REMEDIAL INVESTIGATION M. WALLACE AND SON, INC. SCRAPYARD

SAMPLING AND ANALYSIS PLAN VOLUME 2: QUALITY ASSURANCE PROJECT PLAN

Niagara Mohawk Power Corporation

Syracuse, New York



BLASLAND & BOUCK ENGINEERS, P.C. BLASLAND, BOUCK & LEE ENGINEERS & GEOSCIENTISTS

August 1992

Remedial Investigation M. Wallace and Son, Inc. Scrapyard Sampling and Analysis Plan Volume II: Quality Assurance Project Plan

Niagara Mohawk Power Corporation Syracuse, New York

August 1992



BLASLAND & BOUCK ENGINEERS, P.C. 6723 TOWPATH ROAD SYRACUSE, NEW YORK 13214

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1.0 - PROJECT DESCRIPTION

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) is part of the Sampling and Analysis Plan (SAP) which supports the Remedial Investigation/Feasibility Study (RI/FS) Work Plan for the M. Wallace and Son Scrapyard Site located in Cobleskill, New York. The M. Wallace & Son, Inc. Scrapyard Site Remedial Investigation will include the investigation of ground water, surface water, soils, and sediments. The QAPP presents the analytical methods and procedures to be used during implementation of the RI. Related documents include the RI/FS Work Plan and the Field Sampling Plan (FSP) which is Volume I of the SAP.

This QAPP sets forth the analytical methods and procedures to be used in the RI, while the Field Sampling Plan (FSP) component of the SAP sets forth the RI field procedures. The FSP and this QAPP are integrated and cross-referenced where applicable to minimize redundancy.

This QAPP was prepared in a manner consistent with the United States Environmental Protection Agency (USEPA) reference document, Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA - Interim Final (EPA/540/G-89/004).

Section	Content
1	Project Description
2 Project Organization and Responsibilities	
3	Quality Assurance Objectives for Measurement Data
4	Sampling Procedures
5	Sample and Document Custody
6	Calibration Procedures and Frequency
7	Analytical Procedures
8	Data Reduction, Validation, and Reporting
9	Field and Laboratory Quality Control Checks
10	Performance and System Audits
11	Preventive Maintenance
12	Data Assessment Procedures
13	Corrective Action
14	Quality Assurance Reports to Management

Details are provided in the subsequent sections. This document also contains pertinent information from the RI/FS Work Plan and the FSP related to the measurement and evaluation of RI analytical data.

1.2 RI OBJECTIVES

The purpose of the QAPP is to present the quality assurance/quality control (QA/QC) procedures to be implemented during the RI. The QAPP has been developed to provide data quality which is sufficient to meet the RI objectives. The overall objective of the M. Wallace and Son Inc. Scrapyard Site RI is to obtain the information necessary to a) determine the presence and extent of chemical constituents in environmental media present at the site; b) determine the presence and extent of chemical constituents in sediments and surface water in the quarry pond outlet channel north of the railroad embankment; c) assess the risks, if any, to human health and the environment; and d) support the development, evaluation, and selection of appropriate remedial/response alternatives.

1.3 RI DATA QUALITY OBJECTIVES

<u>1.3.1 General</u>

Data quality objectives (DQOs) are statements, in either qualitative or quantitative terms, regarding the appropriate data quality for an investigation. DQOs are typically determined through an iterative process and are refined as additional information becomes available, and established based on the intended end use of the data to be obtained. General project DQOs for the M. Wallace and Son, Inc. Scrapyard Site RI are summarized in this section, with detailed information provided throughout the QAPP, FSP and the RI/FS Work Plan.

Generally, the data generated during the RI will be used to determine the distribution of chemical constituents to: 1) determine the presence and extent of chemical constituents in environmental media present at the site; 2) determine whether constituents identified at the site are present in the sediments and surface water in the quarry pond outlet channel north of the railroad embankment; 3) assess the risks, if any, to human health and the environment; and 4) support the development, evaluation, and selection of appropriate remedial/response alternatives.

To obtain information necessary to meet the RI objectives stated above, the following four field sampling investigations will be conducted:

- 1. Soil Investigation;
- 2. Sediment Investigation;
- 3. Surface Water Investigation; and
- 4. Ground-Water Investigation.

Preliminary DQOs were identified during the M. Wallace and Son Inc. Scrapyard Site RI/FS scoping and incorporated into the development of the Work Plan, FSP, and QAPP to ensure that the data generated during field investigations will be of adequate quality and sufficient quantity to form a sound basis for decision making purposes relative to the above objectives. Data quality objectives have been specified for each data collection activity or investigation. The DQOs presented herein address investigation efforts only and do not cover health and safety issues, which are addressed in detail in the Health and Safety Plan (HASP) for this project.

A DQO summary for each of the five investigation efforts is presented below. The summary consists of stated DQOs relative to the following items:

- A. Data Uses;
- B. Data Types;
- C. Data Quality;
- D. Data Quantity;
- E. Sampling and Analytical Methods; and

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- F. Data Precision, Accuracy, Representativeness, Completeness, Comparability and Sensitivity (PARCC Parameters).

The analytical levels discussed in the following sections with regard to data quality are defined as follows:

- Level I field screening or analysis using portable instruments. Results are often not compound specific and not quantitative but results are available in real time.
- Level II field analyses using more sophisticated portable analytical instruments. In some cases, the instruments may be set up in a mobile laboratory on site. There is a wide range in the quality of data that can be generated, depending on the use of suitable calibration standards, reference materials, and sample preparation equipment. Results are available in real-time or several hours of sample collection.
- Level III all analyses performed in an off-site analytical laboratory. Level III analyses may or may not use CLP procedures, but do not usually utilize the validation or documentation procedures required of CLP Level IV analysis. The laboratory may or may not be a CLP laboratory.
- Level IV CLP routine analytical services (RAS). All analyses are performed in an office CLP analytical laboratory following CLP protocols. Level IV is characterized by rigorous QA/QC protocols and documentation.
- Level V analysis by non-standard methods. All analyses are performed in an off-site analytical laboratory which may or may not be a CLP laboratory. Method development or method modification may be required for specific constituents or detection limits. CLP special analytical services (SAS) are Level V.
- 1.3.1.1 Soil Investigation

Data Uses

The soil investigation is designed to generate data to support the following evaluations:

- 1. Determine the presence and horizontal extent of chemical constituents in soil at the site; and
- 2. Characterize surface and subsurface soils at the site.

The primary intent of the soil investigation is to characterize the nature and extent of chemical constituents in the site soils. The soil data will also be used to assess the risks to human health and the environment associated with the level of constituents detected in the soils and to evaluate remedial/response alternatives, if necessary.

Data Types

The soil investigations will include the collection and analysis of soil samples for polychlorinated biphenyls (PCBs), Target Compound List (TCL) volatile and semi-volatile organic compounds, and Target Analyte List (TAL) inorganics. Table 1 of this QAPP presents the number of soil samples to be collected for analysis. Table 2 of this QAPP presents the specific chemical parameters for which the soil samples will be analyzed. Visual examination and photoionization device (PID) screening of soil samples from various depth

intervals will also be conducted to evaluate subsurface conditions at the site and to select soil samples for laboratory analysis as described in the RI/FS Work Plan and FSP.

The RI/FS Work Plan, as well as the FSP, provide for the rationale for the soil chemical parameters selected for analysis.

Data Quality

Analytical Level IV is considered appropriate for Contract Laboratory Program (CLP) procedure analyses for volatile organic and semi-volatile organic compounds and for inorganics. Analytical Level III is appropriate for PCB analysis using USEPA SW-846 Method 8080; however, a CLP-type data package, including laboratory documentation will be developed for data generated using Method 8080.

Analytical Level I is appropriate for the field screening of soil samples.

Data Quantity

Soil samples will be collected from 42 locations at the site. The soil sample locations are uniformly distributed throughout the site on a grid basis. As described in the RI/FS Work Plan and FSP, one surface soil sample will be selected from each sampling location for laboratory analysis and additional subsurface soil samples will be selected for analysis based on visual assessment and above background PID readings, as appropriate. The quantity of soil analytical data, including the required field and analytical QA/QC samples is summarized in Table 1. In addition, a background surface soil sample and background subsurface soil samples will be collected during the installation of ground-water monitoring well MW-7 located north of the site. These background soil samples will be analyzed for TAL inorganics.

Sampling and Analytical Methods

The FSP contains a description of the soil sampling procedures to be employed during the RI. The laboratory analytical methods to be utilized are listed in Table 2 of this QAPP.

PARCC Parameters

Precision and accuracy quality control (QC) limits for chemical constituents which are used during data validation to assess analytical performance, are included on Table 3. Published guidance QC limits are identified except as noted on Table 2.

Data representativeness is addressed by the sample quantities and locations identified in the RI/FS Work Plan and FSP. Data comparability is intended to be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the RI.

1.3.1.2 Sediment Investigation

Data Uses

The sediment investigation is designed to generate data to support the following evaluations:

1. Determine the presence and extent of chemical constituents in the on-site quarry pond sediments;

- 2. Determine the presence of chemical constituents in the quarry pond outlet channel sediments north of the railroad embankment; and
- 3. Determine the extent to which sediments act as source areas for chemical constituents.

The primary intent of the sediment investigation is to characterize the nature and extent of chemical constituents in sediments. The sediment data will also be used to assess the risks to human health and the environment associated with the level of constituents detected in sediments and to evaluate remedial/response alternatives, if necessary.

Data Types

The sediment investigation will include the collection and analysis of sediment samples for PCBs, TCL volatile and semi-volatile organics, TAL inorganics, total organic carbon, and particle size distribution (see Table 1 and Table 2 for number of samples and specific constituents). Sediment total organic carbon (TOC) and particle size distribution data will be obtained for use in evaluating constituent distribution and transport and will be used in evaluating potential remedial/response alternatives (if necessary).

The RI/FS Work Plan, as well as the FSP, provide further rationale for the sediment physical and chemical parameters selected for analysis.

Data Quality

Analytical Level IV is considered appropriate for CLP procedure analyses for volatile organic compounds semi-volatile organic compounds, and inorganics. Analytical Level III is appropriate for PCB analysis using USEPA SW-846 Method 8080; however, a CLP-type data package, including laboratory documentation will be developed for data generated using Method 8080.

For TOC and particle size distribution, Analytical Level III is considered appropriate because these data will be used to support the chemical constituent data.

Data Quantity

Seventeen surface (0-to 6-inch) sediment samples will be collected from the on-site quarry pond. These surface sediment sample locations have been uniformly distributed throughout the quarry pond on a grid basis. Six full-core samples will also be collected as described in the FSP. At each full-core location, a visual examination will be conducted and sediment samples will be selected for analysis at each 1 foot of sample depth. In addition, two sediment samples will be collected from the quarry pond outlet channel. Two surface sediment samples from the quarry pond outlet channel will be collected from 0- to 6-inch depths. The estimated quantity of sediment analytical data, including the required field and laboratory QA/QC samples that will be collected during the RI is summarized on Table 1.

Sampling and Analytical Methods

The FSP contains a description of the sediment sampling procedures to be employed during the RI. The laboratory analytical methods to be utilized are listed in Table 2 of this QAPP.

PARCC Parameters

Project analytical precision and accuracy QA limits for sediments, identified on Table 3, have been established to incorporate data quality objectives. A discussion of the general approach to evaluate sediment PARCC parameters is provided in the PARCC parameter discussion for soil.

1.3.1.3 Surface Water Investigation

Data Uses

The surface water investigation is designed to generate hydrologic and water quality data to support the following evaluations:

- 1. Determine the extent to which the surface water is a migration pathway for constituents associated with the site; and
- 2. Investigate the spatial distribution of chemical constituents in the quarry pond and the quarry pond outlet channel water column north of the railroad embankment.

The data obtained will be used to characterize the nature and extent of constituents in surface water associated with the site. The surface water data will also be used to assess the risks to human health and the environment associated with the level of constituents detected in the surface water and to evaluate applicable remedial/response alternatives, if necessary.

Data Types

The surface water will be analyzed for PCBs, TCL volatile organics and semi-volatile organics, and TAL inorganics (see Table 2 of this QAPP for specific constituents). These parameters will aid in characterizing the nature and extent of these target constituents in the site surface water. Surface water velocity and cross-section area measurements will be made during the quarry pond outlet sampling activities to calculate the instantaneous quarry pond outlet flow rate. The rationale for selection of the specific physical (i.e., total suspended solids) and chemical surface water parameters is discussed in detail in the RI/FS Work Plan, and the FSP, and in Section 3.0 of this QAPP. Water quality field parameters such as temperature, conductivity, pH, and dissolved oxygen will also be determined during the RI.

Data Quality

Analytical Level IV is considered appropriate for CLP procedure analyses for volatile organic compounds, semi-volatile organic compounds, and inorganics. Analytical Level III is appropriate for PCB analysis using USEPA SW-846 Method 8080; however, a CLP-type data package, including laboratory documentation will be developed for data generated using Method 8080.

For total suspended solids analysis, Analytical Level III is considered appropriate because these data will be used to support chemical constituent data.

Analytical Level I is appropriate for the surface water flow measurements and water quality field parameters.

Data Quantity

As described in the RI/FS Work Plan and FSP, 5 surface water samples will be collected from the quarry pond. Two surface water samples will be collected from the quarry pond outlet channel during normal flow conditions and two during a precipitation event. The quarry pond surface water sample locations have been selected to provide a uniform distribution of surface water data. Surface water samples will be collected in the quarry pond in such a manner as to characterize the water column at each sampling location as described in the RI/FS Work Plan and FSP. The number of surface water samples, including the required field and analytical QA/QC samples that will be collected during the RI is summarized on Table 1.

Sampling and Analytical Methods

The FSP contains a description of the surface water sampling procedures and methods for calculating surface water flow rates. The laboratory analytical methods being utilized for the chemical and physical parameters are listed in Table 2 of this QAPP.

PARCC Parameters

Precision and accuracy quality control (QC) limits for chemical constituents which are used during data validation to assess analytical performance are included on Table 4.

Data representativeness is addressed by the sample quantities and locations identified in the RI/FS Work Plan and FSP. Data comparability is intended to be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the RI.

1.1.3.4 Ground-Water Investigation

Data Uses

The ground-water investigation is designed to generate hydrogeologic and water quality data to support the following evaluations:

- 1. Determine ground-water quality at the site (including hydraulically upgradient, sidegradient and downgradient water quality);
- 2. Characterize the ground-water flow system at the site, including flow directions, gradients, and velocities; and
- 3. Determine the geological characteristics of overburden and bedrock at the site which could affect the migration of constituents from the site.

The data obtained will be used primarily to characterize the nature and extent of the chemical constituents in the ground water. These data will also be used to assess the risks to human health and the environment associated with the level of constituents detected in the ground water and to evaluate applicable remedial/response alternatives, if necessary.

Data Types

As set forth in the RI/FS Work Plan and above, both hydrogeologic and water quality data are required to meet the objective of the ground-water investigation and subsequently, to use the ground-water data for its intended purposes. Hydrogeologic data will consist of

water level information which will be used to calculate other hydrogeologic parameters. Water quality data will consist of field parameters, including: pH, temperature, conductivity and dissolved oxygen, as well as laboratory parameters, including: PCBs, volatile, semivolatile, and inorganic constituents (see Table 1 for parameters and Table 2 of this QAPP for specific constituents). The rationale for the selection of these parameters is discussed in detail in the RI/FS Work Plan, the FSP, and Section 3.0, herein.

Hydraulic conductivity testing will also be performed during the ground-water investigation. This will consist of obtaining water level measurements over time after a known volume of water has been added or removed from each ground-water monitoring well.

In addition, two soil/bedrock cores will be installed on-site as described in the RI/FS Work Plan and FSP. During the soil/bedrock core installations and new ground-water monitoring well installations, overburden soil samples will be obtained for visual characterization for color, texture, moisture, and soil types. Bedrock cores collected during the soil/bedrock core activities will be visually characterized for color, rock type, fractures, and weathering. These assessments will be used to aid in meeting the objectives of the ground-water investigation.

Data Quality

Analytical Level IV is considered appropriate for CLP procedure analyses for volatile organic compounds, semi-volatile organic compounds, and inorganics. Analytical Level III is appropriate for PCB analysis using USEPA SW-846 Method 8080; however, a CLP-type data package, including laboratory documentation will be developed for data generated using Method 8080.

Analytical Level I is appropriate for the surface water flow measurements and water quality field parameters.

Data Quantity

The ground-water investigation will involve the collection of ground-water samples from 4 existing monitoring wells and from 3 new monitoring wells (to be installed as part of the RI) on and near the M. Wallace and Son Scrapyard Site for field and laboratory analyses, as well as the measurement of ground-water levels in those wells. The existing and new well locations were selected to provide information on the water quality and movement of ground water through the site bedrock. Two additional wells may be installed based on the results of the soil/bedrock coring investigation south of the site (as described above). These additional wells, if installed, will be constructed to screen ground water in the overburden or weathered bedrock depending upon the coring results. The quantity of ground-water analytical data, including QA/QC samples, that will be collected during the RI is summarized in Table 1.

Data quantity related to the water level measurements, hydraulic conductivity testing, and water quality measurements are described in the FSP.

Sampling and Analytical Methods

The ground-water level measurement procedures, water quality measurement procedures, hydraulic conductivity testing procedures, and ground-water sampling procedures are provided in the FSP. The laboratory analytical methods for ground-water samples are listed in Table 2.

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PARCC Parameters

PARCC parameters for ground water are the same as those specified for surface water.

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2.0 - PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 PROJECT ORGANIZATION

The M. Wallace and Son, Inc. Scrapyard Site RI will require integration of personnel from the organizations identified below, collectively referred to as the project team. A project organization chart depicting the project team personnel is included as Figure 1. A detailed description of the responsibilities of each member of the project team is presented below.

2.1.1 Overall Project Management

Blasland & Bouck Engineers, P.C., (Blasland & Bouck) on behalf of Niagara Mohawk Power Corporation (NMPC), has overall responsibility for the M. Wallace and Son, Inc. Scrapyard Site RI/FS. Blasland & Bouck personnel will perform the ground water, surface water, soil, and sediment investigations; the risk assessment; the air emissions assessment; the potential interim remedial measures assessment and the feasibility study. In addition, Blasland & Bouck will be responsible for evaluating resultant investigation data, and preparing the RI/FS deliverables specified in the RI/FS Work Plan and FSP. Project direction and oversight will be provided by NMPC personnel. Oversight in the field may also be provided by NMPC. A listing of key project management personnel is provided below.

Project Title	Company/Organization	Name	Phone Number
Project Manager	Niagara Mohawk Power Corporation	Mr. James F. Morgan	(315) 428-3101
Project Officer	Blasland & Bouck	Edward R. Lynch, P.E.	(315) 446-9120
Project Manager	Blasland & Bouck	David J. Ulm	(315) 446-9120
Project Coordinator	New York State Department of Law	Albert M. Bronson, Esq.	(518) 474-8480
Project Coordinator	New York State Department of Environmental Conservation	Daniel R. Lightsey	(518) 382-6680

2.1.2 Task Managers

The staff performing the investigative and engineering activities of the RI/FS will be directed by representatives of Blasland & Bouck. The personnel responsible for each of the RI/FS tasks are listed below.

Project Title	Company	Name	Phone Number
Environmental Media Investigation Task Manager	Blasland & Bouck	Nancy E. Gensky	(315) 446-9120
Risk Assessment/Biota Investigation Task Manager	Blasland & Bouck	Michele A. Anatra-Cordone, Ph.D.	(315) 446-9120
Feasibility Study Task Manager	Blasland & Bouck	David J. Ulm	(315) 446-9120
Health and Safety Manager	Blasland & Bouck	Marc B. Evans, C.I.H, C.S.P	(315) 446-9120

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2.1.3 Analytical Laboratory and Data Validation Services

Laboratory analytical services for environmental media samples associated with the M. Wallace and Son Scrapyard RI will be provided by Aquatec, Incorporated (Aquatec).

Analytical data identified in Section 8.2.3 of this QAPP will be transmitted to OBG Laboratories, Inc., personnel for independent data validation. Laboratory and data validation management personnel are listed below.

Title	Company	Name	Phone Number
Laboratory Project Manager	Aquatec	Pauline T. Malik	(802) 655-1203
Independent Data Validator	OBG Laboratories, Inc.	Robert A. Martin	(315) 437-0200

2.1.4 Quality Assurance Staff

The QA aspects of the RI/FS will be conducted by Blasland & Bouck, Aquatec, OBG Laboratories, Inc., and representatives of the New York State Department of Environmental Conservation (NYSDEC). To date, the following personnel have been assigned to this project component:

Title	Company/Organization	Name	Phone Number
Quality Assurance Officer	Biasiand & Bouck	Frederick J. Kirschenheiter	(315) 446-9120
Quality Assurance Officer	Aquatec	Karen Chirgain	(802) 655-1203
Independent Data Validator	OBG Laboratories, Inc.	Robert A. Martin	(315) 437-0200
Quality Assurance Officer	NYSDEC	To be assigned by NYSDEC	

2.2 TEAM MEMBER RESPONSIBILITIES

This section of the QAPP discusses the responsibilities and duties of the project team members.

2.2.1 Niagara Mohawk Power Corporation

Project Manager

Responsibilities and duties include:

- 1. Overall direction of the RI/FS;
- 2. Direction of Blasland & Bouck and coordination with regulatory agencies; and
- 3. Review of Blasland & Bouck work products, including data, memoranda, letters, and reports and all documents transmitted to the New York State Department of Law (NYSDOL) and NYSDEC.

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2.2.2 Blasland & Bouck Engineers, P.C.

Project Officer

Responsibilities and duties include:

- 1. Oversight of the Blasland & Bouck RI/FS work products; and
- 2. Provide Blasland & Bouck approval for major project deliverables.

Project Manager

Responsibilities and duties include:

- 1. Management and coordination of all aspects of the project as defined in the RI/FS Work Plan with an emphasis on adhering to the objectives of the RI;
- 2. Review RI Report and all documents prepared by Blasland & Bouck; and
- 3. Assure corrective actions are taken for deficiencies cited during audits of RI/FS activities.

Task Managers

The M. Wallace and Son, Inc. Scrapyard Site RI will be managed by Task Managers as set forth in Section 2.1.2. Responsibilities and duties of each Task Manager include:

- 1. Manage day-to-day relevant RI activities;
- 2. Develop, establish, and maintain files on relevant RI activities;
- 3. Review data reductions from the relevant RI activities;
- 4. Perform final data review of field data reductions and reports on relevant RI activities;
- 5. Assure corrective actions are taken for deficiencies cited during audits of relevant RI activities;
- 6. Overall QA/QC of the relevant portions of the RI;
- 7. Review all relevant field records and logs;
- 8. Instruct personnel working on relevant RI activities;
- 9. Coordinate field and laboratory schedules pertaining to relevant RI activities;
- 10. Request sample bottles from laboratory;
- 11. Review the field instrumentation, maintenance, and calibration to meet quality objectives;
- 12. Prepare sections of RI report pertaining to relevant RI activities; and
- 13. Maintain field and laboratory files of notebooks and logs, data reductions and calculations, and transmit originals to the Project Manager.

Field Personnel

Responsibilities and duties include:

- 1. Perform field procedures associated with the ground-water, surface water, sediment, soil, and biota investigations as set forth in the FSP;
- 2. Perform field analyses and collect QA samples;
- 3. Calibrate, operate, and maintain field equipment;
- 4. Reduce field data;
- 5. Maintain sample custody; and
- 6. Prepare field records and logs.

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Quality Assurance Officer (QAO)

Responsibilities and duties include:

- 1. Review laboratory data packages;
- 2. Oversee and interface with the analytical laboratories;
- 3. Oversee and interface with the independent data validator;
- 4. Coordinate field QA/QC activities with task managers, including audits of RI activities, concentrating on field analytical measurements and practices to meet data quality objectives;
- 5. Review field reports;
- 6. Review audit reports;
- 7. Prepare interim QA/QC compliance reports; and
- 8. Prepare QA/QC report which includes an evaluation of field and laboratory data and data validation reports.

2.2.3 Aquatec, Incorporated

General responsibilities and duties of Aquatec include:

- 1. Perform sample analyses and associated laboratory QA/QC procedures;
- 2. Supply sampling containers and shipping cartons;
- 3. Maintain laboratory custody of sample; and
- 4. Strictly adhere to all protocols in the QAPP.

Project Manager

Responsibilities and duties include:

- 1. Serve as primary communication link between Blasland & Bouck and laboratory technical staff;
- 2. Monitor work loads and ensure availability of resources;
- 3. Oversee preparation of analytical reports; and
- 4. Supervise in-house chain-of-custody.

Quality Assurance Officer

Responsibilities and duties include:

- 1. Supervise the group which reviews and inspects all project-related laboratory activities; and
- 2. Conduct audits of all laboratory activities.

Sample Custodian

Responsibilities and duties include:

- 1. Receive all samples; and
- 2. Maintain custody of the samples and all documentation.

Laboratory Data Reviewer

Responsibilities and duties include:

1. Verify final analytical data prior to transmittal to Blasland & Bouck.

2.2.4 OBG Laboratories, Inc.

Responsibilities and duties include:

- 1. Provide independent validation of analytical data; and
- 2. Prepare validation report for incorporation into RI Report.

2.2.5 Parratt-Wolff, Inc.

General responsibilities and duties include:

- 1. Performance of RI ground-water monitoring well installations, test pits, and soil/rock borings in accordance with the RI protocols in the FSP;
- 2. Decontamination of drilling equipment; and
- 3. Well development.

2.2.6 New York State Department of Law (NYSDOL)

Project Coordinator

Responsibilities and duties include:

- 1. Provide NYSDOL approval of the RI Work Plan, SAP, supporting documents, and future RI/FS deliverables;
- 2. Provide oversight during performance of the RI/FS.

2.2.7 New York State Department of Environmental Conservation (NYSDEC)

Project Coordinator

Responsibilities and duties include:

- 1. Provide NYSDEC approval of RI/FS Work Plan, SAP, supporting documents and future RI/FS deliverables; and
- 2. Provide oversight during performance of the RI/FS.

Quality Assurance Officer

Responsibilities and duties include:

- 1. Review and approval of the QAPP;
- 2. Review of the QA/QC portion of the RI Report; and
- 3. Field and laboratory audit responsibilities, if determined necessary.

3.0 - QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT OF DATA

3.1 SELECTION OF MEASUREMENT PARAMETERS, LABORATORY METHODS, AND FIELD TESTING METHODS

3.1.1 Field Parameters and Methods

3.1.1.1 Field Parameters

During the ground-water and surface water investigations, field parameters consisting of pH, conductivity, dissolved oxygen and temperature will be measured to provide general water quality information. Field test methods to measure pH, conductivity, dissolved oxygen and temperature are presented in Appendix G of the FSP.

Soil samples collected as part of the soil investigation will be screened with a PID to determine the presence and approximate levels of volatile organic compounds in the site soil. PID measurement protocols are presented in Appendix G of the FSP.

During the RI, a site topographic survey will be conducted with the accuracy and precision requirements discussed in the FSP. In addition, site soil samples, sediment samples, and surface water samples will be surveyed to the nearest foot. Top of monitoring site well casing elevations will be obtained to the nearest 0.1 of a foot.

3.1.1.2 Hydrogeologic Measurements

As described in the FSP, ground-water levels will be measured prior to sampling. In-situ hydraulic conductivity measurements will be performed as described in the FSP. Ground-water levels will be measured using the procedures presented in Appendix K of the FSP.

3.1.1.3 Surface Water Measurements

Surface water flow rate measurements will be collected during the surface water sampling events as described in the FSP. Velocity measurements will be made using electronic equipment and channelized surface water channel dimensions will be obtained at the frequencies described in the surface water flow measurement protocols presented in Appendix H of the FSP.

3.1.2 Laboratory Parameters and Methods

As described in the RI/FS Work Plan and FSP, laboratory analyses of ground water, surface water, soil, and sediment will be performed as set forth in Table 1. The analytical parameters selected for each media are described in the RI/FS Work Plan and FSP. Table 2 presents the chemical constituents identified by matrix, along with the selected analytical methods and reporting limits. If other constituents are detected during the performance of the selected analytical methods, they will be identified in the laboratory report.

In order to support the risk assessment, aid in determining the potential for off-site chemical constituent migration and to aid in evaluating appropriate remedial/response alternatives, filtered and unfiltered ground water and surface water samples will be collected at each proposed sampling location for PCB analysis and inorganic analysis as described in the RI/FS Work Plan and FSP.

Supplemental parameters will be analyzed to provide additional information regarding the on-site media as discussed in the RI/FS Work Plan and FSP at the frequency set forth in Table 1. Table 2 presents these supplemental parameters identified by matrix, with the selected analytical methods and reporting limits, if applicable. These parameters were selected to provide ancillary data to support the chemical constituent data.

For sediments, the supplemental parameters include proportion of organic carbon (also referred to as TOC) and particle size distribution. For surface water samples, total suspended solids analysis will be performed on all collected samples.

3.2 QUALITY ASSURANCE OBJECTIVES

The overall quality assurance objective for this RI/FS is to develop and implement procedures for sampling, chain-of-custody, laboratory analysis, instrument calibration, data reduction and reporting, internal quality control, audits, preventive maintenance, and corrective action, such that valid data will be generated. These procedures are presented or referenced in the following sections of the QAPP. Specific QC checks are discussed in Section 9.0 of this QAPP.

Quality assurance objectives are generally defined in terms of five parameters:

- 1. Representativeness;
- 2. Comparability;
- 3. Completeness;
- 4. Precision; and
- 5. Accuracy.

Each parameter is defined below. Specific objectives for this RI are set forth in other sections of this QAPP as referenced below.

3.2.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of the site. The RI has been designed to assess the presence of the chemical constituents and supplemental parameters at the time of sampling. The RI Work Plan and FSP presents the rationale for sample quantities and location. The FSP and this QAPP present field sampling methodologies and laboratory analytical methodologies, respectively. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data. Further discussion of QC checks is presented in Section 9.0 of this QAPP.

3.2.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between phases of the RI, and to the extent possible, with existing data will be maintained through consistent sampling and analytical methodologies set forth in this QAPP, the FSP through the use of established QA/QC procedures, and through utilization of appropriately trained personnel. The comparability of RI data with existing data is limited by uncertainties associated with sampling and analytical differences.

3.2.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 12.0 of this QAPP.

3.2.4 Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the RI. To maximize precision, sampling and analytical procedures will be followed. All work for this RI will adhere to established protocols presented in the QAPP and FSP. Checks for analytical precision will include the analysis of matrix spike, matrix spike duplicates, laboratory duplicates and field duplicates. Checks for field measurement precision will include obtaining duplicate field measurements. Further discussion of precision QC checks is provided in Sections 9.0 and 12.0 of this QAPP.

3.2.5 Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, matrix spikes, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data. Further discussion of these QC samples is provided in Sections 9.0 and 12.0 of this QAPP.

4.0 - SAMPLING PROCEDURES

Ground water, surface water, soil, and sediment samples will be collected as described in the FSP. In addition, the FSP contains the procedures for installing monitoring wells; measuring ground-water levels; performing field measurements; calculating in-situ hydraulic conductivity; and handling, packing, and shipping of RI samples.

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5.0 - SAMPLE AND DOCUMENT CUSTODY

5.1 FIELD PROCEDURES

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through time of transport to the analytical laboratory. Persons will have "custody of samples" when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel. A discussion of sample custody and directions for the field use of chain-of-custody forms are provided in the FSP. An example field chain-of-custody form is also provided in Appendix N of the FSP.

5.2 LABORATORY PROCEDURES

5.2.1 General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain-of-custody form will accompany all samples requiring laboratory analysis. The laboratory will use chain-of-custody guidelines described in the CLP-SOW for organic analysis, Exhibit F. Requirements which specifically pertain to EPA contracts (i.e., EPA Traffic Reports, etc.) are not relevant to this project. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

5.2.2 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the package seal, open the package, and compare the contents against the field chain-of-custody. At this time, the laboratory sample custodian will also be responsible for logging the samples in, assigning a unique laboratory identification number to each, and labelling the sample bottle with the laboratory identification number. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory logbook. If a sample container is broken, the sample is in an inappropriate container, or has not been preserved by appropriate means, Blasland & Bouck will be notified.

5.2.3 Sample Analysis

Analysis of an acceptable sample will be initiated by a work sheet which will contain all pertinent information for analysis. The routing sheet will be forwarded to the analyst, and the sample will be moved into an appropriate storage location to await analysis. The analyst will sign and date the laboratory chain-of-custody form when removing the samples from storage. The document control officer will file all chain-of-custody forms in the project file.

Samples will be organized into sample delivery groups (SDGs) by the laboratory according to both matrix and analysis parameter. A SDG may contain up to 20 field samples (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG must be received by the laboratory over a maximum of 7 calendar days (less, when 7-day holding times for extraction must be met), and must be processed through the laboratory (preparation, analysis, and reporting) as a group. Every SDG must include a minimum of one MS/MSD (or MS/lab dup) pair.

Each SDG will therefore be self-contained for all of the required quality control samples. All parameters within an SDG will be extracted and analyzed together in the laboratory. At no time will the laboratory be allowed to run any sample (including QC samples) at an earlier or later time than the rest of the SDG. An entire SDG for any single parameter will be analyzed on a single instrument within the laboratory. These rules for analysis will ensure that the quality control samples for an SDG are applicable to the field samples of the same SDG, and that the best possible comparisons may be made.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded in a laboratory notebook by the analyst. These notes will be dated, and also identify the analyst, the instrument used, and the instrument conditions.

5.2.4 Laboratory Project Files

During the RI, Aquatec will establish a file for all pertinent data. The file will include the chain-ofcustody forms, raw data, chromatograms (required for all constituents analyzed by chromatography), and sample preparation information. Aquatec will retain project records until the conclusion of the RI, at which time they will be transferred to Blasland & Bouck or NMPC for continued storage, as necessary.

5.2.5 Laboratory Documentation

5.2.5.1 Aquatec Procedures

Documentation

Workbooks, bench sheets, instrument logbooks, and instrument printouts, are used to trace the history of samples through the analytical process, and document and relate important aspects of the work, including the associated quality controls. All logbooks, bench sheets, instrument logs, and instrument printouts are part of the permanent record of the laboratory. Completed workbooks and instrument logbooks are submitted to Aquatec's internal data review groups for review and storage (Aquatec, Inc., 1992).

As required, each page or entry is to be dated and initialed by the analyst at the time the record is made. Entries in the standards logbooks and runlogs are made in duplicate using carbon sheets. Errors in entry are to be crossed out in indelible ink with a single stroke and corrected without the use of white-out or by obliterating or writing directly over the erroneous entry. All corrections are to be initialed and dated by the individual making the correction. Pages inserted into logbooks are to be stapled to a clean, bound page. The analyst's initials are to be recorded in such a manner that the initials overlap the inserted page and the bound page. A piece of nonremovable transparent tape is then to be placed over the initials as a seal. Pages of logbooks that are not completed as part of normal record keeping should be completed by lining out unused portions (Aquatec, Inc., 1992).

Laboratory notebooks are periodically reviewed by the laboratory section leaders for accuracy, completeness, and compliance to this QAPP. All entries and calculations are verified by the laboratory section leader. If all entries on the pages are correct, then the laboratory section leader initials and dates the pages. Corrective action is taken for incorrect entries before the laboratory section leader signs (Aquatec, Inc., 1992).

Computer Tape Storage

Magnetic computer tapes are stored in the computer room, and corresponding tape streamer logbooks are maintained for a minimum of seven years (Aquatec, Inc., 1992).

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Sample Storage Following Analysis

Once an analysis is complete, the unused portion of sample and all identifying tags and laboratory records will be maintained by the laboratory. Samples will be retained at Aquatec for a period of three months, after which Blasland & Bouck, NMPC, and New York State personnel will determine the need for continued storage.

5.3 PROJECT FILE

RI documentation will be placed in a single project file at the Blasland & Bouck office in Syracuse, New York. This file will consist of the following components:

- 1. Agreements (filed chronologically);
- 2. Correspondence (filed chronologically);
- 3. Memos (filed chronologically); and
- 4. Notes and Data (filed by topic).

Reports (including QA reports) will be filed with correspondence. Analytical laboratory documentation (when received) and field data will be filed with notes and data. Filed materials may be removed and signed out by personnel on a temporary basis only.



6.0 - CALIBRATION PROCEDURES AND FREQUENCY

6.1 FIELD EQUIPMENT CALIBRATION PROCEDURES AND FREQUENCY

Specific procedures for performing and documenting calibration and maintenance for the equipment measuring conductivity, temperature, dissolved oxygen, pH, surface water velocity, and ground-water level and organic vapors are provided in Appendix G of the FSP. Calibration checks will be performed daily when measuring conductivity, temperature, dissolved oxygen, water velocity, and pH. For ground-water sampling, the pH meter will be calibrated at each sampling location. Field equipment, frequency of calibration, and calibration standards are provided in Table 5.

6.2 LABORATORY EQUIPMENT CALIBRATION PROCEDURES AND FREQUENCY

Instrument calibration will follow the specifications provided by the instrument manufacturer or specific analytical method used. The analytical methods for chemical constituents and supplemental parameters are identified separately below.

6.2.1 Chemical Constituents

CLP-TCL/TAL

Initial and continuing instrument equipment calibration will follow, at a minimum, CLP guidelines (SOW OLM 01.8 and SOW ILM 02.1).

PCBs

Instrument calibration procedures will follow guidelines presented in SW-846 Method 8080.

6.2.2 Supplemental Parameters

Analysis of the supplementary parameters identified below will require use of calibration procedures and frequencies as specified in the respective methods outlined in Table 2:

Surface Water

Total Suspended Solids

Sediment Samples

Total Organic Carbon Particle Size Distribution

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7.0 - ANALYTICAL PROCEDURES

7.1 FIELD ANALYTICAL PROCEDURES

Field analytical procedures will include the measurement of temperature, conductivity, dissolved oxygen, pH, organic vapors, surface water flow velocity, and ground-water levels. Specific field measurement protocols are provided in the Appendices of the FSP. Field measurement quality control limits in terms of precision and accuracy are presented in Table 6.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analytical requirements presented in the sub-sections below include a general summary of requirements, specifics related to each sample medium to be analyzed, and details of the methods to be used for this project. Current CLP methods will be used with the following exceptions: PCBs, TOC, particle size distribution, and total suspended solids.

7.2.1 General

The following tables summarize general analytical requirements:

Table	Title
Table 1	Environmental and Quality Control Sample Analyses
Table 2	Parameters, Methods and Reporting Limits
Table 7	Sample Containers, Preservation Methods, and Holding Times Requirements

7.2.2 RI Sample Matrices

7.2.2.1 Water

Matrices in this category consist of surface water and ground water. For samples requiring filtering, samples will be filtered in the field using a 0.45-micron pore glass fiber filter, or equivalent as described in the FSP. Analytical results for all analyses will be reported in units identified in Table 2.

7.2.2.2 Soil/Sediment

Analyses in this category relates to sediment and soil samples. Results will be reported as dry weight, in units presented in Table 2. Moisture content will be reported separately. QC limits cited in Table 3 are intended for soil analyses and are generally applied to sediment analyses, as well. However, matrix differences between soils and sediments (i.e., higher moisture content of sediments) may affect method performance. Therefore, the QC limits are considered advisory for sediment analyses.

7.2.3 Analytical Requirements

The primary sources for methods used for this investigation are provided in the following documents:

Statement of Work for Organic Analysis EPA Contract Laboratory Program (CLP), Document Number OLM 01.8 (3/90, revisions through 8/91).

Statement of Work for Inorganic Analysis EPA Contract Laboratory Program (CLP), Document Number ILM 02.0, (3/90).

Test Methods for Evaluating Solid Waste, SW-846 Third Edition, Revision 1, EPA, November 1990.

Methods for Chemical Analysis of Water and Wastes (MCAWW), EPA, 1983.

All analyses will be performed by Aquatec, Inc.

Tables summarizing QC limits required to evaluate analytical performance are provided as follows:

Table	Title	
3	Soil/Sediment Analyses Quality Control Limits	
4	Water Analyses Quality Control Limits	

As identified in Tables 3 and 4, matrix spike/matrix spike duplicate precision for applicable organic analyses will be evaluated as noted on the tables. Also, assessment of the supplemental parameters will generally be based on duplicate sample results.

7.2.3.1 Chemical Constituents

Organic and inorganic analyses will be performed by CLP-SOW methods, and will be reported as complete data validation packages using CLP forms.

Organic analyses performed by SW-846 methods (i.e., PCBs) will be reported to the reporting limits identified in Table 2. If additional constituents are detected by the laboratory during the analysis of chemical constituents, they will be reported, as well. A brief summary of the non-CLP analytical methods to be used during this project for chemical constituents is provided below.

PCBs

Soil, sediment, ground water, and surface water samples will be analyzed using USEPA SW-846 Method 8080 (USEPA 1990). Appendix A of this QAPP presents the Method 8080 procedures.

7.2.3.2 Supplemental Parameters

Total Suspended Solids

Surface water samples will be analyzed for total suspended solids by USEPA Method 160.2 as described in the USEPA document title Methods for Chemical Analysis of Water and Wastes (USEPA 1983).



Total Organic Carbon

Sediment samples will be analyzed for TOC according to the Lloyd Kahn Method, USEPA Region II (7/88).

Particle Size Distribution

Sediment samples will be analyzed according to American Society for Testing and Materials (ASTM) Procedure D-422.

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8.0 - DATA REDUCTION, VALIDATION, AND REPORTING

After field and laboratory data are obtained, these data will be subject to:

- 1. Validation;
- 2. Reduction or manipulation mathematically or otherwise into meaningful and useful forms; and
- 3. Organization, interpretation, and reporting.

8.1 FIELD DATA REDUCTION, VALIDATION, AND REPORTING

8.1.1 Field Data Reduction

Information which is collected in the field through visual observation, manual measurement and/or field instrumentation will be recorded in field notebooks, datasheets, and/or forms. Such data will be reviewed by the appropriate Task Manager for adherence to the FSP and consistency. Any concerns identified as a result of this review will be discussed with the field personnel corrected if possible, and as necessary incorporated into the data evaluation process.

8.1.1.1 Sediment Investigation

Specific data reduction activities which will be performed for the sediment investigation include:

1. Calculation and mapping of sediment deposition areas and depths based on sediment sampling activities.

8.1.1.2 Surface Water Investigation

Field data reduction activities which will be performed during the surface water investigation include:

1. Calculation of quarry pond outlet channel surface water flow based on velocity measurements and drainage channel characteristics.

8.1.1.3 Ground-Water Investigation

Reduction of the field data collected during the ground-water investigation will include:

- 1. Calculation of water elevations by subtracting the depth-to-water data from the surveyed elevation of the measuring point;
- 2. Calculation of in-situ hydraulic conductivities;
- 3. Production of hydrogeologic contour maps by contouring lines of equal water elevations using linear interpolation through known elevation points; and
- 4. Addition of ground-water elevations to database of hydrogeologic measurements.
8.1.2 Field Data Validation

Field data calculations, transfers, and interpretations will be conducted by the field personnel and reviewed for accuracy by the appropriate Task Manager and the QAO. Task Managers will recalculate at least five percent of all data reductions. All logs and documents will be checked for:

- 1. General completeness;
- 2. Readability;
- 3. Usage of appropriate procedures;
- 4. Appropriate instrument calibration and maintenance;
- 5. Reasonableness in comparison to present and past data collected;
- 6. Correct sample locations; and
- 7. Correct calculations and interpretations.

8.1.3 Field Data Reporting

Where appropriate, field data forms and calculations will be processed and included in appendices to the RI Report. The original field logs, documents, and data reductions will be kept in the project file at the Blasland & Bouck office in Syracuse, New York.

8.2 LABORATORY DATA REDUCTION, REVIEW, AND REPORTING

8.2.1 ____ Laboratory Data Reduction

Laboratory analytical data will be directly transferred from the instrument to the computer or the data reporting form (as applicable) by the analyst. Calculation of sample concentrations will be performed using the calculation procedures specified by the analytical method used including, as applicable, regression analysis, response factors, and dilution factors.

8.2.2 Laboratory Data Review

8.2.2.1 Aquatec Review Procedures

Each Aquatec laboratory section provides extensive data review according to the methods used, prior to reporting results to Blasland & Bouck. In general, there are three levels of review as outlined below.

The analyst is responsible for primary review of data generated from sample analysis. If recoveries of all QC samples are within specified QC limits, then the data are presented to data review groups for secondary review. If recoveries of any QC samples exceed specified QC limits, then affected samples are reanalyzed (Aquatec, Inc., 1992).

Secondary review is conducted by data review groups to determine if analytical results are within established QC limits. If recoveries of all QC samples are within specified tolerances, then the data are presented to the Aquatec Project Manager for final review. If recoveries of any QC samples exceed specified tolerances, then affected samples are submitted for reanalysis (Aquatec, Inc., 1992).

The Aquatec Project Manager determines if all analytical results of a sample(s) are consistent. If so, then the data are presented in a final report. If discrepancies or deficiencies exist in the analytical results, then corrective action is taken (Aquatec, Inc., 1992) as discussed in Section 13. Deficiencies discovered as a result of internal data validation, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form (Appendix B). This form will be submitted to the Blasland & Bouck Project Manager.

8.2.3 Laboratory Data Reporting

The laboratory is responsible for reporting the data in tabular form. Data will be tabulated by method and sample with reference to the sample by both field and laboratory identifications. The data tables will provide a cross-reference between each sample and the appropriate QC data package. In addition, the laboratory will provide documentation backup (laboratory calculation sheets, chain-of-custody documentation, etc.).

For the laboratory analyses identified below, a full CLP data package and case narrative will also be provided for each sample delivery group.

Matrix		Data Type	•	
Water, Soil, and Sediment	TCL Volatile Org	anics, TCL Se	mi-Volatile ituents	e Organics,

A CLP-type data package will be provided for each sample delivery group analyzed for PCBs.

In addition, sample preparation records including extraction sheets, digestion sheets, percent solids, and logbook pages will also be provided in the data package.

8.3 INDEPENDENT DATA VALIDATION

Data validation entails a review of the QC data and the raw data to verify that the laboratory was operating within required limits, the analytical results are correctly transcribed from the instrument read outs, and which, if any, environmental samples are related to any out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

An independent data validator, OBG Laboratories, Inc. has been selected to validate the laboratory data for CLP and non-CLP analyses. OBG Laboratories, Inc., is not directly associated with the RI work efforts or laboratory analyses, and as such OBG Laboratories, Inc. responsibility will be to objectively review the analytical data. Data validation will consist of data editing, screening, checking, auditing, review, and interpretation to document analytical data quality and determine if the quality is sufficient to meet the data quality objectives. In addition, data validation will include a review of completeness and compliance, including the elements provided in Table 8, as well as the actual validation.

The independent data validator will use the most recent versions of the following EPA documents, available at the time of project initiation and for the entire duration of the project, as guidance, where appropriate:

- 1. Laboratory Data Validation, Functional Guidelines for Evaluating Inorganic Analyses ("Draft", July 1988), USEPA;
- 2. National Functional Guidelines for Organic Analyses (Draft, December 1990, Rev. June 1991);
- 3. Evaluation of Organics Data for the CLP; EPA Region II, (HW-6, Rev 8, January 1992); and
- 4. Evaluation of Metals Data for the CLP; EPA Region II (NW-2 Rev. II, January 1992).

OBG Laboratories, Inc., will verify reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method (i.e., perform laboratory calculations in accordance with the method-specific procedure) and/or as specified in this QAPP. Any deviations from the analytical method will be delineated on chain of custody forms. Any special reporting requirements apart from this QAPP will also be detailed on chain of custody forms. The data

quality will be evaluated by application of the Functional Guidelines procedures and criteria modified as necessary to address project-specific and method-specific criteria, control limits, and procedures.

Upon receipt of the laboratory data, the following reduction, validation and reporting scheme will be executed by OBG Laboratories, Inc.:

- 1. Laboratory data will be screened to ensure that the necessary QC procedures (detection limit verification, initial calibration, continuing calibration, duplicates, spikes, reagent blanks, etc.) have been performed. QC information not included or of insufficient frequency will be identified in the validation report along with a discussion of the implications.
- 2. QC supporting information will subsequently be screened to identify QC data outside established control limits. If out-of-control data are discovered, documentation of appropriate corrective action will be reviewed. Certain out-of-control data without appropriate corrective action shall result in designation of the affected data as qualified or rejected.

It should be noted that the existence of qualified results does not automatically invalidate data. This point is repeatedly emphasized in the EPA "Functional Guidelines for Inorganics Analysis" and is inherently acknowledged by the very existence of the data validation/flagging guidelines. The goal to produce the best possible data does not necessarily mean producing data without QC qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between OBG Laboratories, Inc., and the Blasland & Bouck QAO. Suggestions for reanalysis may be made to the Blasland & Bouck QAO at this point.

Upon completion of the validation of each sample delivery group/parameter, a report addressing the following topics as applicable to each method will be prepared.

- 1. Assessment of the data package;
- 2. Description of any protocol deviations;
- 3. Failures to reconcile reported and/or raw data;
- 4. Assessment of any compromised data;
- 5. Laboratory case narrative;
- 6. Overall appraisal of the analytical data; and
- 7. Table of site name, sample quantities, data submitted to the laboratory, year of protocol used, matrix, and fractions analyzed.

The data validation reports will be included as an appendix to the RI Report, if appropriate, and kept in the project file at the Blasland & Bouck office in Syracuse, New York.

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9.0 - FIELD AND LABORATORY QUALITY CONTROL CHECKS

Both field and laboratory quality control checks are proposed for the M. Wallace and Son, Inc. Scrapyard Site RI. In the event that there are any deviations from these checks, the Blasland & Bouck QAO will be notified. The proposed field and laboratory control checks are discussed below.

9.1 FIELD QUALITY CONTROL CHECKS

9.1.1 Field Measurements

To verify the quality of data using field instrumentation, duplicate measurements will be obtained and reported for all field measurements. A duplicate measurement will involve obtaining measurements a second time at the same sampling location.

9.1.2 Sample Containers

Certified-clean sample containers (I-Chem 300 series or equivalent) will be supplied by Aquatec, Inc. Certificates of analysis will be filed in the project file.

9.1.3 Field Duplicates

Field duplicates will be collected for water and soil/sediment samples to check reproducibility of the sampling methods. Field duplicates will be prepared as discussed in the FSP. In general, soil/sediment. surface water, and ground-water sample field duplicates will be analyzed at a 5 percent frequency (every 20 samples) for both the chemical constituents and the supplemental parameters. Table 1 provides an estimated number of field duplicates to be prepared for each applicable parameter and matrix.

9.1.4 Rinse Blanks

Rinse blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Rinse blanks will be prepared and submitted for analysis at a frequency of one per day (when sample equipment cleaning occurs) or once for every 20 samples collected, whichever is more. Rinse blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) which has been routed through a cleaned sampling device. When dedicated sampling devices are used or sample containers are used to collect the samples, rinse blanks will not be necessary. Table 1 provides an estimated number of rinse blanks for environmental media samples to be collected during the RI.

9.1.5 Trip Blanks

Trip blanks will be used to assess whether site samples have been exposed to non-site-related volatile constituents during sample storage and transport. Trip blanks will be analyzed at a frequency of once per day, per cooler containing surface water and/or ground-water samples to be analyzed for volatile organic constituents. A trip blank will consist of a container filled with analyte-free water (supplied by the laboratory) which remains unopened with field samples throughout the sampling event. Trip blanks will only be analyzed for volatile organic constituents. Table 1 provides an estimated number of trip blanks to be collected for each matrix and parameter during the RI.

9.1.6 Background Samples

Background samples are used to identify constituents which are non-site-related. Background samples will be obtained as described in the RI/FS Work Plan for the sampling media listed below:

Media	Background Samples
Ground Water	One upgradient ground-water monitoring well.
Soil	One surface soil sample and one subsurface soil sample from the installation of ground water monitoring well MW-7 north of the site.

9.1.7 Other Field Quality Control Checks

One sample of the potable water to be used during the test pit/drilling activities will be collected and analyzed for CLP TCL/TAL chemical constituents (except pesticides/PCBs) and for PCBs using Method 8080 to ensure contaminants are not present in the water supply. In addition, a sample of distilled water used for equipment cleaning will be collected and analyzed for CLP/TCL/TAL constituents and for PCBs.

9.2 ANALYTICAL LABORATORY QUALITY CONTROL CHECKS

9.2.1 Aquatec Procedures

Internal laboratory quality control checks will be used to monitor data integrity. These checks will include method blanks, matrix spikes (and matrix spike duplicates), spike blanks, internal standards, surrogate samples, calibration standards, and reference standards. Project QC limits for duplicates and matrix spikes are identified in Tables 3 and 4. Laboratory control charts will be used to determine long-term instrument trends.

9.2.1.1 Method Blanks

Sources of contamination in the analytical process, whether specific analytes or interferences, need to be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank is initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources which could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples (Aquatec, 1992). CLP guidelines for acceptance will be used. Guidelines for non-standard methods are provided in the appropriate protocols.

9.2.1.2 Matrix Spikes/Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates will be used to measure the accuracy of organic analyte recovery from the sample matrices. All matrix spikes and matrix spike duplicates will be site-specific. For organic constituents, matrix spike/matrix spike duplicate pairs will be analyzed at a 5 percent frequency (every 20 samples). For inorganics, a matrix spike will be analyzed at a 5% frequency.

For water, soil, and sediment organic matrix spike data, results will be examined in conjunction with spike blanks (Section 9.2.1.3 of this QAPP) data and surrogate spike (Section 9.2.1.5) data to assess the accuracy of the analytical method. When matrix spike recoveries are outside QC

limits, associated spike blank and surrogate recoveries will be evaluated to attempt to verify the reason for the variance(s), and determine the effect on the reported sample results. Table 1 presents an estimated number of matrix spike and matrix spike duplicate analyses for each applicable matrix and parameter.

9.2.1.3 Spike Blanks

For water, soil, and sediment organic analyses, spike blanks will be included to provide an additional assessment of data accuracy. The spike blanks provide an assessment of method performance without interferences which may be present in environmental samples. Spike blanks will be analyzed at a frequency of one blank associated with no more than 20 samples. For spike blank analyses, clean matrix is spiked and recoveries are calculated similar to matrix spike recoveries. The clean matrix will consist of laboratory reagent water and clean, dried sand for water and soil/sediment analyses, respectively. Matrix spike blank data will be assessed in conjunction with matrix spike data, as discussed in Section 9.2.1.2 of this QAPP. Table 1 presents an estimated number of matrix spike blanks for each matrix and parameter.

9.2.1.4 Surrogate Spikes

Surrogates are compounds unlikely to be found in nature that have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by GC/MS and GC methods and is added to the samples prior to purging or extraction. The surrogate spike is utilized to provide broader insight into the proficiency and efficiency of an analytical method on a sample specific basis. This control reflects analytical conditions which may not be attributable to sample matrix (Aquatec 1992).

If surrogate spike recoveries exceed specified QC limits, then the analytical results need to be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures (i.e., internal standard and matrix spikes), the integrity of the data may be verifiable and reanalysis of the sample with additional controls would be necessary (Aquatec 1992).

Surrogate spike compounds will be selected utilizing the guidance provided in the analytical methods summarized in Table 2.

9.2.1.5 Laboratory Duplicates

For inorganics and other supplemental parameters, laboratory duplicates will be analyzed to assess laboratory precision. Laboratory duplicates are defined as a second aliquot of an individual sample which is analyzed as a separate sample. Table 1 provides an estimated number of laboratory duplicates for each applicable matrix and parameter.

9.2.1.6 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding the instruments' stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 10 samples, or more frequently as specified in the applicable analytical method. In analyses where internal standards are used, a calibration check standard will only be analyzed in the beginning of an analytical series. If results of the calibration check standard exceed specified tolerances, then all samples analyzed since the last acceptable calibration check standard will be reanalyzed (Aquatec 1992).

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods summarized in Table 2.

9.2.1.7 Internal Standards

Internal standard areas and retention times are monitored for organic analyses performed by GC/MS methods. Method-specified internal standard compounds are spiked into all field samples, calibration standards and QC samples after preparation and prior to analysis. The response of each internal standard is plotted on a control chart. In general, Aquatec applies the following criteria for internal standards; the area of any compound cannot fall below 50 percent of its value in the preceding check standard nor can it rise above 100 percent of its value. If internal standard areas in one or more samples exceeds the specified tolerances, then the instrument will be recalibrated and all affected samples reanalyzed (Aquatec, Inc., 1992).

The use and frequency of internal standard analyses will be determined using the guidance provided within the analytical methods summarized in Table 2.

9.2.1.8 Reference Standards

Reference standards are standards of known concentration, and independent in origin from the calibration standards. Reference standards, are generally available through the EPA, the National Bureau of Standards, or are specified in analytical methods. Reference standards are included in the analytical process, although in some aspects of sample handling and preparation, these standards may not reflect the analytical process. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes the preparation of calibration standards, the validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards are utilized in every analytical series with the exception of GC/MS and certain GC methods for which reference standards do not exist. Reference standards will be analyzed at the frequencies specified within the analytical methods summary in Table 2.

9.3 SEDIMENT CHARACTERIZATION QUALITY CONTROL CHECKS

Analyses of sediment particle size and TOC will be performed in duplicate for 5 percent (every 20 samples) of the total samples in each matrix.



10.0 - PERFORMANCE AND SYSTEMS AUDITS

Performance and systems audits will be completed in the field and the laboratory during the RI as described below.

10.1 FIELD AUDITS

The following field performance and systems audits will be completed during this project.

10.1.1 Performance Audits

The appropriate Task Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field measurements and field meter calibrations to verify that measurements are taken according to established protocols. The Blasland & Bouck QAO will review all field reports and communicate concerns to the Blasland & Bouck Project Manager and/or Task Managers, as appropriate. In addition, the Blasland & Bouck QAO will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures.

10.1.2 Internal Systems Audits

A field internal systems audit is a qualitative evaluation of all components of field QA/QC. The systems audit compares scheduled QA/QC activities from this document with actual QA/QC activities completed. The appropriate Task Manager and QAO will periodically confirm that work is being performed consistent with this QAPP, the RI Work Plan, FSP and HASP.

10.1.3 External Audits

New York State representatives may conduct audits of field operations, if determined necessary.

10.2 LABORATORY AUDITS

The following laboratory performance and systems audits will be completed during this project.

10.2.1 Aquatec Procedures

10.2.1.1 Internal Systems Audits

The internal quality control program will consist of two key segments:

- 1. Documented procedures for daily operation of the laboratory; and
- 2. Inspection and review of laboratory procedures by the Aquatec QAO.

Examples of laboratory procedures that are required for daily operation include:

1. <u>Instruments and Equipment</u>: All instruments and equipment are operated according to laboratory SOPs (on record at the laboratory) which include details of calibration, operation, and maintenance of these devices. The Aquatec QAO observes the use of instruments and the adherence to the SOPs as part of regular inspection activities (Aquatec, Inc., 1992).

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BLASLAND & BOUCK ENGINEERS, P.C. Engineers & Geoscientists 2. <u>Reagents</u>: All reagents are labeled according to laboratory SOPs. This procedure requires labeling of name, concentration, expiration data, storage condition, date of preparation, and name of person who prepared the reagent. The Aquatec QAO also includes reagents in the regular inspection program (Aquatec, Inc., 1992).

The assessment of laboratory analytical data is initiated at the bench level. The analyst directly responsible for the test understands the current operating acceptance limits. The analyst can directly accept or reject the data generated and consult the section leader for any corrective action. Data reported by the analyst is entered into a central data retrieval system. All data is subject to review by the Aquatec Project Manager, who is also responsible for monitoring quality control and analytical procedures (Aquatec, Inc., 1992).

A comprehensive QA/QC program is coordinated by the Aquatec QAO, who is independent of all operating departments and reports directly to management. The Aquatec QAO reviews, approves, and distributes all technical and administrative methods and procedures used in project work. These written methods and SOPs, including an updated file, are part of the official records (Aquatec, Inc., 1992).

The Aquatec QAO conducts semi-annual inspections. The following items are typically inspected:

- 1. Sample handling;
- 2. Chemical assay procedures and validation;
- 3. Reagent preparation and labeling;
- 4. Analytical controls and standards;
- 5. Instrument calibration and maintenance;
- 6. Results of analyses;
- 7. Data recording and analysis;
- 8. Data archiving procedures;
- 9. Preventative maintenance procedures for laboratory instruments; and
- 10. Training, documentation, and personnel qualifications.

Inspection reports are issued to management for all inspections and kept on file by the Aquatec QAO. Adverse findings must be addressed to the Aquatec Project Manager as well as the Laboratory Director. Adverse findings and steps taken to correct deficiencies will be documented in the Corrective Action Form (Appendix B). Once final, the Aquatec QAO inspection records will be made available to Blasland & Bouck. Data units and final report reviews are also part of the Aquatec QAO inspection program.

10.2.1.2 External Audits

There are three mechanisms by which external laboratory audits may be conducted.

- 1. The independent data validator (OBG Laboratories, Inc.) will provide an evaluation of laboratory performance for all data packages submitted for review.
- 2. The State may conduct audits of laboratory operations, if deemed necessary.

3. As a participant in State and federal certification programs, the laboratory sections at Aquatec are audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary (Aquatec, Inc., 1992).

11.0 - PREVENTIVE MAINTENANCE

Preventive maintenance schedules have been developed for both field and laboratory instruments. A summary of the maintenance activities to be performed is presented below.

11.1 FIELD INSTRUMENTS AND EQUIPMENT

Prior to any field sampling, each piece of field equipment will be inspected to assure it is operational. If the equipment is not operational, it must be serviced prior to use. All meters which require charging or batteries will be fully charged or have fresh batteries. If instrument servicing is required, it is the responsibility of the appropriate Task Manager or designer to follow the maintenance schedule and arrange for prompt service.

Field instrumentation to be used in this study includes meters to measure water velocity, conductivity, temperature, pH, dissolved oxygen, water level, organic vapors, and water flow. Field equipment also includes sediment samplers, sediment traps, vacuum pumps, and sampling devices for ground water. A logbook will be kept for each field instrument. Each logbook contains records of operation, maintenance, calibration, and any problems and repairs. The Blasland & Bouck Task Managers will review calibration and maintenance logs.

Field equipment returned from a site will be inspected to confirm it is in working order. This inspection will be recorded in the logbook or field notebooks as appropriate. It will also be the obligation of the last user to record any equipment problems in the logbook.

Non-operational field equipment will be either repaired or replaced. Appropriate spare parts will be made available for field meters. A summary of preventive maintenance requirements for field instruments is provided in Table 9. Details regarding field equipment maintenance, operation, and calibration, are provided in the FSP.

11.2 LABORATORY INSTRUMENTS AND EQUIPMENT

11.2.1 General

Laboratory instrument and equipment documentation procedures are provided in SOPs. Documentation includes details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (which will include information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer. Specific procedures used by Aquatec are discussed below.

11.2.2 Aquatec Procedures

11.2.2.1 Instrument Maintenance

Analytical instrumentation are maintained and serviced according to the manufacturer specifications. Each analytical instrument has a specific maintenance logbook. All routine maintenance and repair work is recorded with the date and the initials of the individual performing the maintenance task. Reports from outside service work are incorporated into the

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instrument logbooks. For GC/MS instrumentation, all performance checks (decafluorotriphenylphosphine and p-bromofluorobenzene) associated with instrument tune for a particular instrument are to be maintained in a separate loose-leaf notebook for that instrument (Aquatec, Inc., 1992).

11.2.2.2 Equipment Monitoring

On a daily basis, the operation of balances, incubators, refrigerators, the high purity water system, furnaces, ovens, and air conditioning, are documented on Aquatec Monitoring Worksheets. Any discrepancies are immediately reported to the appropriate laboratory or technical services personnel for resolution. All analytical balances are checked with Class "S" weights and a thermometer is present in each refrigerator/freezer (Aquatec, Inc., 1992).

The temperatures inside the refrigerator/freezer units are manually recorded on a daily basis through thermometer readings. A computer based system is also connected to the refrigerator/freezer units, which monitors temperature on a continual basis. Acceptable temperature limits have been established and set within computerized program. Each temperature reading is immediately compared to the limits, and for values falling outside of the established limits, a buzzer will sound and corrective action will be initiated immediately. Provisions have been made to contact technical services personnel during off hours to ensure that the refrigeration systems are not out of control for more than 20 minutes (Aquatec, Inc., 1992).

11.2.2.3 Maintenance Control Charts

In addition to routine and preventative maintenance, control charts are maintained for several instruments as an indicator of when maintenance may be necessary. In the GC/MS laboratory, instrument sensitivity is monitored using internal standards. The internal standard solution is injected into every standard, blank, and sample analyzed on the GC/MS. The area of the internal standard compounds are plotted on control charts that can serve as an indicator of the overall condition of the instrument. Instrumentation problems may be diagnosed and remedied by tracking the response patterns on the control charts. The control charts are updated following each analysis (Aquatec, Inc., 1992).

12.0 - DATA ASSESSMENT PROCEDURES

The analytical data generated during the M. Wallace and Son Scrapyard RI will be evaluated with respect to precision, accuracy, and completeness and compared to the data quality objectives set forth in Sections 1.0 and 3.0 of this QAPP. The following tables summarize QC limits required to evaluate analytical performance:

Table	Title
3	Soil/Sediment Analyses Quality Control Limits
4	Water Analyses Quality Control Limits

The procedures utilized when assessing data precision, accuracy, and completeness are presented below.

12.1 DATA PRECISION ASSESSMENT PROCEDURES

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters, and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision for organic analyses will be monitored through the use of matrix spike/matrix spike duplicate sample analyses. For other parameters, laboratory data precision will be monitored through the use of field duplicates and/or laboratory duplicates as identified in Table 1.

The precision of data will be measured by calculation of the relative percent difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements B = Analytical result from the second measurement.

Precision objectives for duplicate analyses are identified in Tables 3 and 4.

12.2 DATA ACCURACY ASSESSMENT PROCEDURES

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of matrix spikes, surrogate spikes and reference standards. Where available and appropriate, QA performance standards will be analyzed periodically to assess laboratory accuracy. Accuracy will be calculated in terms of percent recovery as follows:

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$$\%$$
 Recovery = $\frac{A-X}{B} \times 100$

Where:

- A = Value measured in spiked sample or standard
- X = Value measured in original sample
- B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy between the original and spiked measurements. Accuracy objectives for matrix spike recoveries are identified in Tables 3 and 4.

12.3 DATA COMPLETENESS ASSESSMENT PROCEDURES

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

Completeness = <u>No Valid Results</u> Total number of results generated x 100

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgement to determine data useability for intended purposes.

13.0 - CORRECTIVE ACTION

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP, the Work Plan, or the FSP. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the M. Wallace and Son, Inc. Scrapyard Site RI are described below.

13.1 FIELD PROCEDURES

When conducting the RI field work, if a condition is noted that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented will be documented on a Corrective Action Form (Appendix B) and reported to the appropriate Blasland & Bouck Task Manager, QAO, and Project Manager.

Examples of situations which would require corrective actions are provided below:

- 1. Protocols as defined by the QAPP and FSP have not been followed;
- 2. Equipment is not in proper working order or properly calibrated;
- 3. QC requirements have not been met; and
- 4. Issues resulting from performance or systems audits.

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

13.2 LABORATORY PROCEDURES

13.2.1 General

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action to be taken will be documented, and reported to the appropriate project manager and QAO.

Corrective action may be initiated, at a minimum, under the following conditions:

- 1. Protocols as defined by this QAPP have not been followed;
- 2. Predetermined data acceptance standards are not obtained;
- 3. Equipment is not in proper working order or calibrated;
- 4. Sample and test results are not completely traceable;
- 5. QC requirements have not been met; and
- 6. Issues resulting from performance or systems audits.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Additional details of corrective action procedures used by Aquatec are provided below.

13.2.2 Aquatec Procedures

When deficiencies or out-of-control situations exist, the samples analyzed during out-of-control situations will be reanalyzed prior to reporting of results. There are several levels of out-of-control situations that may occur in the laboratory during analysis. (Aquatec, Inc., 1992).

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13.2.2.1 Bench Level

Corrective action procedures are often handled at the bench level. If an analyst finds a nonlinear response during calibration of an instrument, then the problem is often corrected by a careful examination of the preparation or extraction procedure, spike and calibration mixes, or instrument sensitivity. If the problem persists, it is brought to the attention of the management level. (Aquatec, Inc., 1992).

13.2.2.2 Laboratory Management Level

If resolution at the bench level was not achieved or a deficiency is detected after the data has left the bench level, then corrective action becomes the responsibility of the Aquatec section leader. Unacceptable matrix or surrogate spike recoveries detected by data review are reported to the Aquatec section leader. A decision to reanalyze the sample or report the results is made depending on the circumstances. Documentation procedures for sample reanalysis are initiated at this point if necessary. (Aquatec, Inc., 1992).

13.2.2.3 Receiving Level

If discrepancies exist in either the documentation of a sample or its container, a corrective action decision must be made after consulting with the appropriate management personnel. All decisions will be fully documented. Some examples of container discrepancies are broken samples, inappropriate containers, or improper preservation. In these cases, corrective action will involve the Aquatec Project Manager contacting the Blasland & Bouck Project Manager or QAO to resolve the problems. (Aquatec, Inc., 1992).

13.2.2.4 Statistical Events

An out-of-control situation is defined as data exceeding control limits, unacceptable trends detected in charts, or unusual changes in the instrument detection limits. When these situations arise, it is brought to the attention of the Aquatec Project Manager and the Laboratory Director who will initiate corrective action. (Aquatec, Inc., 1992).



14.0 - QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 **INTERNAL REPORTING**

The independent data validator (OBG Laboratories, Inc.) will submit validation report(s) to the Blasland & Bouck QAO, consistent with the requirements presented in Section 8.3. The Blasland & Bouck QAO will review analytical concerns identified by the independent data validator with the laboratory. For data qualified by the data validator, data useability will be assessed by data users relative to project decisionmaking requirements. Supporting data (i.e., historic data, related field or laboratory data) will be reviewed to assist determining data quality, as appropriate. The Blasland & Bouck QAO will incorporate results of data validation reports and assessments of data useability into a summary report that will be submitted to the Blasland & Bouck Project Manager and appropriate Task Managers. This report will be filed in the project file at Blasland & Bouck's office and will include the following:

- Assessment of data accuracy, precision, and completeness for both field and laboratory data; 1.
- 2. Results of the performance and systems audits;
- Significant QA/QC problems, solutions, corrections, and potential consequences; and Analytical data validation report. 3.
- 4.

14.2 **RI REPORTING**

The RI Report prepared by Blasland & Bouck will contain a separate QA/QC section(s) summarizing the quality of data collected and/or used as appropriate to the project data quality objectives which are discussed in Section 1.3 of this QAPP. Additional details of data quality objectives are provided in the Work Plan and FSP. The Blasland & Bouck QAO will prepare the QA/QC summaries using reports and memoranda documenting the data assessment and validation.

In addition, records will be maintained to provide evidence of the QA activities. A QA records index will be initiated at the beginning of the project, and all information received from outside sources or developed during the project will be retained by Blasland & Bouck. Upon termination of an individual task or work assignment, working files will be forwarded to the project files.

	DEFEDENCES
	REFERENCES
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ENVIRONMENTAL AND QUALITY CONTROL ANALYSES

	Est.			Fleid	QC Ani	alyses	_				Lab	oralory	QC Analy	'ses			
Atrix/ Laboratory Parameters	Environmental Sample Quantity	Trip Bl	ank	Fiel Duplik	d xate	Rinse	Blank	Est.	M	3	MS	D	S	B	Lab Du	plicate	Est. Overall Total
		Freq	No.	Freq	No.	Freq.	No.	Matrix Total	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Soll Samples																	
Total PCBs TCL Volatile Organics TCL Serni-Volatile Organics TAL Inorganics	42 10 10 12 ¹	 		1/20 1/20 1/20 1/20	3 1 1 2	1/20 1/20 1/20 1/20	3 1 1 2	48 12 12 16	1/20 1/20 1/20 1/20	3 1 1	1/20 1/20 1/20 	3 1 1 	1/20 1/20 1/20 1/20	3 1 1 1	 1/20	 1	57 15 15 19
Sediment Samples																	
Total PCBs TCL Volatile Organics TCL Semi-Volatile Organics TAL Inorganics Total Organic Carbon Particle Size Distribution	31 ² 12 ² 12 ² 31 ² 31 ²			1/20 1/20 1/20 1/20 1/20 1/20	2 1 1 2 2	1/20 1/20 1/20 1/20 	2 1 1 	35 16 16 33 33	1/20 1/20 1/20 1/20 	2 1 1 	1/20 1/20 1/20 	2 1 1 	1/20 1/20 1/20 1/20 	2 1 1 	 1/20 	 1 	41 19 19 33 33
Surface Water Samples																	
Total PCBs - Filtered Total PCBs - Unfiltered TCL Volatile Organics TCL Semi-Volatile Organics TAL Inorganics - Filtered TAL Inorganics - Unfiltered Total Suspended Solids	9 9 9 9 9 9 9	 1/day ³ 	 3 	1/20 1/20 1/20 1/20 1/20 1/20 1/20	1 1 1 1 1	1/20 1/20 1/20 1/20 1/20 1/20 	1 1 1 1 1 1 	11 11 14 11 11 11 10	1/20 1/20 1/20 1/20 1/20 1/20	1 1 1 1 1 	1/20 1/20 1/20 1/20 	1 1 1 	1/20 1/20 1/20 1/20 1/20 1/20 	1 1 1 1 1 	 1/20 1/20 	 1 1	14 14 17 14 14 14 14 10
Ground-Water Samples																	
Total PCBs - Filtered Total PCBs - Unfiltered TCL Volatile Organics TCL Semi-Volatile Organics TAL Inorganics - Filtered TAL Inorganics - Unfiltered	7 7 7 7 7 7	 1/day³ 	 2 	1/20 1/20 1/20 1/20 1/20 1/20	1 1 1 1	 	 	8 8 10 8 8 8	1/20 1/20 1/20 1/20 1/20 1/20	1 1 1 1 1	1/20 1/20 1/20 1/20 	1 1 1 	1/20 1/20 1/20 1/20 1/20 1/20	1 1 1 1 1	 1/20 1/20	 1	11 11 13 11 11 11

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ENVIRONMENTAL AND QUALITY CONTROL ANALYSES

Notes:

- 1. Quantity includes two background soil samples from MW-7 installation for TAL inorganic analysis. Table 1 does not include additional subsurface samples that may be collected from test pits. Additional samples may require additional QC analyses based on additional sample quantity compared to QC sample frequencies shown on table.
- 2. Quantity assumes that two samples for analysis for PCBs, TCL volatiles/semi-volatiles, and TAL inorganics will be collected from each of the six sediment core samples (12 total sample).
- 3. 1/day = One trip blank per day of volatile organic sampling of aqueous media. One rinse blank per day of sampling with sampling device which requires field cleaning.
- 4. MS = Matrix spike
- 5. MSD = Matrix spike duplicate
- 6. SB = spike blank
- 7. PCBs = Polychlorinated biphenyls
- 8. Field Dup = field duplicate
- 9. Lab Dup = laboratory duplicate
- 10. TCL = Target Compound List per USEPA Contract Laboratory Program (CLP) Statement of Work (SOW).
- 11. TAL = Target Analyte List per USEPA CLP-SOW
- 12. One sample of tap water and one sample of distilled water used to clean equipment in the field will be collected for analysis for total PCBs, TCL volatile organics, TCL semi-volatile organics, and TAL inorganics.
- 13. Table assumes that samples will be processed in groups of 20 samples for QC analyses. If smaller sample groups are processed, then one MS/MSD (or MS/lab dup) per sample delivery group (up to 20 samples) will be prepared for each sample delivery group.

Page 2 of 2

PARAMETERS, METHODS, AND REPORTING LIMITS

Water, Soil, and Sediment Samples Chemical Constituents Target Compound List (TCL) CLP-SOW Analytical Procedures					
Constituent	Rep	orting Limits'	(ppb)		
	Water	Low Soil	Medium Soil		
Volatile Organics					
Chloromethane	10	10	1,200		
Bromomethane	10	10	1,200		
Vinyl Chloride	10	10	1,200		
Chloroethane	10	10	1,200		
Methylene Chloride	10	10	1,200		
Acetone	10	10	1,200		
Carbon Disulfide	10	10	1,200		
1,1-Dichloroethene	10	10	1,200		
1,1-Dichloroethane	10	10	1,200		
1,2-Dichloroethene (total)	10	10	1,200		
Chloroform	10	10	1,200		
1,2-Dichloroethane	10	10	1,200		
2-Butanone	10	10	1,200		
1,1,1-Trichloroethane	10	10	1,200		
Carbon Tetrachloride	10	10	1,200		
Bromodichloromethane	10	10	1,200		
1,2-Dichloropropane	10	10	1,200		
cis-1,3-Dichloropropene	10	10	1,200		
Trichloroethene	10	10	1,200		
Dibromochloromethane	10	10	1,200		
1,1,2-Trichloroethane	10	10	1,200		
Benzene	10	10	1,200		
trans-1,3-Dichloropropene	10	10	1,200		
Bromoform	10	10	1,200		
4-Methyl-2-pentanone	10	10	1,200		
2-Hexanone	10	10	1,200		
Tetrachioroethene	10	10	1,200		
Toluene	10	10	1,200		
1,1,2,2-Tetrachloroethane	10	10	1,200		

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PARAMETERS, METHODS, AND REPORTING LIMITS

Target Compound List (TCL) CLP-SOW A	nalytical Proce	dures
Constituent	Hep		(ppo)
	vvater	Low Soll	Medidin Son
Chlorobenzene	10	10	1,200
Ethylbenzene		10	1,200
Styrene		10	1,200
Total Xylenes	10	10	1,200
Semi-Volatile Organics			· · · · · · · · · · · · · · · · · · ·
Phenol	10	330	10,000
bis(2-chloroethyl) ether	10	330	10,000
2-Chlorophenol	10	330	10,000
1,3-Dichlorobenzene	10	330	10,000
1,4-Dichlorobenzene	10	330	10,000
1,2-Dichlorobenzene	10	330	10,000
2-Methylphenol	10	330	10,000
2,2'-oxybis (1-chloropropane)	10	330	10,000
4-Methylphenol	10	330	10,000
N-Nitroso-di-n-propylamine	10	330	10,000
Hexachloroethane	10	330	10,000
Nitrobenzene	10	330	10,000
Isophorone	10	330	10,000
2-Nitrophenol	10	330	10,000
2,4-Dimethylphenol	10	330	10,000
bis(2-chloroethoxy)methane	10	330	10,000
2,4-Dichlorophenol	10	330	10,000
1,2,4-Trichlorobenzene	10	330	10,000
Naphthalene	10	330	10,000
4-Chloroaniline	10	330	10,000
Hexachlorobutadiene	10	330	10,000
4-Chloro-3-methylphenol	10	330	10,000
2-Methylnapthalene	10	330	10,000
Hexachlorocyclopentadiene	10	330	10,000
2,4,6-Trichlorophenol	10	330	10,000

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PARAMETERS, METHODS, AND REPORTING LIMITS

Water, Soil, and Sediment Samples Chemical Constituents Target Compound List (TCL) CLP-SOW Analytical Procedures						
Constituent	Reporting Limits' (ppb)					
	Water	Low Soil	Medium Soil			
2,4,5-Trichlorophenol	25	800	25,000			
2-Chloronaphthalene	10	330	10,000			
2-Nitroaniline	25	800	25,000			
Dimethylphthalate	10	330	10,000			
Acenaphthylene	10	330	10,000			
2,6-Dinitrotoluene	10	330	10,000			
3-Nitroaniline	25	800	25,000			
Acenaphthene	10	330	10,000			
2,4-Dinitrophenol	25	800	25,000			
4-Nitrophenol	25	800	25,000			
Dibenzofuran	10	330	10,000			
2,4-Dinitrotoluene	10	330	10,000			
Diethylphthalate	10	330	10,000			
4-Chlorophenyl phenyl ether	10	330	10,000			
Fluorene	10	330	10,000			
4-Nitroaniline	25	800	25,000			
4,6-Dinitro-2-methylphenol	25	800	25,000			
N-nitrosodiphenylamine	10	330	10,000			
4-Bromophenyl phenyl ether	10	330	10,000			
Hexachlorobenzene	10	330	10,000			
Pentachlorophenol	25	800	25,000			
Phenanthrene	10	330	10,000			
Anthracene	10	330	10,000			
Carbazole	10	330	10,000			
Di-n-butyl phthalate	10	330	10,000			
Fluoranthene	10	330	10,000			
Pyrene	10	330	10,000			
Butyl benzyl phthaiate	10	330	10,000			
3,3'-Dichlorobenzidine	10	330	10,000			
Benz(a)anthracene	10	330	10,000			

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PARAMETERS, METHODS, AND REPORTING LIMITS

Water, Soll, and Sediment Samples Chemical Constituents Target Compound List (TCL) CLP-SOW Analytical Procedures							
Constituent	Reporting Limits' (ppb)						
	Water	Low Soil	Medium Soil				
Chrysene	10	330	10,000				
bis(2-Ethylhexyl)phthalate	10	330	10,000				
Di-n-octyl phthalate	10	330	10,000				
Benzo(b)fluoranthene	10	330	10,000				
Benzo(k)fluoranthene	10	330	10,000				
Benzo(a)pyrene	10	330	10,000				
Indeno(1,2,3-cd)pyrene	10	330	10,000				
Dibenzo(a,h)anthracene	10	330	10,000				
<u>Benzo(g,h,i)perylene</u>	10	330	10,000				

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PARAMETERS, METHODS, AND REPORTING LIMITS

Water, Soil, and Sediment Samples Chemical Constituents Target Analyte List (TAL) CLP-SOW Analytical Procedures						
Analyte	Reportin	Reporting Limit ²				
	Water (ppb)	Soil (ppb)				
Aluminum	200	200				
Antimony	60	60				
Arsenic	10	10				
Barium	200	200				
Beryllium	5	5				
Cadmium	5	5				
Calcium	5,000	5,000				
Chromium	10	10				
Cobalt	50	50				
Copper	25	25				
Iron	100	100				
Lead	5	5				
Magnesium	5,000	5,000				
Manganese	15	15				
Mercury	0.2	0.2				
Nickel	40	40				
Potassium	5,000	5,000				
Selenium	5	5				
Silver	10	10				
Sodium	5,000	5,000				
Thallium	10	10				
Vanadium	50	50				
Zinc	20	20				
Cyanide	10	10				

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PARAMETERS, METHODS, AND REPORTING LIMITS

Water, Soil, and Sediment Samples Chemical Constituents PCB Analysis Using SW-846 Method 8080						
	Reporting	Limit ^a (ppb)				
Constituent	Water (ppb)	Soil (ppb)				
Aroclor 1016	0.05	0.050				
Arocior 1221	0.05	0.050				
Aroclor 1232	0.05	0.050				
Aroclor 1242	0.05	0.050				
Aroclor 1248	0.05	0.050				
Aroclor 1254	0.05	0.050				
Aroclor 1260	0.05	0.050				
Total PCBs ⁴	0.35	0.350				

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PARAMETERS, METHODS, AND REPORTING LIMITS

Analyses	Method	Reporting Limit
Sediment Supplemental Parame	iters	
Total Organic Carbon Particle Size Distribution	Lloyd Kahn Method ASTM-D-422	100 ppm
Surface Water Supplemental P.	arameters	
Total Suspended Solids	USEPA Method 160.2	4 ppm⁵

<u>Notes</u>:

- ¹ Reporting limits presented are CLP-SOW (3/90 rev. through 8/91) contract required quantitation limits (CQRLs). Quantitation limits for soil and sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil and sediment calculated on dry weight basis will be higher. Specific quantitation limits are highly matrix dependent. The quantitation limits shown are provided for guidance and may not always be achievable.
- ² Reporting limits presented are based on CLP-SOW (3/90) CRQLs for inorganics and are subject to restrictions specified in CLP-SOW for Inorganic Analysis Part G Section IV of Exhibit D. Higher detection limits may be used if conditions warrant in accordance with CLP-SOWs. CRQLs shown for inorganics are provided for guidance and may not always be achievable.
- ³ Reporting limits shown are based on CLP-SOW (2/88) CRQLs and are for guidance purposes. The quantitation limits calculated by the laboratory for soil and sediments, calculated on dry weight basis, will be higher.
- ⁴ Reporting limits shown for total PCBs are the summation of the reporting limits for each aroclor listed.
- ⁵ Reporting limits are method detection limits based on USEPA for supplemental parameters. Methods contained in Methods for Chemical Analysis of Water and Wastes, USEPA-6001/4-79-020, except as noted.
- ⁶ PCBs = polychlorinated biphenyls.
- ⁷ ppb = parts per billion.
- ppm = parts per million.
 - -- = not applicable.

SOIL/SEDIMENT ANALYSES QUALITY CONTROL LIMITS

Constituent	Method	Accuracy, % Recovery	Precision, RPD
Volatile Organics ^{1,2}			
1,1-Dichloroethane	CLP/TCL	59-172	22
Trichloroethane	CLP/TCL	62-137	24
Benzene	CLP/TCL	66-142	21
Toluene	CLP/TCL	59-139	21
Chlorobenzene	CLP/TCL	60-133	21
Semi-Volatile Organics ^{1,2}			
2-Chlorophenol	CLP/TCL	25-102	50
1,4-Dichlorobenzene	CLP/TCL	28-104	27
N-nitroso-di-n-propylamine	CLP/TCL	41-126	38
1,2,4-Trichlorobenzene	CLP/TCL	38-107	23
4-chloro-3-methylphenol	CLP/TCL	26-103	33
Acenapthene	CLP/TCL	31-137	19
4-nitrophenol	CLP/TCL	11-114	50
2,4-dinitrotoluene	CLP/TCL	28-89	47
Pentachlorophenol	CLP/TCL	17-109	47
Pyrene	CLP/TCL	35-142	36
PCBs ^{1,2,3}			
Aroclor 1242	Method 8080	39-159	
Arclor 1254	Method 8080	29-131	
Inorganics ^{3,4}			
All TAL Inorganics	CLP/TAL	75-120	30

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1 Available QC limits are presented from CLP-SOW (3/90 rev. 8/91) for organic analyses. These limits may be used for guidance on QC limits for other CLP/TCL volatiles or semi-volatiles.

2 QC limits shown on table are only advisory, however, frequent failures to meet the QC limits warrant investigation of the laboratory. з

QC limits are presented for aroclors 1242 and 1254 from CLP-SOW dated 2/88 for organic analysis. QC limits obtained from CLP-SOW for Inorganics Analyses 3/90. 4

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QC limits for supplemental soil/sediment parameters consist of 25% relative percent difference in duplicate samples. 6

RPD = relative percent difference.

7 MS = matrix spike 8

MSD = matrix spike duplicate ۵

SB = spike blank 10

Lab dup = laboratory duplicate

WATER ANALYSES QUALITY CONTROL LIMITS

Constituent	Method	Accuracy, % Recovery	Precision, RPD
Volatile Organics			
1,1-Dichloroethane	CLP/TCL ^{1,2}	61-145	14
Trichloroethane	CLP/TCL ^{1.2}	71-120	14
Benzene	CLP/TCL ^{1,2}	76-127	11
Toluene	CLP/TCL ^{1,2}	76-125	13
Chlorobenzene	CLP/TCL ^{1,2}	75-130	13
Semi-Volatile Organics ²			
2-Chlorophenol	CLP/TCL	12-110	40
1,4-Dichlorobenzene	CLP/TCL	27-123	28
N-nitroso-di-n-propylamine	CLP/TCL	41-116	38
1,2,4-Trichlorobenzene	CLP/TCL	39-98	28
4-chloro-3-methylphenol	CLP/TCL	23-97	42
Acenapthene	CLP/TCL	46-118	31
4-nitrophenol	CLP/TCL	10-80	50
2,4-dinitrotoluene	CLP/TCL	24-96	38
Pentachlorophenol	CLP/TCL	9-103	50
Pyrene	CLP/TCL	26-127	31
PCBs ²			
Aroclor 1242	Method 8080	39-150	
Aroclor 1254	Method 8080	39-150	
Inorganics ³			
All TAL Inorganics	CLP/TAL	75-120	20

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Available QC limits are presented from CLP-SOW (3/90 rev. 8/91) for organic analysis. These limits may be used for guidance on QC limits for other CLP/TCL volatiles or semi-volatiles.

² QC limits presented for aroclors 1242 and 1254 are from CLP-SOW dated 2/88 for organic analysis.

³ QC limits obtained from CLP-SOW for inorganics analysis 3/90.

⁴ QC limits shown on table are only advisory, however, frequent failures to meet the QC limits warrant investigation of the laboratory.

⁵ QC limits for supplemental surface water parameters (i.e., TSS) consist of 25% relative percent difference in duplicate samples.

⁶ RPD = relative percent difference.

MS = matrix spike

⁸ MSD = matrix spike duplicate

SB = spike blank

¹⁰ Lab dup = laboratory duplicate

FIELD CALIBRATION FREQUENCY

Equipment	Calibration Check	Calibration Standard	Calibration Standard Holding Time
pH Meter	Prior to use - daily'	рН 4.0 рН 7.0 рН 10.0	One Month
Conductivity Meter	Prior to use - daily	1,000 mg/l Sodium Chloride	One Month
Flow Meter	Prior to use - daily	N/A	N/A
Water Level Meter Prior to implementing field work		100-foot engineer's tape	N/A
Dissolved Oxygen Meter	Per sampling event	Air	N/A
Turbidity	Prior to use - daily	Formazin 0.5 NTU, 5.0 NTU, 40.0 NTU	N/A

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- The pH meter will also be calibrated at each well prior to ground-water sampling. N/A = not applicable. NTU = nephelometric turbidity units. 1
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FIELD MEASUREMENTS QUALITY CONTROL

Field Parameter	Matrix	Precision ¹	Accuracy
Water Temperature	Ground Water Surface Water	± 1°C	± 1°C instrument capability
рН	Ground Water Surface Water	± 1°C pH S.U.	± 1°C pH S.U. (instrument capability)
Conductivity	Ground Water Surface Water	± 1°C mS/cm	± 5% standard
Dissolved Oxygen	Ground Water Surface Water	± 0.02 mg/l	± 5%
Turbidity	Ground Water	± 1.0 NTU	± 2% standard
Water Velocity	Surface Water	N/A	± 2% standard
Water Level	Ground Water	± 0.1 foot	± 0.01 foot

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Precision units presented in applicable significant figures. N/A = not applicable. 1 2

M. WALLACE AND SON, INC. SCRAPYARD SITE

SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Water Samples*					
Parameter	Reference	Sample Container	Sample Volume	Preservation	Maximum Holding Time from Collection
Volatile Organics	CLP-SOW ¹ , Organics	two 40-ml glass vials with teflon-lined septum cap	80-ml	no head space, 4 drops concentrated HCI, cool 4°C	14 days²
Semi-Volatile Organics	CLP-SOW ¹ , Organics	four liter amber glass with telfon- lined cap	4 liters	cool 4ºC	extract within 7 days, analyze within 40 days following the start of extraction
PCBs	SW-846, Method 8080	One 2 liter amber glass with teflon- lined cap	2 liters	cool, 4ºC	extract within 7 days, analyze within 40 days following the start of extraction
Inorganics*	CLP-SOW ¹ , Inorganics	One 1 liter plastic	1 liter		180 days (28 days for Mercury)
Cyanide	CLP-SOW ¹ inorganics	One 1 liter glass	1 liter	NAOH	14 days
TSS	Method 160.1	polyethylene or glass	500 m!	cool, 4°C	begin analysis as soon as possible
рн	Method 150.1	plastic or glass	25 ml	None required	Analyze immediately

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CLP-SOW = Contract Laboratory Program, Statement of Work.

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7 days if not properly preserved. * = filtered surface water and ground-water samples to be analyzed for inorganics and PCBs will be field filtered prior to the addition of preservatives, all other water samples will not be filtered.

TABLE 7 (Cont'd)

M. WALLACE AND SON, INC. SCRAPYARD SITE

SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

	Soil and Sediment				
Parameter	Reference	Sample Container	Sample Volume	Preservation	Maximum Holdin from Collecti
Volatile Organics	CLP-SOW ¹ , Organics	two 125 ml widemouth glass vial, caps lined with teflon	250 ml	minimize head space, cool 4ºC	14 days
Semi-Volatile Organics	CLP-SOW ¹ , Organics	one 250 ml widemouth glass, caps lined with teflon	250 ml	cool, 4°C	extract within 7 da analyze within 40 following start of extraction
PCBs	SW-846 Method 8080	One 250 ml widemouth amber glass, caps lined with teflon	250 ml	cooi, 4ºC	extract within 7 da analyze within 40 following start of extraction
Inorganics	CLP-SOW ¹ , Inorganics	One 16 ounce widemouth glass	16 oz.	cool, 4ºC	180 days (28 days Mercury)
Cyanide	CLP-SOW ¹ inorganics	One 16 oz. widemouth glass	16 oz.	cool, 4ºC	14 days
Particle Size Distribution	ASTM, D-422	One 8 oz glass or plastic	8 oz.	-	-
Total Organic Carbon	Lloyd Kahn	One 8 oz. glass	8 oz.	Cool, 4°C	14 days

<u>Note</u>:

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CLP-SOW = Contract Laboratory Program, Statement of Work.

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DATA VALIDATION CHECKLIST - LABORATORY ANALYTICAL DATA

REV	IEW FOR COMPLETENESS
1.	Chain-of-custody forms included.
2.	Sample preparation and analysis summary tables included.
з.	QA/QC summaries of analytical data included.
4.	Relevant calibration data included with analytical data.
_5.	Instrument and method performance data included.
6.	Method detection limits documented.
7.	Data report forms of examples for calculations of concentrations.
8.	Raw data used in identification and quantification of the analysis required.
REV	IEW OF COMPLIANCE
1.	Data package completed.
2.	QAPP requirements for data met.
3.	QA/QC criteria met.
4.	Instrument type and calibration procedures met.
5.	initial and continuing calibration met.
6.	Data reporting forms completed.
7.	Problems and corrective actions documented.

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PREVENTIVE MAINTENANCE SUMMARY

Maintenance	Frequency
<u>Turbidity Meter</u> -Store in protective casing -Inspect equipment after use -Clean sample cells -Clean lens -Check and recharge batteries -Keep log book on instrument -Have replacement meter available -Return to manufacturer for service -Calibration	D D M or X D D D X D
Conductivity, pH, Dissolved Oxygen Meters -Store in protective casing -Inspect equipment after use -Clean probe -Keep log book in instrument -Have replacement meter available -Replace probes -Return to manufacturer for service -Calibration	D D D D X X X D
<u>Velocity Meter</u> -Store in protective casing -Inspect equipment after use -Check and recharge batteries -Keep log book on instrument -Have replacement meter available -Return to manufacturer -Calibration	D D D D D X D
<u>Thermometer</u> -Store in protective casing -Inspect equipment after use -Have a replacement thermometer available	D D D
<u>Water Level Meter</u> -Store in protective covering -Inspected equipment after use -Check indicators/batteries -Keep log book on instrument -Have a replacement meter available	D D D D X
Photoionization Detector -Store in protective casing -Inspect equipment after use -Check and recharge batteries -Clean UV lamp and ion chamber -Keep log book on instrument -Have replacement meter available -Return to manufacturer for service -Calibration	D D M or X D D X D

<u>Notes</u>:

D = Daily M = Monthly X = Operator's discretion

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100 **APPENDICES** ---1

APPENDIX A SW-846 METHOD 8080

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METHOD 8080A

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

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1.1 Method 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). The following compounds can be determined by this method:

Compound Name	CAS No.ª	
Aldrin	309-00-2	
α-BHC	319-84-6	
в-BHC	319-85-7	
б-BHC	319-86-8	
γ -BHC (Lindane)	58-89-9	
Chlordane (technical)	12789-03-6	
4.4'-DDD	72-54-8	
4.4'-DDE	72-55-9	
4,4'-DDT	50-29-3	
Dieldrin	60-57-1	
Endosulfan I	959-98-8	
Endosulfan II	33212-65-9	
Endosulfan sulfate	1031-07-8	
Endrin	72-20-8	
Endrin aldehyde	7421-93-4	
Heptachlor	76-44-8	
Heptachlor epoxide	1024-57-3	
4,4'-Methoxychlor	72-43-5	
Toxaphene	8001-35-2	
Aroclor-1016	12674-11-2	
Aroclor-1221	1104-28-2	
Aroclor-1232	11141-16-5	
Aroclor-1242	53469-21-9	
Aroclor-1248	12672-29-6	
Aroclor-1254	11097-69-1	
Aroclor-1260	11096-82-5	

a Chemical Abstract Services Registry Number.

1.1 Table 1 lists the method detection limit for each compound in organicfree reagent water. Table 2 lists the estimated quantitation limit (EQL) for other matrices.

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2.0 SUMMARY OF METHOD

2.1 Method 8080 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides and PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2 to 5 μ L sample is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD) or an electrolytic conductivity detector (HECD).

2.2 The sensitivity of Method 8080 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8080 may also be performed on samples that have undergone cleanup. Method 3620, Florisil Column Cleanup, by itself or followed by Method 3660, Sulfur Cleanup, may be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Refer to Methods 3500, 3600, and 8000.

3.2 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks, especially in the 15% and 50% fractions from the Florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. The contamination from phthalate esters can be completely eliminated with a microcoulometric or electrolytic conductivity detector.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

4.1.2 Columns

4.1.2.1 Column 1: Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m x 4 mm ID glass column or equivalent.

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4.1.2.2 Column 2: Supelcoport (100/120 mesh) coated with 3% OV-1 in a 1.8 m x 4 mm ID glass column or equivalent.

4.1.3 Detectors: Electron capture (ECD) or electrolytic conductivity detector (HECD).

4.2 Kuderna-Danish (K-D) apparatus:

4.2.1 Concentrator tube: 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

4.2.2 Evaporation flask: 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

4.2.3 Snyder column: Three ball macro (Kontes K-503000-0121 or equivalent).

4.2.4 Snyder column: Two ball micro (Kontes K-569001-0219 or equivalent).

4.2.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

4.3 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.4 Water bath: Heated, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}$ C). The bath should be used in a hood.

4.5 Volumetric flasks, Class A: 10, 50, and 100 mL, ground-glass stopper.

4.6 Microsyringe: 10 μ L.

4.7 Syringe: 5 mL.

4.8 Vials: Glass, 2, 10, and 20 mL capacity with Teflon-lined screw caps or crimp tops.

4.9 Balances: Analytical, 0.0001 g and Top loading, 0.01 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

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5.3 Solvents

5.3.1 Hexane, $C_{6}H_{14}$ ~ Pesticide quality or equivalent.

5.3.2 Acetone, CH_3COCH_3 - Pesticide quality or equivalent.

5.3.3 Toluene, $C_{e}H_{s}CH_{3}$ - Pesticide quality or equivalent.

5.3.4 Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$ - Pesticide quality or equivalent.

5.4 Stock standard solutions:

5.4.1 Prepare stock standard solutions at a concentration of 1000 mg/L by dissolving 0.0100 g of assayed reference material in isooctane and diluting to volume in a 10 mL volumetric flask. A small volume of toluene may be necessary to put some pesticides in solution. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.4.2 Transfer the stock standard solutions into vials with Teflonlined screw caps or crimp tops. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.4.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.5 Calibration standards: Calibration standards at a minimum of five concentrations for each parameter of interest are prepared through dilution of the stock standards with isooctane. One of the concentrations should be at a concentration near, but above, the method detection limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

5.6 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.6.1 Prepare calibration standards at a minimum of five concentrations for each analyte of interest as described in Section 5.5.

5.6.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane.

5.6.3 Analyze each calibration standard according to Section 7.0.

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5.7 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and organic-free reagent water blank with pesticide surrogates. Because GC/ECD data are much more subject to interference than GC/MS, a secondary surrogate is to be used when sample interference is apparent. Two surrogate standards (tetrachloro-m-xylene (TCMX) and decachlorobiphenyl) are added to each sample; however, only one need be calculated for recovery. Proceed with corrective action when both surrogates are out of limits for a sample (Section 8.3). Method 3500 indicates the proper procedure for preparing these surrogates.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral, or as is, pH with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using either Method 3540 or 3550.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. The exchange is performed during the K-D procedures listed in all of the extraction methods. The exchange is performed as follows.

7.1.2.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.

7.1.2.2 Increase the temperature of the hot water bath to about 90°C. Momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the macro-Snyder column. Concentrate the extract using 1 mL of hexane to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.1.2.3 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane. A 5 mL syringe is recommended for this operation. Adjust the extract volume to 10.0 mL. Stopper the concentrator tube and store refrigerated

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at 4°C, if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon-lined screw cap or crimp top. Proceed with gas chromatographic analysis if further cleanup is not required.

7.2 Gas chromatography conditions (Recommended):

7.2.1 Column 1: Carrier gas (5% methane/95% argon) flow rate: 60 mL/min Column temperature: 200°C isothermal

When analyzing for the low molecular weight PCBs (PCB 1221-PCB 1248), it is advisable to set the oven temperature to 160° C.

7.2.2 Column 2: Carrier gas (5% methane/95% argon) flow rate: 60 mL/min Column temperature: 200°C isothermal

When analyzing for the low molecular weight PCBs (PCB 1221-PCB 1248), it is advisable to set the oven temperature to 140°C.

7.2.3 When analyzing for most or all of the analytes in this method, adjust the oven temperature and column gas flow so that 4,4'-DDT has a retention time of approximately 12 min.

7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.3.2 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column should be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this prior to beginning initial or daily calibration.

7.4 Gas chromatographic analysis:

7.4.1 Refer to Method 8000. If the internal standard calibration technique is used, add 10 μL of internal standard to the sample prior to injection.

7.4.2 Method 8000 provides instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-concentration standard after each group of 10 samples in the analysis sequence.

<u>Note:</u> A 72 hour sequence is not required with this method.

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7.4.3 Examples of GC/ECD chromatograms for various pesticides and PCBs are shown in Figures 1 through 5.

7.4.4 Prime the column as per Section 7.3.2.

7.4.5 DDT and endrin are easily degraded in the injection port if the injection port or front of the column is dirty. This is the result of buildup of high boiling residue from sample injection. Check for degradation problems by injecting a mid-concentration standard containing only 4,4'-DDT and endrin. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). If degradation of either DDT or endrin exceeds 20%, take corrective action before proceeding with calibration, by following the GC system maintenance outlined in of Method 8000. Calculate percent breakdown as follows:

% breakdown = for 4,4'-DDT	Total DDT degradation peak area (DDE + DDD)				
	Total DDT peak area (DDT + DDE + DDD)				
9 haalidaya	Total endrin degradation peak area (endrin aldehyde + endrin ketone)				
for Endrin	Total endrin peak area (endrin +				

endrin aldehyde + endrin ketone)

7.4.6 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).

7.4.7 Using either the internal or external calibration procedure (Method 8000), determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo cleanup using Method 3620. The resultant extract(s) may be analyzed by GC directly or may undergo further cleanup to remove sulfur using Method 3660.

7.5 Cleanup:

7.5.1 Proceed with Method 3620, followed by, if necessary, Method 3660, using the 10 mL hexane extracts obtained from Section 7.1.2.3.

7.5.2 Following cleanup, the extracts should be analyzed by GC, as described in the previous sections and in Method 8000.

7.6 Calculations (excerpted from U.S. FDA, PAM):

7.6.1 Calculation of Certain Residues: Residues which are mixtures of two or more components present problems in measurement. When they are found together, e.g., toxaphene and DDT, the problem of quantitation becomes even more difficult. In the following sections suggestions are

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offered for handling toxaphene, chlordane, PCB, DDT, and BHC. A 10% DC-200 stationary phase column was used to obtain the chromatograms in Figures 6-9.

7.6.2 Toxaphene: Quantitative calculation of toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene on GC/ECD: (a) adjust sample size so that toxaphene major peaks are 10-30% full-scale deflection (FSD); (b) inject a toxaphene standard that is estimated to be within ± 10 ng of the sample; (c) construct the baseline of standard toxaphene between its extremities; and (d) construct the baseline under the sample, using the distances of the peak troughs to baseline on the standard as a guide (Figures 7, 8, and 9). This procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard. A toxaphene standard that has been passed through a Florisil column will show a shorter retention time for peak X and an enlargement of peak Y.

7.6.3 Toxaphene and DDT: If DDT is present, it will superimpose itself on toxaphene peak V. To determine the approximate baseline of the DDT, draw a line connecting the trough of peaks U and V with the trough of peaks W and X and construct another line parallel to this line which will just cut the top of peak W (Figure 61). This procedure was tested with ratios of standard toxaphene-DDT mixtures from 1:10 to 2:1 and the results of added and calculated DDT and toxaphene by the "parallel lines" method of baseline construction were within 10% of the actual values in all cases.

7.6.3.1 A series of toxaphene residues have been calculated using total peak area for comparison to the standard and also using area of the last four peaks only in both sample and standard. The agreement between the results obtained by the two methods justifies the use of the latter method for calculating toxaphene in a sample where the early eluting portion of the toxaphene chromatogram is interfered with by other substances.

7.6.3.2 The baseline for methoxychlor superimposed on toxaphene (Figure 8b) was constructed by overlaying the samples on a toxaphene standard of approximately the same concentration (Figure 8a) and viewing the charts against a lighted background.

7.6.4 Chlordane is a technical mixture of at least 11 major components and 30 or more minor ones. Gas chromatography-mass spectrometry and nuclear magnetic resonance analytical techniques have been applied to the elucidation of the chemical structures of the many chlordane constituents. Figure 9a is a chromatogram of standard chlordane. Peaks E and F are responses to trans- and cis-chlordane, respectively. These are the two major components of technical chlordane, but the exact percentage of each in the technical material is not completely defined and is not consistent from batch to batch. Other labelled peaks in Figure 9a are thought to represent: Α, monochlorinated adduct of pentachlorocyclopentadiene with cyclopentadiene; B, coelution of heptachlor and α -chlordene; C, coelution of β -chlordene and γ -chlordene; D, a chlordane analog; G, coelution of cis-nonachlor and "Compound K," a

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chlordane isomer. The right "shoulder" of peak F is caused by transnonachlor.

7.6.4.1 The GC pattern of a chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of chlordane can consist of almost any combination of constituents from the technical chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight. Only limited information is available on which residue GC patterns are likely to occur in which samples types, and even this information may not be applicable to a situation where the route of exposure is unusual. For example, fish exposed to a recent spill of technical chlordane will contain a residue drastically different from a fish whose chlordane residue was accumulated by ingestion of smaller fish or of vegetation, which in turn had accumulated residues because chlordane was in the water from agricultural runoff.

7.6.4.2 Because of this inability to predict a chlordane residue GC pattern, it is not possible to prescribe a single method for the quantitation of chlordane residues. The analyst must judge whether or not the residue's GC pattern is sufficiently similar to that of a technical chlordane reference material to use the latter as a reference standard for quantitation.

7.6.4.3 When the chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, quantitate each peak separately against the appropriate reference materials and report the individual residues. (Reference materials are available for at least 11 chlordane constituents, metabolites or degradation products which may occur in the residue.)

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7.6.4.4 When the GC pattern of the residue resembles that of technical chlordane, quantitate chlordane residues by comparing the total area of the chlordane chromatogram from peaks A through F (Figure 9a) in the sample versus the same part of the standard chromatogram. Peak G may be obscured in a sample by the presence of other pesticides. If G is not obscured, include it in the measurement for both standard and sample. If the heptachlor epoxide peak is relatively small, include it as part of the total chlordane area for calculation of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion as in Figure 6j, calculate these separately and subtract their areas from total area to give a corrected chlordane area. (Note that octachlor epoxide, a metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.)

7.6.4.5 To measure the total area of the chlordane chromatogram, proceed as in Section 7.6.2 on toxaphene. Inject an amount of technical chlordane standard which will produce a chromatogram in which peaks E and F are approximately the same size as those in the sample chromatograms. Construct the baseline beneath the standard from the beginning of peak A to the end of peak F as

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shown in Figure 9a. Use the distance from the trough between peaks E and F to the baseline in the chromatogram of the standard to construct the baseline in the chromatogram of the sample. Figure 9b shows how the presence of toxaphene causes the baseline under chlordane to take an upward angle. When the size of peaks E and F in standard and sample chromatograms are the same, the distance from the trough of the peaks to the baselines should be the same. Measurement of chlordane area should be done by total peak area if possible.

NOTE: A comparison has been made of the total peak area integration method and the addition of peak heights method for several samples containing chlordane. The peak heights A, B, C, D, E, and F were measured in millimeters from peak maximum of each to the baseline constructed under the total chlordane area and were then added together. These results obtained by the two techniques are too close to ignore this method of "peak height addition" as a means of calculating chlordane. The technique has inherent difficulties because not all the peaks are symmetrical and not all are present in the same ratio in standard and in sample. This method does offer a means of calculating results if no means of measuring total area is practical.

7.6.5 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, Strobane, and chlordane. In each case, the chemical is made up of numerous compounds. So the chromatograms are multi-peak. Also in each case, the chromatogram of the residue may not match that of the standard.

7.6.5.1 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the tradename Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish.

7.6.5.2 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue. A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue. This may also involve a judgment about what proportion of the different Aroclors to combine to produce the appropriate reference material.

7.6.5.3 Quantitate PCB residues by comparing total area or height of residue peaks to total area of height of peaks from appropriate Aroclor(s) reference materials. Measure total area or height response from common baseline under all peaks. Use only those peaks from the sample that can be attributed to chlorobiphenyls. These peaks must also be present in the chromatogram of the reference materials. Mixtures of Aroclors may be required to provide the best match of GC patterns of sample and reference.

7.6.6 DDT: DDT found in samples often consists of both o,p'- and

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p,p'-DDT. Residues of DDE and TDE are also frequently present. Each isomer of DDT and its metabolites should be quantitated using the pure standard of that compound and reported as such.

7.6.7 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor; it consists of a mixture of six chemically distinct isomers and one or more heptachloro-cyclohexanes and octachloro-cyclohexanes.

7.6.7.1 Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. The elimination rate of the isomers fed to rats was 3 weeks for the α -, τ -, and δ isomers and 14 weeks for the β -isomer. Thus it may be possible to have any combination of the various isomers in different food commodities. BHC found in dairy products usually has a large percentage of β -isomer.

7.6.7.2 Individual isomers $(\alpha, \beta, \tau, \text{ and } \delta)$ were injected into gas chromatographs equipped with flame ionization, microcoulometric, and electron capture detectors. Response for the four isomers is very nearly the same whether flame ionization or microcoulometric GLC is used. The α -, τ -, and δ -isomers show equal electron affinity. β -BHC shows a much weaker electron affinity compared to the other isomers.

7.6.7.3 Quantitate each isomer $(\alpha, \beta, \tau, \text{ and } \delta)$ separately against a standard of the respective pure isomer, using a GC column which separates all the isomers from one another.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

8.2 Mandatory quality control to evaluate the GC system operation is found in Method 8000.

8.2.1 The quality control check sample concentrate (Method 8000) should contain each single-component parameter of interest at the following concentrations in acetone: 4,4'-DDD, 10 mg/L; 4,4'-DDT, 10 mg/L; endosulfan II, 10 mg/L; endosulfan sulfate, 10 mg/L; endrin, 10 mg/L; and any other single-component pesticide, 2 mg/L. If this method is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multi-component parameter at a concentration of 50 mg/L in acetone.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables

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should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000).

8.3.1 If recovery is not within limits, the following are required.

8.3.1.1 Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

8.3.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

8.3.1.3 If no problem is found, re-extract and re-analyze the sample.

8.3.1.4 If, upon re-analysis, the recovery is again not within limits, flag the data as "estimated concentration".

8.4 <u>GC/MS confirmation</u>: Any compounds confirmed by two columns may also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory generated detection limits.

8.4.1 The GC/MS would normally require a minimum concentration of 10 ng/ μ L in the final extract, for each single-component compound.

8.4.2 The pesticide extract and associated blank should be analyzed by GC/MS as per Section 7.0 of Method 8270.

8.4.3 The confirmation may be from the GC/MS analysis of the base/neutral-acid extractables extracts (sample and blank). However, if the compounds are not detected in the base/neutral-acid extract even though the concentration is high enough, a GC/MS analysis of the pesticide extract should be performed.

8.4.4 A reference standard of the compound must also be analyzed by GC/MS. The concentration of the reference standard must be at a level that would demonstrate the ability to confirm the pesticides/PCBs identified by GC/ECD.

9.0 METHOD PERFORMANCE

9.1 The method was tested by 20 laboratories using organic-free reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations. Concentrations used in the study ranged from 0.5 to 30 μ g/L for single-component pesticides and from 8.5 to 400 μ g/L for multi-component parameters. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter

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and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, optional cleanup techniques, and calibration procedures used.

10.0 REFERENCES

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	<u>Retentio</u>	<u>n time (min)</u>	Method
Analyte	Col. 1	Col. 2	limit (µg/L)
Aldrin —	2.40	4.10	0.004
α-BHC	1.35	1.82	0.003
β-BHC	1.90	1.97 👘	0.006
δ-BHC	2.15	2.20	0.009
γ -BHC (Lindane)	1.70	2.13	0.004
Chlordane (technical)	е	е	0.014
4,4'-DDD	7.83	9.08	0.011
4,4'-DDE	5.13	7.15	0.004
4,4'-DDT	9.40	11.75	0.012
Dieldrin	5.45	7.23	0.002
Endosulfan I	4.50	6.20	0.014
Endosulfan II	8.00	8.28	0.004
Endosulfan sulfate	14.22	10.70	0.066
Endrin	6.55	8.10	0.006
Endrin aldehyde	11.82	9.30	0.023
Heptachlor	2.00	3.35	0.003
Heptachlor epoxide	3.50	5.00	0.083
Methoxychlor	18.20	26.60	0.176
Toxaphene	е	е	0.24
PCB-1016	e	e	nd
PCB-1221	е	e	nd
PCB-1232	е	e	nd
PCB-1242	е	e	0.065
PCB-1248	е	e	nd
PCB-1254	е	e	nd
PCB-1260	e	е	nd

TABLE 1. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs*

^aU.S. EPA. Method 617. Organochlorine Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

e = Multiple peak response.

nd = not determined.

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TABLE 2. DETERMINATION OF ESTIMATED QUANTITATION LIMITS (EQLs) FOR VARIOUS MATRICES^a

Matrix	Factor⁵
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

- a Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable.
- b EQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

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Analyte	Test conc. (µg/L)	Limit for s (µg/L)	Rang <u>e</u> for x (µg/L)	Range P, P, (%)
Aldrin α -BHC β -BHC δ -BHC δ -BHC γ -BHC Chlordane 4,4'-DDD 4,4'-DDE 4,4'-DDT Dieldrin Endosulfan II Endosulfan Sulfate Endrin Heptachlor He	2.0 2.0 2.0 2.0 50 10 2.0 10 2.0 2.0 10 10 10 2.0 2.0 50 50 50 50 50 50 50	0.42 0.48 0.64 0.72 0.46 10.0 2.8 0.55 3.6 0.76 0.49 6.1 2.7 3.7 0.40 0.41 12.7 10.0 24.4 17.9 12.2 15.9 13.8 10.4	1.08-2.24 $0.98-2.44$ $0.78-2.60$ $1.01-2.37$ $0.86-2.32$ $27.6-54.3$ $4.8-12.6$ $1.08-2.60$ $4.6-13.7$ $1.15-2.49$ $1.14-2.82$ $2.2-17.1$ $3.8-13.2$ $5.1-12.6$ $0.86-2.00$ $1.13-2.63$ $27.8-55.6$ $30.5-51.5$ $22.1-75.2$ $14.0-98.5$ $24.8-69.6$ $29.0-70.2$ $22.2-57.9$ $18.7-54.9$	42-122 37-134 17-147 19-140 32-127 45-119 31-141 30-145 25-160 36-146 45-153 D-202 26-144 30-147 34-111 37-142 41-126 50-114 15-178 10-215 39-150 38-158 29-131 8-127

TABLE 3. QC ACCEPTANCE CRITERIA[®]

s = Standard deviation of four recovery measurements, in $\mu g/L$.

 \overline{x} = Average recovery for four recovery measurements, in $\mu g/L$.

 $P, P_s = Percent recovery measured.$

D = Detected; result must be greater than zero.

^aCriteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

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Analyte	Accuracy, as recovery, x' (µg/L)	Single analyst precision, s,' (µg/L)	Overall precision, S' (µg/L)
Analyte Aldrin α -BHC β -BHC δ -BHC γ -BHC Chlordane 4,4'-DDD 4,4'-DDT Dieldrin Endosulfan I Endosulfan II Endosulfan Sulfate Endrin Heptachlor Heptachlor epoxide Toyanbene	0.81C+0.04 0.84C+0.03 0.81C+0.07 0.81C+0.07 0.81C+0.07 0.82C-0.05 0.82C-0.04 0.84C+0.30 0.85C+0.14 0.93C-0.13 0.90C+0.02 0.97C+0.04 0.93C+0.34 0.89C-0.37 0.89C-0.04 0.69C+0.10 0.80C+1.74	$(\mu g/L)$ $0.16\bar{x}-0.04$ $0.13\bar{x}+0.04$ $0.22\bar{x}+0.02$ $0.18\bar{x}+0.09$ $0.12\bar{x}+0.06$ $0.13\bar{x}+0.13$ $0.20\bar{x}-0.18$ $0.13\bar{x}+0.06$ $0.17\bar{x}+0.39$ $0.12\bar{x}+0.19$ $0.10\bar{x}+0.07$ $0.41\bar{x}-0.65$ $0.13\bar{x}+0.33$ $0.20\bar{x}+0.25$ $0.06\bar{x}+0.13$ $0.18\bar{x}-0.11$ $0.09\bar{x}+3.20$	S' $(\mu g/L)$ 0.20 \overline{x} -0.01 0.23 \overline{x} -0.00 0.33 \overline{x} -0.95 0.25 \overline{x} +0.03 0.22 \overline{x} +0.04 0.18 \overline{x} +0.18 0.27 \overline{x} -0.14 0.28 \overline{x} -0.09 0.31 \overline{x} -0.21 0.16 \overline{x} +0.16 0.18 \overline{x} +0.08 0.47 \overline{x} -0.20 0.24 \overline{x} +0.35 0.24 \overline{x} +0.25 0.16 \overline{x} +0.08 0.25 \overline{x} -0.08 0.25 \overline{x} -0.08 0.25 \overline{x} -0.22
PCB-1016 PCB-1221 PCB-1232 PCB-1242 PCB-1248 PCB-1254 PCB-1260	0.80C+1.74 0.81C+0.50 0.96C+0.65 0.91C+10.79 0.91C+10.79 0.91C+10.79 0.91C+10.79 0.91C+10.79	$\begin{array}{c} 0.09\underline{x}+3.20\\ 0.13\underline{x}+0.15\\ 0.29\underline{x}-0.76\\ 0.21\underline{x}-1.93\\ 0.21\underline{x}-1.93\\ 0.21\underline{x}-1.93\\ 0.21\underline{x}-1.93\\ 0.21\underline{x}-1.93\\ 0.21\underline{x}-1.93\end{array}$	0.20 <u>x</u> +0.22 0.15 <u>x</u> +0.45 0.35 <u>x</u> -0.62 0.31 <u>x</u> +3.50 0.31 <u>x</u> +3.50 0.31 <u>x</u> +3.50 0.31 <u>x</u> +3.50 0.31 <u>x</u> +3.50

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION*

x' = Expected recovery for one or more measurements of a sample containing concentration C, in $\mu g/L.$

s,' = Expected single analyst standard deviation of measurements at an average concentration of \bar{x} , in $\mu g/L$.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in μ g/L.

C = True value for the concentration, in μ g/L.

m.

 \bar{x} = Average recovery found for measurements of samples containing a concentration of C, in μ g/L.

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Figure 1 Gas Chromatogram of Pesticides

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Figure 5 Gas Chromatogram of Aroclor 1260

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Fig. 6--Baseline construction for some typical gas chromatographic peeks, a, symmetrical separated flat baseline; b and c, overlapping flat baseline; d, separated (pen does not return to baseline between peaks); e, separated sloping baseline; f, separated (pen goes below baseline between peaks); g, α - and γ -BHC sloping baseline; h, α -, β -, and γ -BHC sloping baseline; j, heptachlor and heptachlor epoxide superimposed on chlordane; k, chair-shaped peaks, unsymmetrical peak; l, p,p'-DUT superimposed on toxaphene,

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Figure 7

Fig. 7a--Baseline construction for multiple residues with standard toxaphene.



Fig. 7b--Baseline construction for multiple residues with toxaphene. DDE and 0,p⁴-, and p.p⁴-DDT.

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APPENDIX B CORRECTIVE ACTION FORM

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CORRECTIVE ACTION FORM

CAR No.	<u> </u>	D	ate:	
То:		a	:	Task Manager
You are hereby reque condition, and (B) to (QAM).	sted to take corrective and prevent it from reoccur.	ctions indicated below and as other ring. Your written response is to l	wise dete be returne	rmined by you (A) to resolve the note ed to the Quality Assurance Manage
Condition				
Reference Docurnent	S			
Recommended Corre	ctive Actions			
			:	
Originator	Date	QAM Approval Date	P.N	I. Approval Date
Response		<u> </u>		
A. Resolution B. Pretention C. Affected Docu	iments			
Signature		Date:		
Followup				
Corrective Action Veri	fied:			
Ву:		Date:		

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