for the compounds of interest.

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during on-site evaluations. If the alumina deactivated with 7% (v/w) reagent water does not prove adequate to remove the BNA surrogates and other interferences, the alumina may be deactivated with less water, so long as the criteria for the recovery of all single component pesticides can be met.

- Sample Cleanup on Alumina:
- Pack Kontes Disposaflex glass columns with luer connection with pesticide grade rinsed glass wool and 3g of pretested activity III alumina.
 - Mount in Supelco Solid Phase Extraction (SPE) Apparatus.
 - Place 12 ml collection tube under outlet valve.
 - Dilute 0.5 ml hexane extract with 0.5 ml acetone and mount on alumina. Let liquid pass through column by gravity without vacuum.
 - Add rinses before column "runs dry".
 - Rinse concentrator tube with 1ml hexane, transfer rinse to column.
 - Elute column with 9ml of hexane.
 - Blown down the collected eluate with nitrogen with nitrogen manifold accessory on the Supelco SPE Apparatus to final volume of 1ml.
 - Transfer extract to labeled vial for storage at 4°C.

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- Alumina - Prepare AEC spiking solution: In small
Equivalency vial mix 1 ml of a 1/10 methanol dilution
Check of the BNA surrogate standard (100 ug/ml
(AEC): for base-neutral and 200 ug/ml for acid
 surrogate spikes) and 0.5 ml each of the
 "INDA" and "INDB" pesticides calibration
 standards in hexane.
- Pack Kontes Disposaflex glass column with pesticide- grade rinsed glass wool and 3g of the prepared activity III alumina.
 - Attach column to Supelco SPE apparatus.
 - Place 12 ml testtube under outlet valve.
 - Mount 1ml of AEC solution.
 - Let solution (and rises) pass through packing by gravity; do not use vacuum.
 - Rinse with 1ml hexane and add to alumina column, when liquid surface reaches alumina. Do not let run dry!
 - Rinse with 9ml hexane.
 - Concentrate collected eluates to <1ml with N₂ manifold attachment.
 - Bring to 1ml and store in autosampler vial at 4°C.
 - Analyze on GC.

The dibromophenol surrogate should not be detected by GC/ECD, if the deactivation of the alumina is acceptable. Also the percent recovery for all single component pesticides must be 80%, except for endosulfan sulfate, which must be >60%. Endrin aldehyde is not recovered. The data must be retained by the laboratory and made available for inspection

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5.4 INSTRUMENT ANALYSIS

Retention Time Windows: For each new column analyze three standards over the course of 72 hours and determine the relative standard deviation (RSD) of the retention times for all compounds. "Windows" for identification will be based on 3 times RSD. Note: The minimum window for wide bore columns 0.75%.

Initial Calibration: The instrument is calibrated with external standard procedure. Every time major instrument changes occur, e.g. detector service or column change, a new multipoint calibration is performed. Response is established for all compounds of interest with standard mixes at three levels. The lowest standard concentration is at, or slightly above, CRQL. The other two are higher by a factor of 5 to 50. The calibration curves are filed for each instrument. Unlike for routine sample analysis these factors are not used for quantification. Instead, a calibration for all compounds is performed at the beginning of the analyses of a group of samples and the factors from these standard runs are used. Linearity is established over a range from CRQL to 10 x CRQL with the three evaluation mixes at the beginning of each sequence. The relative standard deviation for aldrin, endrin and DBC must be less than equal to 10%.

72-Hour Sequence: Verification of stability of response is done throughout a group of samples. The intervals of this verification is specified in the 72 hour sequence mandated for the order of runs of calibration standards and samples. The sequence also includes the runs of a evaluation standard at three levels. These three evaluation mix analyses provide a linearity check for selected compounds. If a column is used for quantification, the evaluation standards have to meet the linearity criterium of a standard deviation of 10% or less.

The 72-Hour Sequence for Pesticide/PCB Analysis:

- 1) evaluation standard mix A
- 2) evaluation standard mix B
- 3) evaluation standard mix C
- 4) individual standard mix A
- 5) individual standard mix B
- 6) toxaphene
- 7) aroclor 1016/1260
- 8) aroclor 1221 *
- 9) aroclor 1232 *
- 10) aroclor 1242
- 11) aroclor 1248
- 12) aroclor 1254
- 13) five samples
- 14) evaluation standard mix B

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- 15) five samples
- 16) individual standard mix A and B
- 17) five samples
- 18) evaluation standard mix B
- 19) five samples
- 20) individual standard mix A and B (alternate!)
- 21) five samples
- 22) starting with step 14,
repeat sequence with evaluation standard B
- 23) end with individual standard mix A and B

** Include in only one sequence in a month per column and instrument.*

The relative percent difference (RPD) for the calibration factors for the later standards of the sequence compared to the first standards must not exceed 15% for quantification nor exceed 20% for confirmation during the 72-hour period. If deviations are greater than 15%, the samples following the quantification standard that exceeded the criterion have to be reanalyzed.

System Performance Monitoring: Rigid standards are set for the performance of the analytical system:

- The DDT retention time must be larger than 12 minutes.
- Valleys between peaks must show a resolution of at least 25% for quantification.
- Degradation of Endrin and 4,4'-DDT is monitored throughout the sequence. The evaluation mix B runs are checked for the breakdown products, endrin aldehyde and ketone and 4,4'-DDD and DDE. The percent breakdown for endrin and 4,4'-DDT, as well as the combined breakdown of endrin and 4,4'-DDT, must not exceed 20%.
- Shifts of retention times are monitored throughout the sequence. The elution time of the surrogate standard DBC is compared with that of the first standard. The shift must not exceed 2% for packed column and 1% for wide bore columns.
- All retention times (RT) of standards in the sequence have to fall within the calculated RT windows. These windows are computed from the RT of the first standards plus/minus 3 times the standard deviation determined for each column.
- If any parameter during performance monitoring falls outside acceptable limits, remedial action must be

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taken and reanalysis of the standard must meet criteria before analyses can continue.

- If violations are discovered after the sequence is completed, samples analyzed before and after the non compliant standards have to be reanalyzed in a new sequence. Samples with DBC shifts above limits also require reanalysis.

Identification: Empirical retention time windows, based on three standard deviations of observed shifts, are applied to the retention times of the individual standards. If an unknown falls within the RT window, it constitutes a tentative positive identification. This also applies for identification of a PCB. The major peak of PCB has to fall in the calculated RT window. Additionally pattern recognition is followed to determine the presence of a PCB (aroclor). All major peaks of a particular aroclor have to be present at the retention times, i.e., they have to be in the "windows" of the peaks present in the standard. The peaks also have to have correct ratios to each other.

Quantification: If the response of any identified pesticide or PCB is above reporting limit, the compound is quantified with the response factors derived from the first calibration standard injections of the 72-hour CLP sequence. Quantification is normally performed on the primary column, except for toxaphene. If DDT exceeds the 10% RSD linearity criteria, then quantification must be done on the confirmatory column. The confirmatory column results are also used, if the peak is not resolved better than 25% on the primary column. 10% RSD linearity criterium applies for any column for quantification.

The following equations is used for computations of concentrations:

$$\text{Water: conc. (ug/l)} = \frac{(Ax) (Vs)}{(Rf) (Vi) (Vs)}$$

where: Ax = peak area
Rf = response factor; area of standard
divided by amount (ng) injected
Vt = 10000 ul (multiply with factor
for dilution)
Vi = volume of extract injected (ul)
Vs = volume of water extracted (ml)

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Sediment/Soil:

$$\text{conc. (ug/kg)} = \frac{(Ax) (Vt)}{(Rf) (Vi) (Ws) (D)}$$

where: Ax = peak area
Rf = response factor; area of standard
divided by amount (ng) injected
Vi = volume of extract injected (ul)
Vt (low level)
= 20,000ul (multiply with factor
for
dilution
D = percent moisture/100
Ws = weight of sample extracted (g)

If the response exceeds the linear range, the extract is diluted and reanalyzed. Data for the undiluted analysis have to also be reported, to provide information, if lower level compounds are present.

For quantification of PCB's, the major peaks in the standard are summed to establish a response factor by dividing the total area by the amount injected. The same group of peaks is then used for quantification of the PCB in the sample. Interfering peaks that do not match the retention times and ratios are omitted for summing the area, resulting in a "partial area". A "partial" response factor is then used. CLP specifies that at least 50% of the total area must be used for computation.

Confirmation on Secondary Column: Confirmation has to be performed on a column of different polarity. A capillary column is particularly useful for complex matrices, where the compounds have to be resolved from many interferences. For CLP, again the specific sequence of calibration standards, samples and evaluation mixes has to be observed. However, the only standards required are the evaluation standard mixes and the standards containing compounds to be confirmed. The linearity criterium of 10% has to be met only if the sample is to be quantified on the confirmatory column. For confirmation acceptance limit for linearity is 15%.

GC/MS Confirmation: If the concentration of an analyte exceeds the detection limit of the GC/MS instrument, this compound has to be confirmed by GC/MS analysis. Analyze a reference pesticide standard of the compounds of interest and compare retention time and spectrum. If GC/MS does not confirm the compound, it is reported as "non detected" and the reporting limit is elevated to account for interferences. For CLP reporting, the documentation for GC/MS analysis should include standard and sample spectra to confirm identification.

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5.5 QUALITY CONTROL

Surrogate Recoveries: Recoveries for the DBC surrogate standard are generally calculated from the primary column. If DBC coelutes with an interfering compound on the primary column, it can be quantified on the confirmatory column, provided QC criteria for quantification can be met. Note: The limits on the Form II are advisory limits. Samples exceeding these limits do not have to be reextracted.

Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank: For each group of samples (up to 20) received, a sample is spiked in duplicate with a mix containing six targeted pesticides and subjected to the sample preparation procedure. (For mix, see section for sample preparation). To provide information about the efficiency of sample preparation, independent of the matrix, laboratory clean water or Ottawa Sand is spiked and extracted in the same fashion (MSB). These spiked samples and blanks are analyzed, evaluated and reported on Form III and I. Note: The limits on Form III are advisory. Samples exceeding the limits do not have to be reextracted.

Method Blank: With each case comprising SDGs samples collected within 14 days and for up to 20 samples extracted together for a particular matrix, a method blank is extracted and analyzed with the samples on both columns. The blank consists of either nanopure water or 30g of Ottawa sand. The blank extracts undergo the same treatment as the sample extracts, including all cleanup procedures. If not all samples are subject to sulfur cleanup, hexane spiked with the surrogate standard is cleaned as a "sulfur blank". If GPC is performed on only a portion of the samples a separate GPC blank must be prepared. If two blanks are extracted with the samples, one can be dedicated as a GPC blank. Otherwise a "GPC blank" has to be started.

TABLE 5A

GC OPERATING CONDITIONS FOR
ORGANOCHLORINE PESTICIDES AND PCBS

Instrument: HP5890#1

Column: Primary Column (A):
TX-5, Fused Silica 30 m, 0.53 mm ID

Secondary Column (B):
RTX-35, Fused Silica 30 m, 0.53 mm ID

Temp. Program: Initial Temp. 160°C for 0.75 min.

	<u>Rate</u>	<u>Fin. Temp.</u>	<u>Fin. Time</u>
Ramp A	5.5	220	0.00
Ramp B	5.0	250	13.00
Ramp C	70.0	275	1.99

Total Run Time: 33 minutes

Inlet Temp.: 180°C

Carrier Gas: (A): 4.3 ml/min Helium
(B): 3.5 ml/min Helium

Detector: ECD

Detector Range: -----

Detector Temp.: 300°C

Detector Gas: 45 ml/min Nitrogen

Integrator: Nelson Turbochrom System

TABLE 5B

**GC OPERATING CONDITIONS FOR
ORGANOCHLORINE PESTICIDES AND PCBS**

Instrument: HP5890#0

Column: Primary Column (A)
Glass, 6 ft x 4 mm (I.D.);
1.5% SP 2250 + 1.95%
SP 2401 on Supelcoport
100/120 Mesh

Secondary Column (B)
Glass 6 ft x 2 mm (I.D.);
3% SP 2100 on Supelcoport
100/120 Mesh

Temp. Program: Initial Temp. 150°C for 2.5 min.

	<u>Rate</u>	<u>Fin. Temp.</u>	<u>Fin. Time</u>
Ramp A	15	200	2.0
Ramp B	4	220	15.0
Ramp C	10	226	5.7

Total Run Time: 34 minutes

Inlet Temp.: 210°C

Carrier Gas: (A): 50 ml/min Nitrogen
(B): 26 ml/min Nitrogen

Detector: ECD

Detector Range: -----

Detector Temp.: 300°C

Detector Gas: None

Integrator: Nelson Turbochrom System

6. NYSDEC ASP Method Cyanide

6 NYSDEC ASP METHOD CYANIDES

6.1 TOTAL CYANIDE ANALYSIS IN WATER

Method 335.2 CLP-M (*Modified for the Contract Laboratory Program*): Manual Spectrophotometric; Semi-Automated Spectrophotometric.

Scope and Application: This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes. The manual colorimetric procedure is used for concentrations below 1 mg/l of cyanide and is sensitive to about 0.02 mg/l (Option B). The working range of the semi-automated spectrophotometric method is 0.005 to 0.200 mg/l. Higher level samples may be diluted to fall within the working range. (Option C).

Summary of Method: The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically. In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCI , by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridinebarbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

Definitions: Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

Sampling Handling and Preservation: All bottles must be thoroughly cleansed and rinsed to remove soluble material from containers. Oxidizing agents such as chloride decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample volume. Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH > 12) at the time of collection (see Exhibit I). Samples must be stored at 4 C (+/- 2 °C) and must be analyzed within the 12 day holding time.

Interferences: Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1. Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test

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paper indicates the presence of sulfides, treat 25 ml more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide as described in section "Sampling Handling and Preservation" previous. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps under alkaline titration conditions, making the end point almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used.

Apparatus: Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber. Microbut, 5.0 mL (for titration). Spectrophotometric suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger (for manual spectrophotometric method). Technical AA II System or equivalent instrumentation, (for automated spectrophotometric method) including: Sampler; Pump III; Cyanide Manifold (Figure 3); SCIS Colorimeter with 15 mm flowcells and 570 nm filters; Recorder; Data System (optional); Glass or plastic tubes for the sampler.

Reagents-Distillation and Preparation Reagents: Sodium hydroxide solution, 1.25 N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water; Cadmium carbonate-powdered; Ascorbic acid-crystals; Sulfuric acid-concentrated. Magnesium chloride solution: Weight 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 ml flask, dissolved and dilute to 1 liter with distilled water.

Reagents-Stock Standards: Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 22 liter of distilled water. Standardize with 0.0192 N $AgNO_3$. Standard cyanide solution, intermediate: Dilute 50.0 mL of stock (1 mL = 1 mg CN) to 1000 mL with distilled water (1 mL = 50.0 ug). Standard cyanide solution: Prepare fresh daily by diluting 100.0 mL of intermediate cyanide solution to 1000 mL with distilled water and store in a glass stoppered bottle. 1 mL = 5.0 ug CN (5.0 mg/L). Sodium hydroxide solution, 0.25 N: Dissolve 10 g of NaOH in distilled water and dilute to 1 liter.

Reagents-Manual Spectrophotometric Reagents: Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in a liter of distilled water. Refrigerate this solution. Chloramine-T solution: Dissolve 1.0 g of white, water soluble

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chloramine-T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly. Color Reagent - one of the following may be used:

- Pyridine-barbituric acid reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCL (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.
- Pyridine-pyrazolone solution: 3-methyl-1-phenyl-2-pyrazolone -5 one reagent, saturated solution. Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolone-5- one to 50 ml of distilled water, heat to 6-°C with stirring. Cool to room temperature.

3,3'Dimethyl-1 1'-diphenyl [4,4'-bi-2 pyrazolone]-5,5'dione (bispyrazaolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine.

Pour solution (7.3.3.2.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (7.3.3.2.2) collecting the filtrate in the same container as filtrate from (7.3.3.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.

Reagents-Semi-Automated Spectrophotometric: Chloramine-T solution: Dissolve 0.40 g of chloramine-T in distilled water and dilute to 100 ml. Prepare fresh daily. Phosphate buffer: Dissolve 238 g of $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ in distilled water and dilute to 1 liter. Add 0.5 ml of Brij-35 (available from Technicon). Store at 4°C. Pyridine-barbituric acid solution: Transfer 15 g of barbituric acid into a 1 liter volumetric flask. Add 74 ml of pyridine and mix. Add 15 ml of concentrated HCl and mix. Dilute to about 900 ml with distilled water and mix until the barbituric acid is dissolved. Dilute to 1 liter with distilled water. Store at 4°C. Sampler wash: Dissolve 10 g of NaOH in distilled water and dilute to 1 liter.

PROCEDURE:

Distillation: Place 500 mL of sample, or an aliquot diluted to 500 mL in the 1 liter boiling flask. Add 50 mL of sodium hydroxide (7.1.1) to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train. Start a slow stream of air

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entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube. (NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.) Slowly add 25 mL concentrated sulfuric acid (7.1.4) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 minutes. Pour 20 mL of magnesium chloride solution (7.1.5) into the air inlet and wash down with a stream of water. Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect and close off the vacuum source.

Manual Spectrophotometric Determination (Option B): Withdraw 50 mL or less of the solution from the flask and transfer to a 100 mL volumetric flask. If less than 50 mL is taken, dilute to 50 mL with 0.25 N sodium hydroxide solution (7.2.6). Add 15.0 mL of sodium phosphate solution (7.3.1) and mix. Pyridine-barbituric acid method: Add 2 mL of chloramine-T (7.3.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-barbituric acid solution (7.3.3.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes. Pyridine-pyrazolone method: Add 0.5 mL chloramine-T (7.3.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-pyrazolone solution (7.3.3.2) and mix. Dilute to mark with distilled water and mix again. After 40 minutes, read absorbance at 620 nm in a 1 cm cell. (NOTE: More than 0.5 mL of chloramine-T will prevent the color from developing with pyridine-pyrazolone.) Prepare a minimum of 3 standards and a blank pipeting suitable volumes of standard solution into 250 mL volumetric flasks. (NOTE: One calibration standard must be at the contract required quantitation limit (CRQL).)

To each standard add 50 mL of 1.25 N sodium hydroxide and dilute to 250 mL with distilled water. Standards must bracket the concentration of the samples. If dilution is required, use the blank solution. As an example, a 1 ug/mL standard solution could be prepared as follows:

<u>mL of Standard Solution</u> <u>(1.0 = 5 ug CN)</u>	<u>Conc. ug CN</u> <u>per 250 mL</u>
0	Blank
1.0	5
2.0	10
5.0	25
10.0	50
15.0	60
20.0	100

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It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid range) must be distilled and compared to similar values on the curve to insure that the distillation technique is reliable.

If distilled standard does not agree within + 15% of the undistilled standards, the operator should find the cause for the apparent error before proceeding. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations (per 250 mL).

Semi-Automated Spectrophotometric Determination (Option C): Set up the manifold as shown in Figure 3. Pump the reagents through the system until a steady baseline is obtained.

Calibration standards: Prepare a blank and at least three calibration standards over the range of the analysis. One calibration standard must be at the CRQL. For a working range of 0-200 ug/L, the following standards may be used:

<u>mL of Standard Solution</u> <u>(7.2.3) diluted to 1 liter</u>	<u>Concentration</u> <u>ug CN/L</u>
0	0
5.0	25
10.0	50
20.0	100
30.0	150
45.0	200

Add 10 g of NaOH to each standard. Store at 4°C (+/- 2°C). Place calibration standards, blanks, and control standards in the sampler tray, followed by distilled samples, distilled duplicates, distilled standards, distilled spikes and distilled blanks. When steady reagent baseline is obtained, and before starting the sampler, adjust the baseline using the appropriate knob on the colorimeter. Aspirate a calibration standard and adjust the STD CAL dial on the colorimeter until the desired signal is obtained. Record the STD CAL values. Re-establish the baseline and proceed to analyze calibration standard, blanks, control standards, distilled sample, and distilled QC audits.

Calculations: If the semi-automated method is used, measure the peak heights of the calibration standards (visually or using a data system) and calculate a linear regression equation. Apply the equation to the samples and QC audits to determine the cyanide concentration in the original sample. MULTIPLY THE RESULTS BY ONE-HALF (since the original volume was 500 ml and the distillate volume was 250 ml). Also, correct for any dilutions which were made before or after distillation. If the colorimetric procedure is used, calculate the cyanide, in ug/L, in the original sample as follows:

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$$\text{CN, ug/L} = \frac{A \times 1,000 \text{ mL/L}}{B} \times \frac{50 \text{ mL}}{C}$$

Where: A = ug CN read from standard curve
(per 250 mL)
B = mL of original sample for
distillation (see section 6.1)
C = mL taken for colormetric analysis
(see sec. 6.1)
50mL = volume of original aliquot
(see sec. 6.1)
1000 mL/L = conversion mL to L

6.2 TOTAL CYANIDE ANALYSIS IN SOIL SEDIMENTS

Method 335.2 CLP-M (*Modified for the Contract Laboratory Program*): Manual Spectrophotometric; Semi-Automated Spectrophotometric

Scope and Application: This method is applicable to the determination of cyanide in sediments and other solids. The quantitation limit is dependent upon the weight of sample taken for analysis.

Summary of Method: The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically. In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCL, by reaction with chloramine-T at a pH less than 8 without hydrolizing to the cyanate. After the reaction is complete, color is formed o the addition of pyridine-pyrazolone or pyridinebarbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. to obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

Definitions: Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

Sampling Handling and Preservation: Samples are stored at 4°C (+/- 2°C) and must be analyzed within the 12 day holding time. Samples are not dried prior to analysis. A separate % solids determination must be made (see Section 6.1).

Interferences: Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1. Sulfides adversely affect the colorimetric and titration procedures. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from

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collecting in the condenser. Fatty acids will distill and form soaps under alkaline titration conditions, making the end point almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used.

Apparatus: Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber. Spectrophotometric suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger (for manual spectrophotometric method). Technicon AA II System or equivalent instrumentation, (for automated spectrophotometric method) including: Sampler; Pump III; Cyanide Manifold (Figure 3); SCIS Colorimeter with 15 mm flowcells and 570 nm filters; Recorder; Data System (optional); Glass or plastic tubes for the sampler.

Reagents-Distillation and Preparation: Sodium hydroxide solution, 1.25 N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water. Cadmium carbonate-powdered; Ascorbic acid-crystals; Sulfuric acid-concentrated. Magnesium chloride solution: Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 mL flask, dissolved and dilute to 1 liter with distilled water.

Reagents-Stock Standards: Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KCH in 22 liter of distilled water. Standardize with 0.0192 N $AgNO_3$. Standard cyanide solution, intermediate: Dilute 50.0 mL of stock (1 mL = 1 mg CN) to 1000 mL with distilled water (1 mL = 50.0 ug). Standard cyanide solution: Prepare fresh daily by diluting 100.0 mL of intermediate cyanide solution to 1000 mL with distilled water and store in a glass stoppered bottle. 1 mL = 5.0 ug CN (5.0 mg/L).

Reagents-Manual Spectrophotometric: Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in a liter of distilled water. Refrigerate this solution. Chloramine-T solution: Dissolve 1.0 g of white, water soluble chloramine-T in 100 mL of distilled water and refrigerate until ready to use. Prepare fresh weekly. Color Reagent - one of the following may be used:

- Pyridine-barbituric acid reagent: Place 15 g of barbituric acid in a 250 mL volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of HCL (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 mL with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.
- Pyridine-pyrazolone solution: 3-methyl-1-phenyl-2-pyrazolone -5 one reagent, saturated solution. Add

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0.25 g of 3-methyl-1-phenyl-2-pyrazolone-5-one to 50 mL of distilled water, heat to 6-°C with stirring. Cool to room temperature. 3,3'-Dimethyl-1,1'-diphenyl [4,4'-bi-2 pyrazolone]-5,5'-dione (bispyrazaolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine. Pour solution (7.3.3.2.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (7.3.3.2.2) collecting the filtrate in the same container as filtrate from (7.3.3.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.

Semi-Automated Spectrophotometric Reagents: Chloramine-T solution: Dissolve 0.40 g of chloramine-T in distilled water and dilute to 100 mL. Prepare fresh daily. Phosphate buffer: Dissolve 238 g of $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ in distilled water and dilute to 1 liter. Add 0.5 mL of Brij-35 (available from Technicon). Store at 4°C. Pyridine-barbituric acid solution: Transfer 15 g of barbituric acid into a 1 liter volumetric flask. Add 74 mL of pyridine and mix. Add 15 mL of concentrated HCL and mix. Dilute to about 900 mL with distilled water and mix until the barbituric acid is dissolved. Dilute to 1 liter with distilled water. Store at 4°C. Sampler wash: Dissolve 10 g of NaOH in distilled water and dilute to 1 liter.

PROCEDURE:

Distillation: Accurately weigh a representative 1-5 g portion of wet sample and transfer it to a boiling flask. Add 500 mL of distilled water. Shake or stir the sample so that it is dispersed. Add 50 mL of sodium hydroxide (7.1.1) to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube. NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube. Slowly add 25 mL concentrated sulfuric acid (7.1.4) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 minutes. Pour 20 ml of magnesium chloride solution (7.1.5) into the air inlet and wash down with a stream of water. Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect and close off the vacuum source. Drain the solution from absorber into a 250 ml volumetric flask and

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being up to volume with distilled water washings from the absorber tube.

Manual Spectrophotometric Determination (Option B): Withdraw 50 mL or less of the solution from the flask and transfer to a 100 mL volumetric flask. If less than 50 mL is taken, dilute to 50 mL with 0.25 N sodium hydroxide solution (7.2.6). Add 15.0 mL of sodium phosphate solution (7.3.1) and mix.

- Pyridine-barbituric acid method: Add 2 mL of chloramine-T (7.3.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-barbituric acid solution (7.3.3.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.
- Pyridine-pyrazolone method: Add 0.5 mL chloramine-T (7.3.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-pyrazolone solution (7.3.3.2) and mix. Dilute to mark with distilled water and mix again. After 40 minutes, read absorbance at 620 nm in a 1 cm cell. NOTE: More than 0.5 mL of chloramine-T will prevent the color from developing with pyridine-pyrazolone.

Prepare a minimum of 3 standards and a blank pipeting suitable volumes of standard solution into 250 mL volumetric flasks. NOTE: One calibration standard must be at the contract required quantitation limit (CRQL). To each standard add 50 mL of 1.25 N sodium hydroxide and dilute to 250 mL with distilled water. Standards must bracket the concentration of the samples. If dilution is required, use the blank solution. As an example, a 1 ug/mL standard solution could be prepared as follows:

<u>mL of Standard Solution</u> <u>(1.0 = 5 ug CN)</u>	<u>Conc. ug CN</u> <u>per 250 mL</u>
0	Blank
1.0	5
2.0	10
5.0	25
10.0	50
15.0	60
20.0	100

It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid range) must be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standard does not agree within +/- 15% of the undistilled standards, the operator should find the cause for the apparent error before proceeding. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations (per 250 mL).

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Semi-Automated Spectrophotometric Determination (Option C):
Set up the manifold as shown in Figure 3. Pump the reagents through the system until a steady baseline is obtained. Calibration standards: Prepare a blank and at least three calibration standards over the range of the analysis. One calibration standard must be at the CRQL. For a working range of 0-200 ug/L, the following standards may be used:

<u>mL of Standard Solution</u> <u>(7.2.3) diluted to 1 liter</u>	<u>Concentration</u> <u>ug CN/l</u>
0	0
5.0	25
10.0	50
20.0	100
30.0	150
45.0	200

Add 10 g of NaOH to each standard. Store at 4°C (+/- 2°C). Place calibration standards, blanks, and control standards in the sampler tray, followed by distilled samples, distilled duplicates, distilled standards, distilled spikes and distilled blanks. When steady reagent baseline is obtained, and before starting the sampler, adjust the baseline using the appropriate knob on the colorimeter. Aspirate a calibration standard and adjust the STD CAL dial on the colorimeter until the desired signal is obtained. Record the STD CAL values. Re-establish the baseline and proceed to analyze calibration standard, blanks, control standards, distilled sample, and distilled QC audits.

Calculations: A separate determination of % solids must be performed.

Manual Spectrophotometric:

$$\text{CN, mg/kg} = \frac{A \times 50 \text{ mL/B}}{C \times \% \text{ Solids}/100}$$

Where: A = ug CN read from standard curve
(per 250 mL)

B = mL of distillate taken for
colorimetric determination
(See 6.1)

C = Wet weight of original sample in
g (See 6.1)

50 mL = volume of standard taken for
colorimetric determination (See
6.1)

% solids = (See part F)

Semi-Automated Spectrophotometric: If the semi-automated method is used, measure the peak heights of the calibration standards (visually or using a data system) and calculate a

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linear regression equation. Apply the equation to the samples and QC audits to determine the cyanide concentration in the distillates.

$$\text{CN, mg/kg} = \frac{A \times .25}{C \times \% \text{solids}/100}$$

Where: A = ug/L determined from standard curve
C = wet weight of original sample in g (see 6.1)
.25 = conversion fractor for distillage volume (See 6.1) and solids (See section VIII)

6.3 PERCENT SOLIDS DETERMINTATION PROCEDURE

- Immediately following the weighing of the sample to be processed for analysis (see Section III, part B-Soil/Sediment Sample Preparation), add 5-10 g of sample to a tared weighing dish. Weigh and record the weight to the nearest 0.01 g.
- Place weighing dish plus sample, with the cover tipped to allow for moisture escape, in a drying oven maintained at 103-105°C. Sample handling and drying should be conducted in a well-ventilated area.
- Dry the sample overnight (12-24 hours) but no longer than 24 hours. If dried less than 12 hours, it must be documented that constant weight was attained. Remove the sample from the oven and cool in a dessicator with the weighing dish cover in place before weighing. Weigh and record weight to nearest 0.01 g. Do not analyze the dried sample.
- Duplicate percent solids determinations are required at the same frequency as are other analytical determinations. Duplicate results are to be recorded on Form VI-IN.
- For the duplicate percent solids deterination, designate one sample aliquot as the "original" sample and the other aliquot as the "duplicate" sample. Calculate dry weight using the results of the "original" sample aliquot.
- Calculate percent solids by the formula below. The value thus obtained will be reported on the appropriate Form I-IN and, where applicable, Form VI-IN. This value will be used for calculating analytical concentration on a dry weight basis.

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$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

For the purpose of paragraph 3, dryingtime is defined as the elapsed time in the oven; thus raw data must record time in and out of the oven to document the 12 hour drying minimum. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weight-dry-dessicat-weigh cycles with a minimum of 1 hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

7. NYSDEC ASP Method Metals

FIGURE 6

DIGESTION QUALITY CONTROL

SPIKE

FLAME LIQUID SPIKE	BOTTLE #2, #3, #4
FLAME SOIL SPIKE	BOTTLE #2, #3
FURNACE LIQUID/SOIL SPIKE	BOTTLE #1

LCS

1. LIQUIDS

A. FLAME: LCS	BOTTLE #7
FLAME: NAK LCS	BOTTLE #8
B. FURNACE: LCS	BOTTLE #9

2. SOILS (Use 1.0 gram of ottowa sand.
Dilute to 200 ml for final volume.)

A. FLAME: ICS A	BOTTLE #2
FLAME: ICSAB	BOTTLE #1
FLAME: 1000 PPM SB	BOTTLE #3
FLAME: NA,K	BOTTLE #4
B. FURNACE: 1000 PPM,AS,SE,PB,TL	BOTTLE #5

All bottles are labelled as to the proper volumes to use. Please check carefully.

THINGS TO REMEMBER:

1. Follow the amounts written on each bottle for spiking and for soil LCS.
2. Fill out description sheets.
3. Fill out the Chain of Custody completely before and after digestion.
4. Use volumetric flasks to bring samples to final volume.
5. Return original sample to metals department after digestion.
6. Write final volume and amount of original sample used on each digestion bottle.

FIGURE 5

LIQUID DIGESTION

Measure aliquot of sample:	Regular - 50 ml CLP and DEC - 100 ml
Add acids	see below
Concentrate on hotplate (with watch glass) or digestor block to desired volume:	Regular - 12½ to 25 ml CLP and DEC - 25 to 50 ml

Filter sample (if needed) and return up to initial volume.

<u>FLAME</u> (Method 3005)	
<u>100 ml</u>	
2 ml	1:1 HNO ₃
10 ml	1:1 HCL
<u>50 ml</u>	
1 ml	1:1 NHO ₃
5 ml	1:1 HCl

<u>FURNACE</u> (Method 3020)	
<u>100 ml</u>	
1 ml	1:1 HNO ₃
2 ml	30% H ₂ O ₂
<u>50 ml</u>	
.5 ml	1:1 NHO ₃
1 ml	30% H ₂ O ₂

<u>FURNACE</u>
Pb
(except Pb solder [flame])
As
Se
Tl

Liquid _____
Soil _____
Lead Solder _____
Paint Chip _____

FLAME
All other matals

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FIGURE 4

DIGS

SOILS - METHOD 3050

Do a duplicate every ten (10) samples
Use watch glass for all heating of samples

1. Weigh out soils, 1-2 grams, write weight on digestion tubes.
2. Add 10 mls Nitric (1:1), add spike after acid has been added.
3. Heat at 90°C for 10 to 15 minutes.
4. Cool for a few minutes, take off digester block.
5. Add 5 mls Nitric (conc.).
6. Cook 35 to 40 minutes.
7. Cool and add 5 mls Nitric (conc.).
8. Cook 35 to 40 minutes.
9. Cool samples, add 2 mls distilled H₂O, and 3 mls 30 percent peroxide, put on digester for 10 minutes.
10. Add 1 ml of peroxide at intervals until effervescence subsides.
11. Cool sample.

FLAME (CLP and NON-CLP)

Add 5 mls HCL (conc.)
Add 10 mls H₂O
Cook 15 minutes
Cool and dilute to 200 ml

FURNACE (NON-CLP, DEC & Regular CLients)

Reduce sample to 5 ml
Cool and dilute to 200 ml

FURNACE (CLP-Modified 3050)

Reduce sample to 2 ml
Cool, add 10 ml water
Warm mixture
Cool and dilute to 200 ml

DIGESTION QC FOR NYSDEC AND CLP CLIENTS (EDG, G&H AND NJDEP)

CLP - Be sure to fill out description sheets for sample before and after digestion.

Be sure to run duplicate every 10 samples.

SOIL

TOTAL VOLUME: 200 ml, (1.0g sample/ottowa sand)

BLANK: Ottawa sand

LSC: Add spike to 1.0g ottowa sand
(Laboratory Control Standard)

Flame:	<u>Bottle #</u>	<u>Amount</u>
	1	1.0 ml
	2	0.4 ml
	3	1.0 ml
NA, K:	<u>Bottle #</u>	<u>Amount</u>
	4	0.6 ml
Furnace:	<u>Bottle #</u>	<u>Amount</u>
	5	1.0 ml
	Any uncommon element	

<u>SPIKES:</u>	Flame:	<u>Bottle #</u>	<u>Amount</u>
		4	0.2 ml
		5	0.2 ml
		11	0.1 ml
		Any uncommon element	
	Furnace:	<u>Bottle #</u>	<u>Amount</u>
		6	0.2 ml
		10	0.2 ml

NOTE: Any uncommon element which is requested for analysis (e.g. B, Au, Sn), must have its own LCS and must be added to the spike elements.

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FIGURE 2

DIGESTION QC FOR NYSDEC AND CLP CLIENTS (EDG, G&H AND NJDEP)

- CLP - Be sure to fill out description sheets for sample before and after digestion.
- DEC - Be sure to run duplicate every 10 samples.

LIQUIDS

TOTAL VOLUME: 100 ml

BLANK: Distilled Water

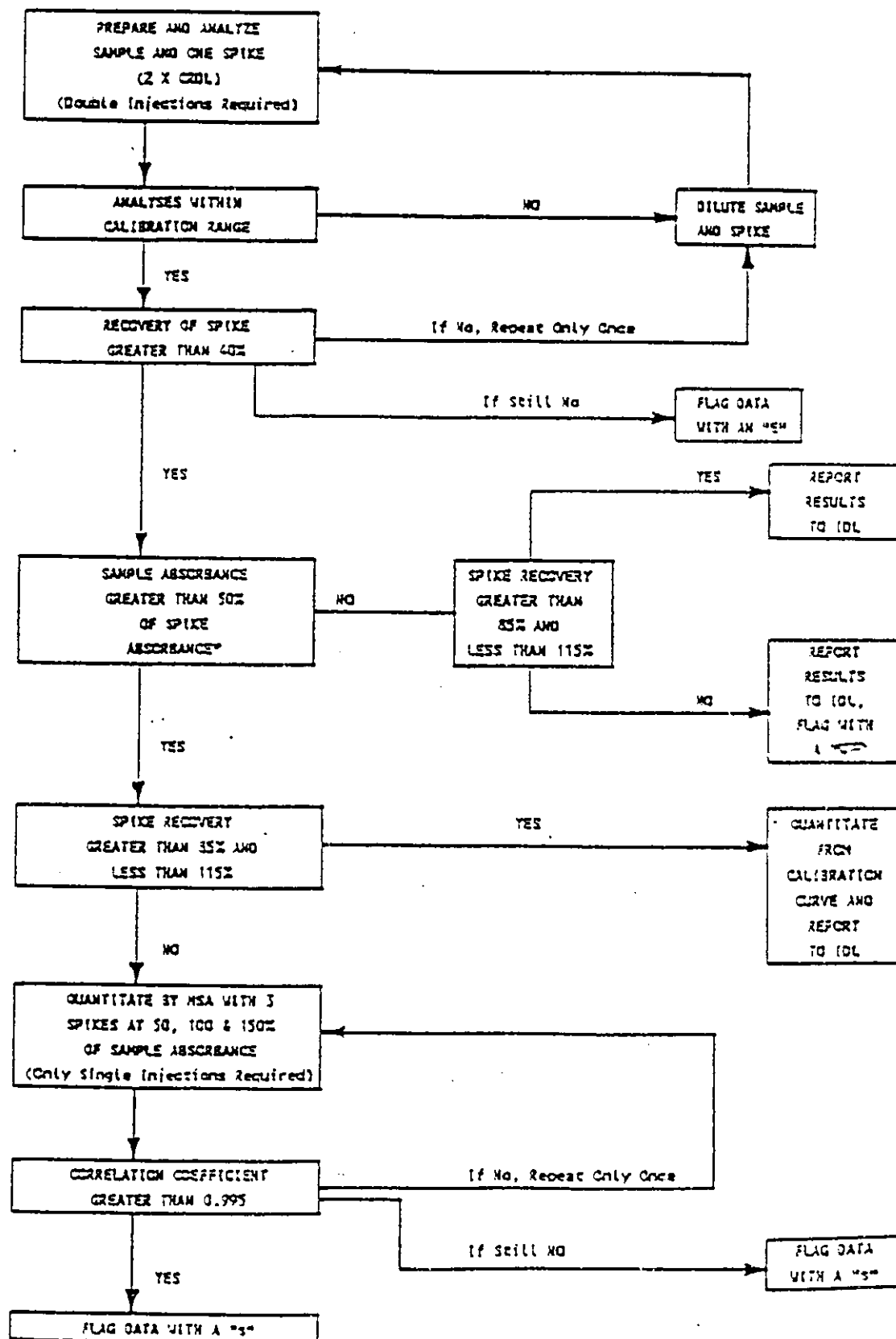
LSC: 100 ml
(Laboratory
Control
Standard)

Flame: A - LCS-fl (multi element), bottle #7
B - LCS-Na, K, bottle #8, (do not run for
priority pollutant metals or heavy
metals)
C - Any common element

Furnace: A - LCS-fu, bottle #9

<u>SPIKES:</u>	<u>Flame:</u>	<u>Bottle #</u>	<u>Amount</u>
		2	0.10 ml
		3	1.00 ml
		4	0.10 ml
		11	0.05 ml
		any common element	

<u>Furnace:</u>	<u>Bottle #</u>	<u>Amount</u>
	1	0.1 ml
	10	1.0 ml



*Spike absorbance is defined as (absorbance of spike sample) minus (absorbance of the sample).

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7.4.12 LABORATORY CONTROL SAMPLE ANALYSIS

The LCS monitors the efficiency of the digestion procedure. An aqueous LCS must be analyzed on a digestion batch basis, a solid LCS is analyzed on a monthly basis. Results of the LCS must be within 20% for all analytes except Sb and Ag. Results which fall outside these limits indicate a sample preparation problem or an instrument error.

7.4.13 SPIKED SAMPLE ANALYSIS

This will provide information about the effect of the sample matrix on the recovery of the analytes in question. The spike is added prior to the digestion step. A spike is performed for each matrix and each concentration range for each SDG. The spiking levels are found on Table 3. The following factors impact the outcome of the spike sample results, these include matrix suppression or enhancement effects, duplicate precision, digestion efficiency, contamination and the concentration level of the sample and the spike. If the spike recovery is outside 75-125% the data associated with the spiked sample is flagged with a "N" qualifier..

7.4.14 DUPLICATE SAMPLE ANALYSIS

A blind duplicate is usually submitted with each SDG. The results of the duplicate are used to monitor the precision of the method. Since many sample types are non-homogeneous, the data is flagged to indicate that the results were outside the quality control limits.

7.4.15 FURNACE ATOMIC ABSORPTION

All furnace samples are injected in duplicate and a post digestion spike is performed.

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7.4.8 ICP LINEAR RANGE ANALYSIS

A standard at two times the CRDL is analyzed at the beginning and end of each sample run or at a minimum of twice per eight hour shift (whichever is more frequent to verify linearity near the CRDL). The upper limit of the linear range must be verified on a quarterly basis. The standard must be within $\pm 5\%$ of the true value. Any sample results that exceed the linear range must be re-analyzed at a dilution to fall within the range.

7.4.9 INSTRUMENT DETECTION LIMITS

On three non-consecutive days the average of the standard deviations multiply by 3 are calculated from the analysis of a standard solution at a concentration 3 to 5 times the IDL with 7 consecutive measurements per day. The updated IDL's are performed starting on the 15th day of January, April, July, and October.

7.4.10 METHOD PERFORMANCE QA/QC

Preparation Blanks - for each batch of samples digested or for each 20 samples received whichever is more frequent, a preparation blank is prepared from distilled water and carried through the entire digestion process to determine if any contaminations are introduced in the preparation step. If the concentration in the blank is above the CRDL, than any samples with less than 10 times the concentration level identified on the blank must be redigested and re-analyzed. The reported sample results are not corrected for the blank results.

7.4.11 ICP SERIAL DILUTION ANALYSIS

One sample from each group of samples of a similar matrix type and concentration from each case must undergo at least one serial dilution to determine whether significant physical or chemical interferences exist due to physical or chemical interferences exists due to sample matrix. If the analyte concentration is a factor of 10 above the IDL after dilution, the analysis must agree within 10% of the original result. If the result is not within 10%, a chemical or physical interference effect should be suspected and the data flagged with a E qualifier.

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7.4.4 INSTRUMENT CALIBRATION

Instruments must be calibrated daily. The number of calibration standards are specified for each analytical method. Calibration standards must be prepared using the same type of acid or combination of acids used in the digestion. Initial Calibration Verification (ICV) is performed immediately following the instrument calibration by analyzing an independent, certified solution to verify the initial calibration. When the ICV results exceed the specified control limits, the analysis is stopped and corrective measures taken, the instrument is recalibrated and the calibration re-verified.

7.4.5 CALIBRATION CONTROL LIMITS

A calibration blank is analyzed each time the instrument is calibrated at the beginning and end of the run and at a frequency of 10 percent during analysis. If the CB result is greater than the CRDL, the analysis is halted and corrective action measures taken and the instrument is re-calibrated.

Metals	90-110
Mercury	80-120
Cyanide	85-115

7.4.6 CONTINUING CALIBRATION VERIFICATION

To assure calibration accuracy and monitor instrument performance during each analysis run, a CCV standard is analyzed at a frequency of 10% or every two hours whichever is more frequent. The concentration level is near the mid-range level of the calibration curve. The following control limits must be achieved before analysis can occur.

Metals	90-110
Mercury	80-120
Cyanide	85-115

7.4.7 ICP INTERFERENCE CHECK SAMPLE ANALYSIS (ICS)

Verifies the interelement and background correction factors. ICS is analyzed at the beginning and end of each analysis run or at a minimum twice per 8 hour day, whichever is more frequent. The ICS must be within +/-20% of the true value or analysis is terminated, corrective action measure taken and the instrument re-calibrated.

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Three to five working standards are prepared by dilution of the stock standard. The concentration of the calibration standards is chosen so as to cover the working range of the instrument. All sample measurements are made within this working range. Once the working standards are prepared, they are analyzed on ICAP or AAS.

The instrument responses of these analyses of the working standards are entered into calibration curves reflecting the absorbance/emission units readout equivalents to the concentration units. These curves then provide direct readout for unknown samples.

7.4.2 VERIFICATION OF CALIBRATION

Once the instrument has been initially calibrated, the analysis of the working standards is repeated during sample analysis and to confirm the calibration settings. If the calibration settings are not confirmed, the instrument has to be recalibrated. During sample analysis, a midpoint standard is analyzed to monitor instrument stability. If the analysis indicates the instrument calibration has changed, the instrument is recalibrated and the analysis is repeated.

7.4.3 CLP CALIBRATION

An initial calibration verification (ICV) is performed. The concentration of the calibration solution, "Initial Calibration True," is compared with the most recent value received from measurement, "Initial Calibration True," is compared with the most recent value received from measurement, "Initial Calibration Found" (ICF). The relative difference is computed as:

$$\%R = \frac{\text{Found (ICV)}}{\text{True (ICV)}} \times 100$$

This verification is repeated in specified intervals in the continuing calibration verification (CCV). Differences are calculated similarly with the formula:

$$\%R = \frac{\text{Found (CCV)}}{\text{True (CCV)}} \times 100$$

The relative deviation %R cannot exceed 10 percent for either ICV or CCV.

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At a frequency of 10%, or every two hours, a continuing calibration verification standard is analyzed to assure accuracy. If this is not within the central limits of 80-120% recovery, the analysis is terminated, the instrument recalibrated and samples reanalyzed. A continuing calibration blank is analyzed every 10% also. If this value is greater than the CRDL, analysis is terminated.

A matrix spike analysis is performed on each group of 10 samples of similar matrix-type, or for each SDG whichever is more frequent. Limits for spike recovery are 75-125%. If this criteria is not met, appropriate qualifiers are noted in the case narrative for the SDG.

A duplicate analysis is performed on each group of 10 samples of similar matrix type, or for each SDG. A control limit of 20% RPD shall be used for sample values $>5 \times \text{CRDL}$. A control limit of $+\text{CRDL}$ shall be use for sample values $<5 \times \text{CRDL}$. If this criteria is not met, appropriate qualifiers are noted in the case narrative.

7.3.5 SAMPLE PREPARATION FOR MERCURY ANALYSIS

Place 100ml of sample in a 300ml BOD bottle. If the sample is a soil, weigh a representative 0.2g portion of wet sample and place in the bottom of a BOD bottle. Add 5mL of sulfuric acid and 2.5mL of concentrated nitric acid mixing after each addition. Heat two minutes in a water bath at 95°C . Cool, add 50ml distilled water, 15mL potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 minutes at 95°C . Cool and add 6mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. Add 55mL of distilled water. Treating each bottle individually, add 5mL of stannous sulfate (6.3) and immediately attach the bottle to the aeration apparatus.

7.4 CALIBRATION: INDUCTIVELY COUPLED ARGON PLASMA SPECTROMETER (ICAP) ATOMIC ABSORPTION SPECTROPHOTOMETER (AAS) FLAME & FURNACE

7.4.1 INITIAL CALIBRATION

The ICAP and AAS are standardized for the metal of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration, or, in accordance with instrument manufacturer's recommendations.

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A preparation blank analytical spike must be analyzed within the control limits of 85-115% recovery. If the criteria is not met, the analysis is terminated, and then repeated.

The following ICP quality control criteria also pertains to furnace analysis: Initial and continuing calibration verification, initial and continuing calibration blank analysis, preparation blank analysis, duplicate sample analysis, LCS and IDL determinations. All requirements and control limits for this criteria is previously stated under ICP analysis.

7.3.2 FLAME ATOMIC ABSORPTION

The procedure for analysis by flame atomic absorption is called direct aspiration. A liquid sample is sprayed into a flame produced by the oxidation of acetylene or nitrous oxide. Subsequent lamp warm-up, alignment of the light beam, the flame and the monochrometer is carried out. The monochrometer is then used to measure the absorption of light, at a pre-set wavelength, caused by the sample in the concentration units.

The quality control criteria for flame analysis are the same as for mercury. The holding time for metals other than mercury is six months.

7.3.3 MERCURY ANALYSIS BY MANUAL COLD VAPOR TECHNIQUE

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7nm by mercury vapor. Organic 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 the AA. Absorbance is measured as a function of mercury concentration and is recorded in the usual manner.

7.3.4 QUALITY CONTROL REQUIREMENTS

Samples are preserved by acidification to a pH of <2 with nitric acid at the time of collection, and are analyzed within a 28-day holding time for plastic containers. The AA instrument is calibrated before and an initial calibration verification standard are run to verify calibration and baseline. This standard is from a different source than that used for calibration, and is of mid-range concentration on the curve fit.

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A pre-digestion spike sample analysis is performed on each group of similar matrix-type samples. Control limits are 75-125% recovery. A post-digested spike sample analysis is performed only when the pre-digestion spike recovery is outside the control limits and the sample result does not exceed four times the spike added (except Ag).

One duplicate analysis is analyzed from each SDG of similar matrix type. A control limit of 20% for relative percent difference shall be used for sample values $>5 \times \text{CRDL}$, and $+ \text{CRDL}$ for sample values $<5 \times \text{CRDL}$. If both values are less than IDC, the RPD is not calculated.

An aqueous Laboratory Control Sample (LCS) is analyzed using the same sample preparations. One LCS must be prepared and analyzed for each SDG. The control limits are 80-120% recovery except (Ag + Sb). If this criteria is not met, the analysis is terminated, samples redigested and reanalyzed. Results are reported on Form VII-IN.

If the analyte concentration is significantly high (minimally a factor of 10 above IDL), an analysis of a five-fold dilution must be performed. This serial dilution must agree within 10% of the original value.

Interelement correction factors for spectral interference due to Al, Ca, Fe and Mg are determined for all wavelengths within six months of the start of the contract and at least annually thereafter.

A linear range verification check standard must be analyzed to determine the upper limit of the ICP range. Results must be within +5% of the true value, analyzed and reported quarterly.

7.3 ATOMIC ABSORPTION

7.3.1 GRAPHITE FURNACE ATOMIC ABSORPTION

The graphite furnace is a flameless sampling device. The energy required for atomicization is supplied by passing a high electrical current through a graphite tube into which the sample has been placed. The furnace is aligned in the sampling area of the spectrophotometer in such a way that the light from the spectral lamp passes through the center of the graphite tube. The atomic vapor generated from a sample when the furnace is fired and absorbs light from the lamp. This absorption is plotted versus concentration and a curve fit is generated. All analysis requires duplicate injections for which the average will be reported.

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possibility of additional interferences are recognized and appropriate corrections made accordingly.

7.2.2 QUALITY CONTROL REQUIREMENTS

Samples are preserved by acidification to a pH of two upon collection, and are analyzed after digestion procedures within six months.

The ICP is calibrated (according to manufacturers instructions) prior to each analysis with freshly prepared standards. Absorbance versus concentration is plotted and a curve fit is produced. A mid-range standard from a different source is analyzed to initially verify the calibration at each wavelength used.

At a frequency of 10%, or every two hours, this standard is analyzed to ensure calibration accuracy during the analysis run. Control limits for this standard is 90-110% of true value. If this criteria is not met, analysis is terminated, instrument recalibrated and samples reanalyzed.

To verify linearity near the CRDL, an ICP standard is at two times the CRDL and is analyzed at the beginning and end of each sample analysis run. Specific acceptance criteria for the standard will be set by EPA.

A calibration blank is analyzed at each wavelength used immediately after every initial and continuing calibration verification. If the values exceed, the CRDL analysis is terminated.

A preparation or reagent blank consisting of deionized distilled water processed through each sample preparation procedure is prepared and analyzed with every sdg. If the analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated Sample Delivery Group must be ten times the blank concentration, otherwise, samples are redigested and reanalyzed. The sample concentration is not corrected for the blank value.

An ICP interference check sample (ICS) consisting of two solutions is analyzed to verify inter-element and background correction factors. Solution A contains interferences, solution AB contains analytes mixed with interferences. An ICS analysis consists of analyzing both solutions consecutively. Results of solution AB must fall within the control limit of +20% of the true value.

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peroxide digestate until the volume has been reduced to approximately 2 ml, add 10 ml of Type II water and warm the mixture. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 ml with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. The diluted digestate solution contains approximately 2 percent (v/v) HNO_3 . Dilute the digestate 1:1 (200 ml final volume) with acidified water to maintain constant acid strength. For analysis, withdraw aliquots of appropriate volume, and add any required reagent or matrix modifier. The sample is now ready for analysis.

Calculations: A separate determination of percent solids must be performed. The concentrations determined in the digest are to be reported on the basis of the dry weight of the sample.

$$\text{Concentration (dry wt.) (mg/kg)} = \frac{C \times V}{W \times S}$$

Where: C = Concentration (mg/L)
V = Final volume in liters after sample preparation
W = Weight in kg of wet sample
S = Percent solids/100

7.2 INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRIC METHOD (200.7 CLP)

7.2.1 TRACE ELEMENT ANALYSIS OF WATER AND WASTES

Simultaneous multielement determinations of trace elements are performed. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer. Background correction techniques are required to compensate for variable background contributions. The

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Procedure:

- Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01g) a 1.0g to 1.5g portion of sample and transfer to a beaker portion.
- Add 10 ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3 , replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 4 ml while maintaining a covering of solution over the bottom of the beaker.
- After the second reflux step has been completed and the sample has cooled, add 2 ml of Type II water and 3 ml of 30 percent hydrogen peroxide (H_2O_2). Return the beaker to the hotplate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the beaker.
- Continue to add 30 percent H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. NOTE: Do not add more than a total of 10 ml 30 percent H_2O_2 .
- If the sample is being prepared for the furnace AA analysis of Sb, the flame AA or ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, Sn, V, and Zn, add 5 ml of 1:1 HCl and 10 ml of Type II water, return the covered beaker to the hotplate, and heat for an additional 10 minutes. After cooling; filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 ml with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5 percent (v/v) HNO_3 . Dilute the digestate 1:1 (200 ml final volume) with acidified water to maintain constant acid strength. The sample is now ready for analysis.
- If the sample is being prepared for the furnace analysis of As, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Ag, Tl, V, and Zn, continue heating the acid-

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making certain the sample does not boil. Cool sample and filter to remove insoluble material. NOTE: In place of filtering, the sample after dilution and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Adjust the volume to 100 ml with deionized distilled water. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

7.1.2 SOIL/SEDIMENT SAMPLE PREPARATION

Scope and Application: This method is an digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace Atomic Absorption Spectroscopy (AAS) or by Inductively Coupled Plasma Spectroscopy (ICP). Samples prepared by this method may be analyzed by AAS or ICP for the following metals.

Aluminum	Chromium	Potassium
Antimony	Cobalt	Selenium
Arsenic	Copper	Silver
Barium	Iron	Sodium
Beryllium	Lead	Thallium
Cadmium	Magnesium	Vanadium
Calcium	Nickel	Zinc

Summary of Method: A representative lg (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Hydrochloric acid is used as the final reflux acid for the furnace AA analysis of Sb, the flame AA or ICP analysis of As, Sb, Ba, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V, and Zn. A separate sample shall be dried for a total solids determination.

Apparatus and Materials: 250 ml beaker or other appropriate vessel; watch glasses; thermometer that covers range of 0° to 200°C; Whatman No. 42 filter paper or equivalent.

Reagents: ASTM Type II water (ASTM D1193) (water must be monitored); concentrated Nitric Acid (sp.gr. 1.41); concentrated hydrochloric acid (sp.gr. 1.19); hydrogen peroxide (30 percent).

Sample Preservation and Handling: Soil/sediment (non-aqueous samples) must be refrigerated at 4°C ($\pm$ 2°C) upon receipt until analysis.

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7. NYSDEC ASP METHOD-METALS

The metals lab is equipped to analyze nearly any aqueous, soil, sludge or sediment sample. A combination of ICP's, furnace AA's, flame AA's and a dedicated AA for cold vapor mercury analysis. Holding times: Mercury - 26 days; Metals - 180 days.

7.1 INORGANIC METHODS - TRACE METALS ANALYSIS

The metals lab is equipped to analyze nearly any aqueous soil, sludge or sediment sample. This capacity is made possible with the following equipment: one ARL Inductively Coupled Plasma (ICP); three furnace equipped Atomic Absorption Spectrometers (AAS) dedicated to the furnace application Perkin-Elmer AA-2380 for flame and cold vapor mercury analyses.

This combination of equipment provides a high degree of versatility, namely rapid turnaround time, low detection limits and back-up support if a unit was to fail.

7.1.1 WATER SAMPLE PREPARATION

Acid Digestion Procedure for Furnace Atomic Absorption Analysis: Shake sample and transfer 100 ml of well mixed sample to a 250 ml Griffin beaker, add 1 ml of (1+1) HNO_3 will provide data for total concentration contained in the sample, and 2 ml 30 percent H_2O_2 . Cover with watch glass and or similar cover, heat on a steambath or hotplate for two hours at 95°C or until the volume is reduced to between 25 and 50 ml, make certain samples do not boil. Cool sample and filter to remove insoluble material. NOTE: In place of filtering, the sample after dilution and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Adjust sample volume to 100 ml with ionized distilled water. The sample is now ready for analysis. Concentrations so determined shall be reported as "total." If Sb is to be determined by furnace, AA use the digestates prepared for ICP/AA analysis.

Acid Digestion Procedure for Inductively Coupled Plasma (ICP) and Flame Atomic Absorption (AA) Analyses: Shake sample and transfer 100 ml of a well mixed, acid preserved sample to a Griffin beaker. Add 2 ml of (1+1) HNO_3 and 10 ml of (1+1) HCl to the sample and heat on a steambath or hotplate for sample and heat on a steambath or hotplate for two at 95°C or until the volume has been reduced to between 25 and 50 ml

8. NYSDEC ASP Method 415.1 Total Organic Carbon

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8. NYSDEC ASP METHOD 415.1 - TOTAL ORGANIC CARBON

Method 415.1 is used for the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes.

A Dohrmann DC-190 carbon analyzer converts the carbon to CO₂ by catalytic combustion. The CO₂ formed is measured directly by infrared detection. The amount of CO₂ is directly proportional to the concentration of carbonaceous material in the sample.

The Dohrmann DC-190 is calibrated per manufacturers specifications. Check standards are analyzed every 10 samples. All analysis are run in duplicate with the average value reported.

It is suggested that the following USEPA Lloyd Kahn "Determination of Total Organic Carbon in Sediment" method be substituted for the EPA 415.1 method when analyzing non-liquid samples. Method 415.1 is not applicable to a soil/sediment matrix. The Lloyd Kahn method for TOC is widely used by the USEPA and the Army Corps of Engineers.

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DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT

JULY 27, 1988

PREPARED BY: Lloyd Kahn, Quality Assurance Specialist

AFFILIATION: U.S. Environmental Protection Agency, Region II
Environmental Services Division
Monitoring Management Branch
Edison, New Jersey 08837

1. Scope and Application

- 1.1 This method describes protocols for the determination of organic carbon in ocean sediments.
- 1.2 Although the detection limit may vary with procedure or instrument, a minimum reporting value of 100 mg/kg will be required for the ocean dumping/dredging program.
- 1.3 Several types of determination, which are considered equivalent are presented.
- 1.4 Data are reported in mg/kg on a dry weight basis.
- 1.5 Wet combustion methods are not considered to be equivalent to the pyrolytic methods herein described.

2. Summary of Method

- 2.1 Inorganic carbon from carbonates and biocarbonates is removed by acid treatment.
- 2.2 The organic compounds are decomposed by pyrolysis in the presence of oxygen or air.
- 2.3 The carbon dioxide that is formal is determined by direct non-dispersive infrared detection, flame ionization gas chromatography after catalytic conversion of the carbon dioxide to methane; thermal conductivity gas chromatography, differential thermal conductivity detection by sequential removal of water with magnesium perchlorate.
- 2.4 Water content is determined on a separate portion of sediment.

3. Sample handling and Preservation

- 3.1 Collect sediments in glass jars with Teflon or aluminum foil. Cool and maintain at 4°C. Analyze within 14 days.

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4. Interferences

- 4.1 Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.
- 4.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4°C, analyzing within the specified holding time, and analyzing the wet sample.

5. Apparatus

5.1 Drying oven maintained at 103° to 105°C.

5.2 Analytical instrument options:

- A. Perkin Elmer Model 240C Elemental Analyzer or equivalent: In this instrument, the sample from Section 7.2 is pyrolyzed under pure oxygen, water is removed by magnesium perchlorate and the carbon dioxide is removed by ascarite. The decrease in signal obtained by differential thermal conductivity detectors placed between the combustion gas stream before and after the ascarite tubs is a measure of the organic carbon content.
- B. Carlo Erba Model 1106 CHN Analyzer, or equivalent: In this apparatus, the sample is pyrolyzed in a induction type furnace, and the resultant carbon dioxide is chromatographically separated and analyzed by a differential thermal conductivity detector.
- C. LECO Models WR-12 and Model WR-112: In the LECO WR-12, the sample is burned in high frequency induction furnace, the carbon dioxide is selectively absorbed at room temperature in a molecular sieve. It is subsequently released by heating and is measured by a thermal conductivity detector. The WR-112 is an upgraded WR-12 employing microprocessor electronic and a printer to replace the electronic digital voltmeter.
- D. LECO Model CR-12 carbon determinator: In the LECO CR-12 carbon determinator, the sample is combusted in oxygen, moisture and dust are removed by appropriate traps and the carbon dioxide is measured by a selective, solid state, infrared detector. The signal from the detector is then processed by microprocessor and the carbon content is displayed on a digital readout and recorded on an integral printer.
- E. LECO Model CHM-600 or CHM-800 analyzers: In the LECO Models CHM-600 and CHM800 elemental analyzers, the sample is burned under oxygen in a resistance furnace and the

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carbon dioxide is measured by a selective infrared detector.

- F. Dohrman Model DC85 Digital High Temperature TOC Analyzer: In this instrument, the sample is burned in resistance furnace under oxygen, the interfering gases are removed by a sparger/scrubber system and the carbon dioxide is measured by a non-dispersive infra-red detector and shown on a digital display in concentration units.

5.3 No specific Analyzer is recommended as superior. The above listing is for information only and is not intended to restrict the use of other unlisted instruments capable of analyzing TOC. The instruments to be used must have the following specifications:

- A. A combustion boat which is heated in a stream of oxygen or air in a resistance or induction-type furnace to completely convert organic substances to CO₂.
- B. A means to physically or by measurement technique to separate water and other interferants from CO₂.
- C. A means to quantitatively determine CO₂ with adequate sensitivity (100 mg/kg), and precision (25 percent at the 95 percent confidence level as demonstrated by repetitive measurements of a well mixed ocean sediment sample).

5.4 A strip chart or other permanent recording device to document the analysis.

6. Reagents

- 6.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.
- 6.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter; Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml. NOTE: Sodium oxalate and acetic acid are not recommended as stock Solutions.
- 6.3 Potassium hydrogen phthalate, standard solution: Prepare standard solutions from the stock solution by dilution with distilled water.
- 6.4 Phosphoric acid solution, 1:1 by volume.

7. Procedure

- 7.1 Weigh the well mixed sample (up to 500 mg) into the combustion boat or cup. Add 1:1 phosphoric acid drop wise until effervescence stops. Heat to 75°C. NOTE: This procedure will

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convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample.

7.2 Analyze the residue according to the instrument manufacturer's instructions.

7.3 Determine percent residue on a separate sample aliquot as follows:

- . Heat a clean 25 ml beaker at 103° to 105°C for one hour. Cool in desiccator, weigh to the nearest mg and store in desiccator until use.
- . Add 1g, weighed to the nearest mg, of an aliquot of the well-mixed sample.
- . Dry and heat in the 103° to 103°C oven for one hour. Cool in desiccator. Weigh to the nearest mg.

8. Calibration

8.1 Follow instrument manufacturer's instructions.

8.2 Prepare calibration curve plotting mg carbon vs. instrument response, using four standards and a blank covering the analytical range of interest.

9. Precision and Accuracy

9.1 The precision and accuracy will differ with the various instruments and matrices and must be determined by the laboratories reporting data. To initiate a control chart, a representative sample of well mixed sediment should be analyzed 15 times to determine the analytical precision. Set up a control chart showing three times the standard deviation limits for precision.

9.2 Subsequently during analysis of environmental samples, take one sample per batch of 20 or less and run in quadruplicate. Calculate standard deviation and report with initial control chart data.

9.3 If the sample being run in quadruplicate exceeds the three standard deviation limit, identify error and rerun environmental samples in that batch along with the quadruplicate sample.

9. Data Generation

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9. DATA GENERATION

All Deliverables submitted shall be in conformance with the requirements of CLP protocol or methods requirements including any additional requirements of the client. If any technical or analytical problems in the laboratory preclude the successful performance of the requirements of the contract, the client will be notified within 24 hours.

9.1 COLLECTION AND DATA REDUCTION

Data validation begins with the processing of data and continues through data review and reporting of analytical results. Data processing can be performed by the analyst who obtained the data or by another analyst. Data review starts with an analyst independent of the data acquisition and processing, reviewing (validating) that the data processing was performed correctly and continues through verifying that the reported analytical results correspond to the data acquired and processed.

As stated, the first step in validation is data processing. In general, data will be processed by an analyst in one of the following manners:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets.
- Input of raw data for computer processing.
- Direct acquisition and processing of raw data by computer.

If data are manually processed by an analyst, all steps in the computation shall be provided, including equations used and the source of input parameters such as response factors, dilution factors and calibration constants. If calculations are not performed directly on the data sheet, calculations should be attached to the data sheets.

For data that are entered by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and

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response factors, and output parameters such as units and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date the resulting output.

The GC/MS analyses are processed on the Aquarius software and the reports generated by the Terwilliger software. The pesticide data is acquired using a Nelson Turbochrom and the reporting package is processed by Finnigan Formaster. The metals data is reported using Telecations software.

9.1.1 REVIEW OF DATA PROCESSING

The following is a discussion of the method to be used for reviewing (checking) data processing. At least 20 percent of all data shall be reviewed in this manner. If, during the review process, errors are determined, a complete review shall be performed for the data set. The data must be reviewed either by a senior analyst or the department supervisor. The analyst has to provide the reviewer with the complete data package. The package shall include, as appropriate: raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors, etc.

The review of the data shall cover the following aspects: Appropriateness of equations used; Correctness of numerical input; Numerical correctness of all calculations. (This should be done by re-performing numerical computations.); Correct interpretation of strip charts, etc.

The review process must be thorough enough to validate the results. If the reviewer disagrees with any part of the computations, he/she shall mark through the number with a single line and place the revised number above it. Any changes made by the reviewer shall be re-reviewed by the originator. If he/she agrees with the change, no action is necessary. If he/she disagrees, the two must resolve their difference so that they agree with the result presented.

9.1.2 DATA VALIDATION

The actual data package is reviewed for correctness and completeness by the organic and inorganic laboratory supervisors or the technical manager. Their review includes validation of method used, as well as compliance with the requirements of the CLP protocol. Following their approval the package goes to typing. The finalized reports undergo a last review by the quality assurance manager.

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9.1.3 DATE REPORTING AND AUTHORIZATION PROCEDURES

Completed data packages are generated in all organic departments through the use of the various software packages in use (e.g. Aquarius, Terwilliger and Formaster). The inorganic traditional data is submitted to the QC department, signed for and reviewed prior to acceptance. The metals raw data is submitted to the QC department which computes the data and, using Telications software, generates the data package. Examples of the order of data packages by the ASP CLP format and NJDEP and USEPA format are included.

All data packages are subjected to review by either the department supervisor, technical manager or quality assurance manager. The review process includes all information included in the organic and inorganic checklists that are included. Any deviations or noncompliances are documented in the case narrative written by the reviewer. Any omissions or errors are listed and the data package rejected and returned to the department for correction. After corrections have been made, the reviewer then verifies the corrections, the case narrative is typed and signed by the reviewer.

The following individuals are authorized to review data packages:

Metals and Inorganic

Maureen Wineberger	Metals Supervisor
Salvatore Badalamenti	Wet Chemistry Supervisor
Vincent Stancampiano	Vice President
Joann Slavin	QA Manager

Pesticides

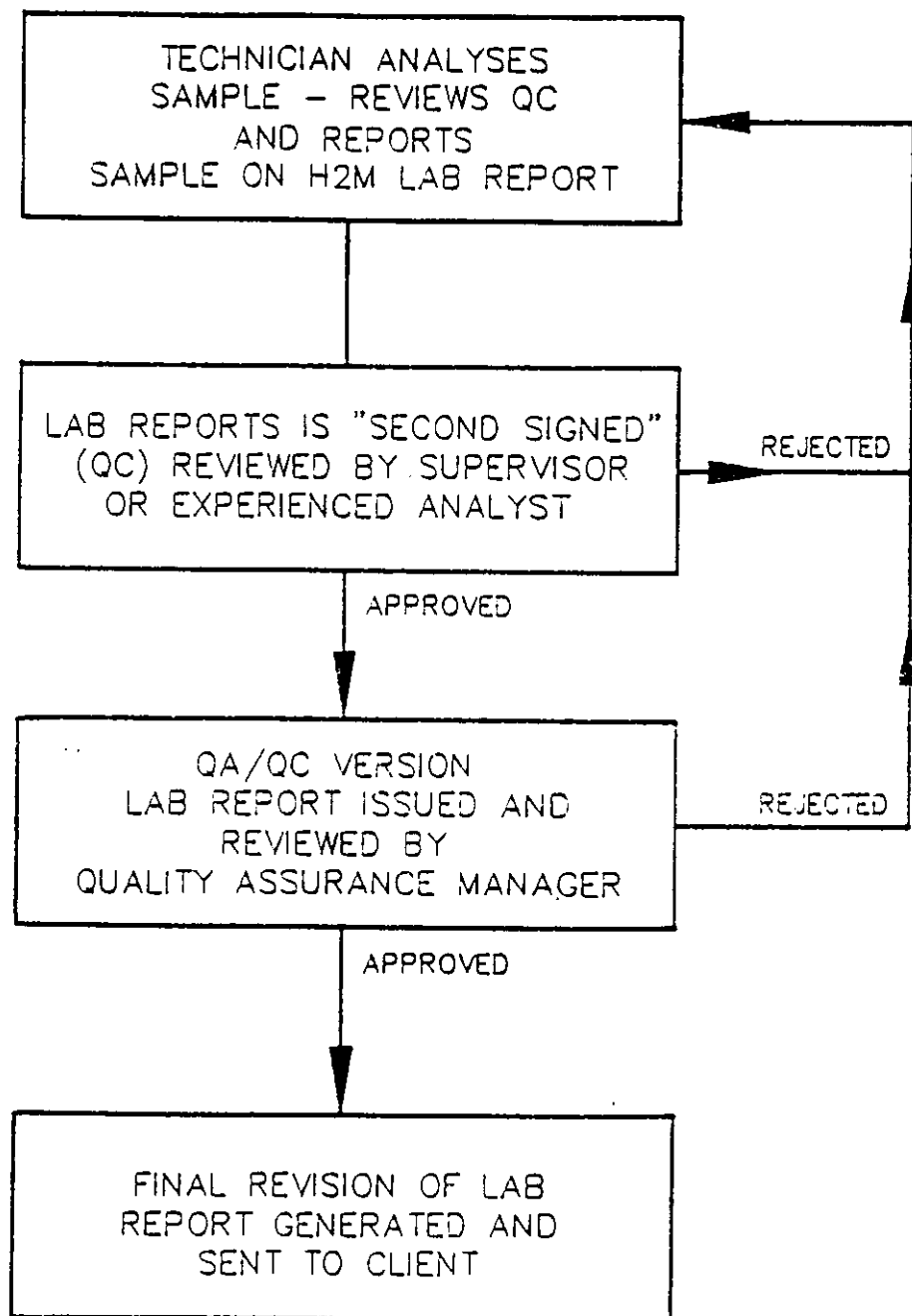
Ursula Middel	Technical Manager
Joann Slavin	QA Manager
Stephan Stefanou	GC Supervisor

GC/MS

Glen Bochicchio	GC/MS Supervisor
Ursula Middel	Technical Manager
Joann Slavin	QA Manager

Only the above mentioned individuals are authorized to perform a review of the data. The review checklist is completed and signed and stored with the laboratory's copy of the data packages. Any questions are forwarded to the individual who performed the review.

LAB REPORT REVIEW PROCEDURE



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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 1 of 9

VOA
BNA
PEST

Site: _____

Project: _____

Summaries

YES NO N/A

1. Are external COC records submitted? _____
2. Are internal COC records submitted? _____
3. Do internal COC records document continuity? _____
4. Does record of samples analyzed match internal and external chain of custody? _____
5. Is sample preparation summary and copy of sample preparation log submitted? _____
6. Is narrative included? _____
7. Are holding times for sample preparation met of 7 days for volatiles and 5 days for extractables? _____
8. Are 40 day limits met for sample analysis for the extractables? _____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

QA/QC PACKAGE

I. Surrogate Percent Recovery

1. Are the forms present for water? _____
soil? _____
2. Are all the samples listed for each matrix? _____
3. Are the outliers correctly marked with an asterisk? _____
4. Are the calculations correct, based on extract volume, GPC, dilution and final volume? _____
5. Do surrogate recoveries meet CLP protocol for all samples? _____
6. Are both reports submitted for samples that do not meet recoveries for both extracts? _____
7. Is recovery for DBC above 5%? _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

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VOA
BNA
PEST

Site: _____

Project: _____

Surrogate Percent Recovery (con't)

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

II. Matrix Spike Duplicates and MSB Recovery

YES NO N/A

1. Is matrix spike summary submitted for a sample of this batch?	_____	_____	_____
2. Is data summary submitted for a sample from another batch?	_____	_____	_____
3. Are frequency requirements met for spike analysis?	_____	_____	_____
4. Are the outliers correctly marked with an asterisk?	_____	_____	_____
5. Are the number of outliers for %REC and %RPD listed correctly at the bottom of the page?	_____	_____	_____
6. Are data calculated and transcribed correctly, based on GPC, dilution, and final volume?	_____	_____	_____
7. Does data meet CLP protocol for VOA and BNA?	_____	_____	_____
8. Is a spiked blank analyzed and summary submitted?	_____	_____	_____
9. Do MSB recoveries meet limits of 75-125%?	_____	_____	_____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 3 of 9

VOA
BNA
PEST

Site: _____

Project: _____

III. Method Blanks

YES NO N/A

1. For VOA's: Has an instrument blank been analyzed at least once every 12 hours?	_____	_____	_____
2. For B/N/A's and Pest/PCB's: Has a method blank been extracted and analyzed for each set of samples and subjected to the same cleanup?	_____	_____	_____
3. Are the method blank summaries present and complete for all blanks?	_____	_____	_____
4. Are the dates correct on the summary table and the reports?	_____	_____	_____
5. Are common laboratory contaminants present? If yes: Under CRDL _____ Between CRDL and 5x CRQL _____ Above 5x CRQL _____	_____	_____	_____
6. Are other TCL contaminants present? If yes: Under CRDL _____ Above CRDL _____	_____	_____	_____
7. Are the contaminants also found in samples qualified with a "B"?	_____	_____	_____
8. Do all surrogate recoveries meet the allowable limits?	_____	_____	_____
9. Are all internal standard areas within the allowable limits?	_____	_____	_____
10. Are blanks analyzed on same instrument and column as samples?	_____	_____	_____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 4 of 9

VOA
BNA
PEST

Site: _____

Project: _____

IV. GC/MS Tuning and Mass Calibration

YES NO N/A

1. Are all the tuning and mass calibration forms present?	_____	_____	_____
2. Has a tuning performance compound been run for every 12 hours of sample analysis?	_____	_____	_____
3. Are bar graph spectra and m/z listings submitted for BFB/DFTPP?	_____	_____	_____
4. Are data transcribed correctly from mass lists to the GC/MS tuning form?	_____	_____	_____
5. Are ratios for abundances calculated correctly?	_____	_____	_____
6. Have the ion abundance criteria been met for all mass calibrations?	_____	_____	_____

Missing information, error or noncompliance for

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

SAMPLE REPORTS

1. Are TCL and TIC (if applicable) forms present for all samples?	_____	_____	_____
2. Are header information and units correct?	_____	_____	_____
3. Are positives calculated and transcribed correctly from raw data to reporting forms?	_____	_____	_____
4. Are correct qualifiers used?	_____	_____	_____
5. Are the reported limits calculated correctly for dry weights, GPC and dilution?	_____	_____	_____
6. If a sample exceeded linearity for a parameter, was it reanalyzed after dilution?	_____	_____	_____
7. Do results for sample duplicates match?	_____	_____	_____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 5 of 9

VOA
BNA
PEST

Site: _____

Project: _____

SAMPLE REPORTS (con't)

YES NO N/A

8. Are all chromatograms, quant lists, mass spectra and computer identifications for TIC's submitted?	_____	_____	_____
9. Are positives and surrogate and internal standards marked on chromatograms?	_____	_____	_____
10. Do positives meet criteria for mass-abundances, compared with standard?	_____	_____	_____
11. Are extraneous ions present greater than 10% of base peak?	_____	_____	_____
12. If GPC was performed, are the GPC calibration check recoveries submitted, including chromatograms for analysis and calibration standard?	_____	_____	_____

Pest/PCB's Only

13. Are the PCB pattern recognizable? Could minimum areas be detected?	_____	_____	_____
14. If positive PCB's are found, are reporting limits raised for interfering pesticides?	_____	_____	_____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
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Item #____: _____ on page _____
see CLP paragraph _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

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VOA
BNA
PEST

Site: _____

Project: _____

STANDARD PACKAGE FOR VOA & BNA

YES NO N/A

Initial Calibration

1. Was a 5-point initial calibration performed per matrix and level and the form submitted? _____
2. Do the initial calibrations meet criteria for CCC's? (25% RSD for VOA and 30% for BNA) _____
3. Do the initial calibrations meet criteria for SPCC's? _____
4. Are all average relative response factors at or above 0.05? _____
5. Are all other RSD's under or equal 35% for VOA and 30% for BNA? _____
6. Did levels used correspond to CLP requirements? _____

Continuous Calibration

7. Was a continuing calibration performed per matrix per level? _____
8. Do all CCC's meet the requirements for continuing calibration (25% D for VOA, 20% for BNA) _____
9. Do all SPCC's meet the requirements for continuing calibration? _____
10. Are all TCL response factors at or above 0.05? _____
11. Is the %D less than 35% for all VOA compounds, and less than 20% for BNA compounds? _____

Internal Standard Area Summary

1. Is summary submitted? _____
2. Do areas comply with requirements? _____
3. Is retention time shift of internal standard less than 30 seconds? _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

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VOA
BNA
PEST

Site: _____
Project: _____

STANDARD PACKAGE FOR VOA & BNA (con't)

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____
Item #____: _____ on page _____
see CLP paragraph _____
Item #____: _____ on page _____
see CLP paragraph _____
Item #____: _____ on page _____
see CLP paragraph _____

STANDARD PACKAGE FOR PESTICIDES/PCB'S

YES NO N/A

Form VIII-1

1. Was sequence completed within 72 hours?	___	___	___
2. Does order of runs comply with sequence requirements?	___	___	___
3. Are all headers filled out completely and correctly?	___	___	___
4. Are all EVALB runs listed?	___	___	___
5. Is breakdown under 20%?	___	___	___
6. Are breakdown peaks identified in the EVALB runs?	___	___	___
7. Does linearity meet criteria of 10% D for primary column?	___	___	___
8. Does linearity meet criteria of 10% D for secondary column?	___	___	___

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

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VOA
BNA
PEST

Site: _____

Project: _____

STANDARD PACKAGE FOR PESTICIDES/PCB'S (con't)

YES NO N/A

Form VIII-2

9. Is DBC shift under 2% for packed 0.3% for cap.
column and 1.5% for widebore cap. columns?

Form IX

10. Are all standards of sequence documented?

11. Was %D under 15% for quantification?

12. Was %D under 20% for confirmation?

Form X

13. Are all reported positives listed on Form X with
correct windows?

14. Do all peaks fall within windows?

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

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CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

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VOA
BNA
PEST

Site: _____

Project: _____

RAW OC PACKAGE

YES NO N/A

1. Are all TCL and TIC (if applicable) report forms present for all blanks, holding blanks, matrix spikes, matrix spike duplicates, and MSB? _____
2. Are copies submitted for chromatograms and quant. lists? _____
3. Are mass spectra and computer identification for TIC included for blanks? _____
4. Do concentrations in MS/MSD reports match concentrations in unspiked sample? _____
5. Are breakdown products for Endrin and DDT found and marked? _____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

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CHECKLIST FOR REVIEW OF INORGANIC CLP PACKAGES

Page 1 of 6

Reviewer: _____ Date: _____ Package ID: _____

Cover Page

YES NO N/A

Are all samples listed with correct corresponding lab number? (check against COC)

☐ ☐ ☐

Is the SDG number properly identified?

☐ ☐ ☐

(1) Reports

Are all form I's present and complete for all samples in the SDG?

☐ ☐ ☐

Are results in correct units (ug/l for waters and mg/kg for soils)?

☐ ☐ ☐

Are sample results for each element corrected for percent solids on soils?

☐ ☐ ☐

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

☐ ☐ ☐

Are all "less than IDL" values properly coded with "U"?

☐ ☐ ☐

Are all values over the IDL and less than the CRDL properly coded with "B"?

☐ ☐ ☐

Is physical description of samples before and after digestion listed?

☐ ☐ ☐

(2A) Initial and Continuing Calibration Verification

Are all elements reported for all ICV's and CCV's?

☐ ☐ ☐

Are the true values listed correctly?

☐ ☐ ☐

Are all %R's within control limits?

☐ ☐ ☐

Metals..... 90-110%

Hg & Sn..... 80-120%

Cn⁻..... 85-115%

If no, stop and find out why.

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

☐ ☐ ☐

Are the method's correctly entered for all elements, including those elements run on more than one instrument?

☐ ☐ ☐

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CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 2 of 6

	YES	NO	N.
<u>(2B) CRDL Standard</u>			
Are all elements reported in AA or ICP column?	☐	☐	☐
Are all ICP elements reported for both CRII and CRIF?	☐	☐	☐
Are the true values entered correctly for all elements?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(3) Blanks</u>			
Are all elements reported for ICB's, CCB's?	☐	☐	☐
Is the proper matrix entered for the Prep Blank?	☐	☐	☐
Are there additional forms if there is more than one matrix?	☐	☐	☐
Is the data for the Soil Prep Blank entered in MG/KG?	☐	☐	☐
Are all blanks (ICB's, CCB's, PB's) less than the CRDL? (Flagged either U or B)	☐	☐	☐
Are the U and B flags used properly (check 10%)?	☐	☐	☐
Check 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(4) ICP Interference Check Samole</u>			
Is one form present for each ICP run?	☐	☐	☐
Are all Interference check sample results inside 80-120%?	☐	☐	☐
Was an Interference check sample analyzed at the beginning and end of each run?	☐	☐	☐
Are the true solution values entered correctly?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐

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CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

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	YES	NO	N
<u>(5A) Spike Sample Recovery</u>			
Is there a form present and complete for all designated MS/MSD's?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
Are all recoveries within control limits 75-125% for samples with concentrations 4 times the spike added?	☐	☐	☐
If no, are the elements properly flagged with "N" on this form, and the associated form I's?	☐	☐	☐
Are sample & spike analysis results, and spike added in correct units?	☐	☐	☐
<u>(5B) Post Digest Spike Recovery</u>			
Is there a form present and complete for all designated MS/MSD's?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
Are recoveries within 75-125% for those elements flagged on the associated form 5A (except Ag)?	☐	☐	☐
<u>(6) Duplicate</u>			
Is there a form present and complete for all designated MS/MSD's?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
Is the RPD column left blank when both sample and duplicate are less than the instrument detection limit?	☐	☐	☐
Does the control limit column have the CRDL (in appropriate units) if the sample or duplicate results are less than 5 times the CRDL?	☐	☐	☐

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CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

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	YES	NO	N/
<u>(6) Duplicate (con't)</u>			
If yes, is the absolute difference between sample and duplicate less than the CRDL?	☐	☐	☐
If no, is it flagged with "*" on this form and associated form I's?	☐	☐	☐
For those element which have a sample or duplicate result above the 5 times the CRDL, is the RPD less than 20%?	☐	☐	☐
<u>(7) Laboratory Control Sample</u>			
Is an LCS form present and complete for every group of samples of similar matrix and each group digested?	☐	☐	☐
Are all percent recoveries between 80-120% (except Ag & Sb)?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(8) Method of Standard Additions</u>			
If MSA's were required, were they properly entered on this form?	☐	☐	☐
If no MSA's were required, is there a blank form with the header properly filled out present?	☐	☐	☐
If the correlation coefficient was < 0.995, was the MSA re-run once?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(9) Serial Dilution</u>			
Was a serial dilution analysis performed for each 20 samples?	☐	☐	☐
each matrix type?	☐	☐	☐
each concentration range?	☐	☐	☐
If no for any of the above, is any sample concentration (undiluted) greater than			

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CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

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	YES	NO	N
<u>(9) Serial Dilution (con't)</u>			
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	___	___	___
Was the field blank used for serial dilution?	___	___	___
Are all serial dilution results within 10% when the original sample concentration is greater than 50 times the IDL?	___	___	___
If no, are the associated data flagged with an "E" on this form and associated form I's?	___	___	___
<u>(10) Holding Times</u>			
Check holding times against raw data and chain of custody for:			
Mercury (26 days).....exceeded?	___	___	___
Cyanide (12 days).....exceeded?	___	___	___
other metals (6 months)....exceeded?	___	___	___
<u>(11) Instrument Detection Limit</u>			
Are all IDL's present for all analytes and all methods?	___	___	___
Is the IDL greater than the CRDL for any analytes?	___	___	___
If yes, is the concentration of any of the samples analyzed on the instrument whose IDL exceeds CRDL, greater than 5 times the IDL?	___	___	___
<u>Raw Data</u>			
Is digestion log present and complete for flame AA/ICP and furnace?	___	___	___
Are all LCS & PB's listed on digestion logs?	___	___	___
Are preparation dates, weights, dilutions and volumes used to obtain values on all digestion logs?	___	___	___
Are total solids sheets for soils present and complete?	___	___	___
Was a continuing calibration performed every 10 samples?	___	___	___

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CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 6 of 6

	YES	NO	N/
<u>Raw Data (con't)</u>			
Was an initial calibration blank analyzed, and a continuing calibration blank analyzed every 10 samples?	☐	☐	☐
Was one preparation blank analyzed for each 20 samples, each matrix type?	☐	☐	☐
Are all raw data to support all ICP sample analyses and QC operation's present?	☐	☐	☐
legible?	☐	☐	☐
properly labeled?	☐	☐	☐
Are all raw data to support all Flame sample analyses and QC operation's present?	☐	☐	☐
legible?	☐	☐	☐
properly labeled?	☐	☐	☐
Are all raw data to support all Furnace sample analyses and QC operation's present?	☐	☐	☐
legible?	☐	☐	☐
properly labeled?	☐	☐	☐
Are all furnace samples which are flagged with a "W" on raw data, flagged on the associated Form I's?	☐	☐	☐
<u>Calibration</u>			
Is a record of at least a 2 point calibration present for ICP?	☐	☐	☐
Is a record of at least a 4 point calibration present for each flame AA?	☐	☐	☐
Furnace AA?	☐	☐	☐
Is a record of a 4 point calibration present for Hg?	☐	☐	☐
Is a record of a 4 point calibration present for Cd?	☐	☐	☐

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B. - SAMPLE DATA SUMMARY PACKAGE

As specified in the Delivery Schedule, one Sample Data Summary Package each shall be delivered to BTSR and the sample collector concurrently with delivery of other required sample data. The Sample Data Summary Package consists of copies of specified items from the Sample Data Package. These items are listed below and described under part C, Sample Data Package.

The Sample Data Summary Package shall be ordered as follows and shall be submitted separately (i.e., separated by rubber bands, clips or other means) directly preceding the Sample Data Package. Sample data forms shall be arranged in increasing NYSDEC sample number order, considering both letters and numbers. E487 is a lower sample number than R187, as E precedes R in the alphabet.

The Sample Data Summary Package shall contain all data for all samples within one Sample Delivery Group of the Case as follows:

1. Case Narrative
2. By fraction (VOA, BNA, PEST, INORG, CONV) and by sample within each fraction - tabulated target compound results (Form I-ORG or Form I-IN) and tentatively identified compounds (Form I-ORG, TIC) (VOA and BNA only)
3. By fraction (VOA, BNA, and PEST) - surrogate spike analysis results (Form II-ORG) by matrix (water and/or soil) and for soil, by concentration (low or medium)
4. By fraction (VOA, BNA, and PEST) - matrix spike/matrix spike duplicate/matrix spike blank results (Form III-ORG) - as required by method.
5. By fraction (VOA, BNA, and PEST) - QC Check Sample/Standard Recovery Summary - If required by method.
6. By fraction (INORG and CONV only) - duplicate sample results (Form VI-IN)
7. By fraction (INORG and CONV only) - spike sample results (Form V-IN)
8. By fraction (VOA, BNA, PEST, INORG, CONV) - blank data (Form IV-ORG and Form III-IN) and tabulated results (Form I-ORG and Form I-IN) including tentatively identified compounds (Form I-ORG, TIC) (VOA and BNA only).
9. By fraction (VOA and BNA only) - internal standard area data (Form VIII-ORG).

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C. - SAMPLE DATA PACKAGE

The Sample Data Package is divided into the eight major units described below. The last six units are each specific to an analytical fraction (volatiles, semivolatiles, pesticides/PCBs, GC organics, inorganics, and conventional wet-chemistry). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable.

The Sample Data Package shall include data for analyses of all samples in one Sample Delivery Group, including field samples, reanalyses, blanks, duplicates, spikes, matrix spikes, and matrix spike duplicates.

All data produced in support of Superfund investigations/remediations as identified by checked boxes under the Contract Laboratory Section of the Contract Laboratory Sample Information Sheet (See Exhibit A) shall be reported as specified for the Superfund Category (paragraph 1.). All data generated in support of the SPDES program as identified by a CASE # beginning with the letter "E" shall be reported using ASP Category-B (paragraph 3.). All other samples shall be reported using either ASP Category A or ASP Category B described in paragraphs 2. and 3. The specific reporting level to be used shall be specified by the Contract Laboratory Sample Information Sheet.

1. SUPERFUND CATEGORY

1.1 Case Narrative

This document shall be clearly labeled "Case Narrative" and shall contain: Laboratory name; Case number; Sample Delivery Group number (SDG); sample numbers in the SDG, differentiating between initial analyses and re-analyses; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.

Whenever data from sample re-analysis are submitted, the Laboratory shall state in the Case Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

The Laboratory must also include documentation of any internal quality control processes used, a summary of corrective actions taken, and the resolution.

The Case Narrative shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions of the contract."

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this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or his designee, as verified by the following signature." This statement shall be directly followed by signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

Additionally, the Case Narrative itself must be signed in original signature by the Laboratory Manager or his designee and dated.

1.2 Contract Lab Sample Information Sheets

A copy of the Contract Lab Sample Information Sheets (CLSIS) for all of the samples in the SDG. The CLSIS shall be arranged in increasing DEC sample number order, considering both letters and numbering in ordering samples.

1.3 Chain-of-Custody Forms

Copies of both the external and internal chain-of-custody sheets for all samples within the SDG.

1.4 Superfund-CLP Volatiles Data

1.4.1 QC Summary

1.4.1.1 Surrogate Percent Recovery Summary (Form II-CLP-VOA)

1.4.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form II-CLP-VOA)

1.4.1.3 Method Blank Summary (Form IV-CLP-VOA)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

1.4.1.4 GC/MS Tuning and Mass Calibration (Form V-CLP-VOA).

BFB in chronological order; by instrument.

1.4.1.5 Instrument Detection Limits

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1.4.2 Sample Data

Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I-CLP-VOA, including Form I-CLP-VOA-TIC), followed by the raw data for volatile samples. These sample packets should then be placed in increasing DEC sample number order, considering both letters and numbers in ordering samples.

1.4.2.1 TCL Results - Organic Analysis Data Sheet (Form I-CLP-VOA).

Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C). The validation and release of these results is authorized by a specific, signed statement in the Case Narrative (reference C.1). In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the Case Narrative.

On Form I-CLP-VOA, the appropriate concentration units shall be entered. For example, $\mu\text{g/L}$ for water samples or $\mu\text{g/Kg}$ for soil/sediment samples. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

1.4.2.2 Tentatively Identified Compounds (Form I-CLP-VOA-TIC).

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found".

Form I-CLP-VOA-TIC is the tabulated list of the highest probable match for up to 10 of the non-surrogate organic compounds not listed in Exhibit C (Superfund-TCL) for the appropriate program under which the samples were submitted, including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentrations. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the

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peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. **NOTE:** The laboratory must be consistent, i.e., use peak height for all comparisons or use total area count for all comparisons.

1.4.2.3 Reconstructed Total Ion Chromatograms (RIC) for each sample or sample extract.

RICs must be normalized to the largest non-solvent component and contain the following header information:

- o NYSDEC sample number
- o Date and time of analysis
- o GC/MS instrument ID
- o Lab file ID

Internal standard and surrogate spiking compounds are to be labeled and identified with their chemical names, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak. If automated system procedures are used for preliminary identification and/or quantification of the Superfund Target Compound List (Superfund-TCL) compounds, the complete data system report must be included in all sample data packages in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet", which contains the following information, must be included in the sample data package in addition to the chromatogram.

- o NYSDEC sample number
- o Date and time of analysis
- o RT or scan number of identified Superfund-TCL compounds

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- o Ion used for quantitation with measured area
- o Copy of area table from data system
- o GC/MS instrument ID
- o Lab file ID

1.4.2.4 For each sample, by each compound identified:

1.4.2.4.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Superfund-TCL) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Spectra must be labeled with NYSDEC sample number, lab file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

1.4.2.4.2 Copies of mass spectra of nonsurrogate organic compounds not listed in Exhibit C (Superfund-TCL) (Tentatively Identified Compounds) with associated best-match spectra (three best matches), labeled as in 1.4.2.4.1 above.

1.4.3 Standards Data

1.4.3.1 Initial Calibration Data (Form IV-CLP-VOA) - in order by instrument if more than one instrument used.

1.4.3.1.1 VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five point) calibration, labeled as in 1.4.2.3 above. Spectra are not required.

1.4.3.1.2 All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

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1.4.3.2 Continuing Calibration (Form VII-CLP-VOA) - in order by instrument, if more than one instrument used.

1.4.3.2.1 VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in 1.4.2.3. Spectra are not required.

1.4.3.2.2 When more than one continuing calibration is performed, forms must be in chronological order, within fraction and instrument.

1.4.3.3 Internal Standard Area Summary (Form VIII-CLP-VOA) - in order by instrument, if more than one instrument used.

When more than one continuing calibration is performed, forms must be in chronological order, by instrument.

1.4.4 Raw QC Data

1.4.4.1 BFB (for each 12-hour period, for each GC/MS system utilized)

1.4.4.1.1 Bar graph spectrum, labeled as in 1.4.2.3.

1.4.4.1.2 Mass listing, labeled as in 1.4.2.3.

1.4.4.2 Blank Data - in chronological order. NOTE: This order is different from that used for samples.

1.4.4.2.1 Tabulated results (Form I-CLP-VOA)

1.4.4.2.2 Tentatively Identified Compounds (Form I-CLP-VOA-TIC) - even if none found.

1.4.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.3.

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1.4.4.2.4 Superfund-TCL spectra with lab generated standard, labeled as in 1.4.2.4. Data systems which are incapable of dual display shall provide spectra in order:

- o Raw Superfund-TCL compound spectra
- o Enhanced or background subtracted spectra
- o Laboratory generated Superfund-TCL standard spectra

1.4.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 1.4.2.4.

1.4.4.2.6 Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations

1.4.4.3 Matrix Spike Blank Data

1.4.4.3.1 Tabulated results (Form I-CLP-VOA) of all Superfund-TCL compounds. Form I-CLP-VOA-TIC is not required.

1.4.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.4. Spectra are not required.

1.4.4.4 Matrix Spike Data

1.4.4.4.1 Tabulated results (Form I-CLP-VOA) of all Superfund-TCL compounds. Form I-CLP-VOA-TIC is not required.

1.4.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.4. Spectra are not required.

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ferences on the reconstructed ion chromatogram.

NOTE: The laboratory must be consistent (i.e., use peak height for all comparisons or use total area count for all comparisons).

1.5.2.3 Reconstructed total ion chromatograms (RIC) for each sample, sample extract, standard, blank, and spiked sample.

RICs must be normalized to the largest nonsolvent component, and must contain the following header information:

- o DEC sample number
- o Date and time of analysis
- o GC/MS instrument ID
- o Lab file ID

Internal standard and surrogate spiking compounds are to be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak. If automated data system procedures are used for preliminary identification and/or quantification of the Superfund Target Compound List (Superfund-TCL) compounds, the complete data system report must be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet," containing the following information, must be included in the sample data package in addition to the chromatogram.

- o DEC sample number
- o Date and time of analysis
- o RT or scan number of identified Superfund-TCL compounds

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1.5.2 Sample Data

Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I-CLP-SV, including Form I SV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing DEC sample number order, considering both letters and numbers in ordering samples.

1.5.2.1 TCL Results - Organic Analysis Data Sheet (Form I-CLP-SV-1, SV-2).

Tabulated results (identification and quantitation) of the specified Superfund-CLP target compounds (Exhibit C). The validation and release of these results is authorized by a specific, signed statement in the Case Narrative (reference C.1). In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the Case Narrative.

On Form I-CLP-SV, the appropriate concentration units shall be entered. For example, $\mu\text{g/L}$ for water samples or $\mu\text{g/Kg}$ for soil/sediment samples. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

1.5.2.2 Tentatively Identified Compounds (Form I-CLP-SV-TIC)

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found".

Form I-CLP-SV-TIC is the tabulated list of the highest probable match for up to 20 of the nonsurrogate organic compounds not listed in Exhibit C (Superfund-TCL), including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of in-

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1.4.4.5. Matrix Spike Duplicate Data

1.4.4.5.1 Tabulated results (Form I-CLP-VOA) of all Superfund-TCL Compounds. Form I-CLP-VOA-TIC is not required.

1.3.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 3.3.2.4. Spectra are not required.

1.4.4.6 Copy of Calculations

The Laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

1.5 Superfund-CLP Semivolatiles Data

1.5.1 QC summary

1.5.1.1 Surrogate Percent Recovery Summary (Form II-CLP-SV)

1.5.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III-CLP-SV)

1.5.1.3 Method Blank Summary (Form IV-CLP-SV)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

1.5.1.4 GC/MS Tuning and Mass Calibration (Form V-CLP-SV)

DFTPP in chronological order; by instrument.

1.5.1.5 Instrument Detection Limits

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- o Ion used for quantitation with measured area
- o Copy of area table from data system
- o GC/MS instrument ID
- o Lab file ID

1.5.2.4 For each sample, by each compound identified:

1.5.2.4.1 Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Superfund-TCL) that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Spectra must be labeled with DEC sample number, lab file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

1.5.2.4.2 Copies of mass spectra of nonsurrogate organic compounds not listed in Exhibit C (Superfund-TCL) (Tentatively Identified Compounds) with associated best-match spectra (three best matches), labeled as in 3.3.

1.5.2.4.3 GPC chromatograms (if GPC performed).

1.5.3 Standards Data

1.5.3.1 Initial Calibration Data (Form VI-CLP-SV-1, SV-2) - in order by instrument, if more than one instrument used.

1.5.3.1.1 BNA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five point) calibration, labeled in 1.4.2.3. Spectra are not required.

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1.5.3.1.2 All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

1.5.3.2 Continuing calibration (Form VII-CLP-SV-1, SV-2) - in order by instrument, if more than one instrument used.

1.5.3.2.1 BNA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in 1.4.2.3. Spectra are not required.

1.5.3.2.2 When more than one continuing calibration is performed, forms must be in chronological order, by instrument.

1.5.3.3 Internal Standard Area Summary (Form VIII-CLP-SV-1, SV-2) in order by instrument, if more than one instrument used.

When more than one continuing calibration is performed, forms must be in chronological order by instrument.

1.5.4 Raw QC Data

1.5.4.1 DFTPP (for each 12-hour period, for each GC/MS system utilized)

1.5.4.1.1 Bar graph spectrum

1.5.4.1.2 Mass listing

1.5.4.2 Blank Data - in chronological order. NOTE: This order is different from that used for samples.

1.5.4.2.1 Tabulated results (Form I-CLP-SV-1, SV-2)

1.5.4.2.2 Tentatively Identified Compounds (Form I-CLP-SV-TIC) - even if none found.

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- 1.5.4.2.3 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.3.
- 1.5.4.2.4 Superfund-TCL spectra with lab generated standard, labeled as in 1.4.2.4. Data systems which are incapable of dual display shall provide spectra in order:
 - o Raw Superfund-TCL compound spectra
 - o Enhanced or background subtracted spectra
 - o Laboratory generated Superfund-TCL standard spectra
- 1.5.4.2.5 GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 1.5.2.4.
- 1.5.4.2.6 Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations
- 1.5.4.3 Matrix Spike Blank Data
 - 1.5.4.3.1 Tabulated results (Form I-CLP-SV-1, SV-2) of all Superfund-TCL compounds. Form I-CLP-SV-TIC not required.
 - 1.5.4.3.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.3. Spectra are not required.
- 1.5.4.4 Matrix Spike Data
 - 1.5.4.4.1 Tabulated results (Form I-CLP-SV-1, SV-2) of all Superfund-TCL compounds. Form I-CLP-SV-TIC not required.

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1.5.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.3. Spectra are not required.

1.5.4.5 Matrix Spike Duplicate Data

1.5.4.4.1 Tabulated results (Form I-CLP-SV-1, SV-2) of all Superfund-TCL compounds. Form I-CLP-SV-TIC not required.

1.5.4.4.2 Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 1.4.2.3. Spectra are not required.

1.6 Superfund-CLP Pesticide/PCB Data

1.6.1 QC Summary

1.6.1.1 Surrogate Percent Recovery Summary (Form II-CLP-PEST)

1.6.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III-CLP-PEST)

1.6.1.3 Method Blank Summary (Form IV-CLP-PEST)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

1.6.2 Sample Data

Sample data shall be arranged in packets with the Traffic Report copy, the Organic Analysis Data Sheet (Form I-CLP-PEST), followed by the raw data for pesticide samples. The sample packets should then be placed in increasing DEC sample number order, considering both letters and numbers in ordering samples.

1.6.2.1 Superfund-TCL Results - Organic Analysis Data Sheet (Form I-CLP-PEST).

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Tabulated results (identification and quantitation) of the specified Superfund target compounds (Exhibit C). The validation and release of these results is authorized by a specific, signed statement in the Case Narrative (reference E.1). In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the Case Narrative.

On Form I-CLP-PEST, the appropriate concentration units shall be entered. For example, $\mu\text{g/L}$ for water samples or $\mu\text{g/Kg}$ for soil/sediment samples. No other units are acceptable.

NOTE: Report analytical results to two significant figures for all pesticide/PCB samples.

1.6.2.2 Copies of pesticide chromatograms.

All chromatograms must be labeled with the following information.

- o DEC sample number
- o Volume injected (μl)
- o Date and time of injection
- o GC column identification (by stationary phase)
- o GC instrument identification
- o Positively identified compounds must be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

1.6.2.3 Copies of pesticide chromatograms from second GC column confirmation. Chromatograms to be labeled as in 1.6.2.2 above.

1.6.2.4 GC integration report or data system printout and calibration plots (area vs. concentration) for 4-4'-DDT, 4,4'-DDD, 4,4'-DDE or toxaphene (where appropriate).

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1.6.2.5 Manual work sheets.

1.6.2.6 UV traces from GPC (if GPC performed).

1.6.2.7 If pesticide/PCBs are confirmed by GC/MS, the Laboratory shall submit copies of raw spectra and copies of background-subtracted mass spectra of Superfund target compounds listed in Exhibit C (Superfund-TCL) that are identified in the sample and corresponding background-subtracted Superfund-TCL standard mass spectra. Compound names must be clearly marked on all spectra. For multicomponent pesticides/PCBs confirmed by GC/MS, the Laboratory shall submit mass spectra of 3 major peaks of multicomponent compounds from samples and standards.

1.6.3 Standards Data

1.6.3.1 Form VII-CLP-PEST - Pesticide Evaluation Standards Summary (all GC columns)

1.6.3.2 Form IX-CLP-PEST - Pesticide/PCB Standards Summary (all GC columns)

1.6.3.3 Form X-CLP-PEST - Pesticide/PCB Identification (only required for positive results)

1.6.3.4 Pesticide standard chromatograms and data system printouts for all standards to include:

- o Evaluation Standard Mix A
- o Evaluation Standard Mix B
- o Evaluation Standard Mix C
- o Individual Standard Mix A
- o Individual Standard Mix B
- o All multiresponse pesticides/PCBs
- o All quantitation standards

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- o A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

1.6.3.4.1 All chromatograms are required to have the following:

- o Label all chromatograms with the "NYSDEC Sample Number" for standards, i.e., EVALA, EVALB, etc. (See Forms Instructions for details).
- o Label all standard peaks for all individual compounds either directly out from the peak or on the printout of retention times if retention times are printed over the peak.
- o List total ng injected for each standard.
- o A printout of retention times and corresponding peak areas must accompany each chromatogram.
- o Date and time of injection.
- o GC column identification (by stationary phase).
- o GC instrument identification.

1.6.4 Raw QC Data

1.6.4.1 Blank Data - in chronological order. NOTE: This order is different from that used for samples.

1.6.4.1.1 Tabulated results (Form I-CLP-PEST).

1.6.4.1.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis.

1.6.4.2 Matrix Spike Data

1.6.4.2.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

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1.6.4.2.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.6.4.3 Matrix Spike Duplicate Data

1.6.4.3.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

1.6.4.3.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.6.4.4 Matrix Spike Blank Data

1.6.4.4.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

1.6.4.4.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.7 GC Organic Data

1.7.1 QC Summary

1.7.1.1 Surrogate Percent Recovery Summary (Form II-GC)

1.7.1.2 Matrix Spike/Matrix Spike Duplicate/Matrix Spike Blank Summary (Form III-GC)

1.7.1.3 Method Blank Summary (Form IV-GC)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

1.7.1.4 Instrument Detection Limits

1.7.2 Sample Data

Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I GC), followed by the raw data for pesticide samples. These sample packets should then be placed in increasing DEC sample number order, considering both letters and numbers in ordering samples.

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1.7.4 Raw QC Data

1.7.4.1 Blank Data - in chronological order. NOTE: This order is different from that used for samples.

1.7.4.1.1 Tabulated results (Form I-CLP-PEST).

1.7.4.1.2 Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis.

1.7.4.2 Matrix Spike Data

1.7.4.2.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

1.7.4.2.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.7.4.3 Matrix Spike Duplicate Data

1.7.4.3.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

1.7.4.3.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.7.4.4 Matrix Spike Blank Data

1.7.4.4.1 Tabulated results (Form I-CLP-PEST) of all Superfund-TCL compounds.

1.7.4.4.2 Chromatogram(s) and data system printout(s) (GC), labeled as in 1.4.2.3.

1.8 Inorganic Data

Sample data shall be submitted with the Inorganic Analysis Data Reporting Forms for all samples in the SDG, arranged in increasing alphanumeric DEC sample number order, followed by the QC analyses data, Quarterly Verification of Instrument Parameters forms, raw data, and copies of the digestion and distillation logs.

1.8.1 Results – Inorganic Analysis Data Sheet (FORM I-CLP-IN)

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Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C). The validation and release of these results is authorized by a specific, signed statement on the Cover Page. If the Laboratory Manager cannot validate all data reported for each sample, he/she must provide a detailed description of the problems associated with the sample(s) on the Cover Page.

Appropriate concentration units must be specified and entered on Form I-CLP-IN. The quantitative values shall be reported in units of micrograms per liter ($\mu\text{g/L}$) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. No other units are acceptable. Results for solid sample must be reported on a dry weight basis. Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10. Results for percent solids must be reported to one decimal place.

1.8.2 Quality Control Data

1.8.2.1 Initial and Continuing Calibration Verification [FORM II-CLP-IN (Part 1)]

1.8.2.2 CRDL Standard for AA and Linear Range Analysis for ICP [FORM II-CLP-IN (PART 2)]

1.8.2.3 Blanks [FORM III-CLP-IN]

1.8.2.4 ICP Interference Check Sample [FORM IV-CLP-IN]

1.8.2.5 Spike Sample Recovery [FORM V-CLP-IN (PART 1)]

1.8.2.6 Post Digest Spike Sample Recovery [FORM V-CLP-IN (PART 2)]

1.8.2.7 Duplicates [FORM VI-CLP-IN]

1.8.2.8 Laboratory Control Sample [FORM VII-CLP-IN]

1.8.2.9 Standard Addition Results [FORM VIII-CLP-IN]

1.8.2.10 ICP Serial Dilutions [FORM IX-CLP-IN]

1.8.2.11 Holding Times [FORM X-CLP-IN]

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1.8.3 Verification of Instrument Parameters

1.8.3.1 Instrument Detection Limits (Quarterly) [FORM XI-CLP-IN]

1.8.3.2 ICP Interelement Correction Factors (Annually) [FORM XII-CLP-IN (PART 1)]

1.8.3.3 ICP Interelement Correction Factors (Annually) [FORM XII-CLP-IN (PART 2)]

1.8.3.4 ICP Linear Ranges (Quarterly) [FORM XIII-CLP-IN]

(Note that copies of Verification of Instrument Parameters forms for the current quarter must be submitted with each data package.)

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1.8.4 Raw Data

For each reported value, the Laboratory shall include in the data package all raw data from the instrument used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as sample results. This statement does not apply to the Verifications of Instrument Parameters submitted as part of each data package. Raw data must contain all instrument readouts used for the sample results, including those readouts that may fall below the IDL. All AA and ICP instruments must provide a legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the direct sequential instrument readout must be included. A hardcopy of the instrument's direct instrument readout for cyanide must be included if the instrumentation has the capability.

The order of raw data in the data package shall be: ICP, Flame AA, Furnace AA, Mercury, and Cyanide. All raw data shall include intensities (ICP) and absorbances with concentration units for flame AA, furnace AA, Mercury and Cyanide. All flame and furnace AA data shall be grouped by element.

Raw data must be labeled with DEC sample number and appropriate codes, shown in Table 1 following to unequivocally identify:

- 1.8.4.1 Calibration standards, including source and prep date.
- 1.8.4.2 Initial and continuing calibration blanks and preparation blanks.
- 1.8.4.3 Initial and continuing calibration verification standards, interference check samples, ICP serial dilution samples, CRDL Standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike.
- 1.8.4.4 Diluted and undiluted samples (by DEC sample number) and all weights, dilutions and volumes used to obtain the reported values. (If the volumes, weights and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient).
- 1.8.4.5 Duplicates.

III. Instructions for Report Format and Deliverables Requirements

All final data reports shall contain the following information in the following order:

A. Title Page

The NJDEP case name and/or number, field sample numbers, laboratory sample numbers, sample location, date and time of sample collection, date of data report, name and signature of the Laboratory Quality Assurance Officer and name and signature of the laboratory manager must be listed on this page. The laboratory shall use NJDEP Form A-1 or equivalent.

B. Table of Contents

This table must list with a page reference all topic headings in Section II "Order of Data Deliverables" of this attachment marked with an asterisk (*).

C. Sample Analysis Request Form

Form DEP-059 will be completed by NJDEP and submitted with the samples to be analyzed. The laboratory must immediately notify NJDEP if these forms are not received or if the forms contain errors.

D. Chain of Custody Document(s)

Field and internal laboratory chain of custody documents must be provided. Form DEP-060 (With Shuttle System) and/or DEP-009 (Without Shuttle System) shall be completed by NJDEP field personnel for all samples collected and submitted to the laboratory and shall serve as field chain of custody documentation. The laboratory shall properly complete these forms. The laboratory must notify NJDEP if these forms are not received or if the forms contain errors. In addition, a Receipt for Property form (DEP-011) is submitted with the samples a copy of that form shall also be included in the data report.

The laboratory shall use DEP Form 077 for internal laboratory chain of custody. The internal chain of custody is initiated when the laboratory receives a sample(s) from NJDEP. Each sample submitted will consist of one or more sample aliquots (i.e., portions of a sample collected in separate containers). Distinct sample aliquots are used in the analysis of a specific analytical fraction (e.g., sample aliquots stored in 40 ml vials with teflon lined septum screw caps are analyzed for volatiles). Internal chain of custody must be documented for each sample aliquot representing a specific analytical fraction (e.g., volatiles, semi-volatiles, metals, etc.). For a given case (group) of samples submitted to the laboratory, internal chain of custody documentation for all sample aliquots for a given analytical fraction may be listed on one (1) form.

The chain of custody must clearly trace the movement of the sample aliquot(s) through the laboratory by showing the time and date of the relinquishing and accepting of the sample aliquot(s) by each

H2M LABS, INC.

- 1.8.4.6 Spikes (indicating standard solutions used, final spike concentrations, volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient.
- 1.8.4.7 Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation.
- 1.8.4.8 All information for furnace analysis clearly and sequentially identified on the raw data, including DEC sample number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, full MSA data, MSA correlation coefficient, slope and intercepts of linear fit, final sample concentration (standard addition concentration), and type of background correction used: BS for Smith-Heitje, BD for deuterium Arc, or BZ for Zeeman.
- 1.8.4.9 Time and date of each analysis. Instrument run logs can be submitted if they contain this information. If the instrument does not automatically provide times of analysis, these must be manually entered on all raw data for initial and continuing calibration verification and blanks, as well as interference check samples and linear range analysis.

1.8.4.10 Integration times for AA analyses.

1.8.5 Digestion Logs

Logs shall be submitted in the following order: digestion logs for ICP, flame AA, furnace AA and mercury preparations, followed by a copy of the distillation log for cyanide. These logs must include: (1) date, (2) sample weights and volumes, (3) sufficient information to unequivocally identify which QC samples (i.e. laboratory control sample, preparation blank) correspond to each batch digested, (4) comments describing any significant sample changes or reactions which occur during preparation, and (5) indication of pH <2 or >12, as applicable.

10. Document Control

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10. DOCUMENT CONTROL

10.1 LABORATORY NOTEBOOK POLICY

The analyst log books are bound books with all pages numbered consecutively. Analysts are required to sign (in full signature) and date next to all analyses performed. For GC and GC/MS the instrument program is to be listed as well as sample I.D., amount injected and reason if any for re-analysis (under comments). For wet chemistry tests all raw data used in calculations is to be recorded in the log book. For sample preparation all weights to the closest 0.1 gram and/or exact volume of milliliter of sample extracted is to be listed as well as type of cleanup performed and date extracted. Examples of log book pages are included in figure 1 and figure 2.

10.2 SAMPLE TRACKING AND CUSTODY PROCEDURES

Upon delivery to the H2M sample custodian, the samples are logged into the system as described in the Sample Custody Section. The sample custodian gives a copy of the external COC, and internal COC for each department as well as any information received with the sample to the document control section of the QA/QC department. All paperwork is reviewed and checked for any transcription errors (example: comparing the sample request to the external COC; the computer printout as compared to the internal COC). If there is any transcription errors the sample custodian is contacted as well as any affected departments. Verification that corrections were made properly is also the responsibility of the document control section.

The samples are then entered into a sample tracking logbook and an SDG folder is prepared including all pertinent information (such as the sample request form and external COC). The folder is labeled with the SDG #, date received, date due, number and type of samples and types of analysis.

All external and internal COC is labeled with a group number. Usually an abbreviation of Client and the number of packages alphanumerically e.g. H2M001. The sample tracking book contains the following information: header information, client test I.D.'s, lab #'s and samples.

On a daily basis holding times are monitored as a check on the different departments and the supervisors are notified if holding times are drawing near. (At least 2 days in advance). The COC procedure is audited weekly. Weekly the

H2M LABS, INC.

document control section meets with the supervisors to update the status of the project. A status report is generated every week and distributed to all managerial and supervisory personnel.

Figure 3 is an example of a status report. Upon completion of a project, the QA/QC section co-ordinates collation of the data package and reviews that all required forms are included and that the package is mailed within the required time frame.

10.3 LOGBOOK MAINTENANCE AND ARCHIVING PROCEDURES

Recently a new approach to archiving has been implemented in the laboratory. Upon completion of a logbook, the book and associated raw data (e.g. chromatograms), are archived by the QA/QC department.

The binder of the book is labeled with the test, start and completion data and run #. This data is stored in book cases in the QA/QC department for 1 year and then placed in labeled boxes and put in storage. The boxes are labeled with pertinent information as to the test and date and this information is listed in a logbook.

10.4 CASE FILE ORGANIZATION, PREPARATION, AND REVIEW PROCEDURES

The document control coordinator of the QA/QC department organizes the case file by monitoring and accumulating information from receipt to mailing. The data package is collated in this department. Each department receives the necessary information in the internal COC to analyze the samples in a data package. Each department sends out a complete package. (software is available and the reports are computer generated). The data package (each individual fraction: (VOA, BNA, PEST, METALS) is reviewed by an individual determined by the QA/QC department depending upon workload. The reviewer is a technically qualified individual such as the QA Manager, Technical Manager or Department Supervisor.

The information listed in the forms in Attachment 1 and 2 is checked in the review procedure. A case narrative is prepared by the reviewer and if necessary the package will be sent to the department for correction.

Each fraction of the data package is now compiled in the QA/QC department with Summary forms, and external COC

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included. The summary package is pulled and the packages are copied and bound. A copy of the data package is maintained on file in the QA/QC department.

10.5 PREPARATION, APPROVAL, REVIEW, REVISION AND DISTRIBUTION OF SOP'S AND TECHNICAL PROCEDURES MANUALS

The laboratory SOP is prepared by the Quality Assurance Manager and the Technical Manager. The methods and/or procedures are written and then reviewed by the department for correctness. Revisions are monitored by a revision date on the title page of the manual.

Final review and approval is completed by the Lab Director and QA Manager. A limited number of copies are made at this time. The distribution of the manual is controlled by document numbers of each copy of the manual and if a revision is released, a recall of the outdated manual is issued.

H2M LABS, INC.

CLP/COC
SAMPLE PREPARATION LOG

LAB #
EXTRACTION DATE
CLIENT
CLIENT ID #
SCANS
INITIAL pH
ANALYST
METHOD
SURROGATE/ SPIKE VOL.
MATRIX SPIKE/ SPIKE VOL.
INITIAL SAMPLE VOLUME
CONCENTRATED/ DATE/INITIALS
ALUMINA CLEAN-UP REQUIRED (Y/N)
G.P.C./ REQUIRED (Y/N)
FINAL VOLUME OF:
BN
AE
PP
REMARKS

[illegible]

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H2M LABS, INC.

TO: JJM, SI, SWM, VIS, URM, JED, SB, MAW, HJB, GKB, SS, AMM, NTH
FROM: LTD, JED

Revised: MARCH 25, 1991

SUMMARY OF CLP SAMPLE STATUS

SITE	SDG#	LAB NUMBER	DATE REC'D	ANALYSES DUE BY +	MAIL DATE	SAMPLE TYPE	X.PU	X.BN X.AE	X.PP	TCL METAL	WET CHEM	
	B	NSI001	9104200-04, 4216	2/12/91	3/14/91	3/24/91	SLUDGE	PR	PR	PR	A	A, N
	B	NSI002	9105950-54, 5962	2/27/91	3/25/91	4/8/91	SLUDGE	PR	PR	A	A	A, N
	C	EDG021	9107323-29	3/12/91	3/27/91	4/14/91	WATER	R	A	-	RD	C
SMITH.	C	GIB081	9107439-7443	3/13/91	3/28/91	4/12/91	GW-FILT.	-	-	-	X	-
CONST.	C	GIB082	9107444-47	3/13/91	3/28/91	4/12/91	S-EPTOX	-	-	-	X	-
	C	EDG022	9107461-75	3/13/91	3/28/91	4/14/91	WATER	R	A	-	A	A, N
	C	EDG023	9107606-21	3/14/91	3/29/91	4/14/91	WATER	R	A	-	A	A, N
	C	EDG024	9107679-82	3/14/91	3/29/91	4/14/91	WATER	A	A	-	A	A, N
	C	CRA003	9108314-8320	3/22/91	4/4/91	4/20/91	SOIL, R.B.	X	X	X	X	A, N

SUMMARY OF NTPDES (J) & NJDEP (D) SAMPLE STATUS

J/D	SDG#	LAB NUMBER	DATE REC'D	ANALYSES DUE BY +	MAIL DATE	SAMPLE TYPE	X.PU	X.BN X.AE	PEST	METAL	WET CHEM
B	GAH002	9104439, 4440	2/19/91	3/15/91	3/21/91	SLUDGE	PR	PR	A	A	A, N
B	GAH003	9108026-8027	3/19/91	4/3/91	4/18/91	SLUDGE	R	A	A	X	A, N

KEY

X = Samples in house
A = Extracted/digested - analysis required
R = Analysis complete - reporting in progress
PR = Preliminary review before typing required
T = Typing in progress or needed
FR = Final review after typing required
TC = typing corrections needed
C = Completed - needs copying and binding
B = Bound and ready to go
* = Partial redigestion &/or re-analysis required
+ = This is the date analyses should be completed and sent to preliminary review

CL = Clean-up of sample in progress
R-CL = Re-extracted and clean-up in progress
R-O = Redigestion in progress
R-E = Re-extraction in progress
R-A = Re-analyses of samples in progress
Under SITE - "I" = incomplete site
Under SITE - "C" = complete site
Under SITE - "I" = incomplete site, informational
N = Analysis complete, NEED data handed in
Xp = Chlorinated Hydrocarbons to be run in C

DATA VALIDATIONS (V) & RESPONSES (R) IN HOUSE

V/R	CLIENT	REC'D	VOA	PEST/PCB	BNA	METALS
R	CIB041, 60	2/12/91	B	B	B	B
R	CIB052	2/19/91	T	URM	T	T
R	CIB050, S1, S5	2/26/91	JMS	URM	JMS	JMS
R	CIB069	3/22/91	JMS	URM	JMS	JMS

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 1 of 9

VOA
BNA
PEST

Site: _____

Project: _____

Summaries

YES NO N/A

1. Are external COC records submitted? _____
2. Are internal COC records submitted? _____
3. Do internal COC records document continuity? _____
4. Does record of samples analyzed match internal and external chain of custody? _____
5. Is sample preparation summary and copy of sample preparation log submitted? _____
6. Is narrative included? _____
7. Are holding times for sample preparation met of 7 days for volatiles and 5 days for extractables? _____
8. Are 40 day limits met for sample analysis for the extractables? _____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

QA/QC PACKAGE

I. Surrogate Percent Recovery

1. Are the forms present for water?
soil? _____
2. Are all the samples listed for each matrix? _____
3. Are the outliers correctly marked with an asterisk? _____
4. Are the calculations correct, based on extract volume, GPC, dilution and final volume? _____
5. Do surrogate recoveries meet CLP protocol for all samples? _____
6. Are both reports submitted for samples that do not meet recoveries for both extracts? _____
7. Is recovery for DBC above 5%? _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 2 of 9

VOA
BNA
PEST

Site: _____

Project: _____

Surrogate Percent Recovery (con't)

Missing information, error or noncompliance for

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

II. Matrix Spike Duplicates and MSB Recovery

YES NO N/A

1. Is matrix spike summary submitted for a sample of this batch?	_____	_____	_____
2. Is data summary submitted for a sample from another batch?	_____	_____	_____
3. Are frequency requirements met for spike analysis?	_____	_____	_____
4. Are the outliers correctly marked with an asterisk?	_____	_____	_____
5. Are the number of outliers for %REC and %RPD listed correctly at the bottom of the page?	_____	_____	_____
6. Are data calculated and transcribed correctly, based on GPC, dilution, and final volume?	_____	_____	_____
7. Does data meet CLP protocol for VOA and BNA?	_____	_____	_____
8. Is a spiked blank analyzed and summary submitted?	_____	_____	_____
9. Do MSB recoveries meet limits of 75-125%?	_____	_____	_____

Missing information, error or noncompliance for

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 4 of 9

VOA
BNA
PEST

Site: _____

Project: _____

IV. GC/MS Tuning and Mass Calibration

YES NO N/A

1. Are all the tuning and mass calibration forms present? _____
2. Has a tuning performance compound been run for every 12 hours of sample analysis? _____
3. Are bar graph spectra and m/z listings submitted for BFB/DFTPP? _____
4. Are data transcribed correctly from mass lists to the GC/MS tuning form? _____
5. Are ratios for abundances calculated correctly? _____
6. Have the ion abundance criteria been met for all mass calibrations? _____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

SAMPLE REPORTS

1. Are TCL and TIC (if applicable) forms present for all samples? _____
2. Are header information and units correct? _____
3. Are positives calculated and transcribed correctly from raw data to reporting forms? _____
4. Are correct qualifiers used? _____
5. Are the reported limits calculated correctly for dry weights, GPC and dilution? _____
6. If a sample exceeded linearity for a parameter, was it reanalyzed after dilution? _____
7. Do results for sample duplicates match? _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 5 of 9

VOA
BNA
PEST

Site: _____

Project: _____

SAMPLE REPORTS(con't)

YES NO N/

8.	Are all chromatograms, quant lists, mass spectra and computer identifications for TIC's submitted?	_____	_____	_____
9.	Are positives and surrogate and internal standards marked on chromatograms?	_____	_____	_____
10.	Do positives meet criteria for mass-abundances, compared with standard?	_____	_____	_____
11.	Are extraneous ions present greater than 10% of base peak?	_____	_____	_____
12.	If GPC was performed, are the GPC calibration check recoveries submitted, including chromatograms for analysis and calibration standard?	_____	_____	_____

Pest/PCB's Only

13. Are the PCB pattern recognizable? Could minimum areas be detected? _____
14. If positive PCB's are found, are reporting limits raised for interfering pesticides? _____

Missing information, error or noncompliance for

Item # _____ on page _____
see CLP paragraph _____

Item # _____ on page _____
see CLP paragraph _____

Item # _____ on page _____
see CLP paragraph _____

Item # ____: _____ on Page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # _____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 6 of 9

VOA
BNA
PEST

Site: _____

Project: _____

STANDARD PACKAGE FOR VOA & BNA

YES NO N/A

Initial Calibration

1. Was a 5-point initial calibration performed per matrix and level and the form submitted? _____
2. Do the initial calibrations meet criteria for CCC's? (25% RSD for VOA and 30% for BNA) _____
3. Do the initial calibrations meet criteria for SPCC's? _____
4. Are all average relative response factors at or above 0.05? _____
5. Are all other RSD's under or equal 35% for VOA and 30% for BNA? _____
6. Did levels used correspond to CLP requirements? _____

Continuous Calibration

7. Was a continuing calibration performed per matrix per level? _____
8. Do all CCC's meet the requirements for continuing calibration (25% D for VOA, 20% for BNA) _____
9. Do all SPCC's meet the requirements for continuing calibration? _____
10. Are all TCL response factors at or above 0.05? _____
11. Is the %D less than 35% for all VOA compounds, and less than 20% for BNA compounds? _____

Internal Standard Area Summary

1. Is summary submitted? _____
2. Do areas comply with requirements? _____
3. Is retention time shift of internal standard less than 30 seconds? _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 7 of 9

VOA
BNA
PEST

Site: _____

Project: _____

STANDARD PACKAGE FOR VOA & BNA (con't)

Missing information, error or noncompliance for

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

Item # ____: _____ on page _____
see CLP paragraph _____

YES NO N/A

STANDARD PACKAGE FOR PESTICIDES/PCB'S

Form VIII-1

1. Was sequence completed within 72 hours?	_____	_____	_____
2. Does order of runs comply with sequence requirements?	_____	_____	_____
3. Are all headers filled out completely and correctly?	_____	_____	_____
4. Are all EVALB runs listed?	_____	_____	_____
5. Is breakdown under 20%?	_____	_____	_____
6. Are breakdown peaks identified in the EVALB runs?	_____	_____	_____
7. Does linearity meet criteria of 10% D for primary column?	_____	_____	_____
8. Does linearity meet criteria of 10% D for secondary column?	_____	_____	_____

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 8 of 9

VOA
BNA
PEST

Site: _____

Project: _____

STANDARD PACKAGE FOR PESTICIDES/PCB'S (con't)

YES NO N/A

Form VIII-2

9. Is OBC shift under 2% for packed 0.3% for cap.
column and 1.5% for widebore cap. columns?

Form IX

10. Are all standards of sequence documented?

11. Was %D under 15% for quantification?

12. Was %D under 20% for confirmation?

Form X

13. Are all reported positives listed on Form X with
correct windows?

14. Do all peaks fall within windows?

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

H2M LABS, INC.

CHECKLIST FOR VALIDATION OF ORGANIC CLP PACKAGES

Page 9 of 9

VOA
BNA
PEST

Site: _____

Project: _____

RAW QC PACKAGE

YES NO N/A

1. Are all TCL and TIC (if applicable) report forms present for all blanks, holding blanks, matrix spikes, matrix spike duplicates, and MSB? _____
2. Are copies submitted for chromatograms and quant lists? _____
3. Are mass spectra and computer identification for TIC included for blanks? _____
4. Do concentrations in MS/MSD reports match concentrations in unspiked sample? _____
5. Are breakdown products for Endrin and DDT found and marked? _____

Missing information, error or noncompliance for

Item #____: _____ on page _____
see CLP paragraph _____

Item #____: _____ on page _____
see CLP paragraph _____

H2M LABS, INC.

CHECKLIST FOR REVIEW OF INORGANIC CLP PACKAGES

Page 1 of 6

Reviewer: _____ Date: _____ Package ID: _____

Cover Page

YES NO N/

Are all samples listed with correct corresponding lab number? (check against COC)

Is the SDG number properly identified?

(1) Reports

Are all form I's present and complete for all samples in the SDG?

Are results in correct units (ug/l for waters and mg/kg for soils)?

Are sample results for each element corrected for percent solids on soils?

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

Are all "less than IDL" values properly coded with "U"?

Are all values over the IDL and less than the CRDL properly coded with "G"?

Is physical description of samples before and after digestion listed?

(2A) Initial and Continuing Calibration Verification

Are all elements reported for all ICV's and CCV's?

Are the true values listed correctly?

Are all %R's within control limits?

Metals..... 90-110%

Hg & Sn..... 80-120%

Cu..... 85-115%

If no, stop and find out why.

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

Are the method's correctly entered for all elements, including those elements run on more than one instrument?

H2M LABS, INC.

CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 2 of 6

	YES	NO	N/
<u>(2B) CRDL Standard</u>			
Are all elements reported in AA or ICP column?	☐	☐	☐
Are all ICP elements reported for both CRII and CRIF?	☐	☐	☐
Are the true values entered correctly for all elements?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(3) Blanks</u>			
Are all elements reported for ICB's, CC3's?	☐	☐	☐
Is the proper matrix entered for the Prep Blank?	☐	☐	☐
Are there additional forms if there is more than one matrix?	☐	☐	☐
Is the data for the Soil Prep Blank entered in MG/KG?	☐	☐	☐
Are all blanks (ICB's, CC3's, PB's) less than the CRDL? (Flagged either U or B)	☐	☐	☐
Are the U and B flags used properly (check 10%)?	☐	☐	☐
Check 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(4) ICP Interference Check Sample</u>			
Is one form present for each ICP run?	☐	☐	☐
Are all Interference check sample results inside 80-120%?	☐	☐	☐
Was an Interference check sample analyzed at the beginning and end of each run?	☐	☐	☐
Are the true solution values entered correctly?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐

H2M LABS, INC.

CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 3 of 6

(5A) Spike Sample Recovery

YES NO N/A

Is there a form present and complete for all designated MS/MSD's?

☐ ☐ ☐

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

☐ ☐ ☐

Are all recoveries within control limits 75-125% for samples with concentrations 4 times the spike added?

☐ ☐ ☐

If no, are the elements properly flagged with "N" on this form, and the associated form I's?

☐ ☐ ☐

Are sample & spike analysis results, and spike added in correct units?

☐ ☐ ☐

(5B) Post Digest Spike Recovery

Is there a form present and complete for all designated MS/MSD's?

☐ ☐ ☐

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

☐ ☐ ☐

Are recoveries within 75-125% for those elements flagged on the associated form 5A (except Ag)?

☐ ☐ ☐

(6) Duplicate

Is there a form present and complete for all designated MS/MSD's?

☐ ☐ ☐

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.

☐ ☐ ☐

Is the RPD column left blank when both sample and duplicate are less than the instrument detection limit?

☐ ☐ ☐

Does the control limit column have the CRDL (in appropriate units) if the sample or duplicate results are less than 5 times the CRDL?

☐ ☐ ☐

H2M LABS, INC.

CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 4 of 6

	YES	NO	N/
<u>(6) Duplicate (con't)</u>			
If yes, is the absolute difference between sample and duplicate less than the CRDL?	☐	☐	☐
If no, is it flagged with "*" on this form and associated form I's?	☐	☐	☐
For those element which have a sample or duplicate result above the 5 times the CRDL, is the RPD less than 20%?	☐	☐	☐
<u>(7) Laboratory Control Sample</u>			
Is an LCS form present and complete for every group of samples of similar matrix and each group digested?	☐	☐	☐
Are all percent recoveries between 80-120% (except Ag & Sb)?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(8) Method of Standard Additions</u>			
If MSA's were required, were they properly entered on this form?	☐	☐	☐
If no MSA's were required, is there a blank form with the header properly filled out present?	☐	☐	☐
If the correlation coefficient was < 0.995, was the MSA re-run once?	☐	☐	☐
Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors? If yes, do a complete review.	☐	☐	☐
<u>(9) Serial Dilution</u>			
Was a serial dilution analysis performed for each 20 samples?	☐	☐	☐
each matrix type?	☐	☐	☐
each concentration range?	☐	☐	☐
If no for any of the above, is any sample concentration (undiluted) greater than 50 times the IDL?	☐	☐	☐

H2M LABS, INC.

CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 5 of 6

(9) Serial Dilution (con't)

YES NO N/

Check 10% for calculation errors, and 10% for transcription errors from raw data. Any errors?
If yes, do a complete review.

___ :___: ___

Was the field blank used for serial dilution?

___ :___: ___

Are all serial dilution results within 10% when the original sample concentration is greater than 50 times the IDL?

:___: ___

If no, are the associated data flagged with an "E" on this form and associated form I's?

:___: ___

(10) Holding Times

Check holding times against raw data and chain of custody for:

Mercury (26 days).....exceeded?

___ :___: ___

Cyanide (12 days).....exceeded?

___ :___: ___

other metals (6 months)....exceeded?

___ :___: ___

(11) Instrument Detection Limit

Are all IDL's present for all analytes and all methods?

:___: ___

Is the IDL greater than the CRDL for any analytes?

:___: ___

If yes, is the concentration of any of the samples analyzed on the instrument whose IDL exceeds CRDL, greater than 5 times the IDL?

___ :___: ___

Raw Data

Is digestion log present and complete for flame AA/ICP and furnace?

:___: ___

Are all LCS & PB's listed on digestion logs?

:___: ___

Are preparation dates, weights, dilutions and volumes used to obtain values on all digestion logs?

:___: ___

Are total solids sheets for soils present and complete?

:___: ___

Was a continuing calibration performed every 10 samples?

:___: ___

H2M LABS, INC.

CHECKLIST FOR PRELIMINARY REVIEW OF INORGANIC CLP PACKAGES

Page 6 of 6

	YES	NO	N/
<u>Raw Data (con't)</u>			
Was an initial calibration blank analyzed, and a continuing calibration blank analyzed every 10 samples?	☐	___	___
Was one preparation blank analyzed for each 20 samples, each matrix type?	☐	___	___
Are all raw data to support all ICP sample analyses and QC operation's present?	☐	___	___
legible?	☐	___	___
properly labeled?	☐	___	___
Are all raw data to support all Flame sample analyses and QC operation's present?	☐	___	___
legible?	☐	___	___
properly labeled?	☐	___	___
Are all raw data to support all Furnace sample analyses and QC operation's present?	☐	___	___
legible?	☐	___	___
properly labeled?	☐	___	___
Are all furnace samples which are flagged with a "W" on raw data, flagged on the associated Form I's?	☐	___	___
<u>Calibration</u>			
Is a record of at least a 2 point calibration present for ICP?	☐	___	___
Is a record of at least a 4 point calibration present for each flame AA?	☐	___	___
Furnace AA?	☐	___	___
Is a record of a 4 point calibration present for Hg?	☐	___	___
Is a record of a 4 point calibration present for Cd?	☐	___	___

H2M LABS, INC.

575 Broad Hollow Road, Melville, N.Y. 11747
(516) 694-3040 FAX: (516) 694-4122

██████████ LANDFILL LOTUS SPREADSHEETS

1991 GROUNDWATER MONITORING

1st QUARTER

CONTRACT

COMC. UNITS: UG/L

ANALYTE	FIRST QUARTER 1991 DATE COLLECTED: 3/13/91 H2M LAB #:	SECOND QUARTER 1991 DATE COLLECTED: H2M LAB #:	THIRD QUARTER 1991 DATE COLLECTED: H2M LAB #:	FOURTH QUARTER 1991 DATE COLLECTED: H2M LAB #:	AVERAGE 1991
	CONCENTRATION :QUALIFIER:	CONCENTRATION :QUALIFIER:	CONCENTRATION :QUALIFIER:	CONCENTRATION :QUALIFIER:	CONCENTRATION :QUALIFIER:
VOLATILE DATA					
TOTAL VOLATILE ORGANICS	157				
TOTAL HALOGENATED HYDROCARBONS	78				
ACETONE	12	B			
TRICHLOROETHENE	1	J			
CARBON TETRACHLORIDE	1	J			
BENZENE	52				
STYRENE	2	J			
TETRACHLOROETHENE	2	J			
TOLUENE	2	J			
CHLOROBENZENE	74	J			
ETHYLBENZENE	3	B			
XYLENE (TOTAL)	8	B			
SEMI-VOLATILE DATA					
NAPHTHALENE	20				
2-METHYLNAPHTHALENE	8	J			
ACENAPHTHENE	24				
DIBENZOFURAN	9	J			
FLUORENE	9	J			
PHENANTHRENE	2	J			
BIS(2-ETHYLHEXYL)PHTHALATE	36	B			

BAR CHART

EXISTING WELLS
GROUNDWATER
FIRST ROUND

METCALF & EDDY PROJECT 28-DAY SCHEDULE

23-DAYS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
DAY OF THE WEEK	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED
VOLATILE DATA DUE DATES	5 MH ANALYSIS							4 MH REPORTING										3 MH FINAL REVIEW			1 MH PREP. COPY&BIND							PK3 DUE
SEMI-VOLATILE DATA DUE DATES	2 MH EXTRACTION/CLEAN-UP					5 MH ANALYSIS										4 MH REPORTING			3 MH FINAL REVIEW-PRER. COPY&BIND			1 MH FINAL REVIEW-PRER. COPY&BIND						PK3 DUE
PESTICIDES DATA DUE DATES	2 MH EXTRACTION/CLEAN-UP					5 MH ANALYSIS											5 MH REPORTING			3 MH FINAL REVIEW-PRER. COPY&BIND			1 MH FINAL REVIEW-PRER. COPY&BIND					PK3 DUE
METALS DUE DATES	5 MH DIGESTION					12 MH ANALYSIS					2 MH REPORTING								1 MH FINAL REVIEW-PRER. COPY&BIND									PK3 DUE
WET CHEM DUE DATES																												PK3 DUE

MILESTONES			
VOLATILE ANALYSIS	7 DAY ANALYTICAL HOLDING TIME		
SEMI-VOLATILE ANALYSIS	5 DAY EXTRACTION HOLDING TIME		
PESTICIDES/PCB ANALYSIS	5 DAY EXTRACTION HOLDING TIME		
METALS ANALYSIS	12 DAY 2ND 26 DAY 11g ANALYTICAL HOLDING TIME		

MH = MAJOR HOURS

NEW WELLS
GROUNDWATER
SECOND ROUND

METCALF & EDDY PROJECT 28-DAY SCHEDULE

28-DAYS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
DAY OF THE WEEK	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED
VOLATILE DATA	4 MH																											
DUE DATES	ANALYSIS						3 MH	REPORTING										2 MH	FINAL REVIEW		PREP-COPY&BIND							
SEMI-VOLATILE DATA																												
DUE DATES	EXTRACTION/CLEAN-UP					ANALYSIS												REPORTING		FINAL REVIEW- PREP-COPY&BIND								PKG DUE
PESTICIDE/PCB DATA																												
DUE DATES	EXTRACTION/CLEAN-UP					ANALYSIS												REPORTING		FINAL REVIEW- PREP-COPY&BIND								PKG DUE
METALS																												
DUE DATES	DIGESTION					ANALYSIS							REPORTING							FINAL REVIEW- PREP-COPY&BIND								PKG DUE
WET CHEM																												
DUE DATES	ANALYSIS																			HAND IN DATA-PRELIM REVIEW								PKG DUE

NO. SAMPLES
3

ANALYSIS

TOL VOLATILE ORGANICS

MILESTONES

VOLATILE ANALYSIS 7 DAY ANALYTICAL HOLDING TIME

MH = MANHOURS

METCALF & EDDY PROJECT 28-DAY SCHEDULE

ANALYSIS
TCL VOLATILE ORGANICS

MILESTONES
VOLATILE ANALYSIS T.D.

YH = MAX(HOURS

WYNANTSILL CREEK
SURFACE WATER/SEDIMENT
SEEP/WATER/BOREHOLE SOILS

METCALF & EDDY PROJECT 28-DAY SCHEDULE

28-DAYS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
DAY OF THE WEEK	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED	THURS	FRI	SAT	SUN	MON	TUES	WED
VOLATILE DATA	4.5 MH	ANALYSIS																										
DUE DATES								3.5 MH	REPORTING									3.5 MH	FINAL REVIEW		PREP-COPY&BIND	1 MH	PREP-COPY&BIND			MAIL		PKG DUE
SEMI-VOLATILE DATA																												
DUE DATES							ANALYSIS										REPORTING			FINAL REVIEW- PREP-COPY&BIND						MAIL		PKG DUE
PESTICIDE/PCB DATA																												
DUE DATES							ANALYSIS										REPORTING			FINAL REVIEW- PREP-COPY&BIND						MAIL		PKG DUE
METALS																												
DUE DATES							ANALYSIS					REPORTING								FINAL REVIEW- PREP-COPY&BIND						MAIL		PKG DUE
WET CHEM	4 MH	ANALYSIS																										
DUE DATES																				0.5 MH	0.5 MH	FINAL REVIEW- PREP-COPY&BIND				MAIL		PKG DUE

NO SAMPLES
1 WATER
3 SEDIMENT
2 SEEP/WATER
3 BOREHOLE SOILS
4 BOREHOLE SOILS

ANALYSIS
TCL VOLATILE ORGANICS
TCL VOLATILE ORGANICS
TOTAL ORGANIC CARBON
TCL VOLATILE ORGANICS
TCL VOLATILE ORGANICS
TOTAL ORGANIC CARBON

MILESTONES
VOLATILE ANALYSIS
TOC ANALYSIS
7 DAY ANALYTICAL HOLDING TIME
36 DAY ANALYTICAL HOLDING TIME

MH = MANHOURS

FACILITIES AND EQUIPMENT

H2M LABS, INC.

FACILITIES AND EQUIPMENT

H2M Labs, Inc. is located at 575 Broad Hollow Road, Melville, New York 11747. Broad Hollow Road is also identified as Route 110. It is conveniently located at Exit 49 South of the Long Island Expressway (Route 495).

The laboratory occupies approximately 10,000 square feet. It is staffed with 60 technically qualified people whose educational backgrounds vary, depending on specific job functions.

The laboratory is subdivided into five sections:

1. Shipping/Receiving
2. Inorganic Chemistry
3. Organic Chemistry (GC)
4. Organic Chemistry (GC/MS)
5. Special Process Lab

In a continuing effort to extend our capabilities and timeliness, H2M has recently completed an expansion and remodeling of the laboratory.

The recent acquisition of the Nelson Turbochrone Data System coupled with the Finnegan Formaster Data Software for Pesticide/PCB, completes our automated reporting system for the CLP reporting requirements. This, coupled with the Telecations Software for Metals Data and the Aquarius software for the GC/MS data maximizes the output for the data reporting process.

The laboratory currently operates on a staggered shift: Monday to Friday, double shift, 6 a.m. to midnight; Saturday/Sunday, single shift, 9 a.m. to 5 p.m. (except for bacteriology, which is a seven day-per week operation).

The sample receiving section is open 7 a.m. to 6 p.m. Monday through Friday. Arrangements for receipt of samples on weekends or after normal hours are made upon request.

HANDLING AND DISPOSAL OF HAZARDOUS MATERIALS

Our laboratory is aware of the potential hazards associated with handling highly toxic materials. Therefore, a special process laboratory was designed and built to accommodate this type of sample. This area of the laboratory was designed so as to handle these samples with no significant threat to the safety and health of our staff.

H2M LABS, INC.

All employees working in this laboratory will receive sufficient instruction to allow them to work safely. There will always be at least two employees working in this area at the same time.

This facility is equipped with double sets of doors between this section and the rest of the facility. Access is limited to authorized personnel only. Separate areas are provided within the special process laboratory for changing street clothing to special disposable protective clothing. A washer/dryer is used to launder work clothes to prevent transfer of any contamination. A shower area is provided to be utilized at the end of the work day.

The laboratory area was designed to be a self-contained laboratory to minimize the possibility of contamination.

The laboratory is supplied with a continuous fresh air supply. Hoods are available for use and each is designed for specific functions:

- Opening of coolers with sample containers
- Prescreening of samples aliquots taken
- Extraction of sample aliquots
- Evaporation and concentration

The performance of the hoods is monitored on a routine basis. The hoods are manufactured by Kewune and are stainless steel, coved-corner hoods to minimize any absorption of hazardous materials on the work surface. The hood exhausts are filtered through activated carbon filters to prevent contamination of the environment. The water supply of certain hoods is collected and analyzed to assure proper disposal in accordance with government regulations.

Flammable cabinets are equipped for storage of reagents for use in this area, and an additional exhaust vent is provided. The walls and floors of this area are sealed with an epoxy-coated covering to minimize absorption of the surface.

All standard precautions are taken when analyzing samples, such as goggles, gloves and, in certain cases, face masks.

Safety equipment provided in the laboratory includes safety showers, emergency eye washes, fire extinguishers, fire blankets, spill control kits, emergency lighting and self-contained breathing devices.

The laboratory was designed so that the personnel could be viewed while analyses are performed in all areas of the

H2M LABS, INC.

facility. Support equipment is provided outside the laboratory for rescue operations, if necessary.

Samples collected from drum or hazardous waste sites must be disposed of in a proper manner. After analyses, samples that have high concentrations of targeted analytes are segregated and disposed of in the same manner as general lab wastes. This procedure is as follows:

All lab wastes are held in the laboratory in the Article XII room. The wastes are segregated by classification, i.e. flammable solvents, corrosive waste E.P. toxic metal wastes, and reactive wastes. Waste logs are maintained for each waste container. Lab personnel indicate in the log the specific waste being deposited, the volume and date. Each log should be initialed. The wastes are held in a DOT approved container. The container is properly labeled. A waste profile sheet is prepared for each waste shipment.

H2M LABS, INC.

MAJOR LABORATORY EQUIPMENT

<u>EQUIPMENT</u>	<u>MANUFACTURER</u>	<u>AMOUNT</u>	<u>MODEL</u>	<u>YEAR PURCHASED</u>
<u>ORGANIC ANALYSIS (GC)</u>				
Gas Chromatographs	Tracor	(1)	540	1984
	Tracor	(2)	550	1977,1988
	Tracor	(2)	560	1980,1982
	Perkin Elmer	(1)	8500	1988
	Hewlett Packard	(2)	5890A	1987,1989
Electrolytic Conductivity Detectors	Tracor	(3)	700-A	1984
		(1)	1000	1988
Flame Ionization Detectors	Tracor	(3)	12016A	1977,1982
PID Photo Ionization Detectors	Tracor	(1)		1982
	HNU	(1)		1988
Electron Capture Detectors	Tracor	(2)	113550-3201	1978,1981
Flame Photometric Detector	Tracor	(1)	113550-3400	1980
Computing Integrators/Data Systems	IBM Multichannel	(1)	9000	1984
			Update	1986
	Hewlett Packard	(2)	3392A 2-Channel	1987,1989
	Spectrophysics	(1)	SP4290	1986
	Nelson	(1)	Turbochrom 4-Channel	1989
Autoinjector	Perkin Elmer	(1)	AS100	1982
	Hewlett Packard	(2)	HP7673A	1987,1989
Formaster for Automated CLP Reporting	Finnegan Mat	(1)	Formaster 3.1	1989
Purging Apparatus	Tekmar	(3)	LSC-2	1981,1983
	Tekmar	(1)	LSC-2000	1988
Automatic Liquid Samplers	Tekmar	(2)	ALS10	1980,1983
	Tekmar	(1)	ALS2050	1989

ORGANIC ANALYSIS (HPLC)

High Pressure Liquid Chromatography:

-Quaternary Solvent Pump	Hewlett Packard		1050	1989
-Progressive Fluorescent Detector	Hewlett Packard		1046A	1989
-Detector Autosampler	Hewlett Packard		1050	1989

Water Purification	Barnsteadt		Nanopure II	1989

H2M LABS, INC.

MAJOR LABORATORY EQUIPMENT

EQUIPMENT	MANUFACTURER	AMOUNT	MODEL	YEAR PURCHASED
ORGANIC ANALYSIS (GC/MS)				
Gas Chromatograph/Mass Spectrometer:	Finnegan Mat	(1)	OWA 30	1980
-Gas Chromatograph	Perkin Elmer		Sigma 3	1980
-9 track tape drive	Perkin Elmer			1980
-Computer	Data General		Nova 4	1980
-Printer	Printronic		MVP150	1989
-CRT	Graphon			1988
-Real-time display Oscilloscope	Finnegan Mat			1989
-Winchester 70 Mb Disk Drive	Priam			1986
-1050 Superincos Upgrade	Finnegan Mat			1986
-Capillary interface	Hewlett Packard	(1)	5996A	1984
-Graphics display terminal/printer	Hewlett Packard			1984
-200 CPS printer	Hewlett Packard		HP2623A	1984
-Combined Wiley & NBS Data Base w/Wiswiser Line Notation	Hewlett Packard		HP2933A	1984
-Aquarius Software	Hewlett Packard		HP59868A	1984
-Winchester Disk Drive	Hewlett Packard			1984
-Graphics Display Terminal	Hewlett Packard		HP2632A	1984
GC/MS A Series Data System w/Micro 24 SPU & 304 Mb Disk Drive	Hewlett Packard	(1)	HP59870C	1990
-GC/LC/MS Software	Hewlett Packard	(1)	HP59872C	1990
-Mass Spectral Library	Hewlett Packard	(1)	HP59868C	1990
GC Mainframes	Hewlett Packard	(2)	HP5890A	1987,1988
	Hewlett Packard	(1)	HP5970 Series II	1990
MSD Quadropole Bench Top MS	Hewlett Packard	(3)	HP5970B	1988,1990
Graphics Display Terminals	Hewlett Packard	(2)	HP2393A	1987,1988
Printers	Hewlett Packard	(1)	HP2934A	1987
	Hewlett Packard	(2)	HP2937A	1989
Autoinjector	Hewlett Packard	(2)	HP7673A	1989,1990
	HP Injector Modules	(2)	18593A	
Automatic Liquid Samplers	Tekmar	(1)	ALS10	1985
		(2)	ALS2016	1988,1989
Cryogenic Cap. Interface	Tekmar	(1)	M2000	1987
Liquid Sample Concentrators	Tekmar	(2)	LSC2000	1988,1989
Moisture Control Module	Tekmar	(1)	14-4700	1990
Unacon Sample Concentrator	Envirochem	(1)	810	1984

H2M LABS, INC.

MAJOR LABORATORY EQUIPMENT

<u>EQUIPMENT</u>	<u>MANUFACTURER</u>	<u>AMOUNT</u>	<u>MODEL</u>	<u>YEAR PURCHASED</u>
<u>METAL ANALYSIS</u>				
Atomic Absorption Spectrophotometer:	Perkin Elmer	(1)	AA5000	1979
	Perkin Elmer	(1)	AA2380	1985
	Perkin Elmer	(1)	AA5100	1988
	Varian	(1)	30	1987
Graphite Furnace	Perkin Elmer	(1)	HGA5000	1979
	Perkin Elmer	(1)	HGA5100	
	Varian	(1)	30 Furnace	
Flame Auto Sampler	Perkin Elmer	(1)	AS-50	1979
Chart Recorder	Perkin Elmer	(1)	56	1971
Automatic Burner Control Module	Perkin Elmer	(1)	5000	1979
Printer Sequencer	Perkin Elmer		PRS-10	1979
Auto Sampler for Graphite Furnace	Perkin Elmer	(1)	AS-1	1979
	Perkin Elmer	(1)	AS-60	1989
	Varian	(1)	AS-30	1987
Hydride Generator	Perkin Elmer	(1)	MHS-10	1979
Mercury Analysis System	Perkin Elmer	(1)	CV	1971
Mercury Cold Vapor System	Varian	(1)	VGA-76	1990
Burner Controller	Perkin Elmer	(1)	5000	
Inductively Coupled Plasma Spectrometer Systems	Applied Research Labs	(1)	3410 w/IBM XTC	1986
	Sequential Applied Research Labs	(1)	Computer Package 3560 w/IBM 30	1989
	Simultaneous		Computer Package	
EDL Lamp & Supply	Perkin Elmer	(3)	EDL	1985, 1988
	Varian	(2)	EDI/Westinghouse	1987

INORGANIC ANALYSIS

Balance	Ohaus	(2)	C501	1989
Autoanalyzer	Technicon	(1)	II 4-Channel	1973
Block Digestor	Technicon	(1)	BD40	1977
	Tecator	(1)	1015	1989

H2M LABS, INC.

MAJOR LABORATORY EQUIPMENT

EQUIPMENT	MANUFACTURER	AMOUNT	MODEL	YEAR PURCHASED
INORGANIC ANALYSIS (cont'd)				
Halide Analyzer (TOX):				
-Purgeable Organic Halide(POX)	Dohrmann Xertex	(1)	DX-20	1984
-Adsorption Module		(1)	AD-2	1984
-Microcoulometrix Analyzer Module		(1)	MC-1	1984
Refrigerator Locking (no spark interior)	Kelvinator	(4)		1984
	Fisher Scientific	(3)		1989
Envirotech Organic Analyzer (TOC):	Dohrmann	(1)	DC190	1991
-Boat Sampling Module		(1)	183	1991
Dissolved Oxygen Meter	Yellow	(2)	51-A	1975
	Springs Instruments	(2)	57	1984
COD Apparatus	Glas-col	(2)		1977
	Hach	(1)	Micro Block	1988
Centrifuge	Fisher Scientific	(2)		1957,1984
Drying Oven	Precision Scientific	(1)	THELCO18	1980
Kjeltec Autoanalyzer	Tecator	(1)	1030	1983
Dessicator	Fisher Scientific	(2)		1978
Balance	Sartorius	(1)	1612	1971
		(1)	2602	1974
		(1)	2462	1984
Auto Titrator	Fisher Scientific	(1)	380,381,385,395	1983
Muffle Furnace	Thermoline	(2)	1500	1971,1983
pH Meters	Corning	(4)	103	1984,1988
	Beckman	(1)	4500	1976
	Fisher Scientific	(1)	640	1982
	Gallenkamp	(2)	pit stick	1985
Spectrophotometer	Sequoia-Turner	(2)	340	1983,1989
IR Spectrophotometer	Buck	(1)	HC404	1990
Analytical Nephelometer	Hach	(1)	2424	1977
Glass Stills	Belco	(2)		1975,1977

H2M LABS, INC.

MAJOR LABORATORY EQUIPMENT

<u>EQUIPMENT</u>	<u>MANUFACTURER</u>	<u>AMOUNT</u>	<u>MODEL</u>	<u>YEAR PURCHASED</u>
<u>INORGANIC ANALYSIS (cont'd)</u>				
Specific Ion Electrodes	Orion		Various	1983
Pensky-Martens Flash Point Tester	Precision Scientific	(1)		1983
Autoclave	Market Forge	(2)	STM-E Type C	1976,1984
Microscope	Nikon	(1)	Labobot 104	1983
Coliform Incubator Bath	Precision Scientific	(1)	66850	1980
Ambi-Hi-Lo Chamber Incubator	Labline Instruments	(2)	3554-17	1976
Automatic Pipetting Machine	Scientific Equip. Products	(1)	40	1983,1984
Incubators	Fisher Scientific	(2)	307	1988

SPECIAL PROCESS/SAMPLE PREPARATION

GPC Apparatus	In-House	(4)		1987,1990
TCLP Tumbler	Environ Express	(1)	10-position	1990
	Analytical Testing...	(1)	4-position	1987
	In-House	(1)	8-position	1986
Zero Headspace Extractor	Environ Express	(6)		1990
	Analytical Testing...	(4)	C-102	1987,1989
UV Detector	Rainin	(1)		1991
Continuous Liquid/Liquid Extractor	Organomotion	(1)	Rot-X-Troeth	1991
Agitator	Analytical Testing...	(2)	DC-18	1987
Sonic Disruptor	Tekmar	(1)	TSDB-500	1986
ICC Clinical Centrifuge	Int'l Equipment Co.		ICC Clinical	1985
Sartorius Balance	Brinkmann Instruments	(2)	1002	1987
Corrosivity Tester	In-House	(2)		1987
Blue M Oven	General Signal	(1)		1986
Thelco Water Bath	Precision Scientific	(1)	84	1984
Turbo-Vap Evaporation Workstation	Zymark	(1)	200	1989
Kiln	Cress	(1)	Firemate FE27	1989

APPENDIX B

PHOTOVAC 10S70

OPERATING MANUAL

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WARNING

THIS EQUIPMENT GENERATES, USES AND CAN RADIATE RADIO FREQUENCY ENERGY AND IF NOT INSTALLED AND USED IN ACCORDANCE WITH THE INSTRUCTIONS MANUAL, MAY CAUSE INTERFERENCE TO RADIO COMMUNICATIONS. IT HAS BEEN TESTED AND FOUND TO COMPLY WITH THE LIMITS FOR CLASS A COMPUTING DEVICE PURSUANT TO SUBPART J OF PART 15 OF FCC RULES, WHICH ARE DESIGNED TO PROVIDE REASONABLE PROTECTION AGAINST SUCH INTERFERENCE WHEN OPERATED IN A COMMERCIAL ENVIRONMENT. OPERATION OF THIS EQUIPMENT IN A RESIDENTIAL AREA IS LIKELY TO CAUSE INTERFERENCE IN WHICH CASE THE USER AT HIS OWN EXPENSE WILL BE REQUIRED TO TAKE WHATEVER MEASURES MAY BE REQUIRED TO CORRECT THE INTERFERENCE.

THE 10S70 CONCEPT

Photovac's Model 10S70 portable gas chromatograph is the most advanced member of the 10S series. In terms of the sensitivity of its detector and the range of different types of chromatography offered, the 10S70 is identical to the 10S30 and the 10S50. The 10S70 has virtually the same data reduction power as the 10S50, but there are some subtle differences which will be discussed later.

The 10S70 is primarily a communications instrument. It is equipped with an RS232 interface and can be connected to a remote computer terminal or to a specially programmed computer and can be controlled through the computer terminal. All functions which can be performed on the 10S70's own keyboard, can similarly be performed remotely with some additions. All data displayed on the 10S70 LCD and printer can be presented at the remote location. It is therefore possible to re-program the 10S70, through the terminal, to optimize the required performance.

The 10S70 is also equipped with its own modem unit so that remote communications can be established over a telephone line. A second modem will be required to serve the receiving terminal or computer. An auto-dial feature has been included so that the 10S70 will automatically "call in" if, for example, the level of a particular contaminant exceeds a predetermined limit. Access to the 10S70 by unauthorized personnel is restricted by the use of a password system.

A typical use of the 10S70 could involve an emergency situation where there has been a spill of toxic chemicals and where this has to be continuously monitored. Let us imagine that this has occurred over a weekend or similarly inconvenient time. A 10S70 is deployed to the site and is set up in a shelter having reasonable environmental control. A sample line is run from the instrument to the chosen monitoring point. The 10S70 is provided with a sufficient supply of carrier gas and suitable calibrant and is also connected to an external power supply, such as AC mains or a 12 volt DC. The 10S70 is put into operation and commences the generation of data. Let us suppose that the person responsible now wants to go home! The instrument is programmed with the appropriate telephone number and the individual sets up a terminal and modem at home.

The instrument can be contacted at any time in order to obtain the latest reading and to determine the Time Weighted Average (TWA), accumulated to date, together with the maximum level encountered. Adjustments to the analysis information can also be made if required.

If level should suddenly increase, the 10S70 will report the fact over the telephone line and the message will be displayed on the terminal.

Such a formidable capability provides the environmental specialist with an extremely valuable time-saving in terms of hours spent on the site. A similar time saving could be imagined in the case of Industrial Hygiene monitoring. Here an instrument can be set up in the plant area and the situation monitored from the Industrial Hygiene office. Again, the option exists to set up the terminal off site, at home or at another facility from which the situation can still be monitored.

Instrument Configuration

The top panel layout and the designation of the keys in the 10S70 is virtually identical to that of the 10S50. See Figure 1. There are just two differences:

1. There is a connector mounted on the panel which is marked Phone Serial; this is the point of connection for the telephone line or for the remote terminal.
2. The key in the top left corner of the key cluster is marked "PHONE" (instead of "TEST" as in the 10S50). Upon pressing this key results in a different menu of commands being displayed and the arrow keys are used to view the menu. The items appearing are:

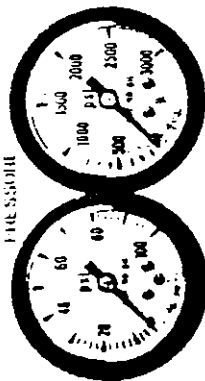
STATUS REPORT
TEST CODES
SYSTEM OPERATIONS
ALARM MESSAGES
CHANGE PASSWORD

"STATUS REPORT" and "TEST CODES" are functions which are available on the 10S50 and the use of these functions is the same, except there are some additional test codes. "SYSTEM OPERATION", "ALARM MESSAGE" and "CHANGE PASSWORD" are functions unique to the 10S70.

There are a number of differences in software between the 10S70 and the 10S50 and the manifest themselves through new LCD messages and different performance characteristics relating to the communications function.

The 10S70 includes a local alarm as a standard feature. When a pre-set limit is exceeded the instrument will respond with a loud tone of varying frequency and will continue for a programmable period of time.

115 V
60 Hz
0.5 A



DELIVERY

CONILNIS

EXT DC

FLOW

MANUAL
INJECTION

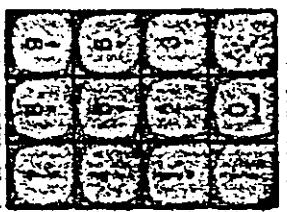
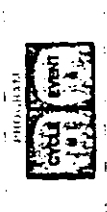
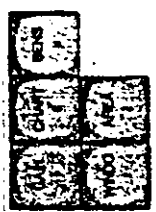
PHOTOVAC

PORTABLE PHOTOIONIZATION GAS CHROMATOGRAPH

INLET

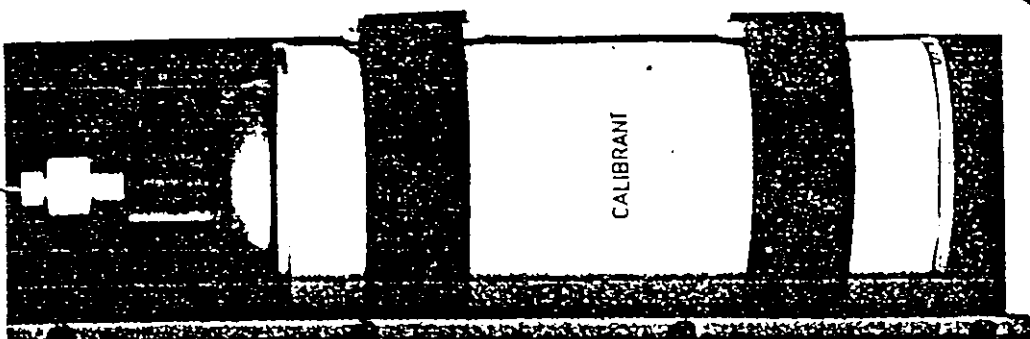
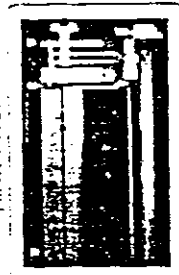
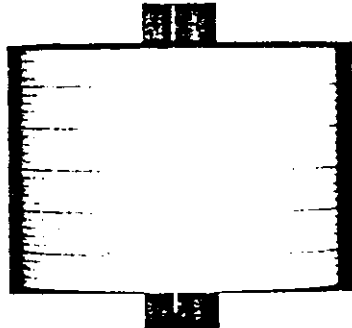
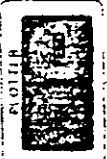
EXTERNAL
CARRIER IN

OUTPUT



PHOTOVAC 10570

CHROMATOGRAPH



OPERATING INSTRUCTIONS

It is first necessary to thoroughly understand the operation of the 10S70 computer and gas chromatograph. We recommend that you begin by running the instrument as though it was a 10S50 and that you work through as many of the 10S50 exercises as possible. Turn to the 10S50 Operating Manual found later in this binder. Remember that the "TEST" key described in the 10S50 manual is replaced with the "PHONE" key in the 10S70. As mentioned in the preceding section the "PHONE" key incorporates additional functions but the two functions of the "TEST" key are still retained.

Once you have studied "local" operation of your 10S70 proceed to the following section which describes "remote" operation.

Operation of the 10S70 Through a Remote Terminal

Connection:

Your 10S70 is supplied with a dual function communications cable which attaches to the receptacle on the 10S70 electronic module. This cable terminates in two types of connector: A DB-25 connector which is wired for RS232 transmission, using ASCII codes and a RJ11C (telephone) connector for direct connection to the telephone system.

The connections to the DB-25 are listed in Appendix 1. The 10S70 is not currently compatible with any other transmission codes.

At the present time, the 10S70 is supplied with a modem which is only approved for use in the United States. Use in other countries may be against the law or in contravention of local telephone company rules. If you purchased your 10S70 from any country other than the United States, for delivery to that country, this will have been made clear to you. If you are planning to travel abroad with your 10S70 you are advised to make careful enquiries in advance. Photovac Incorporated cannot be liable for any consequences of unauthorized use in such a situation.

We will assume that you wish to begin by connecting your 10S70 to a video display terminal. We will also assume the terminal has a DB-25 receptacle.

Your 10S70 will have been set up in the factory to transmit data at a rate of 1,200 BAUD. You should ensure that your terminal is adjusted to accept the same transmission speed and the terminal's automatic line feed should be off. The 10S70 can transmit data at other rates. See Appendix 3 for details.

Connect the 10S70 to the terminal with the DB-25 plug. Turn on the 10S70 and the video terminal. Try to arrange to have a standard calibration mixture available for the exercises which follow.

Depress the spacebar four times in rapid succession. The 10S70 will present the following statement on the terminal: "REMOTE INTERRUPT ENTER PASSWORD".

The purpose of the password is to deny unauthorized users access to the 10S70. Your 10S70 has been set up with the password "10S70". Enter this password and press the return key on the terminal. The password can be changed later. The 10S70 will respond: "JAN 22, 1988 14 20 REMOTE LINK ESTABLISHED", and after a pause "READY ENTER COMMAND" will appear.

The 10S70 is now ready to be operated from the terminal. The following table lists the instructions of the 10S70's keyboard. Each instruction has two of its letters in capitals; these two letters when entered on the terminal transmit the instruction.

Off	PHone	ARea	Calibrate
Llist	GAin	CYcle	^ (Cursor up)
EDit	CHart	EVent	(Cursor down)
USE	SEns	INfo	enter (carriage return)
STore	WIndo	SStart/stop	clear (backspace)

Any invalid command will result in the display of a command menu, similar to the above list.

With the exception of OFF and SStart/Stop, we suggest that you execute each command in turn, to become familiar with the appearance of its presentation. Unless you want to turn the instrument off or start an analysis, we advise that you so not execute OFF or SStart/stop until you are ready. Remember that the carriage return key executes the "ENTER" function.

Notice that a chosen function is retained after entry and execution. For example, if you press "Event", followed by "ENTER", followed by 1 (to select event 1) you may select event 2 by simply entering 2 after you have completed event 1. In other words, you do not have to enter "Event" a second time.

Some of the data that will appear may be unfamiliar to you. We will discuss this at later point.

The "Phone" function requires some additional explanation. If you enter "Phone", the response "STATUS REPORT" will appear and if you enter this you will receive the report on the detector and valve set-up.

If you enter "Phone" and use the arrow keys, you can select the following additional functions:

TEST CODES	These are code numbers which are valuable in problem diagnosis and are listed in Appendix 2.
SYSTEM OPERATION	This function yields a subset of additional functions:
LOCAL ONLY	This function breaks communication between the 10S70 and the terminal. Communication is regained by entering the password.
LOCAL AND REMOTE	This is the present mode of operation with the terminal and the 10S70 both displaying the data. Entering this function will result in no change.
TERMINAL MODE	This function enables the terminal user to display any desired message on the printer of the 10S70. The alphabetic keys of the 10S70 may be used to send a message to the terminal. The terminal mode may be exited by depressing the "CONTROL" key on the terminal and simultaneously press N twice. The response will be "LOCAL ONLY" and "LOCAL AND REMOTE" will be selected and entered.
ALARM MESSAGE	When the 10S70 auto-dials the terminal to report and alarm (in modem operation), a message will appear on the terminal. There are 16 characters available for the alarm message.

CHANGE PASSWORD

This function should be used with great caution as it is possible to enter a password and then forget what it is. Provided you have not broken communication, you can determine the present password by pressing "PASSWORD", followed by "ENTER". There are 16 characters available for the password.

There are a number of additional commands available to the terminal user, for which the corresponding keys are not available on the 10S70 keyboard. The following is a list of these commands, with their functions:

PLot	A chromatogram is normally plotted on the terminal or screen as a line of asterisks ***** this may be replaced with an actual numerical value of the analog signal: 27 27 29 35 47, with a range of +/- 1023. To enter or exit this mode enter "PLot" during a run; successive presses of the "ENTER" key will toggle between the two modes.
BAttery/Time	Displays battery voltage when no analysis in progress, or displays elapsed time of analysis in progress.
SiGnal	Displays signal level in mV as on the 10S70 plotter.
LOcal	Turns the 10S70's printer and display off. This is valuable to conserve paper and pens when only remote operation is needed. Use "ENTER" key to toggle between modes.
TEst	Fast access to test codes, skips "PHone" key.
REport	Status report printed, skips "PHone" key.
.PAssword	Changes password, skips "PHone" key. "RECORD THE PASSWORD CAREFULLY FOR FUTURE REFERENCE".
ALarm message	Change alarm message, skips "PHone" key.
ENd	Returns 10S70 to local mode, breaks communication with terminal. Re-entry requires password. Skips the "PHone" key.
Terminal Mode	Enter terminal mode, skips "PHone key". To exit press "CONTROL N" twice and select "LOCAL AND REMOTE" mode.

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Remote operation between the 10S70 and the terminal was initiated from the terminal end by pressing the SPACE BAR, or any other character key, four times in rapid succession. Then upon entering the password the link was established.

Remote operation can be established from the 10S70 end, without the use of the password. The "PHONE" key is pressed "SYSTEM OPERATION" is selected and entered. The terminal will now be activated and control of the 10S70 is enabled

Remote Control of a 10S70 from a Terminal with a Modem Connection

It will first be necessary to read the instructions for dialing with the modem that you have selected. A transmission rate of 1200 BAUD is recommended and a modem with auto-dial and certainly auto-answer capability is required. If you have a modem with a different transmission rate, refer to Appendix 3 to adjust the 10S70's transmission rate to match.

Once the call has been answered by the 10S70, four presses of the SPACE BAR within five seconds will elicit the request for password.

Programming a 10S70 to Auto-Dial

Make sure the 10S70 is in the "LOCAL ONLY" mode. Select "ALARM MESSAGE". Press the "OFF" key, followed by TB or PB (depending upon whether the phone system uses tone or pulse dialing respectively), followed by the number to be called.

The 10S70 should now be left to monitor, with the appropriate limit values in memory. As soon as an alarm condition occurs, the Auto-Dial feature will be initiated and the receiving terminal will display the data as the analysis proceeds.

Control of a 10S70 by another 10S70

If the two instruments are connected together directly, the transmitting instrument should be placed in the "TERMINAL" mode and the receiving instrument in the "LOCAL AND REMOTE" mode. The receiving instrument can then be controlled by the transmitting instrument, using the alphanumeric keys just as if it were a terminal.

If it is desired that the two instruments communicate over a telephone line, each RJIC connector should be connected into the appropriate extension. The 10S70 to be controlled should be switched "ON". The controlling 10S70 should be switched "ON" and placed in the "TERMINAL" mode. Next, the "OFF" key is pressed, followed by the letters TB or PB depending upon whether the phone system being used employs tone or pulsed transmission respectively, followed by the number to be reached. The response "A", appearing on the printer, indicates that the call has been answered; "B" indicates "busy"; "N" generally means "no answer" or no modem answer tone within a 17 second period following the last ring back tone received. An "X" means failure to receive dial tone.

Assuming an "A" has been printed, four successive presses of (for example) the "ENTER" key will elicit a request for password. Entering the password, establishes control as though the two instruments were connected together with the receiving instrument being controlled by the transmitting instrument.

A very convenient feature which may be exploited with two 10S70s connected in this way is the transmission of an entire library, including set-up data, from one instrument to the other. To do this, the following key presses must be made: "OFF" "X" "SPACE" "ENTER" note that the symbol "n" denotes the number of the library being transmitted (1-4). This command requires 32 seconds at 1200 BAUD.

Appendices

Appendix 1 - Hardware Connections

RS232 Cable

DB-25 Connector

Pin #	FN	Description
1	GND	Protective ground.
2	RXD	Received data to 10S70
3	TXD	Transmitted data from 10S70.
4	RTS	Electrically connected together Not used in 10S70
5	CTS	
7	GND	Signal ground
8	RLSD	This output is always RS232 High (+12 V) when the 10S70 is turned on.
11, 20	DTR	When this output is RS232 low (-12 V), 10S70 transmission is disabled. (has internal pull up resistor)

100 Character input buffer

XON @ 20%

XOFF @ 80%

DTR can be used when the 10S70 is connected to a terminal or printer. (Output control only).

XON/XOFF is recommended for use with a personal computer (both input and output control). Disable DTR when using XON/XOFF when the 10S70 input buffer fills up.

CHAPTER (1) - INTRODUCTORY CONCEPTS

When Photovac first introduced its Model 10A10 portable photoionization gas chromatograph (GC), back in 1979, we felt some sense of achievement in having packaged so sensitive an instrument within a total weight under 12 Kg (26 lbs). Of course, there was also the additional weight of the chart recorder - about 5 kg.

The 10S50, which you have now purchased, has virtually the same weight as its predecessor; the difference is that this now includes an improved GC, multiple valves for a wide range of different automated chromatographies, a sample pump, a most sophisticated computer with a 32 character Liquid Crystal Display (LCD), a completely automatic self-calibrating system (which allows the instrument to carry out continuous monitoring) - AND, a miniature chart-recorder/printer!

In short, the 10S50 is a complete self-contained air analyser.

While our own engineers take considerable pride in this achievement, we recognize also that none of this would have been possible without the ingenuity of countless unnamed engineers among our Suppliers. Without the truly wondrous advances in the miniaturization and reliability-engineering of electronic devices and micro-mechanisms, the 10S50 could not have come into being.

We know that many of the people who purchase our 10S50 will be users for whom the measurement of airborne and waterborne contamination is a major preoccupation.

We also recognize that for many others, these issues are only peripheral - a person working on a high technology plastics product may worry about an unknown health effect of one of the new materials involved - these people are not in the air monitoring field, and are likely not interested in it; their prime objective is to do their work without themselves or their colleagues becoming sick!

We submit that the 10S50 is the first instrument, yet introduced, which can thoroughly satisfy both types of user. For those who need it, analytical sophistication is certainly there; many possible applications and configurations will not be found in this Manual - simply because they have not yet been explored, even by ourselves! Where there is a specific application, however, calling for the continued surveillance of a given chemical, the 10S50 can be set up to do this particular task and ONLY this task. The user requires minimal knowledge as to how the instrument works, in order to be able to obtain high quality data.

NOVEMBER 1987

The 10S50 incorporates Photovac's proprietary Photoionization Detector (PID). This detector was thoroughly proved in our 10A10 instruments, and many hundreds of these are now in use in more than 20 countries around the world. A similar, but de-sensitized, version of our PID is also used in our TIP* product; this being a non-specific photoionization screening tool. In independent tests, the Photovac PID has been able to detect Benzene in a 1 ml air sample at a concentration of 0.1 parts-per-billion (or 1 part in 10,000,000,000 parts of air), with a signal-to-noise ratio of 4:1. This capability makes ours the most sensitive commercial detection system, in its class, existing today - and this includes those detectors available with complex laboratory instruments!

At first sight, having such a formidable detection capability may not seem to matter too much in a practical sense - after all, few compounds can pose a health threat at such low levels. The value, however, becomes apparent when one works at higher concentrations. When working in the parts-per-million (ppm) range, or a few tenths thereof, the detector is operating with incredible ease; the data quality (in terms of crispness and absence of noise) is superb. This should be contrasted with portable Infra-red absorption instruments or Flame Ionization GCs, which have to struggle to see much less than 1 ppm.

Conventional approaches to air monitoring at ppm or sub ppm levels call for the sampling of very large volume of air, often many liters. The sample is pumped through an absorbing medium (like activated charcoal), which is subsequently taken back to the laboratory. The almost universal complaint amongst users of this technique is that there are inordinate delays at the laboratory, while the sample waits in the queue for attention. These delays mean that urgent actions may be postponed and, in the absence of "real time" data, the course of an analytical enquiry cannot be changed to the light of findings. Furthermore, the stability and integrity of stored concentrates is the subject of continuing debate; the data, once obtained, are of indifferent quality.

In many situations, a 10S50 instrument can be deployed to a site under investigation and quality data will be available within minutes. Also, because the 10S50 is able to carry out "continuous monitoring" (this means that it repeats each analysis at pre-determined intervals); Time Weighted Averages (TWAs) can be generated. There is a high degree of confidence in the quality of the data obtained because direct calibrations can be performed at regular intervals, completely automatically.

The 10S50 will provide a printed multi-component report of each analysis, and a summary including TWA and the maximum concentration level encountered for each compound under surveillance.

For those who wish to be more involved in the analytical technique, the actual chromatogram can be called up on the printer/plotter.

The PID in your 10S50 operates using very short wavelength ultraviolet (UV) light. This light has quite a high energy - about 11 electron volts (eV). A special lamp produces this light, which is then directed into a tiny chamber, through which a gas stream (usually high purity air) is passing. Inside the chamber are two electrodes, across which an electric potential of about 300 V is maintained. The negative electrode is connected to an extremely sensitive amplifier.

So long as the gas stream is just pure air or an inert gas like nitrogen or helium, all of these gases require a UV energy greater than 11 eV before they can be IONIZED (broken into two charged fragments). Technically, we say that all these gases have IONIZATION POTENTIALS (IP) greater than 11 eV. By a lucky chance of nature, however, an enormous number of gases and vapors which are classed as pollutants have ionization potentials less than 11 eV and, if there is just a trace of such a compound in the air stream, photoionization will occur and the positive ions will be collected and amplified. This technique delivers the kind of sensitivity quoted earlier.

The PID itself is not able to distinguish between the thousands of different compounds to which it responds. It is necessary, therefore, to carry out separation of each component of a mixed sample and this is where the gas chromatography comes in. A stream of (usually) pure air (the carrier gas), passes through a gas chromatograph column and then into the detector. The column is packed with an adsorptive powder or coated with an adhesive coating. There is a huge variety of different coatings and packings available but the basic property we seek is an ability to retain the chemical being studied - not so strongly that it is held for ever, nor so weakly that the retention is hardly noticeable.

Under ideal conditions, when a sample of air contaminated with a mixture of different chemical compounds is introduced in a discrete "packet" at the column head, away from the detector, each chemical takes its own characteristic time - the RETENTION TIME, to be carried through the column to the detector. Let us assume we are working with benzene and have chosen an appropriate column. The mixture of contaminants, including benzene, is introduced to the column. The lighter compounds (for example, propane, butanes and pentanes) are "retained" roughly in

proportion to their molecular mass; they emerge into the detector as discrete "packets" and the detector responds to them by generating "peaks" which are processed by 10S50's internal computer and appear on the printer/plotter. A series of peaks, rather like a spectrum is generated; if we have chosen our column correctly, the benzene should not come out so quickly, that is, gets merged with the earlier peaks, nor so long that the patience of the analyst is strained - if it comes out at all!

The correct retention time for the benzene will depend mostly upon what else is in the air. If there is only benzene, there is hardly any point in doing chromatography at all - there will only be one peak and this can be arranged to appear in a few seconds. If, however, you have a complex mixture, such as gasoline, you will have several compounds which will have retention times very close to that of benzene - and you want to be sure that these are separated. In this event, you will probably need a retention time of 3 to 5 minutes.

At first sight, and particularly if you have never done chromatography before, all this may seem very intimidating. The reality, as so often, is not that bad: probably 80% of the chemicals needed to be detected can be analyzed with just THREE column types; the results obtained may not be optimal; the chromatograms may not be always so very cosmetic, but nevertheless, a competent job can be done.

If, of course, you are one of those users who have a well-specified and dedicated application, the nuances of gas chromatography will be of little interest to you. Your instrument will have been set up to do your task - and you can escape from reading the bulk of this manual! The chapter on Simplified Operation should be enough to equip you to put the instrument to work.

It is important to discuss the matter of column THERMAL STABILITY and the question of heated column operation. Laboratory GCs, and a few portables too for that matter, contain a column chamber which can be heated and maintained at a steady temperature. When the temperature of a column is raised, compounds pass through (ELUTE) more quickly; furthermore, some high boiling point compounds would never get through the column if it were not heated. There are obvious benefits to heating the column.

Even if a column is not heated to a high temperature, there are advantages to maintain a steady operating temperature, a few degrees above ambient. If you do not do this, you will experience changes in retention time as the ambient temperature changes.

These are some sound reasons which can be put forward in favor of heating your column. There are, however, some distinct disadvantages.

An instrument containing an oven, together with the battery to run it, cannot be so portable. However, Photovac has resolved this issue by giving you a choice. We have come up with a low-power isothermal GC capillary column oven which is compatible with all the 10S models, and can be fitted in the column chamber snugly.

A low-power isothermal GC column oven and an encapsulated capillary column assembly make up the unit for this purpose. With this unit, retention time drift is nearly eliminated and retention time is much shortened. Detailed description of the isothermal capillary column is included in Chapter 7.

If a heated capillary column is not desirable in a particular operation, we have another sound alternative to solve retention time drift problems. With packed column operation, retention time drift exists; however, it is corrected with repeated calibration. Let us see how it works:

The 10S50 is equipped with two gas inlets; one to take in the sample and the other to admit a calibration mixture. These two inlets are connected to a CALIBRATION valve, controlled by the computer - which can select from either by turning the valve on and off. There is a "cradle" on the deck of the instrument which will accept a can, containing calibration mixture of pressure under 40 psi. The can is connected to the calibration inlet. Let us imagine that the gas under study is vinyl chloride monomer (VCM) and that the can contains a 1 ppm mixture of VCM in air. The computer is directed to start a series of sample analyses with calibration analysis. Accordingly, a precise and repeatable quantity of VCM is INJECTED onto the GC column. In about 40 seconds, the 1 ppm VCM peak appears and we store the name VINYL CHLORIDE, together with the level, 1 ppm, into the computer. Now, we can set the 10S50 to CYCLE, and to automatically recalibrate itself every few analyses. Whenever 10S50 performs a calibration, it will check what 1 ppm of VCM "looks like" and update the stored values accordingly. We can program it to do this as often as we wish (every other sample, every five, every ten, etc.)

If drift occurs, the computer can always go back and update its memory as to when the VCM peak will occur and what size of peak 1 ppm VCM now presents (because this, too can change). In short, we are all the time comparing the VCM we see in the sample with the calibrant. We submit that this is an extremely practical and common sense approach to an otherwise difficult problem!

CHAPTER (2) - SIMPLIFIED OPERATION

This chapter is intended for use by those users who have had their 10S50 set up for a specific application. Under these circumstances, the instrument will have been equipped with two GC columns - a precolumn and an analytical column - the flow of carrier gas through these will have been pre-set, the type of calibrant gas will have been defined and procured and all of the switching valve settings and your compounds of interest will have been stored in one of 10S50's four libraries.

The tasks of the user are to check the instrument for any apparent physical damage, due to shipment and to report any damage TO PHOTOVAC, within TEN DAYS of receipt. Check also that the following accessories are included:

- (1) Mains cord
- (2) Packet of 12 septa
- (3) Male Quick-connect fitting
- (4) 2 rolls of plotter paper (1 installed, 1 extra)
- (5) 2 sets pens (4-colors, 1 installed, 1 extra)
- (6) Column conditioning adapter
- (7) Calibrant adapter
- (8) Gas bag adapter
- (9) Printer/plotter cover
- (10) This Operating Manual

Check the Packing List to ensure that all specially ordered accessories are present.

Ensure that the voltage and frequency of your local electrical supply matches the values marked on the instrument panel, beside the input receptacle.

You should now know whether you will be operating your instrument from its own internal carrier gas reservoir, or from an external cylinder. If the former, you should refer to the section on reservoir filling procedure in Chapter (10) "RESERVOIR FILLING PROCEDURE"; if the latter you should already have a suitable supply of gas with a regulator to reduce the pressure to 40 psi (280 kPa). The Quick-Connect accessory fitting should be used to make the connection from the gas cylinder to the "EXTERNAL CARRIER IN" fitting. Note that the connection between the cylinder regulator and the instrument should be very clean and made of a material such as Teflon or stainless steel; NEVER use rubber or soft plastic tubes - their outgassing can cause severe difficulties with contamination.

Connect the instrument to the mains electrical supply, using the cord provided. If the cable is not fitted with a plug, please note that the BLACK wire is LIVE; the WHITE wire is NEUTRAL and the GREEN is GROUND (EARTH). We cannot be responsible for errors in connection.

Having established a supply of carrier gas, the next responsibility is to check that all set-up parameters are still correct. Please see the appended Custom Specification Sheet which can be found in the clear plastic folder in the front of your manual.

Refer to the top panel of the instrument (use Figure 2.1) and locate the gas fitting marked "DETECTOR OUT" and "AUX OUT"; the "AUX OUT" fittings will likely have a small needle valve attached to it. Be very careful not to change the setting on this or any other valve. The small needle valve is very sensitive to even a slight touch. Try not to touch it at all unless you want to adjust the flow.

For the next step you require a 2-channel flowmeter, calibrated for measurement in the 0-50 ml/min range (Photovac accessory number SA205 or equivalent). Connect the left channel to the "DETECTOR OUT", using a 1/8" Swagelok fitting and the right channel to "AUX OUT" needle valve in a similar manner.

Compare the readings showing on the flowmeter with those entered in the Specification Sheet. Adjust, if necessary - the left channel measures the flow through the analytical column, and is adjusted via the "RED" color coded valve, the right channel measures the precolumn backflush flow and is adjusted using the small needle valve, attached to the "AUX OUT" port. These valves interact so your adjustments will need to be iterative. Leave the meter in place and continue.

Depress the "ON" key; the instrument LCD should respond with "LAMP NOT READY PLEASE WAIT"; within 2 to 3 minutes this should change to "READY ENTER COMMAND". Depress the "TEST" key, followed by "ENTER" key. The printer will spring into action and a "Status Report" print-out will appear as shown on Specification Sheet. The date and time of the factory set-up will appear at the top (in red), followed by the headings "Field" and "Power". The values of these should be within 20% of those in the set-up. Finally a matrix of numbers will appear, which shows the various valve timing settings used - these should correspond EXACTLY with those in the Specification Sheet.

Press the "LIST" key. The response is "LIST LIBRARY #?". Press the numeric key corresponding to the library on the Specification Sheet, then "ENTER". A List of all the compounds stored in this library is printed, along with ID numbers, retention times and limit values.

Depress the "GAIN" key and the response will be "SET GAIN TO? 2." Press the "UP ARROW" key until the gain number corresponds to the Specification Sheet, then immediately press "ENTER".

If you are using Photovac's isothermal GC capillary column oven assembly, you have to connect a battery pack or external 12 V power supply to the 10S "EXT DC" Top panel receptacle. The oven assembly is located beneath the module, and you should open the module so that you can see the oven unit.

There are a total of 4 temperature settings available, and each selection has a light beside it. The corresponding light will be on if it is selected. Check if it is the temperature that you want; otherwise, change to your desired temperature by turning the switch.

Depress the "CYCLE" key. The response will be "TIMER DELAY?" and giving a time in seconds (check this with the Specification Sheet); press ENTER and a new response "ANALYSIS TIME" - with a value - will appear. Press "ENTER" and "CYCLE TIME? Min 0" will appear. Check all values in memory against the Specification Sheet as you go, use the numerical key-pad and the "CLEAR" key to make any necessary adjustments. Detailed explanations of these functions appear in Chapter (4).

Decide where you want to locate the unit, this should ideally be a low-traffic area, where the instrument will not be tampered with by unauthorized persons and where the temperature is reasonably constant. Because your unit is self-calibrating, there is no need to worry about small temperature changes.

All that now remains to be done is to connect up the supply of calibration gas. It is important that this meets your Specification Sheet requirement. For example, if your routine monitoring is for vinyl chloride (VCM), the Specification Sheet may say "Calibrant is 1.0 ppm of VCM in air"; in this case, you must make sure that this is the calibrant you are going to use. If you are using an external, pressurized tank, this should be equipped with a reducing regulator to deliver the contents at 5 psi (35 kPa); the delivery line (Teflon or stainless steel) should be securely connected to the "CAL IN" port, using Swagelok fittings. IF YOUR CALIBRANT IS TOXIC, MAKE SURE YOU VENT THE EXCESS BY CONNECTING THE "PUMP OUT" PORT TO A VENTED LINE. If possible, purge the calibrant line before connecting.

Press the "USE" key and ensure the library in use is the one listed on the Specification Sheet. Press "ENTER" and put in the correct date and time.

NOTE, the 10S50 uses a 24-hour clock! 8:00 pm will be 20 Hrs.

Allow 20-30 minutes warm up time, especially when the isothermal capillary column oven is used, after initial switch-on of the power. This is because the Ultraviolet light source needs time to get ready, and the heater requires certain time to reach the desired temperature, and to achieve steady state. You are now ready to begin monitoring!

Press the key marked "START STOP P", the message shown will be "INLET ? (USE <>)" followed by "PROBE IN". Change the selection to "CAL IN" by pressing "DOWN ARROW". Then press "ENTER", the analysis will begin.

As the analysis continues, the chromatogram will appear in red on the plotter. You should see a "peak" on the chromatogram which correspond to the calibrant in your calibration gas. 10S50 automatically numbers and integrates the peak.

At the end of the analysis, the library number, date, and time are printed, along with analysis number, internal temperature, and gain. This block of information is called the ANALYSIS INFORMATION.

Beneath it is the SETUP INFORMATION, which lists the settings of 10S50's "SETUP" and "CYCLE" keys.

Last is a block of PEAK INFORMATION, in which the peaks are listed by name (or as "UNKNOWN"), peak number, retention time, and peak area or concentration.

You now need to update the retention times and response factors stored in the library to reflect your actual calibrant peak.

Press "CAL". At "PLOTTER PEAK #? ENTER TO RELIST", type the number which 10S50 has printed in red beside the calibrant peak and press "ENTER". At "ID NUMBER 1-25?", enter the library ID number (from the blue library listing you printed earlier) which corresponds to the name of your calibrant. At "CONC. IN PPM?", enter your calibrant concentration.

10S50 will now update all its retention times and response factors in this library, and print out a new set of analysis, setup, and peak information. Your calibrant is now identified, and its correct concentration is shown.

To start 10S50 cycling, press "CYCLE". Enter the "TIMER DELAY" and "ANALYSIS TIME" as they stand. At "CYCLE TIME? Min", enter the time you wish 10S50 to wait between starting one analysis and another. At "CAL AT ? CYCLES", enter 2 to have 10S50 alternate between sample and calibration analyses. At "ID NUMBER 1-25?", the library ID number of your calibrant appears. Enter it. At "CONC. IN PPM?" the concentration of your calibrant appears.

Enter it. At "TWA AT ? CYCLES", enter the number of samples you wish 10S50 to take before calculating the Time-Weighted-Average and maximum concentration of each compound. At this point 10S50 will print out a dummy TWA report and clear its TWA registers.

Now 10S50 will wait for one cycle time then begin cycling automatically. First it will calibrate itself and update the library information, then it will take a sample and look for your compounds of interest. If a concentration is measured which is higher than the limit value in the library, then the concentration will be printed in red to grab your attention.

After taking the specified number of samples, 10S50 will automatically print a TWA report.

Routine maintenance of your instrument consists of monitoring the consumable (air carrier gas and calibrant) and replacing these periodically. Be prepared to shut the unit down for about 20 mins after you have changed carrier gas, it will need this time to adjust to the new gas, which will generally have a different background-impurity level. You will also need to regularly change paper and pens on the printer/plotter; these are available from Photovac (Catalog No. SA302 - pens, No. SA303 paper). The frequency of this will depend upon your sampling frequency.

The GC columns in your instrument should last indefinitely; the lamp will require periodic replacement and it is a good idea to hold a spare in reserve (Catalog No. SA301).

APPENDIX C

Instruction

MI

611-132
December 1985

Model OVA 128 CENTURY Organic Vapor Analyzer

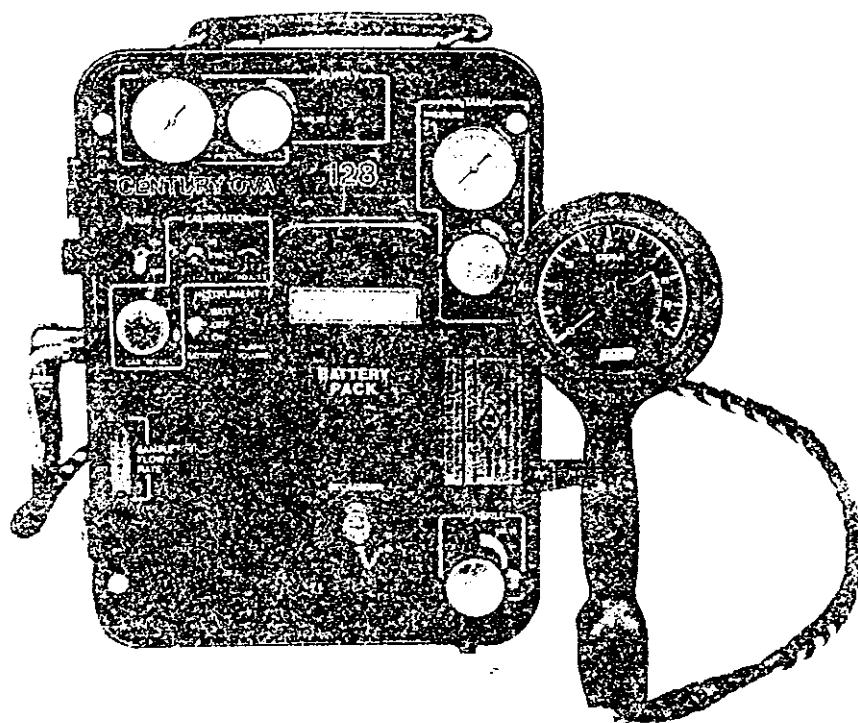


FIGURE 1
PORTABLE ORGANIC VAPOR ANALYZER

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INTRODUCTION

The Model OVA 128 CENTURY Organic Vapor Analyzer (OVA 128) is manufactured in three configurations. These are:

- Basic Flame Ionization Detector for monitoring total hydrocarbons
- Gas chromatograph supplied with two columns
- Gas chromatograph tri-column for Benzene Analysis.

A battery charger can be ordered for either 120 V ac, 60 Hz or for 220 V ac, 50 Hz. Classifications are:

- FM certified for use in Class I, Groups A, B, C, and D, Division I hazardous environments.
- BASEEFA certified intrinsically safe, Ex ib, for IIC, Zone 1, Temperature Class T6. BASEEFA No. 76002/B std. SFA 3007.

Accessories for the OVA 128 are:

- Strip Chart Recorder - either FM or BASEEFA certified.
- Activated Charcoal Filter Assembly - used for zeroing the analyzer in a contaminated environment. Also used with dessicant as a moisture trap.
- Sample Dilutor Assembly for 10:1, 25:1, or 50:1 sample dilution.
- Septum Adapter for direct, on-line injection with the GC.
- Portable Isothermal Pack (PIP) for temperature control of GC columns.

The OVA 128 is a sensitive instrument designed to measure trace quantities of organic materials in air. It is essentially a flame ionization detector such as utilized in laboratory gas chromatographs and has similar analytical capabilities. The flame ionization detector is an almost universal detector for organic compounds with the sensitivity to measure in the parts per million range (V/V) in the presence of atmospheric moisture, nitrogen oxides, carbon monoxide, and carbon dioxide.

The instrument has broad application since it has a chemically resistant air sampling system and can be readily calibrated to measure almost all organic vapors. It has a single linearly scaled readout from 0 ppm to 10 ppm with a X1, X10, and X100 range switch. This range expansion feature provides accurate readings across a wide concentration range with either 10, 100 or 1000 ppm full scale deflection. Designed for use as a portable survey instrument, it can also be readily adapted to fixed remote monitoring or mobile installations. It is ideal for the determination of many organic air pollutants and for monitoring the air in potentially contaminated areas.

The OVA 128 is certified by Factory Mutual Research Corporation (FM) for use in Class I, Groups A, B, C, & D, Division I hazardous locations. Similar foreign certifications have been obtained, including BASEEFA. This requirement is especially significant in industries where volatile flammable petroleum or chemical products are manufactured or used and for instruments which are used in portable surveying or for analyzing concentrations of gases and vapors. Such instruments must be incapable, under normal or abnormal conditions, of causing ignition of hazardous mixtures in the air. In order to maintain the certified safety, it is important that the precautions outlined in this manual be practiced and that no modifications be made to these instruments.

It is highly recommended that the entire manual be read before operating the instrument. It is essential that all portions relating to safety of operation and maintenance be thoroughly understood.

Reference Literature

- MI 611-101 Operation of Tri-Column GC Option
- MI 611-102 Operation of Dilutor Kit
- MI 611-105 Operation of Portable Isothermal Pack

GENERAL DESCRIPTION

The OVA 128 Analyzer is designed to detect and measure hazardous organic vapors and gases found in most industries. It has broad application since it has a chemically resistant sampling system and can be calibrated to almost all organic vapors. It can provide accurate indication of gas concentration in one of three ranges: 0 to 10 ppm; 0 to 100 ppm; or 0 to 1000 ppm. While designed as a lightweight portable instrument, it can be permanently installed to monitor a fixed point.

The instrument utilizes the principle of hydrogen flame ionization for detection and measurement of organic vapors. The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated. During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system.

The sample stream is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber, the sample is exposed to a hydrogen flame which ionizes the organic vapors. When most organic vapors burn, they leave positively charged carbon-containing ions. An electric field drives the ions to a collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated. This current is measured with a linear electrometer preamplifier which has an output signal proportional to the ionization current. A signal conditioning amplifier is used to amplify the signal from the preamp and to condition it for subsequent meter or external recorder display. The display is an integral part of the Probe/Readout Assembly and has 270° scale deflection.

In general, the hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives repeatable results with all types of hydrocarbons; i.e., saturated hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

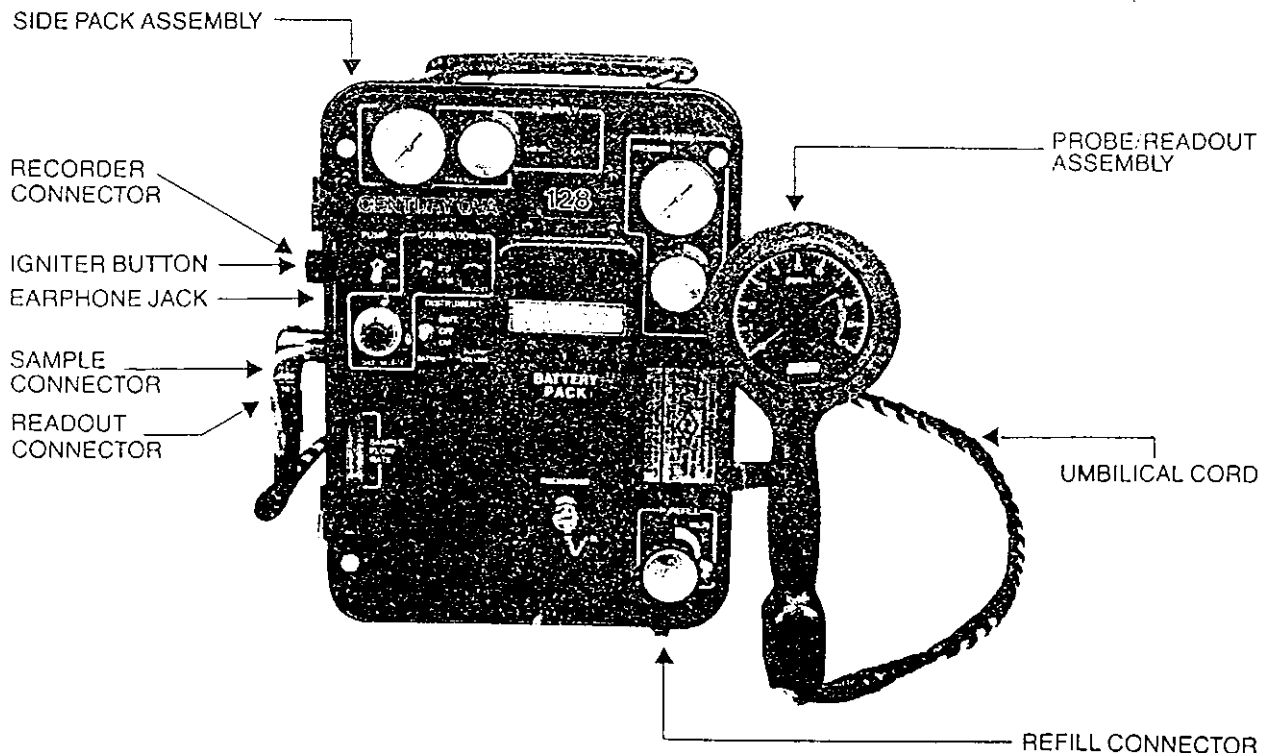


FIGURE 2
PORTABLE ORGANIC VAPOR ANALYZER
Model OVA 128

Typical response of various hydrocarbons, relative to methane is as follows:*

Compound	Relative Response (percent)
Methane	100 (reference)
Hexane	70
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Toluene	120
Ethane	90

Compounds containing oxygen, such as alcohols, ethers, aldehydes, carboxylic acid and esters give a lower response than that observed for hydrocarbons. This is particularly noticeable with compounds having a high ratio of oxygen to carbon such as the lower members of each series which have one, two or three carbons. With compounds containing higher numbers of carbons, the effect is diminished to such an extent that the response is similar to that of the corresponding hydrocarbons.

Nitrogen-containing compounds (i.e., amines, amides, and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Thus, CHCl_3 gives a much higher response than does CCl_4 . As in the other cases, when the carbon to halogen ratio is 5:1 or greater, the response will be similar to that observed for simple hydrocarbons.

*NOTE: Each OVA detector will have slightly different responses for organic vapors relative to methane. The user should determine responses for his individual instrument. The typical response of various compounds relative to methane is as follows:

KETONES	
Acetone	60
Methyl ethyl ketone	80
Methyl isobutyl ketone	100
ALCOHOLS	
Methyl alcohol	15
Ethyl	25
Isopropyl	65
HALOGEN COMPOUNDS	
Carbon tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

The OVA has negligible response to carbon monoxide and carbon dioxide which, due to their structure, do not produce appreciable ions in the detector flame. Thus, other organic materials may be analyzed in the presence of CO and CO_2 .

Applications

- (1) Measurement of most toxic organic vapors present in industry for compliance with Occupational Safety and Health Administration (OSHA) requirements.
- (2) Evaluation and monitoring applications in the air pollution field.
- (3) Source identification and measurement for fugitive emissions (leaks) as defined by EPA.
- (4) Forensic science applications.
- (5) Controlling and monitoring atmospheres in manufacturing and packaging operations.
- (6) Leak detection related to volatile fuel handling equipment.
- (7) Monitoring the background level of organic vapors at hazardous waste sites.
- (8) Quality control procedures geared to leak checking, pressurized system checks, combustion efficiency checks, etc.

Major Features

The basic instrument consists of two major assemblies, the Probe/Readout Assembly and the Side Pack Assembly (See Figure 2). The recorder is optional on all models, but is normally used with all instruments which incorporate the GC Option. The output meter and alarm level adjustments are incorporated in the Probe/Readout Assembly.

The Side Pack Assembly contains the remaining operating controls and indicators, electronic circuitry, detector chamber, hydrogen fuel supply, and electrical power supply.

Other major features are: linear scale readout, approximately two second response time and portable operating time of 8 hours for fuel supply and battery pack. A battery test feature allows charge condition to be read on the meter. Hydrogen flame-out is signified by an audible alarm plus a visual indication on the meter. The instrument contains a frequency modulated detection alarm which can be preset to sound at a desired concentration level. The frequency of the detection alarm varies as a function of detected level giving an audible indication of organic vapor concentration. An earphone is provided to allow the operator to hear the alarm in noisy areas or to avoid disturbing workers.

During use, the Side Pack Assembly can be carried by the operator on either his left or right side or as a back pack. The Probe/Readout Assembly can be detached from the Side Pack Assembly and disassembled for transport and storage.

Standard Accessories

A variety of sampling fixtures can be used. In addition, small diameter tubing can be used for remote sampling or electrically insulated flexible extensions can be used for places that are difficult to reach.

Telescoping Probe

Probe length can be increased or decreased over a 22 to 30 inch range to suit the individual user. A knurled locking nut is used to lock the probe at the desired length. The probe is attached to the Readout Assembly. When appropriate, the probe is replaced with a Close Area Sampler, which is supplied as a standard accessory.

Sampling Accessories

Part Number	Description
510125-1	Close area sampler - Connects directly to the readout assembly.
510035-1	Telescoping wand - Adjustable length - accommodates the probe listed below.
510126-1	Tubular area sampler - Used with the telescoping wand.

Particulate Filters

The primary filter of porous stainless steel is located behind the sample inlet connector (see Side Pack Assembly drawing). In addition, a replaceable porous metal filter is installed in the "close area" sampler.

Carrying Case

An instrument carrying case is provided to transport, ship and store the disassembled Probe/Readout Assembly, the Side Pack Assembly and other equipment.

Specifications

READOUT: 0 to 10, 0 to 100, 0 to 1000 ppm (linear)
SAMPLE FLOW RATE: 1 1/2 to 2 1/2 litre per minute at 22°C, 760 mm, using close area sampler
RESPONSE TIME: Approximately 2 seconds for 90% of final reading.
PRIMARY ELECTRICAL POWER: 12 volt (nominal) battery pack.
FUEL SUPPLY: Approximately 75 mL volume tank of pure hydrogen, maximum pressure 2400 psig, fillable in case.
HYDROGEN FLOW RATE: Factory set 12.5 ± 0.5 mL/min (minus GC option) 11.0 ± 0.5 mL/min (GC models)
PORTABLE OPERATING TIME: Minimum 8 hours with battery fully charged, hydrogen pressure at 1300 psig.
PHYSICAL DIMENSIONS: 9" x 12" x 5" (229 mm x 305 mm x 127 mm) Sidepack only.
WEIGHT: 12 pounds (5.5 kg) (sidepack and hand-held probe assembly)
DETECTION ALARM: Audible alarm plus meter indication. User preset to desired level.
FLAME-OUT ALARM: Audible alarm plus meter indication (needle drops off scale in negative direction).
BATTERY TEST: Battery charge condition indicated on readout meter. Upon activation of momentary contact switch, a meter reading above the indicator line means that there is 4 hours minimum service life remaining (at 22°C).
FILTERS: In-line sintered metal filters will remove particles larger than 10 microns.
OPERATING TEMPERATURE RANGE: 10°C to 40°C.
MINIMUM AMBIENT TEMPERATURE: 15°C for Flame Ignition (coldstart).
ACCURACY: Based on the use of a calibration gas for each range:

Calibration Temp. °C	Operating Temp. °C	Accuracy in % of Individual Full Scale		
		X1	X10	X100
20 to 25	20 to 25	+20	+10	+10
20 to 25	10 to 40	+20	+20	+20

RELATIVE HUMIDITY: 5% to 95%, Effect
on accuracy: $\pm 20\%$ of individual
full scale

RECORDER OUTPUT: 0 to 5 volts

MINIMUM DETECTABLE LIMIT (METHANE):
0.2 ppm

STANDARD ACCESSORIES:

1. Instrument carrying and
storage case
2. Hydrogen fuel filling hose
assembly
3. Battery charger
4. Earphone
5. Various sampling fixtures
6. Maintenance tool kit
7. Operators manual (2 each)
8. Padded leather carrying straps

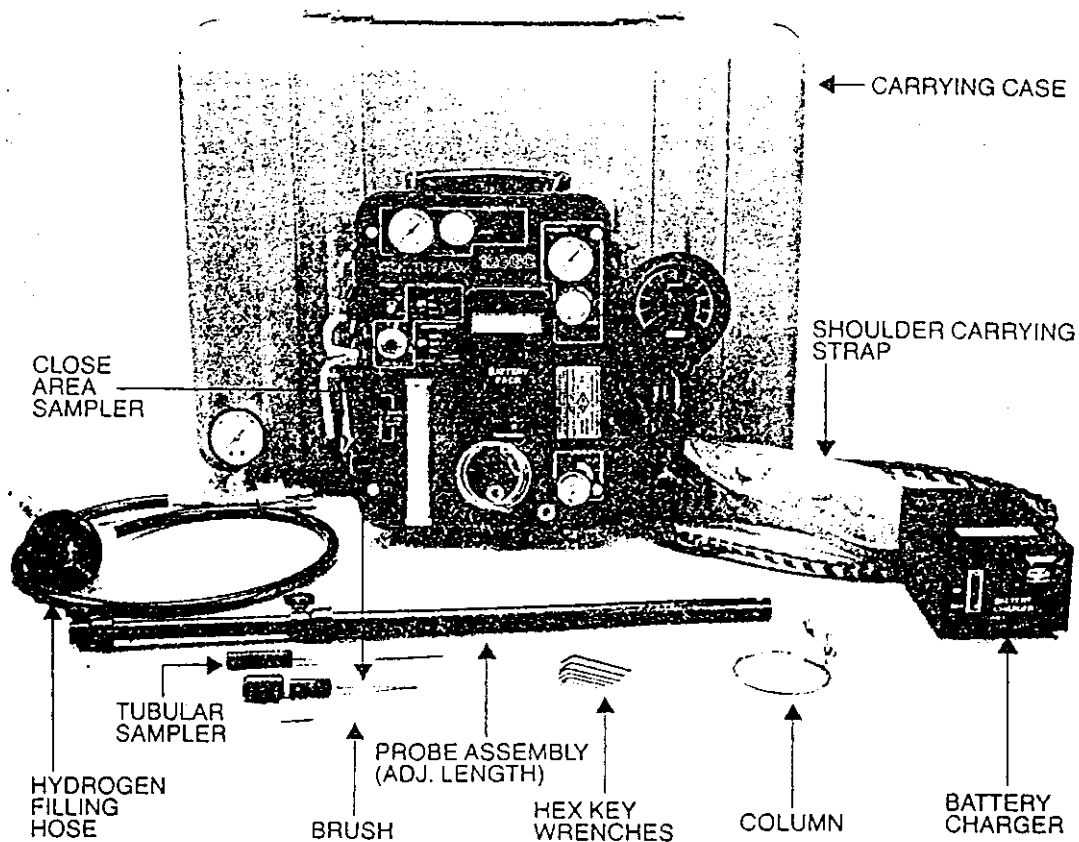


FIGURE 3
OVA-128 ANALYZER COMPONENTS
(Gas Chromatograph Model Shown)

OPERATING PROCEDURES

Controls and Indicators

Sidepack Assembly

- 1) INSTR/BATT Test Switch* - Three position toggle switch controls all instrument electrical power except the pump and alarm power. It also permits display of the battery charge condition on the readout meter.
- 2) PUMP (ON/OFF) Switch* - Toggle switch controls power to the internal pump and audio alarms.
- 3) Igniter Switch - Momentary push button switch connects power to the igniter coil in the detector chamber and simultaneously disconnects power to pump.
- 4) CALIBRATE Switch (range selector) - Selects the desired range: X1 (0 to 10 ppm); X10 (0 to 100 ppm); X100 (0 to 1000 ppm).
- 5) CALIBRATE ADJUST (zero) Knob - Potentiometer used to "zero" the instrument.
- 6) GAS SELECT KNOB (span control) - Ten-turn dial readout potentiometer sets the gain of the instrument (commonly referred to as span control).
- 7) Recorder Connector - Five-pin connector used to connect the instrument to an external recorder with the following pin connections:
 - Pin E - + 12 V dc
 - Pin H - Ground
 - Pin B - Signal 0 to 5 V dc
- 8) Charger Connector - BNC connector used to connect the battery pack to the battery charger.
- 9) HYDROGEN TANK VALVE - Valve used to supply or close off the fuel supply from the hydrogen tank.
- 10) HYDROGEN TANK PRESSURE Indicator - High pressure gauge measures pressure in the hydrogen fuel tank which is an indication of fuel supply.
- 11) HYDROGEN SUPPLY VALVE - Valve used to supply or close off hydrogen fuel to the detector chamber.

- 12) HYDROGEN SUPPLY PRESSURE Indicator - Low pressure gauge used to monitor hydrogen pressure at the capillary restrictor.
- 13) SAMPLE FLOW RATE Indicator - Indicator to monitor the sample flow rate.
- 14) REFILL CONNECTION - $\frac{1}{4}$ in AN fitting to connect the hydrogen refill hose to the instrument.
- 15) REFILL VALVE - Valve to open one end of the instrument fuel tank for refilling with hydrogen.
- 16) EARPHONE JACK - Used to connect the earphone; speaker is disabled when earphone is used.
- 17) VOLUME Knob - Potentiometer adjusts the volume of the internal speaker and earphone.
- 18) Readout and Sample Connectors - Used to connect the sample hose and umbilical cord from the Probe/Readout to the Side Pack.

Controls and Indicators

Probe/Readout Assembly

- 1) Meter - Linear scaled 270° meter displays the output signal level in ppm.
- 2) Alarm Level Adjust Knob - Potentiometer (located on the back of the Readout Assembly) is used to set the concentration level at which the audible alarm is actuated.

*Special Switch - switch handle must be pulled to change position. This prevents accidental movement.

Startup Procedure

- a) Connect the Probe/Readout Assembly to the Sidepack Assembly by attaching the sample line and electronic jack to the Sidepack.
 - b) Select the desired sample probe (close area sampler or telescoping probe) and connect the probe handle. Before tightening the knurled nut, check that the probe accessory is firmly seated against the flat seals in the probe handle and in the tip of the telescoping probe.
 - c) Move the Instr/Batt Switch to the test position. The meter needle should move to a point beyond the white line, indicating that the integral battery has more than 4 hours of operating life before recharging is necessary.
 - d) Move the Instr/Batt Switch to the "ON" position and allow a 5 minute warm-up.
 - e) Turn the Pump Switch on.
 - f) Use the Calibrate Adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the Calibrate Switch must be set to the appropriate range.
 - g) Turn the Volume Knob fully clockwise.
 - h) Using the Alarm Level Adjust knob, turn the knob until the audible alarm is activated.
 - i) Move the Calibrate Switch to X1 and adjust the meter reading to zero using the Calibrate Adjust (zero knob).
 - j) Open the hydrogen Tank Valve 1 or 2 turns and observe the reading on the Hydrogen Tank Pressure Indicator. (Approximately 150 psi of pressure is required for each hour of operation).
 - k) Open the Hydrogen Supply Valve 1 or 2 turns and observe the reading on the Hydrogen Supply Pressure Indicator. The reading should be between 8 and 12 psi.
- 1) After approximately one minute, depress the Igniter Button until the hydrogen flame lights. The meter needle will travel upscale and begin to read "Total Organic Vapors". [Caution: Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.]
- m) The instrument is ready for use. NOTE: If the ambient background organic vapors are "zeroed out" using the Calibrate Adjust knob, the meter needle may move off-scale in the negative direction when the OVA is moved to a location with lower background. If the OVA is to be used in the 0 to 10 ppm range, it should be "zeroed" in an area with very low background. A charcoal filter (Part No. 510095-1) can be used to generate the clean background sample.

Operating Procedures

The following procedure describes operation of the OVA in the "Survey Mode" to detect total organic vapors.

- a) Set the CALIBRATE Switch to the desired range. Survey the areas of interest while observing the meter and/or listening for the audible alarm indication. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. For broad surveys outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative readings or pinpointing, the pickup fixture should be positioned at the point of interest.
- b) When organic vapors are detected, the meter pointer will move upscale and the audible alarm will sound when the setpoint is exceeded. The frequency of the alarm will increase as the detection level increases.

Note: With GC instrument, a column or jumper must be installed.

If the flame-out alarm is actuated, check that the pump is running, then press the igniter button. Under normal conditions, flame-out results from sampling a gas mixture that is above the lower explosive level which causes the hydrogen flame to extinguish. If this is the case, reignition is all that is required to resume monitoring. Another possible cause for flame-out is restriction of the sample flow line which would not allow sufficient air into the chamber to support combustion. The normal cause for such restriction is a clogged particle filter.

It should be noted that the chamber exhaust port is on the bottom of the case and blocking this port with the hand will cause fluctuations and/or flame-out.

Shut Down Procedure

The following procedure should be followed for shut down of the equipment:

- A. Close HYDROGEN TANK VALVE
- B. Close HYDROGEN SUPPLY VALVE
- C. Move INSTR Switch to OFF
- D. Wait 5 seconds and move PUMP Switch to OFF. INSTRUMENT IS NOW IN A SHUT DOWN CONFIGURATION.

Fuel Refilling

NOTE: Use PREPURIFIED or ZERO grade hydrogen (certified total hydrocarbons as methane <0.5 ppm recommended).

- a) The instrument and the charger should be completely shut down during hydrogen tank refilling operations. Refilling should be done in a ventilated area. THERE SHOULD BE NO POTENTIAL IGNITERS OR FLAME IN THE AREA.
- b) If you are making the first filling on the instrument or if the filling hose has been allowed to fill with air, the filling hose should be purged with hydrogen prior to filling the instrument tank. This purging is not required for subsequent fillings.
- c) The filling hose assembly should be left attached to the hydrogen supply tank when possible. Ensure that the FILL/BLEED Valve on the instrument end of the hose is in the OFF position. Connect the hose to the refill connection on the Side Pack Assembly.

- d) Open the hydrogen supply bottle valve slightly. Open the REFILL VALVE and the HYDROGEN TANK VALVE on the instrument panel and place the FILL/BLEED Valve on the filling hose assembly in the FILL position. The pressure in the instrument tank will be indicated on the HYDROGEN TANK PRESSURE Indicator.
- e) After the instrument fuel tank is filled, close the REFILL VALVE on the panel, the FILL/BLEED Valve on the filling hose assembly and the hydrogen supply bottle valve.
- f) The hydrogen trapped in the hose should now be bled off to atmospheric pressure. CAUTION should be used in this operation as described in Step (g) below, since the hose will contain a significant amount of hydrogen at high pressure.
- g) The hose is bled by turning the FILL/BLEED Valve on the filling hose assembly to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED Valve should be turned to the FILL position to allow the hydrogen trapped in the connection fittings to go into the hose assembly. Then, again, turn the FILL/BLEED Valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED Valve to OFF to keep the hydrogen at one atmosphere in the hose so that at the time of the next filling there will be no air trapped in the filling line.
- h) Close the HYDROGEN TANK VALVE.
- i) With the HYDROGEN TANK VALVE and the HYDROGEN SUPPLY VALVE closed, a small amount of HYDROGEN at high pressure will be present in the regulators and plumbing. As a leak check, observe the HYDROGEN TANK PRESSURE Indicator while the remainder of the system is shut down and ensure that the pressure reading does not decrease rapidly (more than 350 psi/h) which would indicate a significant leak in the supply system.

Battery Charging

WARNING: Never charge in a hazardous environment.

- a) Plug charger connector into mating connector on battery cover and insert ac plug into 115 V ac wall outlet.
- b) Move the battery charger switch to the ON position. The lamp above the switch button should illuminate.
- c) Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the left when charging. When fully charged, the pointer will be in line with "charged" marker above the scale.
- d) Approximately one hour of charging time is required for each hour of operation. However, an overnight charge is recommended. The charger can be left on indefinitely without damaging the batteries. When finished, move the battery charger switch to OFF and disconnect from the Side Pack Assembly.

THE FOLLOWING ARE SPECIAL INSTRUCTIONS FOR RECHARGING BATTERIES WHICH HAVE BEEN COMPLETELY DISCHARGED.

It has been established that the above battery charging procedures may not be effective when the operator has allowed the battery to COMPLETELY discharge.

When this happens and the above procedures fail to charge the battery, perform the following additional steps:

- e) Remove the battery from the instrument case.
- f) Connect to any variable dc power supply.
- g) Apply 40 volts at $\frac{1}{2}$ ampere maximum.
- h) Observe the power supply meter. As soon as the battery begins to draw current, gradually reduce the power maintaining $\frac{1}{2}$ A maximum until the meter reads approximately 15 volts.

NOTE: The time required to reach the 15 volt reading will depend on degree of discharge.

- i) Repeat steps (a), (b), (c), and (d) above to complete the charging cycle.

SUMMARY OF OPERATING PROCEDURES

Start Up

- a) Check battery condition by moving the INSTR Switch to the BATT position.
- b) Move INSTR Switch to ON and allow five (5) minutes to warm-up.
- c) Use the Calibrate Adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the Calibrate Switch must be set to the appropriate range.
- d) Turn the Volume Knob fully clockwise.
- e) Using the Alarm Level Adjust knob, turn the knob until the audible alarm is activated.
- f) Set CALIBRATE Switch to X1 position, use CALIBRATE Knob and set meter to read 0.
- g) Move PUMP Switch to ON position, then place instrument panel in vertical position and check SAMPLE FLOW RATE indication. The normal range is 1.5 to 2.5 units. If less, check filters.
- h) Open the HYDROGEN TANK VALVE and the HYDROGEN SUPPLY VALVE. Wait one minute for hydrogen to purge the system.
- i) Depress Igniter Button until burner lights. Do not depress Igniter Button for more than six seconds. (If burner does not ignite, let hydrogen flow for one minute and again attempt ignition.)
- j) Use CALIBRATE Knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set CALIBRATE Switch to X1 and readjust zero on meter. To avoid false flame-out alarm indication, set meter to 1 ppm with CALIBRATE Knob and make differential readings from there.

Shut Down

- a) Close the HYDROGEN SUPPLY VALVE
- b) Close the HYDROGEN TANK VALVE
- c) Move the INSTR Switch and PUMP Switch to OFF
- d) Instrument is now in shut down configuration

CALIBRATION

Recalibration to Various Organic Vapors

The OVA 128 is capable of responding to nearly all organic compounds. At the time of manufacture, the analyzer is calibrated to mixtures of methane in air. For precise analysis it is necessary to recalibrate with the specific compound of interest. The GAS SELECT control is used to set the electronic gain for a particular compound.

The instrument is recalibrated using a mixture of a specific vapor in air, with known concentration. After the instrument is in operation and the normal background is zeroed, draw a sample of the calibration gas into the instrument. The GAS SELECT Knob on the panel is then used to set the read-out meter indication to correspond to the concentration of the calibration gas mixture.

The instrument has now been calibrated to the vapor mixture being used. After this adjustment, the setting on the "digital" should be recorded for that particular organic vapor compound. This exercise can be performed for a large variety of compounds, thereby generating a "library" which can be used for future reference without need for additional calibration standards.

To read a particular compound, the GAS SELECT control is turned to the predetermined setting for the compound. Calibration on any one range automatically calibrates the other two ranges.

Using Empirical Data

Relative response data can be used to estimate the concentration of a vapor without need to recalibrate the analyzer. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The response factor (R) in percent, for that vapor is:

$$R = \frac{\text{Actual Concentration}}{\text{Measured Concentration}}$$

To determine the concentration of an unknown sample of that vapor, multiply the measured concentration by R.

Calibration Standards

Commercial Standards

Commercially available standard samples offer the most convenience and are recommended for the most precise analyses. Always remember to obtain the desired vapor in an air background. Samples should be drawn from the cylinder into a collapsed sample bag, then drawn from the bag by the instrument to prevent a pressure or vacuum at the sample inlet.

Preparation of Standards

The following procedure is for generating calibration standards as an alternative to using commercial mixtures.

Obtain a five (5) gallon glass bottle and determine its volume by measuring the volume of water needed to fill it (use of a 1000 mL graduated cylinder is convenient). Another approach is to weigh the empty bottle, fill it with water and weigh again. The difference between the two values is the weight of water. By multiplying the weight of water in pounds by 0.455, obtain the volume of the bottle in liters. Empty the water and allow the bottle to dry. Place a one-foot piece of Teflon tubing in the flask to aid in mixing the vapors uniformly with the air. The volume of such a bottle should be about 20 liters, which is 20,000 mL. If the volume were 20,000 mL, then a 2 mL sample of a gas would be equivalent to 200 mL per 2 million mL or 100 ppm (V/V). Use of a gas tight Syringe, readable in 0.01 mL, allows the preparation of mixtures in the 1-2 ppm range, which are sufficient for the quantitative estimation of concentrations. A plastic stopper is loosely fitted to the tip of the bottle. The needle of the syringe is placed inside the jug neck and the stopper squeezed against the needle to decrease leakage during sample introduction. Inject the sample into the bottle and withdraw the needle without removing the stopper. Tighten the stopper and shake the bottle for a few minutes with sufficient vigor that the plastic tubing in the bottle moves around to ensure good mixture of the vapors with the air.

Calculations

$$\begin{aligned} \text{Injection} &= \frac{\text{Volume Concentration} \times \text{Molecular Weight} \times \text{System Volume}}{\text{Density} \times \text{Molar Volume at STP}^*} \\ &= \frac{(C) (MW) (V)}{(D) (V)} \end{aligned}$$

Using the Ideal Gas Law, $PV=RT$, the molar volume of any gas at STP (25°C and 1 atm) is:

$$\begin{aligned} V &= \frac{RT}{P} = \frac{\text{Universal Gas Constant} \times \text{Temperature}}{\text{Pressure}} \\ &= \frac{(0.08206 \frac{\text{litre atm}}{\text{mol K}}) (298.15 \text{ K})}{1 \text{ atm}} \\ &= (24.47 \text{ L}) (\text{mol}^{-1}) \end{aligned}$$

Therefore, the injection volume necessary to prepare 1 liter of a 100 ppm sample of hexane would be:

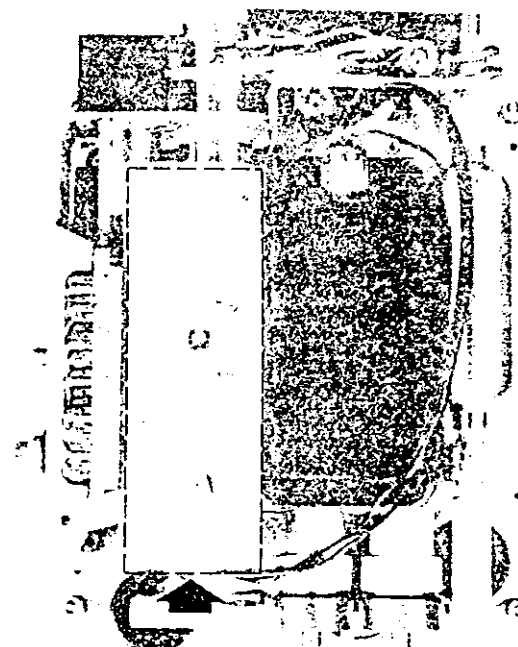
$$\begin{aligned} \text{Injection Volume} &= \frac{(100 \text{ ppm}) [(86.18 \text{ g}) (\text{mol}^{-1})] (1 \text{ liter})}{[(0.659 \text{ g}) (\text{mL}^{-1})] [(24.47 \text{ L}) (\text{mol}^{-1})] [(1000 \text{ mL}) (1^{-1})]} \\ &= 0.534 \mu\text{L} \end{aligned}$$

* STP - Standard Temperature and Pressure

Primary Calibration for Methane

Internal electronic adjustments are provided to calibrate and align the circuits. After initial factory calibration, it should not be necessary to repeat the calibration unless the analyzer undergoes repairs which affect calibration. If the OVA 128 will be extensively used for analysis of a sample other than methane, recalibration of the electronics (after resetting the GAS SELECT CONTROL) may result in better accuracy. See Recalibration to Various Organic Vapors above.

Primary calibration of this instrument is accomplished at the factory using methane-in-air, sample gases.



R-31 R-32 R-33 R-38

FIGURE 4
LOCATION OF ELECTRONIC ADJUSTMENTS

Calibration Using Known Samples for Each Range (Refer to Figure 4)

The accuracy stated under Specifications is obtained when the instrument is calibrated with known concentrations for each range. Prepare separate samples of methane-in-air in these concentration ranges: 7 to 10 ppm, 90 to 100 ppm, and 900 to 1000 ppm. Calibrate the instrument as follows:

- a) Place the instrument in normal operation and allow a minimum of 15 minutes for warm-up and stabilization.
- b) Set the GAS SELECT control to 300.
- c) Set the CALIBRATE Switch to X1.
- d) Set the CALIBRATE ADJUST (zero) Knob so that the meter reads zero.
- e) Check that the meter reads zero on the X10 and X100 ranges.
- f) Set the CALIBRATE Switch to X1 and introduce the sample with known concentration in the 7 to 10 ppm range.
- g) Adjust R31 so that the meter reading corresponds to the sample concentration.
- h) Set the CALIBRATE Switch to X10 and introduce the sample with known concentration in the 90 to 100 ppm range.
- i) Adjust R32 so that the meter reading corresponds to the sample concentration.
- j) Set the CALIBRATE Switch to X100 and introduce the sample with known concentration in the 900 to 1000 ppm range.
- k) Adjust R33 so that the meter reading corresponds to the sample concentration.
- l) The instrument is now calibrated for methane and ready for service.

Calibration Using a Single Sample Calibration (Refer to Figure 4)

Calibration may be accomplished using a single known sample of methane in air in the range of 90 to 100 ppm. This may not provide the accuracy stated under specifications but is adequate for field survey work.

- a) Place instrument in normal operation with CALIBRATE Switch set to X10 and GAS SELECT control set to 300.
- b) Use the CALIBRATE ADJUST (zero) Knob to adjust the meter reading to zero.
- c) Introduce a methane sample of a known concentration (between 90 and 100 ppm not to exceed 100 ppm) and adjust trimpot R-32 so the meter reading corresponds to the known sample.
- d) This sets the instrument gain for methane with the panel mounted gain adjustment (GAS SELECT) set at a reference number of 300.
- e) Turn off HYDROGEN SUPPLY VALVE to put out flame.
- f) Leave CALIBRATE Switch on X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to 4 ppm.
- g) Place CALIBRATE Switch in X1 position and using trimpot R-31 adjust meter reading to 4 ppm.
- h) Move CALIBRATE Switch to X10 position again. Use CALIBRATE ADJUST (zero) Knob to adjust meter to a reading of 40 ppm.
- i) Move CALIBRATE Switch to X100 position and use trimpot R-33 to adjust meter reading to 40 ppm.
- j) Move CALIBRATE ADJUST (zero) Knob to adjust meter reading to zero.
- k) Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

SAFETY PRECAUTIONS

The OVA 128 has been tested and certified by Factory Mutual Research Corporation (FM) as safe for use in Class I, Division 1, Groups A, B, C and D hazardous atmospheres. Similar foreign certifications have been obtained, including BASEEFA. Special restrictions must be strictly adhered to, to ensure the certification is not invalidated by actions of operating or service personnel.

All flame ionization hydrocarbon detectors are potentially hazardous since they use hydrogen or hydrogen mixtures in the detector cell. Mixtures of hydrogen and air are flammable over a wide range of concentrations whether an inert gas such as nitrogen is present or not. Therefore, the recommended precautions and procedures should be followed for maximum safety. Safety considerations were a major factor in the design of the Organic Vapor Analyzer (OVA).

All connections are of the permanent type as opposed to quick disconnect. To protect against external ignition of flammable gas mixtures, the flame detection chamber has porous metal flame arrestors on the sample input and the exhaust ports as well as on the hydrogen inlet connector. The standard battery pack and other circuits are internally current limited to an intrinsically safe level.

No Modifications Permissible

It is imperative that operation and service procedures described in this manual be carefully followed in order to maintain the intrinsic safety which is built into the OVA. NO MODIFICATION TO THIS INSTRUMENT IS PERMISSIBLE. Therefore, component replacement must be accomplished with approved parts.

Electrical Protection

The 12 V battery power supply circuit is current limited to an intrinsically safe level. Fuses are not utilized and all current limiting resistors and other components which are critical to the safety certification are encapsulated to prevent inadvertent replacement with components of the wrong value or specification. Under no circumstances should the encapsulation be removed.

Fuel Supply System

The OVA fuel tank has a volume of approximately 75 cm³ which, when filled to the maximum rated pressure of 2300 psig, holds approximately 5/9 ft³ of gas. The fuel used in the OVA should be PREPURIFIED or ZERO grade hydrogen (certified total hydrocarbons as methane <.5 ppm recommended.)

Hydrogen gas gains heat when expanding and, therefore, should not be rapidly released from a high pressure tank to a low pressure environment. Flow restrictors are incorporated in the hydrogen refill fitting and hydrogen is restricted on the output side of the tank by the low flow rate control system. In addition, a special flow restrictor is incorporated in the FILL/BLEED valve of the hydrogen filling hose assembly. These precautions limit the flow rate of the hydrogen to prevent ignition due to self-heat from expansion.

Precautions should be taken during hydrogen filling or hydrogen emptying operations to ensure that there are no sources of ignition in the immediate area. Since the instrument tank at 2300 psig holds only 5/9 ft³ of hydrogen, the total quantity, if released to the atmosphere, would be quickly diluted to a non-flammable level. There is, however, the possibility of generating flammable mixtures in the immediate vicinity of the instrument during filling or emptying operations if normal care is not exercised.

Detector Chamber

The input and output ports of the flame ionization chamber have sintered metal flame arrestors. The chamber is ruggedly constructed of Teflon such that even if highly explosive mixtures of hydrogen and air are inadvertently created in the chamber and ignited, the chamber would NOT rupture.

MAINTENANCE

This section describes the routine maintenance schedule and provides procedures for trouble-shooting an instrument malfunction.

CAUTION: Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all portions of this manual relating to safety of operation, servicing and maintenance, be thoroughly understood. There should be no potential igniters or flame in the area when filling, emptying or purging the hydrogen system and the instrument should be turned off.

Extreme care should be exercised to ensure that required parts replacement is accomplished with the parts specified by Foxboro. NO MODIFICATIONS ARE PERMITTED. DISASSEMBLE INSTRUMENT ONLY IN A NON-HAZARDOUS ATMOSPHERE.

Routine Maintenance (Refer to Figure 5)

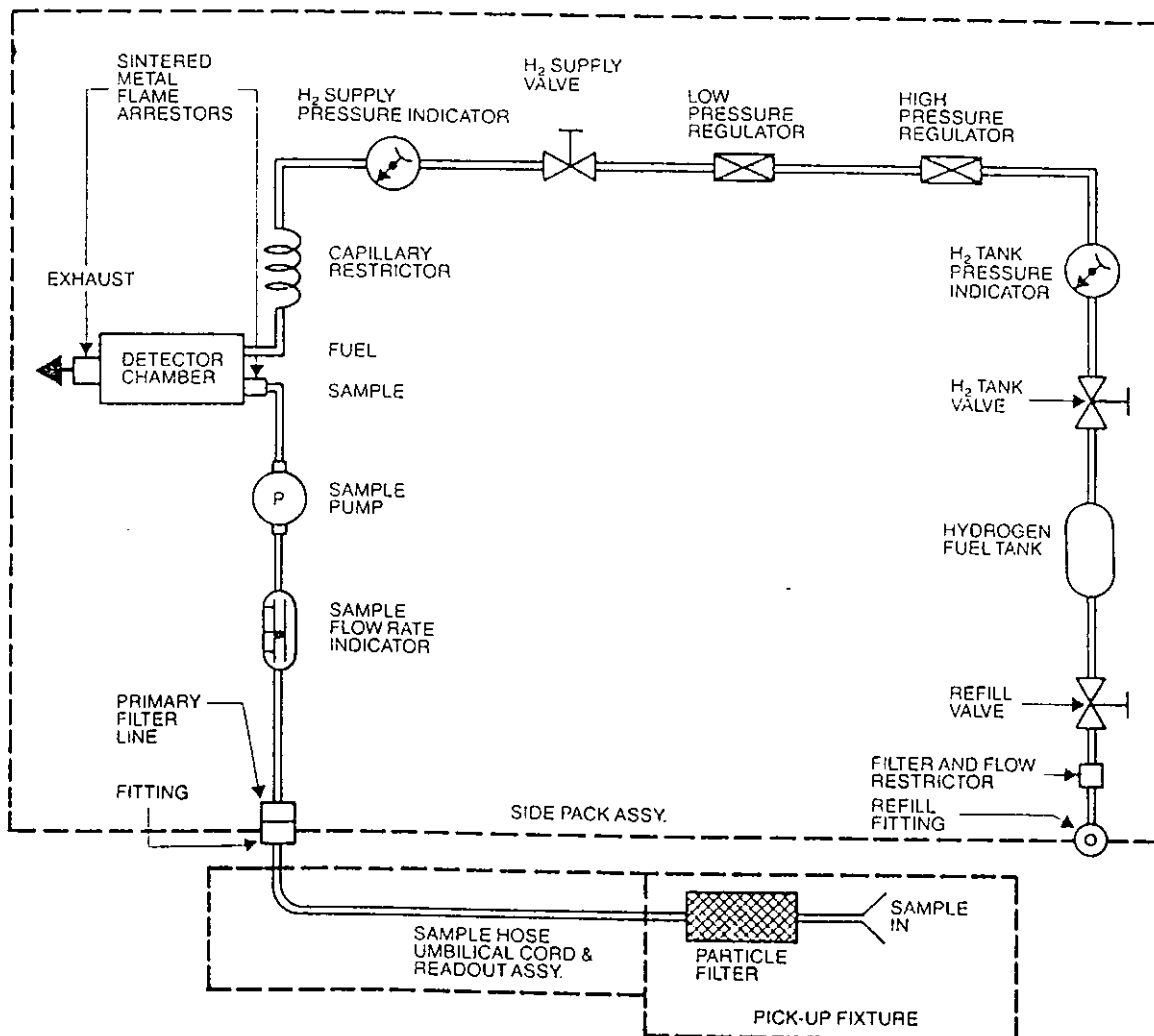


FIGURE 5
BLOCK DIAGRAM - GAS HANDLING SYSTEM

Primary Filter

This filter is located behind the sample inlet connector (Fitting Assembly) on the Side Pack Assembly and is removed for cleaning by using a 7/16 inch thin wall socket to unscrew the Fitting Assembly. The filter cup, "O" ring and loading spring will then come out. The porous stainless filter cup can be cleaned by blowing out or washing in solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter. Reassemble in reverse order ensuring that the "O" ring seal on the Fitting Assembly is intact.

Secondary Filter

A particle filter is located in each pick-up fixture. One of these filters must be in the sample line whenever the instrument is in use. The OVA 123 uses a porous metal filter which can be replaced or cleaned.

Mixer/Burner Assembly Filter

A porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. This filter is used as the sample mixer and inlet flame arrestor in the chamber. The filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

Access to this filter or output surface does not require removing the instrument from the case. For access, remove the safety cover using a hex key wrench (supplied) then unscrew the exhaust port. The Filter Assembly can now be seen on the side of the chamber (Preamp Assembly) and can be cleaned with a small wire brush.

Exhaust Flame Arrestor

A porous metal flame arrestor is located in the exhaust port of the detector chamber (Preamp Assembly). It acts as a particle filter on the chamber output and restricts foreign matter from entering the chamber. This filter may be cleaned by removing the exhaust port. For access, see Mixer/Burner section above. Note that the filter is captive to the exhaust port. Clean the filter with a solvent or detergent and ensure that it is dry and completely baked out at 120°F before reinstalling.

Sampling Fixtures

Sampling fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particle matter.

If a solvent is used, the fixture should be subsequently cleaned with detergent and baked out at 120°F to eliminate residual hydrocarbons from the solvent.

Hydrogen Tank Supply & Refill Valves

After some time, the Teflon washers under each valve packing nut can "cold flow" (move with pressure) and allow hydrogen to leak. Leakage can be determined by using Leak-Tec, Snoop or a soap solution around the valve stems. This leakage can usually be stopped by tightening the compression nut (adapter) as outlined below.

- a) Unscrew the packing nut with a 7/16 inch wrench
- b) Unscrew the valve
- c) Replace the compression rings

This compression is against soft material and only a small amount of force is necessary to sufficiently compress the Teflon washers. If, after tightening, leakage still occurs, it would be advisable to replace the two Teflon washers, as follows:

- a) Drain hydrogen system slowly and to the extent necessary to work on the leaking valve(s). Observe safety precautions. There should be no potential igniters in the area.
- b) Remove all three (3) knob screws and knobs.
- c) Remove the compression nut on the valve that is not sealing properly. Remove the stem by unscrewing it from the valve body. Observe the sandwich of metal and Teflon washers and note their order.
- d) Visually check the Kel-FTM seat on the stem for cracks or foreign material. Wipe clean, if necessary, with a lint free cloth (no solvents or oils) and replace if damaged.
- e) Remove the washers and replace the Teflon washers (the factory procedure is a light wipe of HYDRO-CARBON FREE silicone grease).
- f) Replace the stem assembly in the valve body and tighten lightly.

- g) Push the washers down into the compression area in the same order as noted upon removal. Replace the compression nut and tighten snugly.
- h) Close the low pressure valve and fill the tank assembly. Check valves for leaks. Tighten again, if necessary, and reassemble the unit.

Air Sampling System Maintenance

A potential problem associated with the OVA instrument is that leaks can develop in the air sample pumping system. These leaks can result in dilution or loss of sample, causing low reading of vapor concentration and slow response.

The OVA is equipped with a flow gauge that provides a method to check for air leaks. Assemble the pickup probe selected for use to the readout assembly and then position the sidepack vertically so the flow gauge may be observed. Cover the end of the pickup probe with your finger and observe that the ball in the flow gauge goes to the bottom, indicating no air flow (if ball has slight chatter while on bottom, this is acceptable). Cover the center of the chamber exhaust port with your thumb and again observe the ball going to the bottom. Another simple check is to expose the pickup probe to cigarette smoke or a light vapor (butane) and observe that the meter responds in approximately 2.0 seconds. It should be noted that slow meter response may also indicate a restriction in the air sampling system.

Failure of the ball to go to the bottom when the inlet is blocked indicates a leak in the system between the probe and the pump inlet or the inlet check valve. To isolate the problem, remove parts, one at a time, and again block off the air inlet. Remove the pickup probe(s) and cover the air inlet at the Readout Assembly. If the ball goes to the bottom, check that the "readout to probe" seal washer is in place and replace the probes, holding them back against this seal while tightening the nut. Recheck, and if leakage is still present, it is probably in the probe (pickup fixture), which should be repaired or replaced.

If leakage is indicated as being past the readout handle when the connection to the sidepack is tight, disconnect the sample line at the fitting on the sidepack and cover this inlet with your finger. If the flow gauge ball goes to the bottom, the problem should be a leak in the umbilical cord/Read-

out Assembly, which should be investigated and repaired. There is also the possibility of a leaking check valve in the pump which would not show up on this test. If the leakage is not found in the umbilical cord, it is most likely in the pump check valve. The pump should be replaced.

If the ball does not go to the bottom, the leak will be either in the flow gauge or it's connecting tubing. Visually check that the tubing is connected and if so, the flow gauge should be repaired or replaced. Check the "O" ring installation in the sample inlet connector (Fitting Assembly).

As an alternate approach, leaks on the inlet side of the pump can be detected by using alcohol on a "Q" Tip and lightly swabbing the connections one at a time or by directing organic vapor or smoke at the potential leakage points and observing the meter response or audible alarm.

Leaks (beyond the pump) are easier to locate, as any of the commercially available leak detection solutions can be used. Cover the exhaust port, which will place the exhaust system under pressure, and check each connection, one at a time. Replace the Teflon tubing or retape the threaded connections with Teflon joint tape. Check the igniter and Mixer/Burner Assembly where they screw into the detector, the high voltage terminal screw on the side of the Mixer/Burner and exhaust port itself. If after these checks, the flow gauge ball still will not go to the bottom with the exhaust blocked, the problem is likely a leaking exhaust check valve in the pump, which should be repaired or replaced.

Contaminating Control

On occasion, the background reading may be relatively high under normal ambient conditions. Ambient background readings will vary somewhat depending on the geographical location where the instrument is being used. However, the background reading normally should be in the range of 3 to 5 ppm as methane. The acceptable background reading consists of 1 to 15 ppm of methane which is present in the normal air environment. In addition to the measurement of a normal methane background, there will normally be 2 to 4 ppm of equivalent methane background caused by acceptable levels of contamination in the hydrogen fuel and/or hydrogen fuel handling system resulting in a total equivalent methane reading of 3 to 5 ppm in clean air.

If the background reading goes above 5 ppm to 6 or 7 ppm, this is normally still acceptable since any measurement is additive to that background reading, i.e., 2 ppm on top of 5 or 2 ppm on top of 7 provides the same differential reading, however, the lower background is obviously desirable.

The background reading is zeroed out or nulled - even though in reality the background still exists. The background reading is measured by zeroing the meter with the flame out and noting the meter indication after the flame is on.

The cause for a high background reading is usually associated with contamination in the hydrogen fuel system. This will, of course, cause a background reading since this is the function of the basic detector "to measure contamination entering the detector chamber". In addition, contamination present in the hydrogen will many times leave a small unobservable deposit on the burner face which can continue to generate a background reading when the detector is in operation and the burner assembly is heated.

Another possible cause of contamination is the Mixer/Burner Assembly when the contamination is trapped in the porous bronze sample filter. This is not a common problem and usually only happens when an unusually high level of contaminant is drawn into the assembly. Another possible cause of high background reading is contamination in the air sample line to the detector. This is uncommon but can be the source of the problem.

NOTE: An OVA that has the Chromatograph Option can have high background caused by saturation or contamination of the activated charcoal filter, which is in the line during chromatograph analysis, or of the column which is in the hydrogen line at all times.

Analysis and Correction

Prior to analyzing the problem, the OVA should be checked for proper electronic operation. It should be ensured that the instrument is calibrated to methane as referenced.

If, after checking that the OVA is properly calibrated, the background is still higher than normal for ambient conditions, the following procedure should be followed to isolate the cause of the problem:

- a) Let the OVA run for a period of time (15 to 30 minutes) and see if the background level decreases as a function of time. The background could go down as a result of clearing line contamination which is removable simply by the normal flow of air through the sample line.
- b) Take a reading in a known, relatively clean air environment. Normally, outside air environment is clean enough to assess by comparison whether the background reading is internal to the instrument or is present in the location where the instrument is being used.
- c) If the OVA has the Gas Chromatograph Option, depress the sample inject valve, so that the activated charcoal is in the line, and observe whether the background reading goes down and stays steady after elution of the air peak. The reading should always go down or stay the same but never increase when the sample valve is depressed, since the charcoal filter will remove trace elements of organic vapors in the air sample heavier than C_2 . If another activated charcoal filter is available, this may be attached to the end of the probe to scrub the air so that a clean air sample is supplied to the detector. The external activated charcoal filter can be used on any instrument, with or without chromatograph, for providing a clean air sample to assess background level.
- d) If the background cannot be reduced by any of the previous steps, remove the safety cover and the exhaust port of the detector chamber (on the bottom of the case) and clean the cavity and the electrode using the small wire brush supplied with the analyzer. This will remove any small quantities of contamination which could be the source of the background vapor. After cleaning, replace the exhaust port and safety cover and reignite the OVA. If detector contamination was the cause, the problem should be immediately resolved and the ambient background will drop to an acceptable level.

- e) If the high background is still present, the various parts of the sample flow line such as pickup probes, umbilical cord to the instrument, etc., should be investigated by the process of elimination to see if the contamination can be isolated.

Serious contamination in the air sample line is very uncommon, however, if very large doses of low vapor pressure compounds are sampled, there is a possibility of residual contamination. This would eventually clear itself out but may take a considerable period of time. A typical cause for high background from the sample line is a contaminated Mixer/Burner Assembly. If heavy contamination of the Mixer/Burner is indicated, replace the Mixer/Burner Assembly.

- f) In the event of contamination in the pump or other internal parts of the sample flow lines which cannot be removed, the sample flow components have to be disassembled and cleaned. This is normally a factory operation, however, components such as the pump can be replaced in the field along with any contaminated tubing.
- g) High background readings on OVA's which include the Gas Chromatograph Option can be caused by other sources of contamination. If the charcoal filter mounted on the instrument panel is saturated, contaminated air would be supplied to the detector and raise the ambient level background. To check for this, refill the cartridge with fresh charcoal, Foxboro P/N CSC004. This would determine if the charcoal was the source of the background reading. It is also possible that a high background reading could be due to contamination in the column. This could be caused by compounds slowly eluting from a column which has become contaminated. The easiest way to check for column contamination is to replace the column with a clean column or a short empty piece of column tubing and see if the high background reading drops.

- h) If the above steps do not correct the high background, the cause will normally be contamination in the hydrogen fuel system.

Contamination in the hydrogen fuel system is usually the direct result of contaminated hydrogen gas or contamination introduced during the filling operation. Filling hose contamination can be caused by storing the hose in a contaminated area.

To remove contamination, the fuel system should be purged with hydrogen. Effective purging is accomplished by disconnecting the capillary tube fitting to the manifold block which has the low pressure gauge (Hydrogen Supply Pressure Gauge and Hydrogen Supply Valve). This disconnects the capillary tubing from the hydrogen line so that hydrogen may be purged at a reasonable rate from the tank assembly through the regulators, gauges and valves. After disconnecting the capillary, the hydrogen tank can be filled in the normal manner. The tank valve and hydrogen supply valve can then be opened which will bleed the hydrogen from the tank through the hydrogen fuel system, purging contamination which is in vapor form. There is the possibility that contamination has been introduced into the hydrogen fuel system which is not readily purged by the hydrogen gas, but this is unlikely. After purging with clean hydrogen two or three times, the capillary tube should be reconnected and the background again checked. Five or ten minutes should be allowed before assessing the background reading, since contaminated hydrogen can be trapped in the capillary tube.

If another clean instrument is available, the fuel system from the clean instrument can be connected to the contaminated instrument to verify whether the problem is associated with the hydrogen fuel supply system. The interconnection should be made to the capillary tube of the contaminated instrument.

Troubleshooting

Table 1 presents a summary of field troubleshooting procedures. If necessary, the instrument can be easily removed from the case by unlocking the four (4) $\frac{1}{4}$ turn fasteners on the panel face and removing the refill cap. The battery pack is removed by taking out the four (4) screws on the panel and disconnecting the power connector.

Factory Maintenance

To ensure continuous trouble-free operation, a periodic factory maintenance, overhaul, and recalibration is recommended. The recommended schedule is every six to nine months. This maintenance program includes replacement of plastic seals and parts as required, pump overhaul, motor check, sample line cleaning, hydrogen leak check, recalibration, and detailed examination of the unit for any other required maintenance and repair.

Recommended Spare Parts

Item	Description	Part Number	Recommended Quantity
1	Igniter	510461-1	2
2	Pump Assembly	510223-6	1
3	Cup, Filter (3/8 inch OD, ss)	510318-1 (5/pkg.)	1
4	Mixer/Burner Assembly	510513-1	1
5	Wafer, Teflon, H ₂ Valve	510160-1 (10/pkg.)	1
6	Washer, Brass, H ₂ Valve	510160-2 (10/pkg.)	1
7	Exhaust Port Assembly	510530-1	1
8	Battery Pack Assembly	510542-1	1
9	Sample Line Assembly	510316-1	1
10	Particle Filters	510116-1	1

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator. Nominally 2 units on flow gauge. (See also 6 below)	a) Check primary filter in sidepack and particle filters in the pickup assembly. b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove Readout Assembly, remove primary filter, etc. c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output chamber, etc. NOTE: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when troubleshooting.	Replace or clean filter if clogged. Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required. If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace.
2) Hydrogen flame will not light. (See also 6 below)	a) Check sample flow rate (see 1 above) b) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE BUTTON is depressed. c) Check for rated Hydrogen Supply Pressure. (Listed on calibration plate on pump bracket). d) Check hydrogen flow rate by observing the psi decrease in pressure on the Hydrogen Tank Pressure gauge. The correct flow rate will cause about 130 psi decrease in pressure per hour. (Approximately 12 cm³/min at detector). e) Check all hydrogen plumbing joints for leaks using soap bubble solution. Also, shut off all valves and note pressure decay on hydrogen tank gauge. It should be less than 350 psi per hour.	If sample flow rate is low, follow procedure 1 above. If igniter does not light up, replace the plug. If igniter still does not light, check the battery and wiring. If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap. The most likely cause for hydrogen flow restriction would be a blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the Hydrogen Supply Pressure by one-half or one psi. If flow rate cannot be compensated for, replace capillary tubing. Repair leaking joint.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	f) Check to see if hydrogen supply system is frozen up by taking unit into a warm area. g) Remove exhaust port and check for contamination. h) Check spacing between collecting electrode and burner tip. Spacing should be 0.1 to 0.15 inches.	If there is moisture in the hydrogen supply system and the unit must be operated in subfreezing temperatures, purge the hydrogen system with dry nitrogen and ensure the hydrogen gas used is dry. If the chamber is dirty, clean with ethyl alcohol and dry by running pump for approximately 15 minutes. If hydrogen fuel jet is misaligned, ensure the porous metal flame arrestor is properly seated. Adjust by screwing Mixer/Burner Assembly in or out. This spacing problem should only occur after assembling a Mixer/Burner Assembly to a Preamp Assembly.
3) Hydrogen flame lights but will not stay lighted.	a) Follow procedures 2(a), (c), (d), (e), (g) and (h) above. Also refer to 5 below.	
4) Flame-out alarm will not go on when hydrogen flame is out.	a) Check instrument calibration setting and GAS SELECT control setting. b) Remove exhaust port and check for leakage current path in chamber (probably moisture or dirt in chamber). c) If above procedures do not resolve the problem, the probable cause is a malfunction in the preamp or power board assemblies. d) Check that volume control knob is turned up.	Readjust as required to proper setting. Note that the flame-out alarm is actuated when the meter reading goes below zero. Clean contamination and/or moisture from the chamber using a swab and alcohol, dry chamber by running pump for approximately 15 minutes. Return preamp chamber or power board assembly to the factory for repair. Adjust for desired volume.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
5) False flame-out alarm.	a) Flame-out alarm is actuated when signal goes below electronic zero (with flame on). This can be due to inaccurate initial setting, drift, or a decrease in ambient concentration. Verify if this is the problem by zeroing meter with flame out and reigniting.	When using the X1 range adjust meter to 1 ppm, rather than zero, be sure instrument has been zeroed to "lowest expected ambient background level".
6) Slow response, i.e., time to obtain response after sample is applied to input is too long.	a) Check to ensure that probe is firmly seated on the rubber seal in the readout assembly. b) Check sample flow rate per procedure 1 above.	Reseat by holding the probe firmly against the rubber seat and then lock in position with the knurled locking nut. See 1 above.
7) Slow recovery time, i.e., too long a time for the reading to get back to ambient after exposure to a high concentration or organic vapor.	a) This problem is normally caused by contamination in the sample input line. This requires pumping for a long period to get the system clean of vapors. Charcoal in the lines would be the worst type of contamination. Isolate through the process of elimination. (See 1(b)). b) Check flame chamber for contamination.	Clean or replace contaminated sample line or assembly as required. Clean as required.
8) Ambient background reading in clean environment is too high.	a) A false ambient background reading can be caused by hydrocarbons in the hydrogen fuel supply system. Place finger over sample probe tube restricting sample flow and if meter indication does not go down significantly the contamination is probably in the hydrogen fuel. b) A false ambient background reading can also be caused by a residue of sample building up on the face of the sample inlet filter. If the test in 8(a) above produces a large drop in reading, this is usually the cause.	Use a higher grade of hydrocarbon free hydrogen. Check for contaminated fittings on filling hose assembly. Remove the exhaust port (it is not necessary to remove instrument from case). Use the small wire brush from the tool kit or a knife blade and lightly scrub surface of sample inlet filler.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	c) A false ambient background reading can also be caused by hydrocarbon contamination in the sample input system. The most likely cause would be a contaminant absorbed or condensed in the sample line. NOTE: It should be emphasized that running the instrument tends to keep down the buildup of background vapors. Therefore, run the unit whenever possible and store it with the carrying case open in clean air.	Clean and/or replace the sample input lines. Normally the false reading will clear up with sufficient running.
9) Pump will not run.	a) Check that there is no short circuit in wiring.	If no short circuit, pump motor is defective.
10) No power to electronics but pump runs.	a) Short circuit in electronics.	There is a short in the electronics assembly. Return OVA to factory or authorized repair facility.
11) No power to pump or electronics	a) Place battery on charger and see if power is then available. Recharge in a non-hazardous area only.	If power is available, battery pack is dead or open. Recharge battery pack. If still defective, replace battery pack.

GAS CHROMATOGRAPH (GC) OPTION

The Model OVA 128 CENTURY Organic Vapor Analyzer provides efficient and accurate indication of total organic compound concentrations on a continuous sampling basis. However, in areas where mixtures of organic vapors are present, it often becomes necessary to determine the relative concentration of the components and/or to make quantitative analysis of specific compounds.

To provide this capability, a gas chromatograph (GC) option is available. See Figure 6 for the location of the major components and controls associated with the GC option. When the GC option is used, the capability of the OVA includes both qualitative and on-the-spot quantitative analysis of specific components present in the ambient environment. The Recorder, which is used with the GC option, is described separately.

This section is applicable only to an OVA with the optional gas chromatograph system.

Modes of Operation

The OVA with GC option has two modes of operation. The first mode is the measurement of total organic vapors in the same manner as described for the basic OVA instrument. This mode is referred to as the "Survey Mode". The OVA is in the "Survey Mode" of operation whenever the Sample Inject Valve is in the "out" position.

The second mode of operation is called the "GC Mode". The OVA is in this mode of operation any time a sample has been injected into the GC system and the sample is being transported through the GC column. This section provides a brief description of how a gas chromatograph (GC) operates and specifically, how the model OVA 128 performs the required operations. A comprehensive discussion of gas chromatography theory, column selection, and data analysis is beyond the scope of this manual.

The OVA with GC option can be utilized for many types of analysis in the outdoor or indoor ambient environment or for specific laboratory type analysis. The OVA was not designed to compete with the research or process gas chromatograph but to compliment these instruments or eliminate their need in field applications.

This manual is intended to provide the operator with information to operate and maintain the OVA. Foxboro publishes Application/Technical Notes to assist the operators in applying the instrument to field monitoring situations.

All flame ionization detector (FID) gas chromatographs require certain elements for their operation. These elements include three flow regulated gas supplies as follows: 1) A carrier gas to transport the sample through the column; 2) Hydrogen gas for operation of the FID; 3) A clean air supply to support combustion to the FID. In addition, a method for injecting a known volume of sample air (aliquot) to be analyzed is required.

In standard gas chromatographs these three (3) flow regulated gases are individually supplied from pressurized cylinders equipped with regulators and flow control apparatus. The Model 128 GC system differs in that the hydrogen fuel for the FID is also used as the carrier gas. The clean air supply is simply the normal air sample pumped to the FID. During the GC analysis, this air is scrubbed in a charcoal filter to provide the clean air supply. The end result is that no additional gas supplies are required to add the GC option to the basic OVA instrument.

A valving arrangement is incorporated to provide a method for transferring a fixed volume of air into the GC system for analysis. The sample air injected into the GC column is the same sample being analyzed by the OVA for total organic vapor concentration. Therefore, the instrument provides the unique capability to observe the total organic vapor concentration of the sample prior to injecting it into the GC system. This operating feature is invaluable in field work where the environment is continually changing and where valuable GC analysis time must be expended only on the sample of concern.

OVA Columns

Columns are available in 4, 8, 12, 24, 36 and 48 inch lengths as standard offerings with any of the column packings listed below. Longer lengths are available in 12-inch increments on a non-standard basis. To order a column simply use the general part number for a column which is 510454 followed by a dash (-), the Foxboro packing material designation, a second dash and the desired length in inches. A sample column designation is 510454-G-24. This would represent a 24 inch column with 10% OV 101 on Chromosorb W, HP 60/80 mesh. If a specific application arises which calls for a column material not listed below, please contact Foxboro. We will be happy to check on its availability.

<u>Foxboro Designation</u>	<u>Material</u>
A	20% Dioctyl Phthalate on Chromosorb-P, AW 60/80 Mesh
C	Chromosorb 101, 60/80 Mesh
D	20% Ucon 50 HB 280 on Chromosorb-P, AW 60/80 Mesh
E	20% Carbowax 400 on Chromosorb-P, AW 60/80 Mesh
F	5/1.75% Diethylhexyl Sebacate/Bentone 34 on Chromosorb W, AW 60/80 Mesh
G	10% OV-101 on Chromosorb W, HP 60/80 Mesh
T	10% 1,2,3-Tris (2-cyanoethoxy) Propane on Chromosorb P, AW 60/80 Mesh
B	3% Diisodecyl Phthalate on Chromosorb W, AW 60/80 Mesh
PT	Poropak T, 60/80 Mesh
Q	Poropak Q, 60/80 Mesh
H	20% Carbowax 20M on Chromosorb P, AW 60/80 Mesh
J	n-Octane on Porasil C, 80/100 Mesh
N	Poropak N, 60/80 Mesh

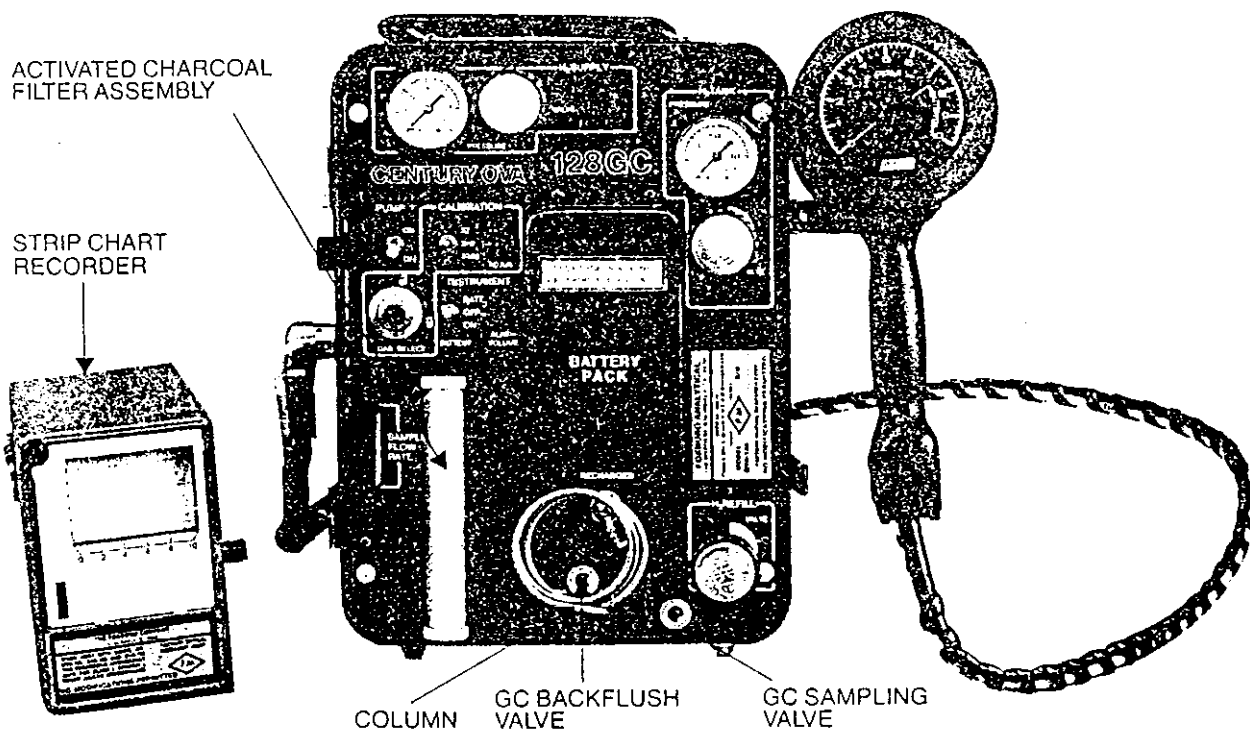


FIGURE 6
ADDITIONAL CONTROLS & COMPONENTS - GC OPTION

Sample Flow

Figure 7 is a flow diagram illustrating the flow paths of the hydrogen fuel, sample air supply, and GC injected sample aliquot.

Two push-pull valves are used in the GC system; the Sample Inject Valve and the Backflush Valve.

Block D illustrates the flow paths with the Sample Inject Valve in the "out" position. With this valve in the "out" position, the OVA functions in its normal manner as a total organic vapor analyzer.

Block C illustrates the flow paths after the Sample Inject Valve is moved to the "in" position to initiate the GC Mode.

The hydrogen flow path is now through the sample loop which enables hydrogen to sweep the air sample from the loop and carry it through the GC column.

Also note that the sample air going to the FID chamber is now routed through the activated charcoal filter where essentially all organic vapor contamination is removed from the air. The activated charcoal filter will effectively absorb most organic vapors with the exception of methane and ethane. The functions of the Sample Inject Valve are, therefore, to transfer a fixed volume sample of the air being monitored into the hydrogen stream and to reroute the sample air supply through a filter (scrubber).

The Backflush Valve has no prepositioning requirement to function. It can be in either the "in" or "out" position at the time a sample is injected into the GC system for analysis. The Backflush Valve simply reverses the direction of the hydrogen flow through the GC column.

Regardless of the operating mode, hydrogen always flows through the column to the FID detector and the sample air supply always flows to the FID detector to provide oxygen for the hydrogen flame.

The recommended hydrogen flow rate is 12 cm³/min for proper FID operation

and as a standard flow rate for generating GC reference/calibration data. This hydrogen flow rate is adjusted by varying the Hydrogen Supply Pressure, which is the hydrogen pressure at the input of the flow control capillary tube of the OVA. The pressure is changed by adjusting the set screw in the bonnet of the low pressure regulator, accessible by removing the battery pack from the instrument panel. To monitor the hydrogen flow rate, connect a bubble flowmeter to an end of the GC column which has been disconnected from the panel fitting and move the Backflush Valve so that hydrogen is flowing out of the column. Primary hydrogen flow control is accomplished by the capillary tube of the OVA. However, the flow restriction of a GC column will also affect the hydrogen rate and the effect will vary with column length, type of packing and packing methods. The nominal Hydrogen Supply Pressure is around 10 psig and the pressure drop across a typical 24 inch long column packed with 60/80 mesh material is approximately 1 to 1.5 psig. Normally, when the hydrogen flow rate is set at 12 cm³/min with a standard 24 inch long column, no adjustment needs to be made when using columns from four (4) inches to four (4) feet long. Longer columns may require hydrogen flow adjustment for proper operation. Adjustment would be required if and when precisely controlled analysis was being conducted or when the hydrogen flow was too low to keep the flame burning.

The sample air flow rate is not adjustable and is nominally 1.0 liter/minute. This flow rate should remain relatively constant. A sample flow gauge is provided on the OVA panel to monitor the sample flow rate. (Note: Panel gauge is not calibrated in L/min). When the Sample Inject Valve is in the "in" position, there may be a slight increase or decrease in sample air flow rate (0 to 15%). This change will normally not affect operation of the instrument as long as the flow rate is consistent from analysis to analysis. Basically, if the flow rate is consistent between calibration and end usage, there will be suitable precision in the measurements.

GC Analysis

1) SAMPLE INJECTION

When the Sample Injection Valve is depressed, the air in the sample loop is injected into the hydrogen stream which transports the sample through the column for separation of its components and to the flame chamber for analysis. This small volume of injected sample is qualitatively analyzed based on the retention time of the individual components of that sample while passing through the column. Quantitative analysis can then be accomplished by peak height or peak area analysis methods.

2) THE COLUMN

The column consists of tubing packed with a material which physically interacts with organic vapors and retards the passage of the vapors through the column. Since the packing material has a different attraction for each organic substance, each component in a mixture of gases will be slowed down to a different extent.

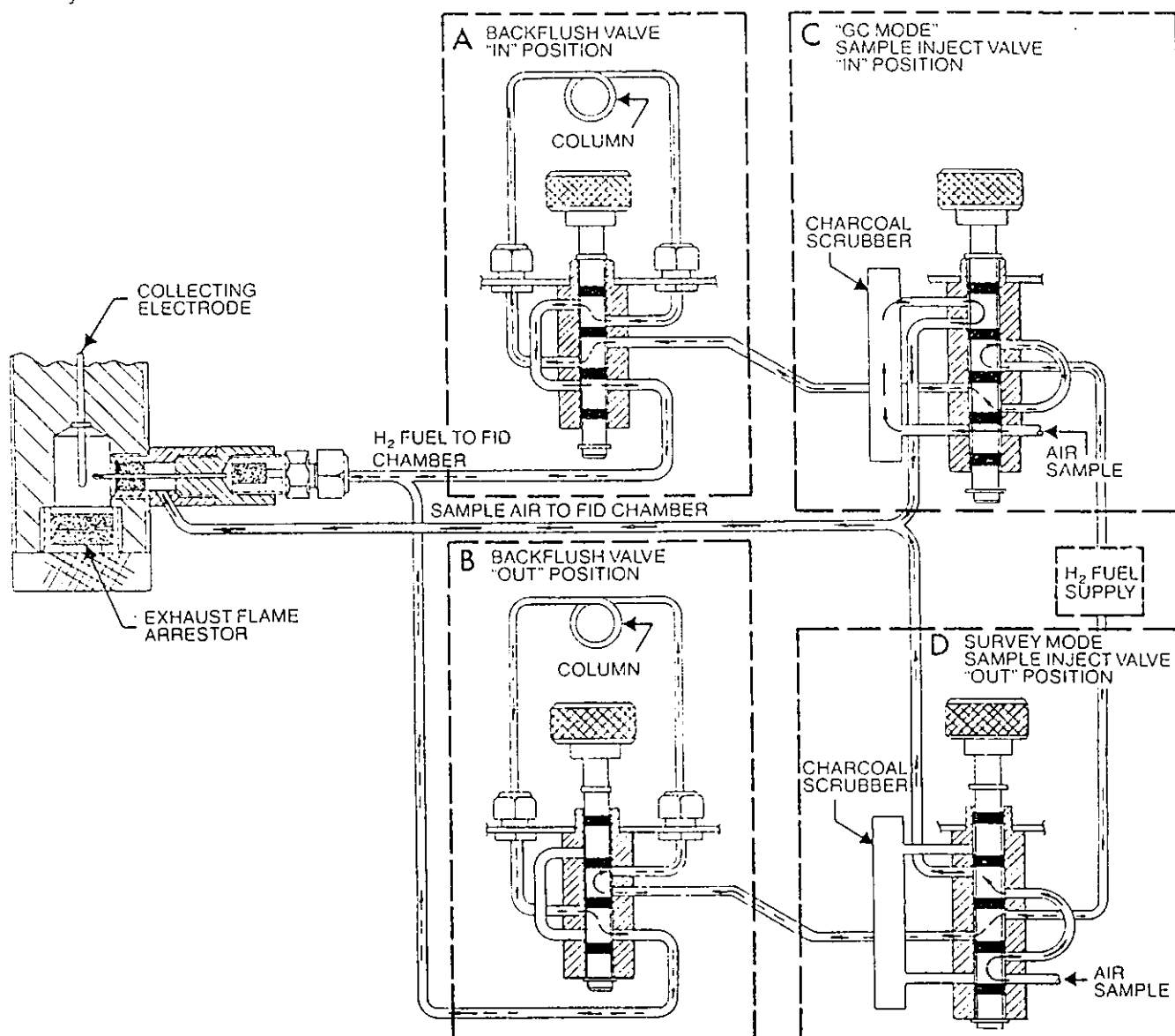


FIGURE 7
FLOW DIAGRAM - GC OPTION

The net effect is that each component elutes from the column at a different time. The components are then fed to the detector which gives a response to the meter or to an external strip chart recorder.

A portable isothermal pack (PIP) can be used for temperature control and/or isothermal analysis. This is described further under PIP kit option.

3) QUALITATIVE ANALYSIS

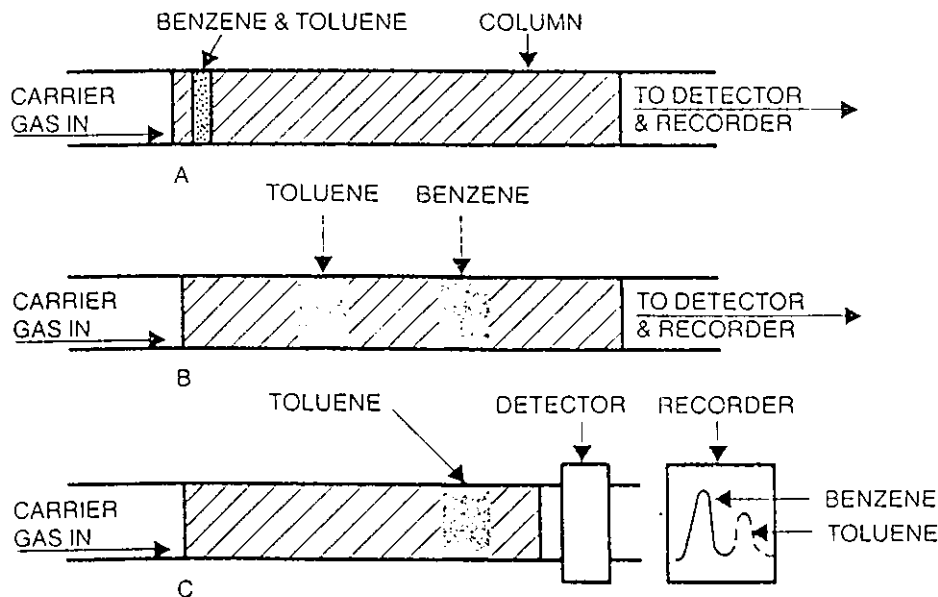
As each organic substance has a unique interaction with the column packing material, the time that the substance is retained on the column is also unique and thus characteristic of that particular substance. The "retention time" (RT) is primarily dependent on the type of packing material, the length of the column, the flow rate of the gas carrying the mixture through the column and the temperature range of the system.

When these variables are controlled, the retention times can be used to identify each of the components in a mixture. Because of these variables, it is usually necessary to establish retention times for each instrument by making a test with the pure substances of interest or to refer to established time data charts prepared in advance for that specific instrument. In those cases where retention times of the components are too close together for a good analysis, an adjustment in one or more of the operating variables will effect a sufficient difference in retention times to enable meaningful analysis.

4) QUANTITATIVE ANALYSIS

The detector response to any organic component is proportional to the quantity of material passing through the detector at a given time. For an eluted component, a plot of concentration vs. time forms a bell-shaped curve.

When using a strip chart recorder, the curve drawn on the paper is triangularly shaped and the area under the peak is related to the amount of substance being analyzed.



PICTORIAL SEPARATION OF BENZENE AND TOLUENE - "A" AT BEGINNING OF SEPARATION; "B" DURING SEPARATION; "C" BENZENE HAS ALREADY PASSED THE DETECTOR AND IS RECORDED. TOLUENE (DOTTED LINES) WILL APPEAR ON RECORDER AS IT PASSES THE DETECTOR.

FIGURE 8
TYPICAL COLUMN SEPARATION SEQUENCE

5) BACKFLUSH

The column Backflush Valve is provided to reverse the flow of the carrier gas (hydrogen) through the column. It is necessary that the column be backflushed after each individual analysis except under certain special conditions. The primary purpose of the backflush function is to clear the column of heavy compounds (with long retention times) which would contaminate the column and cause interferences to future GC analysis. The Backflush Valve has no prepositioning requirement; it is reversed from either position it was in during GC analysis. The Backflush Valve should be actuated immediately after the peak of the last compound of interest elutes. Figure 8 illustrates the function of the Backflush Valve.

In the GC system, the backflush is "to the detector". This is possible because the carrier gas and detector fuel are the same, i.e., hydrogen. It provides a convenient means of quantifying the total compounds in the backflush by simply recording the peak that elutes during the backflush operation. For field instruments, this quantitative backflush information is valuable since it provides a direct means of observing the condition of the column and seeing when the column is clean and the detector response has returned to baseline. The time required for the backflush is usually 1.2 to 1.5 times the GC analysis time.

6) SURVEY TO GC MODE

There is an inherent advantage to integrating the GC system to the basic total Organic Vapor Analyser (OVA). The OVA provides a direct reading of total organic vapors in the air being sampled, which gives the operator information about the sample being injected into the GC system. This information can be used to predict and verify the peaks that result during the GC analysis, including the backflush peak.

This feature eliminates expending valuable GC analysis time where there is no contamination of concern (comparable to taking noise measurements in quiet corners). It also enables the operator to select the most appropriate location to conduct an analysis, normally the area of highest concentration.

GC MODE OPERATING PROCEDURES

The gas chromatographic analysis mode (GC Mode) of operation can be initiated at any time during a survey by simply depressing the Sample Inject Valve. After completion of the analysis and backflush operations, the Sample Inject Valve is pulled out and the survey continued or another sample injected. Note that when the Sample Inject Valve is in the survey mode (out position) the OVA operates in the same manner as an OVA which does not incorporate the GC option.

Controls/Indicators

Refer to Figure 6.

- 1) Sample Inject Valve - This two (2) position valve (shown schematically in Figure 7) is used to select either Survey Mode (valve out) or GC Mode (valve in).
- 2) Backflush Valve - This two (2) position valve (shown schematically in Figure 7) is used to reverse the flow of hydrogen through the column to:
 - a) Backflush the column for cleaning.
 - b) Quantitatively measure total compounds after a selected point. Example: Separation of methane from non-methane hydrocarbons to read total non-methane hydrocarbon level.
- 3) Column - Separates components of a gas mixture so that each component of the mixture elutes from the column at a different time.
- 4) Activated Charcoal Filter Assembly - This assembly functions only in the GC Mode (Sample Inject Valve "in") as shown schematically in Figure 7). It removes organic compounds (except methane and ethane) by absorption from the sample air supply.

Turn on Procedure

Place the Sample Inject Valve in the "out" position and put the OVA instrument in operation per "Operating Procedures" for the survey mode. NOTE: Leave the hydrogen fuel and pump "on" for three (3) to four (4) minutes before attempting ignition to allow time for hydrogen purging of the column.

Survey Mode

When using the OVA in the Survey Mode, ensure that the Sample Inject Valve remains in the full "out" position and that the Backflush Valve is either full "in" or full "out". Note that when changing from the GC Mode to the Survey Mode, the OVA output reading will continue to change until all compounds have been eluted from the GC column. Therefore, under normal field conditions, the GC column should be backflushed for clearing, which takes approximately 1.2 to 1.5 times the forward analysis time. The backflush peak may be observed returning to baseline, after which the Sample Inject Valve may be moved to the Survey Mode (out) position.

When the compound(s) being analyzed are known to be the only compound(s) present in the air sample, backflushing may be omitted.

GC Mode Operation

In normal GC analysis, a strip chart recorder is used to record the output concentration from the OVA as a function of time. This record, called a chromatogram, is utilized for interpretation of the GC data.

a) OPERATION

- 1) Turn on recorder and push Sample Inject Valve "in" with a fast, positive motion. This starts the GC analysis which is automatic up to the point of backflushing. NOTE: Rapid and positive motion should be used when moving either the Sample Inject or Backflush Valves. On occasion, the flame in the FID detector may go out, which would be indicated by a sharp and continued drop of the concentration level. If this occurs, reignite the flame and continue the analysis. NOTE: A negative "air" peak typically occurs shortly after sample injection and should not be confused with flame-out.
- 2) The negative air peak and various positive compound peaks indicated on the OVA readout meter and the strip chart recorder represent the chromatogram.

- 3) After the predetermined time for the analysis has elapsed (normally immediately after the peak of the last compound of concern), rapidly move the Backflush Valve to its alternate position (in or out). Leave the instrument in this condition until the backflush peak returns to baseline, then pull the Sample Inject Valve to the "out" position. If no backflush peak appears, pull the Sample Inject Valve out after being in the backflush condition for a period at least twice as long as the analysis time. The OVA is now in the Survey Mode and ready for survey or injection of another sample into the GC system.

b) INTERPRETATION OF RESULTS

The OVA 128 with GC option is intended for applications where there are a limited number of compounds of interest and the compounds are normally known. Under these conditions, the operator must know the retention time and peak height characteristics of the compounds under specific operating conditions. To calibrate the OVA in the GC Mode, determine, by test, the retention time and peak area (using peak height analysis) for the compounds of concern. These tests should be conducted on the column to be utilized and over the concentration and temperature range of concern. When representative characteristic data is available, such as in the Application/Technical Notes, a spot calibration check is normally all that is required.

It should be noted that under normal field conditions, the vapor concentrations vary continually as a function of time, location, and conditions. Field measurements for industrial hygiene work are normally associated with a threshold level around a pre-established concentration. Surveys for locating fugitive emission sources present a continually varying situation. Under these conditions, it is desirable to have a simple method of interpreting the GC data for on-the-spot analysis and decision making.

High precision is normally not a requirement for these type analyses since the environment is continually changing. The methods presented in this section are designed to provide a means for typical field analysis. When the OVA is used under laboratory conditions, standard laboratory methodology may be used for greater precision.

Technical Discussion

The chromatogram is a chart recorder trace of the organic vapor concentration from the Organic Vapor Analyzer (OVA) as a function of time. A typical chromatogram is illustrated in Figure 9 and is a series of triangular shaped peaks originating from and returning to a fixed baseline. Qualitative interpretation of a chromatogram involves identifying a peak by analyzing the time it took for the peak to appear after initial injection [referred to as retention time (RT)] and comparing this RT to reference data. Quantitative interpretation involves analyzing the area under the peak and relating this area to calibration data of peak area versus concentration for that specific compound under the conditions present during the GC analysis.

It can be seen that interpretation of a chromatogram requires the use of calibration reference data. GC reference data is always generated empirically, i.e., through tests. Foxboro Application/Technical Notes may be used as a reference for selecting columns and interpreting chromatograms. However, simple tests must be conducted to obtain the required reference data.

a) QUALITATIVE ANALYSIS

Under a given set of operating conditions the retention time is characteristic of that particular substance and can be used to identify specific compounds. It will be necessary to calibrate retention times by making tests with the pure compounds of interest.

The retention time (RT) is defined as that period of time from injection until the time of maximum detector response for each substance. Retention time is measured from the time of sample injection to the time the apex of the triangle shaped curve is obtained on the strip chart recorder. (See Figure 9). The strip chart recorder operates on a clock mechanism such that the distance along the baseline is proportional to time. While retention times are characteristic for each compound, it is possible that two materials could have the same retention times. Thus, if there is any question as to the identity of the vapor, it may be necessary to verify identification by retention times on different columns.

Use of a longer column will increase the retention times of those components it is capable of separating. The time between peaks will also be increased. This is especially useful if a component comes through too fast or if desired peaks are so close that they overlap.

b) COLUMN SELECTION

Two columns are supplied with the instrument. These are general purpose columns which are useful in a wide variety of applications. If they do not achieve separations for a particular application, it may be necessary to select other packing materials or longer columns. Foxboro will assist in this selection or prepare a custom column if necessary.

If columns are made by the user or purchased from other sources, ensure that the packing density does not create too large a pressure drop. A large pressure drop can result in flame-out problems.

c) TEMPERATURE EFFECT ON RETENTION TIME

An increase in temperature will decrease column retention time (RT) and vice versa. Normally retention time (RT), as a function of temperature, changes linearly over the range of 0 to 40°C. For complex qualitative analysis, a calibration plot of RT versus temperature will be required. In typical usage, such as inside a factory, the effect of temperature can be compensated for during chromatogram interpretation. A single component tracer compound can be sampled at any time to provide a "key" for other compound identification.

d) CARRIER GAS FLOW RATE AFFECT ON RETENTION TIME

An increase in carrier gas flow rate will decrease retention time. For reproducible data, the carrier gas (hydrogen) flow rate must be recorded in association with a chromatogram. Primary control of the hydrogen flow rate is accomplished in the OVA by regulating the hydrogen pressure across a capillary tube. The hydrogen flow rate is also affected by the restriction of the GC column but most columns have a limited effect. The hydrogen flow rate is factory set at 12 cm /minute with a typical 24 inch column.

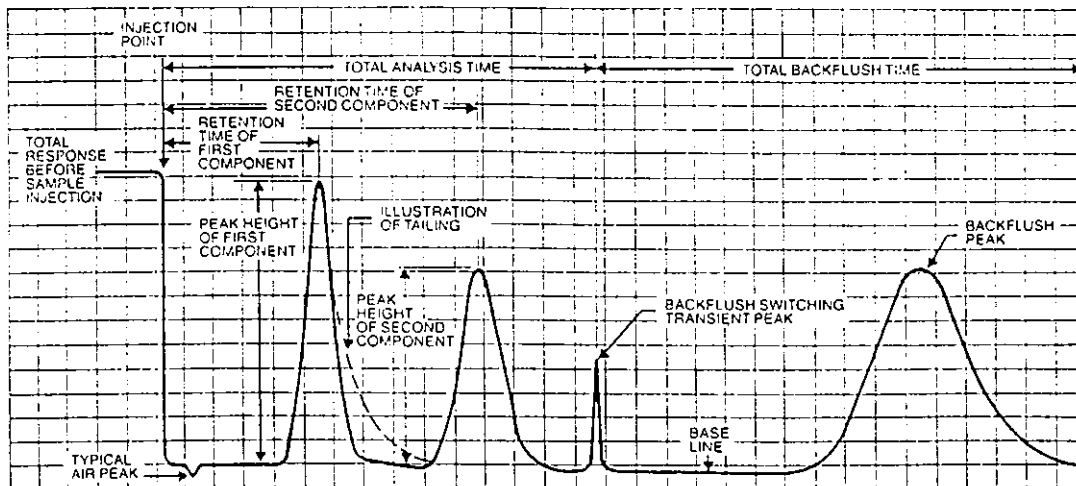


FIGURE 9
TYPICAL CHROMATOGRAM

e) QUANTITATIVE ANALYSIS

In general, the more triangularly symmetrical the peak, the better the peak height analysis capability. However, many GC peaks have "tailing" as illustrated in Figure 9. Peak height calibration is an acceptable method for quantitative analysis as long as the area under the tail is small compared with the total peak area. If severe tailing occurs, empirical calibration data generated through tests may be required to plot the peak height versus the concentration curve.

Only peak height analysis will be discussed in this manual. The method involves injecting a known concentration of the compound and recording the peak height under the test conditions. Peak height characteristics can be established for various columns and various temperatures. Normally, both retention time and peak height characteristics will be measured.

When peak area measurements are desired, the areas may be measured using an integrator on the OVA output signal. Other manual methods may also be used, such as counting squares, weighing curves or simple triangulation. When the GC peaks have good symmetry, triangulation (area equals $1/2$ base x height) is a convenient method.

Calibration Data

When conducting tests to obtain GC calibration data, the following information should be recorded.

- a) Column - description and serial number as applicable.
- b) Temperature - column temperature, normally room ambient.
- c) Chart speed - distance/unit time.
- d) Carrier flow rate - hydrogen flow rate through the column (cm /min).
- e) Sample concentration - ppm for each compound,
- f) Sample volume - OVA by serial number or typically 0.25 cm for standard value.
- g) Recorder scaling - ppm per unit deflection.
- h) Range - range of OVA being used, i.e. X1, X10, X100.
- i) OVA serial number.

To obtain a calibration point, inject a known concentration sample into the GC system and record the resulting chromatogram peak. The retention time for the peak may be scaled from the record or timed with a stop watch. The peak height may be scaled from the record or the OVA readout meter may be observed during the elution of the peak. Figure 10B presents the format of a chart which may be used to record calibration data. Experience has indicated that the peak height response of a compound is linear within the concentration range of 0 to 160 ppm. Therefore, a single calibration point, preferable around the concentration of concern, is normally all that is required to plot peak height response in ppm as a function of compound concentration. Data for other compounds on the same column may also be plotted along with their associated retention times, percent relative response in the total organic Survey Mode, TLV, etc. It is recommended that copies of the actual chromatograms be kept with the charts for observing the peak shapes, peak interferences, etc. It should be noted that a chromatogram can be utilized like a fingerprint for compound identification or peak height and shape comparison. Transparent overlays are an aid in chromatogram analysis.

When temperature variations are anticipated, data should be taken at several points and recorded on the chart as a new curve or as a relative change as a function of temperature as illustrated in Figure 10B.

Preparing and using the calibration chart is very straightforward. As an example, once the elution sequence of a group of compounds is determined, a mixture of 100 ppm of each can be prepared and run on the GC for chart data. The retention time of each compound and the peak height of each can be read directly from the chromatogram and the data put on the chart. If temperature data is to be taken, additional chromatograms may be run with the same sample and the RT and peak height as a function of temperature.

When complex mixtures such as gasoline are analyzed, it may be desirable to keep the record of the backflush peak for future reference and peak area comparison. It is also recommended that the total organic vapor concentration reading on the OVA be recorded for each calibration sample used. This reading is used for arriving at relative response numbers and as a check on sample preparation precision.

Routine Maintenance

a) COLUMN

Any column can be contaminated with compounds having long retention times. This will result in high background readings. This condition can be checked by installing a new column or a blank column (tubing only). If this reduces the background reading, the contaminated column should be baked at 100°C (212°F) for three (3) to four (4) hours in a drying oven while passing nitrogen through the column. Higher temperatures may permanently damage the column packing.

When installing any column, avoid touching the ends, as this may cause contamination. Also, ensure that the fittings are tight to avoid hydrogen leakage.

IMPORTANT: The following simple test may be run to determine whether the GC column is contaminated. While in a clean ambient air background, place the Sample Inject Valve in the "in" (GC Mode) position. Observe the background reading on the meter or recorder. After one (1) to two (2) minutes, change the position of the Backflush Valve and again observe the background reading. If the background reading went down and then started to increase in one to two minutes, the column is probably contaminated and needs to be cleaned. Note that if hydrogen flows into one end of the column for a period of time, the contamination is pushed into the column.

Then when the hydrogen flow is reversed, the exhaust end of the column will be clean until the contamination is again pushed through. Remember that to clean a column the purge gas must be run through the column in one direction until all contamination is removed. **NOTE:** Contaminated columns can be avoided by backflushing the column after every analysis.

b) CHARCOAL FILTER ASSEMBLY

After repeated use, the Charcoal Filter Assembly will become saturated. Periodically, the operator should check the effectiveness of the activated charcoal.

This can easily be done by operating the unit with the Sample Injection Valve "in" and passing the probe near a concentrated sample of the compound being analyzed. The readout should remain nearly steady (should not rise more than 0 to 2 parts per million (ppm)). If rise is more than 2 ppm, replace the old charcoal with new activated charcoal. Care should be taken to completely fill the tube to prevent a path for sample to bypass the charcoal. The life of the charcoal depends on the time (length) of exposure and the concentration level during that exposure. When changing charcoal, be sure that any fine charcoal dust is removed from the assembly.

Another test of the charcoal filter is to note the background reading with the Sample Inject Valve "out" and then note the reading with the valve "in". The level should never be higher when the valve is in the "in" position and the charcoal filter is in the air line. If the reading with the valve in the "in" position is higher, the charcoal filter is probably contaminated and acting like a contamination emitter.

Trouble Shooting

Table 2 presents recommended field trouble shooting procedures which are associated with the GC system. These procedures are in addition to those found in the basic OVA section of the manual.

TABLE 2

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator.	a) Check Teflon tubing on valve assembly for kinks, etc. b) Check flow rate with valve in down position.	Straighten or replace teflon tubing. Check for over restriction of charcoal filter.
2) Hydrogen flame will not light.	a) Check column connections on top of unit to make sure they are tight. b) Check column for sharp bends or kinks. (Hydrogen flows through this column at all times and a sharp bend will compact packing too tightly for proper hydrogen flow). c) Check charcoal filter fittings to make sure they are tight. d) Check hydrogen flow rate from the column. e) Check that the Inject and Back-flush Valves are both completely in or out. A partially activated valve will block the hydrogen and air flow paths. f) If a new column was installed prior to problem identification, check for proper hydrogen flow rate through the column (should be approximately 12 cm /min).	Tighten fittings. Replace column. Tighten fittings. Adjust hydrogen pressure to obtain 12 cm /min flow rate. Ensure both valves are either completely in or out. Increase hydrogen pressure to obtain proper hydrogen flow rate or if column is excessively restrictive, replace or repack the column.
3) Ambient background reading in clean environment is too high.	a) Check for contamination in charcoal filter assembly. This can be detected if ambient reading increases when going in to the chromatographic mode. b) Check for contamination in column. c) Check for contamination in column valve assembly.	Replace activated charcoal in charcoal filter assembly. Replace or clean column. Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
4) Flame-out when operating either valve.	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with a positive motion.

TABLE 2

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	b) Either hydrogen or air may be leaking around one or more of the valve quad rings. Assess by tests and "O" ring inspection.	Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings).
	c) Damaged or worn quad rings causing leak.	Replace quad rings and grease as above.
5) Excessive peak tailing	a) Change or clean GC; see if problem disappears.	Ensure columns are clean prior to use. If one of the same type of column tails are worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessive lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" ring. Do not get grease into the "O" ring grooves.

Recommended Spares

The following spare parts and supplies are recommended to support the GC system and recorder. These are an addition to the spare parts list for the basic OVA described in the "OVA MAINTENANCE" section.

ITEM DESCRIPTION	PART NO.
1) Quad Rings	510496-1 (10/pkg.)
2) Tubing, .148 in ID .020 wall	12942
3) Tubing, Teflon .120 in ID .030 wall	12941
4) Activated Charcoal	CSC-004
5) "O" Ring for Charcoal Scrubber	U0118CE
6) Chart Paper (linear)	CSC-008 (6/rls/pkg)

ACCESSORIES

Recorder Accessory

A portable Strip Chart Recorder is available for use with the OVA (reference Figure 11). The recorder is powered from the OVA battery pack and the output can be scaled to match the OVA readout meter, thereby providing a permanent record for subsequent analysis or reference. P/N 510445-4 is FM certified intrinsically safe. P/N 510445-6 is BASEEPA certified.

The recorder can be used with the OVA to provide a long term monitoring profile of total hydrocarbon or can be used with the Gas Chromatograph Option to provide a chromatogram.

Features

The recorder prints dry (no ink) on pressure sensitive chart paper. The recorder is equipped with two gain ranges and an electronic zero adjustment. The HIGH gain position is normally used to provide a means of scale expansion.

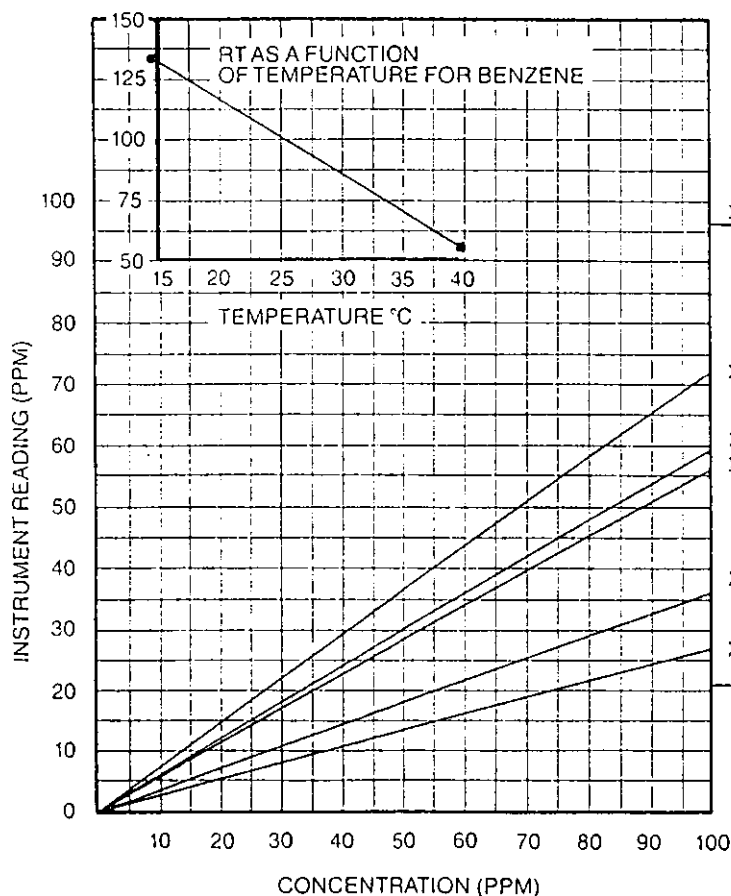
Controls and Connections

Described below are the functions of recorder controls and connectors.

- 1) HIGH-LOW Switch - This switch, located on the right hand side of the recorder, provides 2 ranges. The LOW range is set for the same full scale reading as the OVA readout meter. The HIGH range can be set to give an increased sensitivity to the recorder without effecting the OVA calibration.
- 2) ZERO ADJUST Knob - This potentiometer, located on the right hand side of the recorder, permits "nulling" of the background reading on the recorder without affecting the calibration of the OVA displayed on the OVA readout. In the full clockwise position, the recorder will display the same reading as the OVA meter. Counterclockwise rotation will reduce the reading on the recorder.

- 3) POWER CONNECTOR - This 126 series, 5 pin connector provides power and signal to the recorder, as follows:

PIN	FUNCTION
B	Input Signal
E	pos. 12VDC input
H	Ground



Column G-18 Chromatograph OVA-128
Temperature 25°C Carrier Flow 12 mL/sec.
Injection: Volume 2.5 mL, Type valve

X	Compound	R.R (%)	RT (sec)	TLV (ppm)
X4	Heptane	80	471	400
X2	Pentane	100	90	1000
X2	Hexane	90	198	100
X2	Benzene	150	291	1
X3	Toluene	120	762	200

- NOTE:
1. Concentration used for test is 100 ppm or as indicated.
 2. X = Concentration multiplier

FIGURE 10A
CALIBRATION CHART

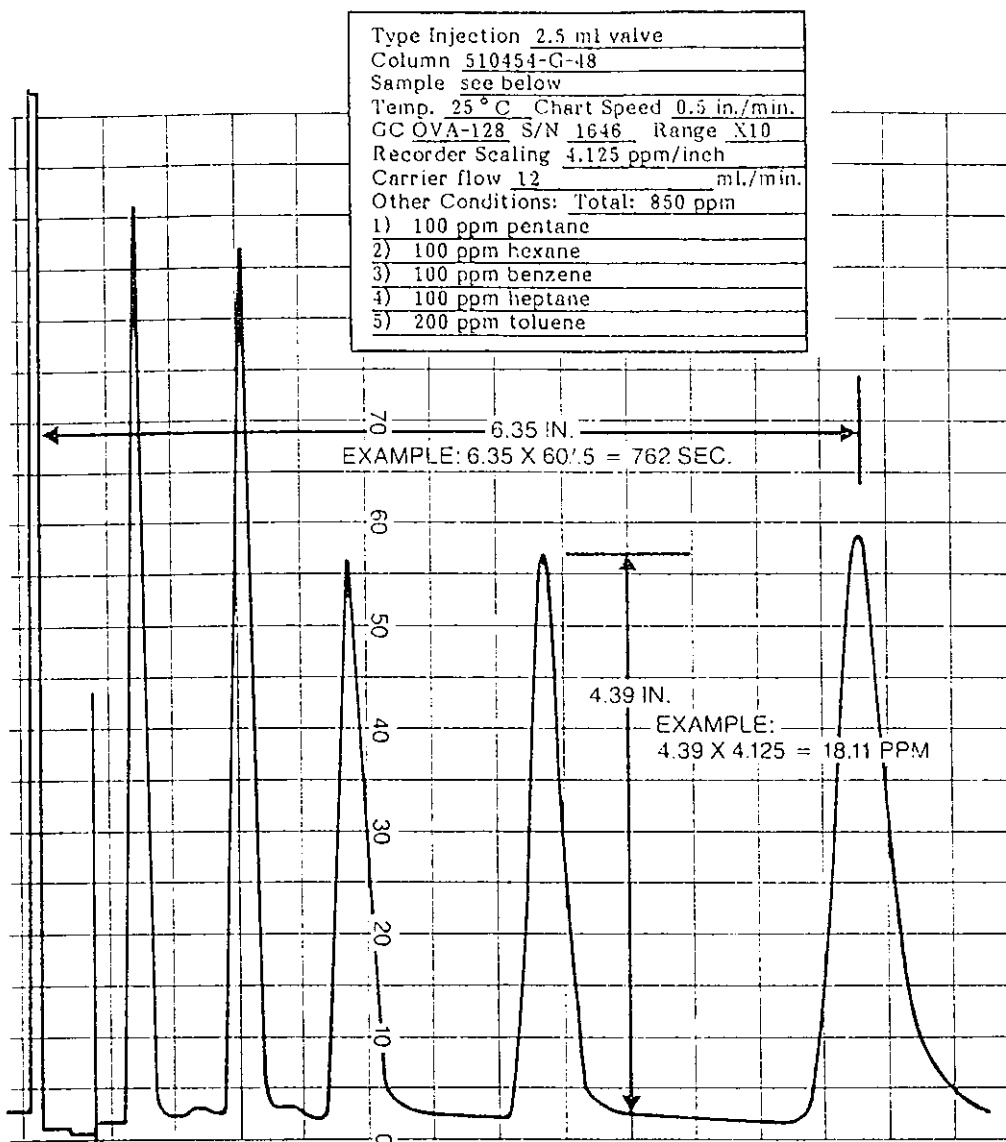


FIGURE 10B
CHROMATOGRAM

Calibration

Electronic and mechanical adjustments, other than the operational adjustments on the side panel, are provided to calibrate and align the recorder. (See Figure 11).

MECHANICAL ZERO ADJUSTMENT

- A) Snap out the front panel nameplate using a small blade screwdriver in the left hand slot) for access to mechanical zero adjust screw, place HIGH-LOW Switch in OFF position.

- B) Unscrew knurled fastener at top of front panel to open recorder. Pull down plastic chassis latch on right side to release sticker bar tension on paper and adjust mechanical zero as required. Replace nameplate, chassis latch and resecure front panel.

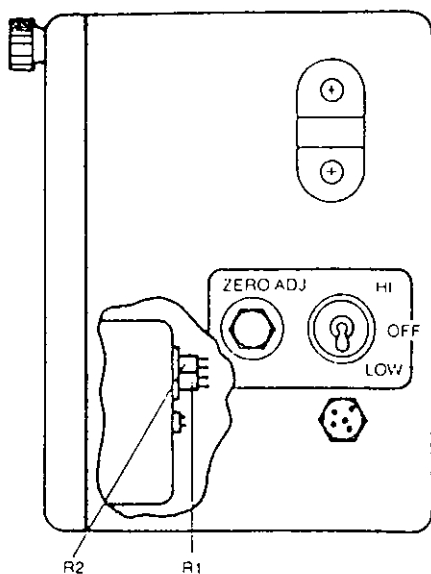


FIGURE 11
RECORDER CONTROLS AND ADJUSTMENTS

GAIN ADJUSTMENT

Separate adjustments are provided for the HIGH and LOW ranges on the recorder. (Refer to Figure 11 for location).

- Connect recorder to OVA and adjust OVA for full scale reading on readout (about 5 VDC).
- Loosen knurled fastener on upper left of the front panel and pull front panel down.
- Place HIGH-LOW Switch in LOW and adjust R1 until recorder prints full scale.
- Place HIGH-LOW Switch in HIGH and adjust OVA to read the desired full scale with front panel CALIBRATE ADJUST Knob, typically half scale on the readout. Adjust R2 until recorder reads full scale. NOTE: Full scale adjustment of the recorder for 1/2 scale on the OVA gives a gain increase of two (2) in the height of the peak on the chromatograms. This is the factory set point for the HIGH gain range; however, other points can be set as desired with a gain of three being the maximum obtainable without amplifier loading.

Maintenance and Routine Operations

Refer to the manufacturer's (Gulton) manual on the recorder which is enclosed with each recorder when shipped.

Changing Chart Speeds

The recorder is equipped with a 16 RPM motor which gives a writing speed of four (4) strikes per second. The chart advance speed is determined by the gear train assembly number used. The inches per hour for each gear train is given in the table on page 9 of the Gulton recorder manual. Refer to the bottom line of the chart adjacent to drive motor 16 and note for example that a number 1 gear train has a chart speed of 8"/hour.

- To change the paper speed, open the recorder, remove gear box spring (on left side), move gear box in direction of arrow on its case and lift out from top. Do not force out from bottom. Insert new gear, bottom first, slide into position against arrow direction. Replace gear box spring.

Activated Charcoal Filter Accessory

The Activated Charcoal Filter Assembly is an accessory which can be installed on the OVA Readout Assembly or attached at the end of the telescoping probe. The filter is typically filled with activated charcoal which acts as an absorbent and effectively filters out organic vapors other than methane or ethane.

A screw cap on the probe end is removed for refilling the filter with activated charcoal or other filtering media.

Applications of the filter include:

- Obtaining a clean air sample for zero baseline check and adjustment.
- Running "blank" chromatograms to assess instrument contamination.
- Rapid screening of methane and non-methane organic vapors.
- Selective screening for natural gas surveys.
- As a moisture filter when filled with a desiccant such as silica gel.

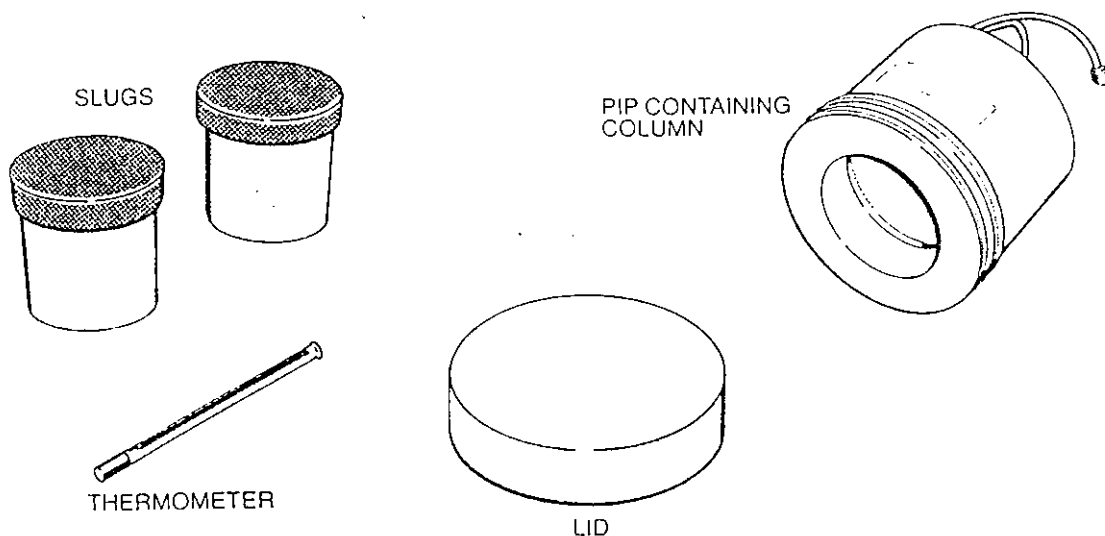


FIGURE 14
PORTABLE ISOTHERMAL PACK

PIP Components & Spare Parts

511800-1	PIP Kit
511805-1	PIP Assembly (specify column length and packing material)
511810-1	40°C Slug (phase-change material)
511830-1	Seeder for 40°C Slug
511815-1	Aluminum Slug
511820-1	Empty Bottles (package of six)
511825-1	Insulating Cover
511826-1	Thermometer

PIP columns can be prepared with any standard column packing material. A temperature control slug is inserted into the PIP slug cavity which has exterior foam insulation. For field operation in extreme ambient temperatures, an additional sheepskin jacket can be installed. The period of temperature control depends upon the temperature difference between ambient and the slug. For a 0°C ice pack and ambient temperature of 27°C, a control period of approximately 10 hours is typical. Additional information on the PIP system will be found in Foxboro document TI611-105.

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Carbowax is a trademark of Union Carbide Corporation.

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Porasil is a trademark of Waters Associates.

APPENDIX D



Standard Test Method for Particle-Size Analysis of Soils¹

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.

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 Constants²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

E 100 Specification for ASTM Hydrometers⁴

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 oper-

ated 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 $\frac{1}{4}$ in. (19.0 mm) nor more than $1\frac{1}{2}$ 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 cup⁵ (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 ft³/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 2 $\frac{1}{2}$ 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.

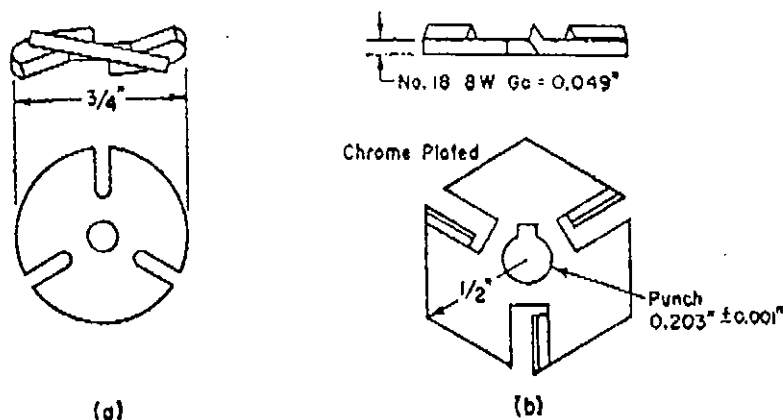
Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 - 62.

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	1/2	3/4
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles

3-in. (75-mm)	No. 10 (2,000-μm)
2-in. (50-mm)	No. 20 (850-μm)
1 1/2-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
3/4-in. (19.0-mm)	No. 140 (106-μm)
1/2-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 (1,180-μm)
1 1/2-in. (37.5-mm)	No. 30 (600-μm)
1-in. (25.0-mm)	No. 50 (300-μm)
3/4-in. (19.0-mm)	No. 100 (150-μm)
1/2-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	
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.

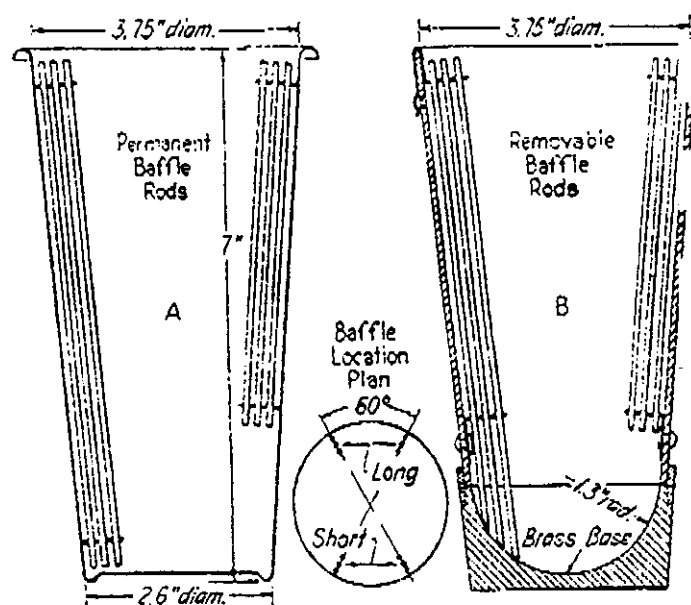
3.9 Timing Device—A watch or clock with a second hand.

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



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

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.

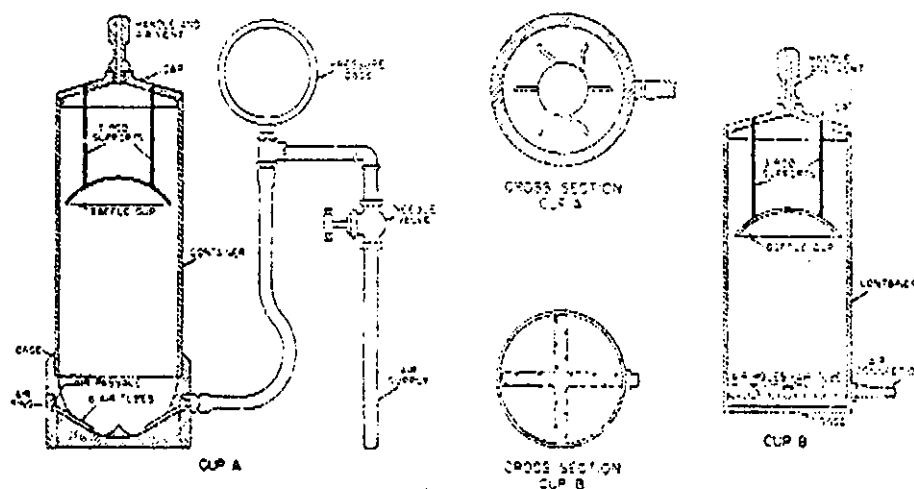


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

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
3/4 (19.0)	500
1 (25.4)	1000
1 1/2 (38.1)	2000
2 (50.8)	3000
3 (76.2)	4000
	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.

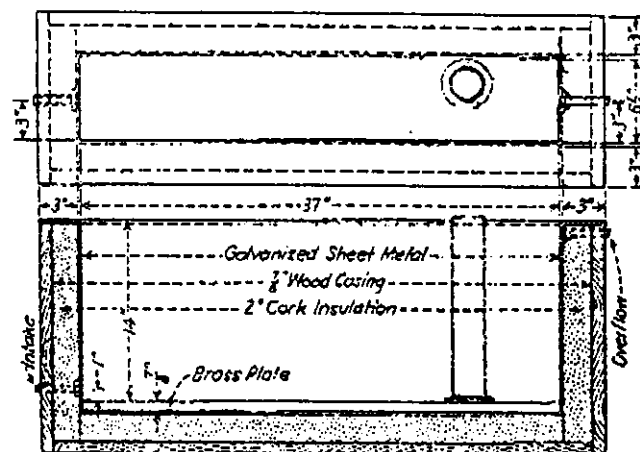
5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages 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),



Metric Equivalents

in.	3/4	1	3	6 1/4	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

2-in. (50-mm), 1 1/2-in. (37.5-mm), 1-in. (25.0-mm), 3/4-in. (19.0-mm), 1/2-in. (12.5-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.

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 cylinder 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 $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$), 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. 1, 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 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 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 or no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent 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 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 read the hydrometer readings at the following intervals (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, 120, 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- μ m) 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 $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) 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 $\frac{1}{8}$ -in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the $\frac{1}{8}$ -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 hygroscopic 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

TABLE 1 Values of Correction Factor, α , 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.

(2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

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:

α = correction fraction 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:

$$D = \sqrt{[30\pi/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,

- n = 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_1 = 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 $C1$ -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

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	8	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:

$$L = L_1 + \frac{1}{2} (L_2 - (V_b/A))$$

where:

L = effective depth, cm.

L_1 = distance along the stem of the hydrometer from the top of the bulb to mark for a hydrometer reading, cm.

L_2 = overall length of the hydrometer bulb, cm.

V_b = volume of hydrometer bulb, cm³, and

A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_1 = 14.0 cm

V_b = 67.0 cm³

A = 27.8 cm²

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

of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and 1 : percentages smaller than the corresponding diameters to all

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
15	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	0.01323	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	0.01225	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	0.01255	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.	
½-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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