## SAMPLING AND ANALYSIS PLAN

**Prepared for:** 

BLACK & DECKER (U.S.) INC. Hampstead, Maryland

**JUNE 1995** 

Prepared by:

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# SECTION 1 INTRODUCTION

Pursuant to the April 1995 Consent Order between MDE and Black & Decker (U.S.) Inc., this Sampling and Analysis Plan (SAP) provides a description of field and laboratory procedures to be followed during investigative activities at the Hampstead, Maryland facility. A description of the Field Sampling Plan is presented in Section 2. The Laboratory Quality Assurance Plan is presented in Section 3. The Site Health and Safety Plan is presented in Section 4.

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# SECTION 2 FIELD SAMPLING PLAN

#### 2.1 <u>SAMPLING LOCATIONS</u>

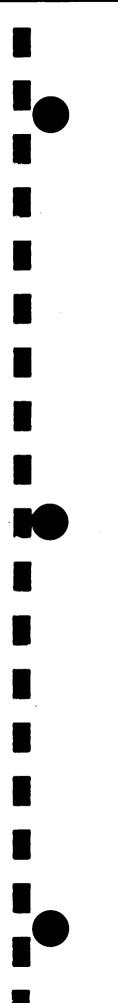
Groundwater sampling will be performed on a quarterly basis. Monitor well locations to be sampled are included in Table 2-1. Surface water and sediment samples will be collected from both the east and west lagoons on a bi-annual basis for a period of one year. In addition, test pits are to be excavated in the brush pile area.

#### 2.2 FIELD OPERATIONS

#### 2.2.1 Soil Investigations

#### 2.2.1.1 Test Pit Excavation

Test pit investigations will be performed using a backhoe. Test pits will be excavated to either the water table, to refusal, or to the maximum reach of the backhoe, whichever is encountered first. Test pit excavation will be supervised by a Weston geoscientist, and complete logs (including visual descriptions of lithology, observations of groundwater occurrence, and instrument readings) will be completed. All descriptions will be made by observations of downhole conditions from the surface, or by examination of material pulled to the surface by the backhoe bucket. The geoscientist will describe the soil after it has been deposited on a plastic sheet situated a minimum of 3 ft from the test pit opening. An HNu photoionization meter or OVA flame ionization detector will be used for air monitoring, and a combustible gas meter will be used to monitor gas emissions in all test pits. If entrance into test pits by Weston personnel is required for soil sampling, the excavation will be sloped or benched in compliance with OSHA requirements. Requirements for sloping are discussed in the HASP.



## Table 2–1 Black and Decker Quarterly Groundwater Sampling Locations

WELL ID NO.
<b>EW</b> -1
<u> </u>
EW-3
<u> </u>
EW-5
EW-6
EW-7
EW-8
EW-9
<b>EW</b> -10
RFW-1A
RFW-1B
RFW-2A
RFW-2B
RFW-3B
RFW-4A
RFW-4B
RFW-5A
RFW-6
RFW-7
RFW-8
RFW-9
RFW-10
RFW-11A
RFW-11B
RFW-12B
RFW-13
RFW-16
RFW-17
RFW-18
RFW-19
TOWN #22
TOWN #23
LEISTER DAIRY WELL
LEISTER RES. WELL #1
LEISTER RES. WELL #2
LEISTER RES. WELL #3
JOS. A BANK PROD. WELL #1
JOS. A BANK PROD. WELL #2
"SHOPPING CENTER" WELL*

\* proposed location

Soil samples will be collected from the identified depths using the backhoe bucket. The backhoe will deposit the material to be sampled on plastic sheeting, and the geoscientist will collect samples using dedicated stainless steel trowels and/or scoopulas. Each test pit will be photographed during excavation and/or upon completion.

If field screening indicates the potential for soil contamination, excavated soils will be segregated into two separate piles, one for potentially contaminated soils and another for soils showing no signs of contamination. Samples will be collected for analysis from each of these piles.

Upon completion, test pits will be backfilled using the soil removed during test pitting pending results of laboratory analysis of those soils, and/or with clean soil from an offsite source. The fill will be compacted with the backhoe to ensure that no cave-ins occur. All test pit locations will be tied horizontally to a coordinate system and vertically to a U.S. Coast and Geodetic Survey (USCGS) or U.S. Geological Survey (USGS) benchmark.

#### 2.2.2 Groundwater Investigation

#### 2.2.2.1 Water Level Measurements

Groundwater level measurements will be taken in wells on a monthly basis and prior to purging or sampling, using an electric water level probe graduated to 0.01 ft. Measurements will be referenced to a surveyed point marked on the top of the PVC or steel riser. The reference point will be described in the records for each well. The total depth of the well will be available from previous measurements. These data will determine the amount of water to be evacuated from each well prior to sampling. Water level measurements will be taken three times per well or until measurements are within  $\pm 0.01$  ft. Measurements will be recorded in the field notebook and on field sampling sheets.

2-3

#### 2.2.2.2 Monitor Well Sampling

Groundwater samples will be collected from monitor wells on a quarterly basis during the months of February, May, August, and November. Upgradient wells will be sampled first and wells suspected of having low levels of contamination will be sampled prior to those suspected of having medium or high levels. Procedures for sampling monitor wells are as follows:

- Water level measurements will be taken to the nearest 0.01 ft with respect to the established survey point located on top of the well casing. All measuring devices used in the well will be decontaminated as specified in Subsection 2.2.4.1 prior to use at each well. The total depth of the well will be measured previously and recorded. The depth to the static water surface will be subtracted from the total casing depth to determine the height and, subsequently, the volume of standing water in the casing.
- To ensure that samples are representative of groundwater quality, a submersible pump will be used to remove a minimum of three times the calculated volume of water in the well casing. The pump will be equipped with a foot-operated check-valve to prevent purged water from flowing back into the well. Wells that become dewatered prior to producing three casing volumes will be sampled as soon as practical once they recover sufficiently.
- Purge water will be containerized if previous analytical data indicate that contaminant levels exceed MCLs.
- Temperature, pH, and electrical conductivity (EC) will be measured and recorded during purging. A visual observation of turbidity (i.e high, medium, low) will also be recorded. A sample will be taken after a minimum of three well volumes have been removed and when the temperature, pH, and EC have stabilized. If these parameters do not stabilize within three purge volumes, a maximum of six well volumes will be removed.

• The groundwater sample will be collected using a clean, dedicated Teflon bailer.

- All samples for chemical analysis will be placed in laboratory-prepared bottles. The bottles will be filled to the top and capped securely. The types of sample containers and preservatives required for VOC analysis are described in Subsection 2.3.1. If required, preservatives will be placed in the sample containers prior to collecting the samples. Extra care will be used in filling VOCs sample vials to ensure that the Teflon liner of the septum is facing inward and that no air bubbles are entrapped. Each sample bottle will be placed in an insulated cooler chest immediately after sampling and maintained at 4°C until extraction.
- All sample equipment will be decontaminated after sampling to prevent cross-contamination between wells, as detailed in Subsection 2.3.4.2. The bailer line will be disposed of at each well and will consist of polypropylene rope. Sampling bailers will be protected from contamination between sampling points by wrapping them in aluminum foil.

#### 2.2.2.3 Extraction Well Sampling

The extraction wells will be sampled on a quarterly basis. Sampling of the extraction wells will be performed by running the spigot located at each well head to purge the lines between sampling. The spigot will be flushed for approximately two minutes prior to sampling. After water quality parameters (pH, temperature, and specific conductance) are measured, VOC samples will be collected directly from the spigot. Each sample bottle will be placed in an insulated cooler immediately after sampling and maintained at 4°C until extraction.

## 2.2.3 Surface Water and Sediment Investigation

#### 2.2.3.1 Surface Water Sampling

A total of three surface water samples will be collected from each of the two surface impoundments located in the southern part of the site. These samples will be collected on a bi-annual basis for one year. Sampling of the impoundment will be performed by

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use of a Kemmer Sampler or a similar device, permitting the collection of water samples from discrete depths in the water column. Samples will be collected at depths of approximately two-thirds of the distance from the surface to the bottom of the lagoon. Each location will be sampled for VOCs.

#### 2.2.3.2 Sediment Sampling

Sediment samples will be collected using a Ponar dredge sampler or equivalent device. These sample devices will allow for collection of discrete samples of the sediment with minimal disturbance to the sediment. Three samples will be collected from each of the two surface impoundments. Each sample will be analyzed for VOCs.

#### 2.2.4 Decontamination Procedures

All material and equipment will arrive at the site in clean condition. Procedures for equipment decontamination are described in the following subsections. Equipment decontamination will be performed at the wastewater treatment plant except as specified below.

#### 2.2.4.1 Water Level Indicator Decontamination

Water level indicators used in wells will be decontaminated after use at the well location by flushing the electrical probe with ASTM Type II reagent water.

#### 2.2.4.2 Water and Soil Sampling Equipment Decontamination

Bailers, bowls, spatulas, trowels, etc., will be decontaminated between sampling points. Pumps used for well purging will be decontaminated by submerging the pump intake in a washing solution (laboratory-grade detergent) and pumping this solution through the pump system. The pump will then be placed in clean potable water and run until the discharge is detergent-free.

The procedure for decontaminating sampling equipment is:

- Rinse equipment in potable water to remove surface dirt and mud, if necessary.
- Scrub equipment with a bristle brush in laboratory-grade detergent and potable water.
- Rinse off soap with potable water.
- Rinse with ASTM Type II reagent water.
- Allow equipment to air dry before use.

## 2.3 <u>SAMPLE HANDLING AND ANALYSES</u>

## 2.3.1 Sample Containers, Sample Volumes, Preservation, and Holding Times

All samples submitted for analyses on this project will be collected by Weston personnel. Sampling containers and preservatives will be provided on request by the contracted analytical laboratory. For water samples, the specific requirements for sample containers, preservatives, and sample volumes for VOCs (Method 8260) are 40 ml clear glass vials with Teflon-lined septum caps, preserved with hydrochloric acid. For soil samples, 120 ml clear glass bottles with Teflon-lined caps, unpreserved, are required for VOC analysis.

Once samples have been collected, field personnel will return to the laboratory with the analytical samples and a completed chain-of-custody record.

The holding times for all required analyses are measured from time of sample collection. Holding time for VOC samples (Method 8260) is 14 days.

Upon sample receipt at the project laboratory, all sample collection dates are noted by the sample custodian. The required dates for completion of analyses (or extractions) are noted on the chain-of-custody record and are keyed to the holding times.

#### 2.3.2 Sample Custody

The purpose of sample custody procedures is to document the history of sample containers and samples (and sample extracts or digestates) from the time of container preparation through sample collection, shipment, and analysis. An item is considered to be in one's custody if:

- It is in the physical possession of the responsible party.
- It is in view of the responsible party.
- It is secured by the responsible party to prevent tampering.
- It is secured by the responsible party in a restricted area.

#### 2.3.2.1 Sample Labels

All samples will be identified with a label that will be attached directly to the container. Sample labels will be completed using waterproof ink. The labels will contain the following information:

- Sample number.
- Time and date of collection.
- Site name (Black & Decker).
- Parameters to be analyzed.
- Preservative (if any).
- Sample source/location.

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As each sample is collected it will be placed in a labeled container.

#### 2.3.2.2 Chain-of-Custody Records

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record will be completed for each sample at each sampling location. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time of transfer, will be documented.

#### 2.3.2.3 Transfer of Custody and Shipment

Prior to shipment of samples, the chain-of-custody record will be signed and dated by a member of the Weston field team who has verified that those samples indicated on the record are indeed being shipped. After packaging has been completed, custody seals, signed and dated by a member of the Weston field team, will be placed on the cooler.

#### 2.3.3 Sampling QA/QC Protocols

Field QA/QC samples will be collected and analyzed as part of all field sampling activities, including surface and subsurface soils and groundwater sampling. The following protocols will be followed for collection of QA/QC samples.

A trip blank consists of a sample bottle filled with ASTM Type II reagent water prepared in the laboratory, brought to the site with the sample bottles, and handled as a sample. One trip blank will be sent to the laboratory each day that VOC samples are collected. The trip blank for soils is also a Type II reagent water.

One equipment blank will be collected during each sampling round for soil and groundwater and analyzed for the same parameters as the corresponding soil or groundwater samples. These equipment blank samples will be collected by pouring ASTM Type II reagent water through the sampling device (e.g., bailer) and into the appropriate sampling container.

Field duplicates will also be collected for water samples and soil samples. The number of field duplicates collected will equal 5% of the total number of samples. A field duplicate will be collected as a separate sample immediately after collection of the field sample that it is intended to duplicate. Collection procedures for field duplicates will be identical to the original samples.

Duplicate groundwater samples will be collected from the same bailer. Duplicate surface water samples will be collected as separate grab samples from the same location, one immediately after another.

#### 2.4 FIELD MEASUREMENTS

#### 2.4.1 Field Parameters

Several parameters will be tested in the field as part of the analytical protocol for water samples. All liquid samples will be tested for temperature, pH, and EC. Turbidity, color and odor will also be noted on the field logs. The following subsections describe the procedures for analysis of field parameters.

#### 2.4.1.1 pH Measurement

The pH of liquid samples will be measured using a Fisher Model No. 107 portable water pH meter (or equivalent). The pH meter will be calibrated and checked against the standard buffer solutions before analyzing a sample. The probe will then be rinsed with distilled water and placed in the sample to be tested. The meter will stabilize for 1 minute before the pH is measured. After the measurement, the probe will be rinsed with

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distilled water and placed in 7.0 buffer solution until the next test. Results will be recorded in the field logbook.

## 2.4.1.2 Electrical Conductivity and Temperature Measurement

Measurements of EC and temperature of liquid samples will be collected using a YSI Model 33 meter (or equivalent). When not in use the meter probe will be placed in a jar of distilled water. Prior to placing the probe in the sample and after measuring the EC and temperature of the sample, the probe will be rinsed with distilled water. The temperature will be taken with the knob set on "temperature" and the EC measured using the appropriate range of the "conductance" setting. A period of 1 minute will be allowed for the instrument to stabilize prior to taking the measurement. Results will be recorded in the field logbook.

## 2.4.2 Equipment Calibration Procedures and Frequency

#### 2.4.2.1 pH Meter

The Fisher Model No. 107 pH Meter, or equivalent, is a portable pH monitoring instrument for determining pH in surface water and groundwater, waste systems, and other water quality applications.

The instrument requires field calibration daily or each time the meter is turned on (if more than once per day). The calibration will be checked at mid-day and at the end of the work day if it is left on all day. Distilled water and buffer solutions (pH 7, pH 4, and pH 10) are required for field calibration. All solutions must be at the same temperature to reduce meter stabilization time and to maintain accuracy. The instrument is calibrated as follows:

1. Rinse the electrode in distilled water.

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- 2. Place the electrode in the pH 7 buffer solution and allow the meter reading to stabilize.
- 3. Adjust the control using the knob on the front panel of the instrument until the meter reads pH 7.
- 4. Rinse the electrode in distilled water.
- 5. Place the electrode in pH 4 solution and allow the meter readout to stabilize.
- 6. Adjust the control knob until the meter reads the correct value of the pH 4 solution.
- 7. Place the electrode in pH 10 solution and allow meter readout to stabilize.
- 8. Adjust the control knob until the meter reads the correct value of the pH 10 solution.
- 9. Record results in logbook.

#### 2.4.2.2 Electrical Conductivity Meter

The YSI Model 33, or equivalent, is a portable, battery-operated, transistorized instrument used to measure salinity, EC, and temperature in surface water, groundwater, and wastewater systems. The meter is calibrated daily or each time the meter is turned on (if more than once per day) by turning the MODE control to REDLINE and adjusting the REDLINE control so that the indicator lines up with the redline on the meter face. The meter will also be calibrated with two solutions with concentrations ranging from 10 to 1,000 µmhos. The calibration will also be checked at the end of the work day if the meter is left on all day.

# SECTION 3 ANALYTICAL LABORATORY QUALITY ASSURANCE PROGRAM PLAN

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OPERATING PRACTICE WESTON Analytics Division: Quality Assurance Program Plan

Eff. Date: 09/01/94

Initiated By: R. J. Frederici Approved By: E. M. Hansen Author

Authorized By: A. M. Henry SP No. 21-06A-001

# ANALYTICAL LABORATORY QUALITY ASSURANCE PROGRAM PLAN (QAPP) ROY F. WESTON, INC.

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OPERATING PRACTICE WESTON Analytics Division: Quality Assurance Program Plan

Eff. Date: 09/01/94

Initiated By: R. J. Frederici Approved By: E. M. Hansen Authorized E

Authorized By: A. M. Henry SP No. 21-06A-001

## QUALITY ASSURANCE PLAN

#### FOR

#### **ROY F. WESTON, INC.**

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## 3.0 <u>QUALITY ASSURANCE POLICY</u>

It is the objective of Roy F. Weston, Inc. (WESTON®) to be acknowledged as an organization that provides high-quality services. WESTON provides high-quality services because it takes pride in its work and because providing such services is a sound business practice.

Quality is the most significant criterion influencing the viability and well-being of WESTON. It is the criterion that distinguishes the best organization from the others.

WESTON is a professional services company and as such the quality of its services and deliverables are dependent on their reception by the client. The quality objective is mutually defined between WESTON and the client and is an inherent part of the quality assurance program.

Within relatively narrow limits, the level of excellence required may vary from project to project and from work element to work element. This is dependent on the technological and the financial risk to either the client or WESTON. There shall be a WESTON "standard of quality" established at a level of excellence to assure attaining a reputation as a quality organization.

The attainment of a reputation as a quality organization does not just happen. It results from a successful concerted effort over a long period of time. Therefore, the effort must be well planned and supported both organizationally and individually. It must be credible and rigorous, yet flexible to respond to the ever-changing needs of WESTON and the client.

Organizational support for accomplishing this quality goal is derived from Policy Directives and Operating Practices. Within these documents, the development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify quality, QA organizations overview the quality achievement and verification process through reviews, audits, and surveillances. Top management leadership, support and/or pressure insures that the Corporate Policies and Practices are appropriately implemented.

Individual support for accomplishing this quality goal is derived from pride and professionalism. The individuals directing, managing, supervising, conducting, reviewing, monitoring, and approving work are individually and collectively accountable for the quality of the work. Pride in one's own effort and professionalism within the organization can assure quality.



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## 3.1 <u>Quality Assurance/Quality Control (QA/QC)</u>

Quality, as the term is used herein, is defined as the level of excellence needed to conform to an established standard. Generally, quality will refer to the excellence of end results and/or the excellence of performance required to attain the established standard.

Quality Assurance (QA) is defined as those planned and systematic actions necessary to provide adequate confidence to WESTON and its clients that the services provided meet mutually accepted quality standards consistent with project scope and budget. Quality assurance is attained through the implementation of a quality control program.

Quality Control (QC) is defined as the operational processes employed to ensure an objective level of excellence. Established performance criteria are defined for all areas, including

- administrative and technical methods and procedures,
- position accountability, duties and authority,
- performance monitoring, and
- peer and supervisory review, check, approval, and sign-off.

QC provides the tools to measure and evaluate the conformance of the operational procedures to criteria.

#### 3.2 Standard of Quality

Standard of quality refers to a specific, defined, pre-established level of excellence. The phrase "standard of quality" will usually be preceded by a descriptive word or phrase such as "WESTON," "EPA," "USATHAMA," "USAFOEHL/TS," "NFESC," "USACE," etc. which references documented descriptions of the level of excellence to be achieved.

A standard of quality shall be established for every Analytics Division project and work element. The vehicle through which this standard is documented and communicated for standard analytical services is this Quality Assurance Program Plan (QAPP) for the Analytics Division, in conjunction with a Project Technical Profile and/or Quality Assurance Project Plan (QAPjP).



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The Project Technical Profile will summarize basic project requirements, such as, QC and deliverables requirements, analyte lists, certification requirements, turn-around-times, and miscellaneous project information (e.g., the project manager, work order number, subcontracting restrictions, hazard restrictions, etc.).

The QAPjP allows for customized, client-specific quality control measures that can be added to or that can supersede the basic laboratory QAPP guidance to satisfy the needs of individual programs with specialized requirements. Laboratory personnel are available to discuss the design, advantages, and disadvantages of other quality control options, and to aid the data user in developing a project specific Quality Assurance Project Plan (QAPjP). Generally, the minimum information to be provided in these project specific plans will include the following:

- background and overall project objectives,
- intended use of the acquired data,
- list of measurement parameters,
- number of samples to be taken,
- kinds of samples to be taken (e.g. soil, surface water, groundwater, biological, sludge, drums, etc.), and
- dates anticipated for project start and completion.

#### 3.3 Document Authorities

This plan has been prepared based on the guidelines and principles set forth in the following documents:

- QAMS-005/80, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80, 29 December 1980 (for format and content),
- ASME NQA-1-1989 Edition, "Quality Assurance Program Requirements for Nuclear Facilities," American Society of Mechanical Engineers, 345 East 47th Street, New York, New York 10017 (for the 18 criteria, but not necessarily all supplements and appendices),



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- WESTON Standard Practices Manual, Chapter 10: "Quality Assurance" (for policies and procedures), and
- "Quality Assurance Manual for Industrial Hygiene Chemistry," prepared by the Analytical Chemistry Committee of the American Industrial Hygiene Association (AIHA), 1988 (for general content).

Tables 3-1 through 3-3 provide matrices showing the correlation of sections in the Analytical Laboratory QAPP with sections of the following documents:

- Table 3-1, QAMS-005/80
- Table 3-2, ANSI/ASME NQA-1
- Table 3-3, AIHA.



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## TABLE 3-1

## Correlation of WESTON'S Analytics Division Quality Assurance Program Plan with EPA QAMS-005/80

EPA QAMS-005/80 REQUIREMENT		ANALYTICS QAPP SECTION	
1.0	Title Page	1.0	Title Page
2.0	Table of Contents	2.0	Table of Contents
3.0	Project Description	3.0	Quality Assurance Policy
4.0	Project Organization and Responsibility	4.0	Organization and Responsibilities
5.0	QA Objectives for PARCC	8.0	Quality Assurance Targets for Precision and Accuracy
6.0	Sampling Procedures	5.0	Sampling Procedures
7.0	Sample Custody	6.0	Chain of Custody
8.0	Calibration Procedures and Frequency	10.0	Calibration Procedures and Frequency
9.0	Analytical Procedures	7.0	Analytical Procedures
10.0	Data Reduction, Validation and Reporting	11.0	Data Reduction and Reporting
11.0	Internal Quality Control Checks	9.0	Internal Quality Control Checks: Laboratory
12.0	Performance and Systems Audits	14.0	Performance and Systems Audits
13.0	Preventative Maintenance Procedures	12.0	Preventative Maintenance
14.0	Specific Routine Procedures to Assess Data PARCC	9.0	Procedures Used to Assess Data Quality
15.0	Corrective Action	13.0	Corrective Action
16.0	Quality Assurance Reports to Management	15.0	Quality Assurance Reports to Management



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## TABLE 3-2

## Correlation of WESTON's Analytics Division Quality Assurance Program Plan with ANSI/ASME NQA-1

	ANSI/ASME NQA-1 CRITERIA*	ANALYTICS QAPP SECTION
1.0	Organization	4.0 Organization and Responsibilities
2.0	Quality Assurance Program	3.0 Quality Assurance Policy
3.0	Design Control	<ul> <li>7.0 Analytical Procedures</li> <li>8.0 Quality Assurance Targets for Precision and Accuracy</li> </ul>
	·	9.0 Internal Quality Control Checks 10.0 Calibration Procedures and Frequency 11.0 Data Reduction and Reporting
4.0	Procurement Document Control	7.0 Document Control
5.0	Instructions, Procedures, and Drawings	(same as NQA-1, item 3.0 Design Control)
6.0	Document Control	1.0 Title Page 2.0 Table of Contents
7.0	Control of Purchased Items and Services	7.0 Document Control
8.0	Identification and Control of Items	7.0 Document Control
9.0	Control of Processes	<ul> <li>3.0 Quality Assurance Policy</li> <li>5.0 Sampling Procedures</li> <li>7.0 Analytical Procedures</li> <li>9.0 Procedures Used to Assess Data Quality</li> <li>11.0 Data Reduction and Reporting</li> </ul>
10.0	Inspection	14.0 Performance and Systems Audits
11.0	Test Control	9.0 Internal Quality Control Checks
12.0	Control of Measuring and Test Equipment	10.0 Calibration Procedures and Frequency 12.0 Preventative Maintenance
13.0	Handling, Storing and Shipping	6.0 Chain of Custody
14.0	Inspection, Test, and Operating Status	12.0 Preventative Maintenance 14.0 Performance and Systems Audits
15.0	Control of Nonconforming Items	<ul> <li>9.0 Internal Quality Control Checks</li> <li>10.0 Calibration Procedures and Frequency</li> <li>12.0 Preventative Maintenance</li> <li>14.0 Performance and Systems Audits</li> </ul>
16.0	Corrective Action	13.0 Corrective Action
17.0	Quality Assurance Records	10.0 Calibration Procedures and Frequency 11.0 Data Reduction and Reporting
18.0	Audits	<ul><li>14.0 Performance and Systems Audits</li><li>15.0 Quality Assurance Reports to Management</li></ul>

\* addresses the intent of NQA-1 as specified in the above 18 criteria, but not necessarily pulling in all the supplements and appendices. Refer to content of referenced section of the Analytics QAPP for extent of implementation of the 18 NQA-1 criteria.



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#### TABLE 3-3

#### Correlation of WESTON'S Analytics Division Quality Assurance Program Plan with AIHA

	AIHA CRITERIA		ANALYTICS QAPP SECTION
1.0	Description of QA Policies	3.0	Quality Assurance Policy
2.0	Organization Chart	4.0	Organization and Responsibilities
3.0	Personnel Qualifications	4.0	Organization and Responsibilities
4.0	Training	15.0	Quality Assurance Reports to Management
5.0	Sampling Considerations	5.0	Sampling Procedures
6.0	Chain of Custody for Samples	6.0	Chain of Custody
7.0	Sample Processing Procedures	5.0	Sampling Procedures
8.0	Reagents	9.0	Internal Quality Control Checks
9.0	Preparation and Storage of Standards and Samples	6.0 10.0	Chain of Custody Calibration Procedures and Frequency
10.0	Analytical Methodology	7.0	Analytical Procedures
11.0	Method Validation	7.0 11.0	Analytical Procedures Data Reduction and Reporting
12.0	Data Reduction Including Statistics	8.0 9.0 11.0	Quality Assurance Targets for Precision and Accuracy Procedures Used to Assess Data Quality Data Reduction and Reporting
13.0	Record Keeping	11.0	Data Reduction and Reporting
14.0	Calibration and Maintenance of Equipment	10.0 12.0	Calibration Procedures and Frequency Preventative Maintenance
15.0	Internal Quality Assurance Procedures	2.0 14.0	Table of Contents Performance and Systems Audits
16.0	External Quality Assurance Procedures	2.0 14.0	Table of Contents Performance and Systems Audits
17.0	Quality Assurance Audit Procedure	14.0	Performance and Systems Audits
18.0	Corrective Action Plan	13.0	Corrective Action



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#### 4.0 ORGANIZATION AND RESPONSIBILITIES

## 4.1 <u>General</u>

Analytics, WESTON's environmental laboratory services division, was established to provide the organizational structure to enhance the attainment of Corporate goals. Corporate goals are to enhance the quality of human life and the physical environment through the creative and sound application of human, economic, and natural resources; advanced science; and applied technology. Our objective is to provide comprehensive and integrated professional services effectively and efficiently. Quality analytical services is a primary WESTON goal.

Within the Analytics Division, the infra-structure provides maximum availability of senior, expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments. Additionally, WESTON Corporate resources in engineering, design and construction, industrial hygiene, geosciences, management and computer sciences, radiological services, air quality management, asbestos monitoring, regulatory compliance, and field operations enhance the Analytics Division capabilities to consult, trouble-shoot, and solve analytical problems. Project specific plans provide the organizational structure for operations with unique environmental situations, as appropriate.

The organization of WESTON's Analytics Division is shown in Figures 4-1 through 4-5. A description of the QA infra-structure describing QA reporting functions outside the laboratory is given in Section 4.2. Line management responsibilities and accountabilities with respect to ensuring that quality goals are met are summarized in Section 4.3. Professional qualifications and experience for the individuals filling these positions are maintained with the laboratory's training records. A description of WESTON's three analytical laboratories are summarized in the following subsections.

#### 4.1.1 WESTON's Lionville Laboratory

The laboratory in Lionville, PA, which is located approximately 25 miles west of Philadelphia and 5 miles from WESTON's corporate offices in West Chester, is staffed by 180 professionals and managed by J. Peter Hershey, Ph.D.. The laboratory has two physical facilities. The first laboratory, located at 208 Welsh Pool Road, is a 39,000 square foot state-of-the-art commercial laboratory capable of performing thousands of environmental chemical analyses a year. The second Lionville laboratory, located at 256 Welsh Pool Road, is designed to handle samples containing high concentrations of contaminants such as PCBs and/or dioxin as well as co-mixed (low level radioactive) waste. It is equipped with a "clean room" designed specifically for handling high-hazard materials such as dioxins.



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All mixed waste samples are received and screened for radioactivity at this facility. Samples containing above-background levels of radioactivity are extracted or digested here. Only non-radioactive extracts/digestates are returned to the 208 facility for analyses; others are completely processed in the 256 facility.

The laboratory is physically divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample Receipt and Refrigerated Storage.
- Organic Sample Preparation.
- Glassware Preparation.
- Metals Digestion.
- Wet Chemistry Laboratory.
- Instrumentation Laboratories.

The main instrumentation laboratories include the following:

- Atomic Absorption Spectroscopy Laboratory.
- Inductively Coupled Plasma Emission Laboratory.
- Gas and Liquid Chromatography Laboratory.
- Gas Chromatography/Mass Spectrometry Laboratory.

Each of these areas has separate heating, ventilation, and air conditioning systems. Nondestructive gas chromatographic detectors, GC autosampler flush solvent, and GC/MS rotary pumps are vented out of the instrumentation laboratories through charcoal filters. The main laboratory at 208 Welsh Pool Road was designed and the construction was managed by the laboratory managers and staff who pooled their accumulated laboratory experience to construct a highly functional, efficient facility which is one of the premier environmental laboratories in the country. It boasts a number of unique features such as the layout of the organic extraction area, the design of the GC/MS laboratories, and an energy-efficient water recirculation system.

The organic sample preparation laboratory, one of the largest in this business, has capacity for performing 80 Soxhlet or 80 continuous liquid-liquid extractions, 100 sonication extractions, and/or 50 separatory funnel extractions each day. The unique configuration of the extractors and the specially constructed fume exhausts were designed by the laboratory staff and managers to facilitate rapid, efficient sample preparation. 

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#### 4.1.2 WESTON's Gulf Coast Laboratory

The WESTON-Gulf Coast facility is located approximately 30 miles south of Chicago in University Park, Illinois, is staffed by 120 professionals and managed by Michael J. Healy. This laboratory has the instrumentation, personnel and expertise to handle almost any analytical requirement. The laboratory, located at 2417 Bond Street, comprises 45,000 square feet of laboratory and office space and provides environmental services to industrial and governmental clients including the Illinois Environmental Protection Agency. WESTON-Gulf Coast, Inc., is also actively involved in field sampling of quarterly groundwater sampling at more than 500 monitoring wells per year.

The laboratory is physically divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample Receipt and Refrigerated Storage.
- Organic Sample Preparation.
- Glassware Preparation.
- Metals Digestion.
- Wet Chemistry Laboratory.
- Instrumentation Laboratories.
- Data Report Preparation and Production.

The main instrumentation laboratories are:

- Atomic Absorption Spectroscopy Laboratory.
- Inductively Coupled Plasma Emission Laboratory.
- Gas and Liquid Chromatography Laboratory.
- Gas Chromatography/Mass Spectrometry Laboratory.

Instrumentation laboratories have separate heating, ventilation, and air conditioning systems. Nondestructive gas chromatographic detectors, GC autosampler flush solvent, and GC/MS rotary pumps are vented out of the instrumentation laboratories through charcoal filters.

#### 4.1.3 WESTON's Stockton Laboratory

The Stockton Laboratory is located approximately 80 miles east of San Francisco and 50 miles south of Sacramento, is staffed by 60 professionals and managed by Bosco M. Ramirez. The laboratory, located at 212 Frank West Circle, Suite A, consists of 25,000 square feet of laboratory and office space and is divided into separate areas for organic sample preparation, inorganic sample preparation; instrumentation rooms for GC/VOA and GC/MS VOA, GC/MS BNA, ICP, HPLC, and AA; sample receipt and storage; and data



# ANALYTICS DIVISION STANDARD PRACTICES MANUAL

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management. This facility is dedicated to provide rapid turnaround of specific analysis to support programs such as remediation. The Stockton Laboratory performs CLP analyses for inorganic and organic TCL constituents. In addition, the Stockton Laboratory has provided analytical services to a variety of other governmental agencies including the U.S. Air Force OEHL, the Bureau of Land Management and numerous industrial clients.

#### 4.2 **Quality Assurance Infra-Structure**

The chief Corporate Quality Assurance (QA) Officer is the Executive Vice President, Quality Assurance/Finance. Corporate QA managerial and implementation responsibilities and authorities are held by the Vice President, Corporate Quality Assurance. The position has the authority to organize, initiate, and monitor quality assurance programs. The Corporate QA Vice President can review and approve/disapprove all Division Quality Assurance Plans; can initiate, man (e.g., teams and committees), and allocate costs of the audit process for the purpose of identifying problems and determining compliance with Corporate policies and practices; and is obligated to recommend corrective action depending upon the situation.

Within the Analytics Division, the Division QA Manager and the QA personnel in the individual laboratories are responsible for implementing and monitoring the divisional QA These individuals have sufficient authority, access to work areas, and Program. organizational freedom (including sufficient independence from cost and schedule considerations) to:

- identify problems affecting quality;
- initiate, recommend, or provide solutions to problems through designated channels;
- verify implementation of solutions; and
- assure that further work is stopped or controlled until proper resolution of a nonconformance, deficiency, or unsatisfactory condition has occurred.

The Quality Assurance Section in each laboratory reports to the Laboratory Manager, where appropriate action can be affected. However, should a situation arise where acceptable resolution of identified problems cannot be agreed upon at the laboratory level, the QA Section has direct access to Divisional and Corporate QA Management. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure that QA policies and procedures are enforced.



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#### 4.3 Description of Laboratory Personnel Responsibilities

The specific duties and responsibilities of the Division Manager, Project Director, Laboratory Manager, Project Manager, Technical Manager, Section Managers and Unit Leaders, Quality Assurance Personnel, Health and Safety Manager, Waste Management Personnel, and Analysts are as follows.

#### 4.3.1 <u>Analytics Division Manager</u>

The Analytics Division Manager is ultimately responsible and accountable for establishing and implementing the Division's quality policy, as well as ensuring performance and profitability of the Division. The Division Manager reports directly to the Chief Operating Officer of the Corporation.

#### 4.3.2 <u>Project Director</u>

The laboratory Project Director (PD) is responsible for the overall direction of a project from a divisional level and has primary responsibility for project quality assurance. The PD is accountable for the following:

- Defining the level of excellence for the project performance and/or results.
- Assuring the preparation of a tailored Project Technical Profile and/or Quality Assurance Project Plan (QAPjP).
- Ensuring peer review of the adequacy of QAPjP's.
- Ensuring allocation of proper quality control budgets.
- Attaining concurrence with Section (e.g., Laboratory) Managers on performance and/or results objectives.
- Achieving acceptable project implementation performance.
- Approving the quality of the project results (e.g., data, reports).

The PD reports to the Division Manager and does not have line authority over those performing the work.



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# 4.3.3 Laboratory Manager

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Manager, who is accountable to the Division Manager for this function. The Laboratory Manager has the authority to effect those policies and procedures to ensure that only data of the highest attainable quality is produced. It is the Laboratory Manager's responsibility to see that all tasks performed in the laboratory are conducted according to the requirements of this Quality Assurance Program Plan (QAPP), the Project Technical Profile and/or the appropriate QAPjP; to assure that the quality of service provided complies with the project's requirements.

The Laboratory Manager supports a QA Section which is not subordinate to or in charge of any person having direct responsibility for sampling and analysis, and that has additional reporting responsibilities to Corporate QA.

### 4.3.4 <u>Project Manager</u>

WESTON recognizes the importance of efficient project management. The laboratory Project Managers (PMs) are responsible for preparing the Project Technical Profile summarizing QA/QC requirements for the project, maintaining the laboratory schedule, ensuring that technical requirements are understood by the laboratory, and advising the Project Director and Laboratory Manager of all variances.

The laboratory PM will provide technical guidance and the necessary laboratory related information to the preparer of project specific Quality Assurance Project Plans (QAPjP) are (generally prepared by the client), and provide peer review of the final document to ensure accuracy of the laboratory information.

# 4.3.5 <u>Technical Manager</u>

Technical Managers report to the Laboratory Manager and serve as the technical expert on assigned projects, provide technical liaison, and assist in resolving any technical issues within the area of their expertise. They are responsible for providing input and review in the development and implementation of the QA/QC program; and for providing the critical review of proposal and project work for programs as directed by the Laboratory Manager. They will coordinate these activities with the Project Manager and Quality Assurance Section.



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#### 4.3.6 <u>Section Managers/Unit Leaders</u>

To assist the Laboratory Manager in achieving section goals, the Laboratory Section Managers and Unit Leaders are responsible for the implementation of established policies and procedures. They possess the authorities commensurate with their responsibilities for the day-to-day enforcement and monitoring of laboratory activities.

Section Managers have the responsibility for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; and that system audits are performed on an as-needed basis. These system audits will include the analysis of external check samples to determine the analyst/instrument capability to identify and quantify routine analyses.

#### 4.3.7 Data Management Section Manager

The Data Management Section Manager is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the Project Technical Profile, and ensuring that data are reported in a timely manner and in the proper format.

#### 4.3.8 <u>Quality Assurance Manager</u>

The Quality Assurance Manager has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in the Quality Assurance Plan. In addition, the QA Manager may assist in the preparation, compilation, and submittal of quality assurance plans. The Quality Assurance Manager reviews program plans for consistency with organizational and contractual requirements and will advise appropriate personnel of deficiencies. The Quality Assurance Manager maintains a sufficient staff to initiate and oversee audits and corrective action procedures, performs data review, and maintain documentation of training. In addition, the Quality Assurance Manager has the authority to stop work on projects if QC problems arise which affect the quality of the data produced.

#### 4.3.9 Quality Assurance Personnel

The Laboratory Quality Assurance Personnel have responsibility for conducting and evaluating results from system audits. In addition, the preparation of operating practices and quality assurance documentation for the laboratory shall be coordinated by the QA Section. The QA Section will review program plans, as requested, for consistency with organizational and contractual requirements and will advise appropriate personnel. The group also performs data review responsibilities, Performance Evaluation Sample Tracking,



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Personnel Training Tracking, Document Control, MDL/IDL Studies, Solvent Check Tracking, and Certifications.

# 4.3.10 <u>Health and Safety/Waste Management</u>

The Health and Safety Manager is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, developing a safety plan that complies with Federal Regulations, conducting laboratory safety tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedure for emergency situations. The Health and Safety Manager or designee is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Manager responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste.

# 4.3.11 <u>Chemists/Technicians</u>

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is in-control before reporting results.

4.4 <u>Personnel Qualification and Training</u>

# 4.4.1 Basic Requirements

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Once on board, each employee participates in a comprehensive training program coordinated by the Laboratory's QA Section. This program is designed to provide an introduction to laboratory policies and procedures, define training mechanisms, and document the processes that provide proof of an analyst's proficiency. Highlights of the program include an orientation process, initial training to attain requisite proficiency, continual monitoring of the employee's progress, opportunity for career enhancement seminars or training courses, and documentation files to track each analyst's progress.



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Orientation provides the employee with a basic introduction to WESTON and to the laboratory. Company and laboratory goals and expectations are presented and explained, applicable QA and Health and Safety items are discussed, key laboratory staff are introduced, available resources are discussed, and the employee receives an orientation manual to store introductory materials and/or Operating Practices (OPs).

Following orientation, training in the specifics of the task to be performed is the first major laboratory assignment. The trainee, under supervision of a qualified analyst, receives onthe-job instruction which culminates in sign off of a method training record to document the training. Details of this training program are in related OPs.

Documentation of training (internal and external) is maintained by the QA Section in the employee's training file.

#### 4.4.2 <u>Project-Specific Requirements</u>

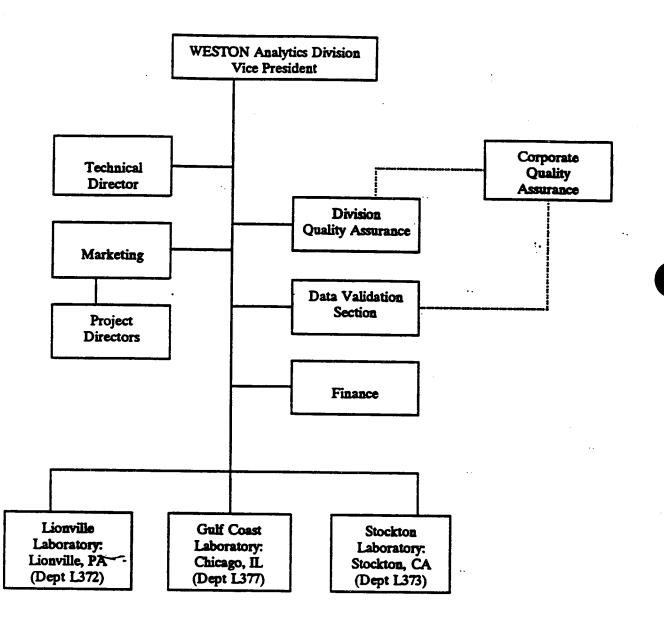
Prior to work on a new project, task specific training will occur to discuss schedules and unique aspects of the project. This may be accomplished at the production unit level, or at a job opening meeting. Items to be discussed may include the project Technical Profile, turn around times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special protocol requirements.



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### FIGURE 4-1

# **Analytics Division Organizational Chart**



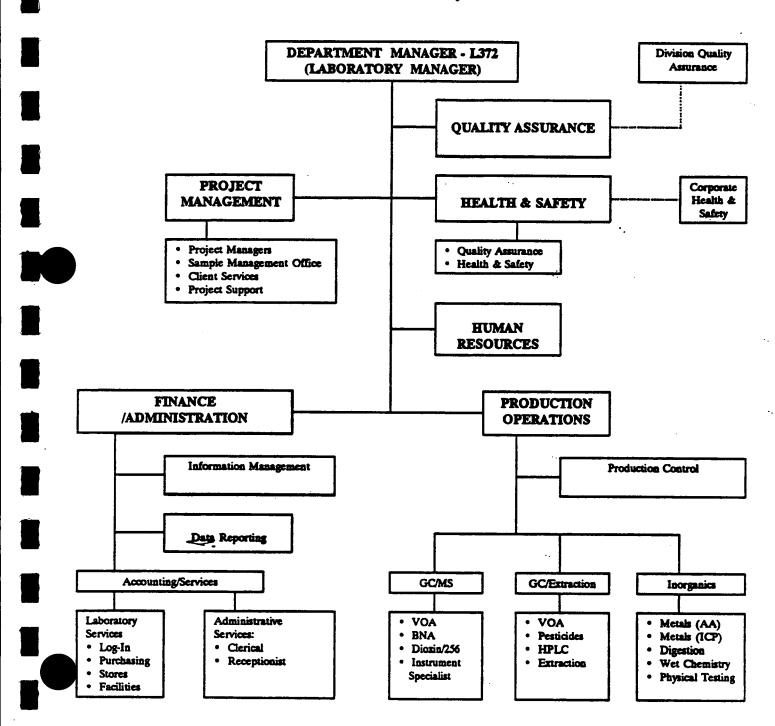


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#### FIGURE 4-2

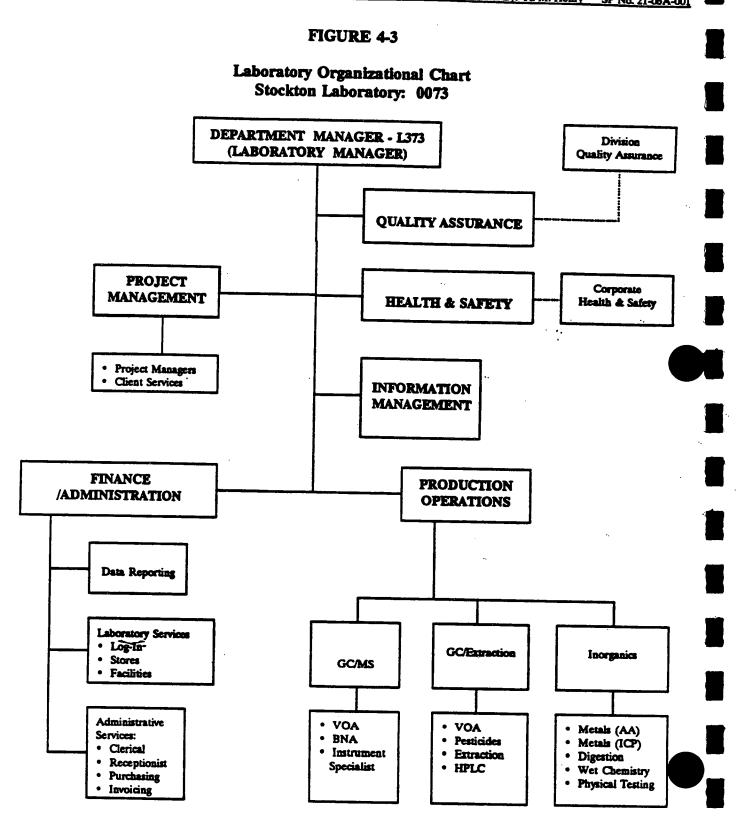
#### Laboratory Organizational Chart Lionville Laboratory: 0072





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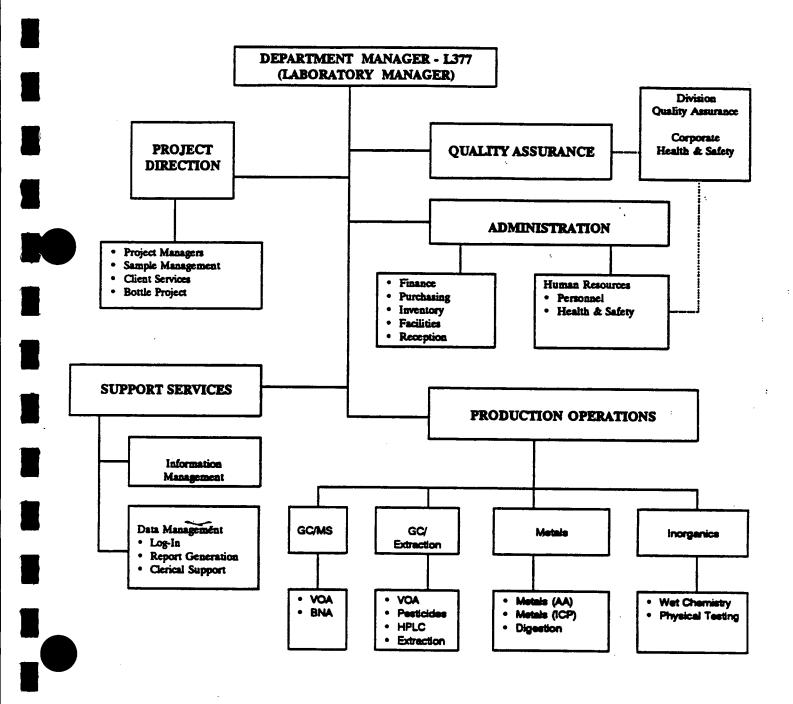


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#### FIGURE 4-4

#### Laboratory Organizational Chart Gulf Coast Laboratory: 0077





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#### 5.0 SAMPLING PROCEDURES

WESTON has the capability to conduct field sampling in accordance with specific agency guidelines, established operating practices, and/or project specific Sampling and Analysis Plans (SAPs). When field sampling is requested the sampling protocols are selected to correspond with the project scope and objectives.

#### 5.1 <u>Sample Preservation and Holding Times</u>

Depending on the selected methods and regulatory program, the Analytics Division adheres to sample preservation requirements as stated in Table II of 40 CFR Part 136, Manual for Certification of Laboratories Analyzing Drinking Water, USEPA CLP Document Nos. OLM01.8 and ILM02.1, as updated, and SW846 3rd Edition, July 1992. All samples are required to be preserved in the field prior to or immediately following sample collection. Upon sample receipt and log-in, the samples are maintained in a temperature controlled environment to insure sample integrity.

The project manager is contacted whenever any sample received is not appropriately preserved. The project manager contacts the client for a decision on preserving the sample in the laboratory or refers to instructions in the Technical Profile/Quote which may delineate the client's requirements regarding actions for sample(s) not appropriately preserved. With either action, the sample's preservation status at the time of laboratory receipt is noted on the chain-of-custody form or other documentation record. For additional information regarding sample receipt and integrity, refer to Section 6.0.

The analysis holding time is the maximum time that may elapse before sample preparation or analysis. It is measured from the date of sample collection in the field, unless the sample analyses are requested for compliance to specific programs (e.g., USEPA CLP) in which holding times are determined by verified time of sample receipt (VTSR). Generally, drinking water, wastewater and RCRA program analyses require holding times measured from sample collection. These holding times are listed in 40 CFR Part 136 and/or the EPA Manual for the Certification of Laboratories Analyzing Drinking Water: Criteria and Procedures, Quality Assurance, Change 2 - September 1992; SW846 3rd Edition, Methods of Chemical Analysis of Water and Waste; Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992; or other established program requirements.

Holding times are tracked in the laboratory with the aid of the Laboratory Information Management System (LIMS). Each laboratory section may have additional tracking systems to ensure that holding times are met. All sample collection dates and receipt dates are recorded on the chain-of-custody. This information is transferred into LIMS by sample login personnel and LIMS calculates the appropriate holding time for each parameter (GC/MS



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Sections monitor their holding times). This information is then available to laboratory personnel for tracking and scheduling purposes.

The LIMS backlog reports list sample preparation and analysis due dates for each method based on the requested reporting date or the holding time, whichever is earlier.

All analyses which have holding times of 48 hours or less are identified by the sample custodian(s). Log-in then notifies the appropriate operating section or unit that short hold time samples have been received by the laboratory and are ready for analysis.

# 5.2 <u>Sample Bottles</u>

WESTON provides sample bottles and preservatives to clients upon request. Each laboratory maintains a bottle preparation section that coordinates the assembly and shipment of sample bottles, preservatives and shipping coolers.

The laboratories may procure sample bottle types with varying levels of supporting quality control (QC). For the most rigorous QC, bottles are cleaned according to USEPA washing procedures and each bottle lot is analyzed for purity. The lot number is labeled for traceability to a certificate of analysis. For less rigorous supporting QC, bottles are cleaned according to USEPA washing procedures, but a certificate of analysis is not provided with the bottles. All bottles used by the Division are washed in accordance with EPA guidance and contain a certificate of analysis, or the laboratory randomly tests and documents the purity of supplied bottles.

Other than sample bottles and preservatives, the following items are included in sample shipping coolers: packing materials, custody seals, chain-of-custody forms and zip-lock bags (for paperwork). Supplies are checked regularly to insure that ample amounts are available to support the Division's analytical projects.

# 5.2.1 Bottle Washing

All sample bottles are cleaned using USEPA guidance. Typically, the bottle suppliers use one of three procedures described further in this section or cleaning procedures which produce equivalent results as determined by the laboratory's QA Manager or designate through the review of the suppliers certificate of analysis.



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# 5.2.1.1 <u>Cleaning Procedure A</u>

Bottles cleaned by Procedure A are Boston Round, amber glass bottles (1-liter) and jugs (1-gallon).

- Bottles, liners and caps are washed in laboratory-grade, non-phosphate detergent.
- Rinsed 3 times with distilled water.
- Rinsed with 1:1 Nitric Acid.
- Rinsed 3 times with ASTM Type 1 organic-free water.
- Oven-dried for 1 hour.
- Rinsed with hexane.
- Oven-dried for 1 hour.

#### 5.2.1.2 <u>Cleaning Procedure B</u>

Bottles cleaned by Procedure B are 40-mL vials; 1000-mL, 4-oz., and 8-oz. clear wide-mouth glass jars.

- Vials, septa and caps are washed in laboratory-grade, non-phosphate detergent.
- Rinsed 3 times with distilled water.
- Rinsed 3 times with ASTM Type 1 organic-free water.
- Oven-dried for 1 hour.

#### 5.2.1.3 -<u>Cleaning Procedure C</u>

Bottles cleaned by Procedure C are the 1-liter, 250-mL, and 500-mL plastic bottles.

- Bottles, liners and caps are washed in laboratory-grade, non-phosphate detergent.
- Rinsed 3 times with distilled water.
- Rinsed with 1:1 Nitric Acid.



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- Rinsed 3 times with ASTM Type 1 organic-free water.
- Air-dried.

# 5.2.2 Bottle Preservatives

The Division's laboratories use preservatives that are prepared internally from reagent-grade chemicals or pre-measured reagents in ampules purchased from suppliers.

All preservatives are shipped in a manner consistent with local and federal shipping regulations.

The internally prepared reagents are added to the sample bottles prior to shipping or premeasured ampules are shipped with the sample bottles and are added at the time of sampling. Ampuled preservatives obtained from suppliers are received with quality control documentation verifying purity. These records are maintained by the laboratory and are available for inspection. Each shipment of preservatives are custody sealed and contain a certificate of analysis. An identifying lot number is on each ampule to assure preservative traceability.

# 5.2.3 <u>Placement of Bottle Orders</u>

Requests for sample bottles are initiated with a bottle request form and then forwarded to the bottle preparation section. All bottle order forms are maintained by the bottle section. Typically, this form includes the following information: Client name, company contact, shipping address, telephone number, date, WESTON contact, courier used, date needed, date sent, number of samples, bottle type(s), parameter requested, preservative needed, shipping costs, facility/I.D., matrix, no. of coolers used, and space for additional comments.

# 5.2.4 Sample Cooler Preparation

Coolers having a unique identification code are pulled from stock and the identification code is recorded in a cooler log book or on the bottle request form and the chain of custody during log-in. When coolers are used, the client name and date are noted in the log book to allow tracking of the cooler.

All bottles are packed and arranged to minimize breakage during transport. Depending on the client need and regulatory program requirements, blue ice or ice is shipped with the cooler. All documentation information is enclosed in a plastic zip-lock bag and placed inside the cooler to prevent water damage. The cooler is then closed and sealed.



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#### 5.2.5 <u>Sample Cooler Shipment</u>

Coolers are shipped to the sampling site by various means: a common courier service may be used; the laboratory's field personnel may take coolers to the site; or the client may pick up coolers at the laboratory. All shipment of sample coolers are performed in accordance with the guidelines established by the International Air Transport Association (IATA) and the Department of Transportation (DOT).

Typically, orders requiring next day air delivery (e.g., internal clients) are shipped via Federal Express. In this event the Federal Express Power Ship II computer tracking system is used to track all shipments. For orders not requiring next day delivery, regular United Parcel Service (UPS) or Federal Express is typically used. (Delivery times vary from one to seven days for receipt of shipment.) UPS or other common couriers may be used to provide next-day air and second-day air delivery.

#### 5.2.6 <u>Sample Cooler Maintenance</u>

Sample coolers returned to the laboratory are emptied of packing materials, ice (both water and blue ice), and any extra or broken bottles or other materials. The coolers are washed and placed back into stock. WESTON marked "blue" ice is washed in tap water before being reused. Typically, empty client coolers are returned via regular UPS or Federal Express.

Each laboratory maintains a tracking system to determine where each cooler is sent. If a cooler has not been returned, the client is contacted via letter or phone call regarding the status of the cooler(s) return. Records are periodically reviewed to locate coolers that have not been returned to the laboratory.



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6.0 <u>SAMPLE CUSTODY</u>

6.1 <u>Sample Receipt</u>

Designated sample custodian(s) and staff are responsible for samples received at the WESTON laboratories. In addition to receiving samples, the sample receipt staff is also responsible for documentation of sample receipt, storage before and after sample analysis. The operating practices for sample receipt are described in detail in Lionville, Stockton, and Gulf Coast OPs 21-08L-002, 21-08S-002 and 21-08G-0002 respectively. A summary of these procedures follows.

Upon receipt, the sample custodian signs, dates, and documents the time of sample receipt on the air bills received from the couriers. The sample custodian signs the chain-of-custody assuming custody of the samples. If a chain-of-custody is not received with a set of samples, a "Custody Transfer/Lab Work Request" form is initiated. Refer to Figure 6-1 for an example of a "Custody Transfer/Lab Work Request". The sample custodian inspects the sample cooler for integrity and then documents the following information: the type of courier, shipped or hand delivered (copies of the airbills are maintained); sample temperature, ambient or chilled; the presence of leaking or broken containers; and documentation of sample preservation.

Additionally, the sample holding time and date collected are checked. If all samples were received within the appropriate holding time, it is documented on the custody transfer record.

Any additional comments are documented in the designated "Notes" section on the chain-ofcustody. Any errors made on the chain-of-custody are corrected by drawing a single line through the incorrect entry, initialing and dating the cross out.

The sample custodian or designee matches the sample container information (e.g., sample tag/label), log book information, chain-of-custody records, and all pertinent information associated with the sample. The sample custodian then verifies sample identity to assure that all information is correct. Any inconsistencies are resolved with the client through the project manager and corrective action measures are documented before sample analysis proceeds.

The sample custodian assigns a unique RFW # (as described in Section 6.4) to each sample received. The RFW # is recorded on the chain-of-custody and on the container labels using a permanent marker. The RFW # is the primary means of tracking a sample through the laboratory.



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The chain-of-custody form is maintained at the laboratory. Copies of the chain-of-custody are provided to the sample custodian or project manager, section managers, and sample preparation personnel. In addition, the sample custodian notifies the appropriate production unit(s) of any analyses requiring immediate attention due to short holding times.

The sample custodian logs the sample information into the Laboratory Information Management System (LIMS). These data include laboratory number, field sample identification, dates collected and received, project or client identification, and parameters requested for analysis.

#### 6.2 <u>Sample Containers</u>

Sample containers are verified against the requested analysis to ensure compatibility. For example, organic extractables must be sampled in glass jars with Teflon@-lined caps, since plastics can contaminate the sample.

# 6.3 <u>Sample Custody</u>

Chain-of-custody procedures document the historical possession of sample containers and samples, sample extracts and sample digestates. The associated documentation provides traceability of sample containers from the time of preparation (e.g., NJDEPE), through collection, shipment, storage, analysis and disposal of the sample. Custody, as defined by this document, is referred to as:

- It is in someone's actual possession, or
- It is in someone's view, after being in their physical possession, or
- It was in someone's possession and then locked, sealed or secured in a manner which prevents unsuspected tampering, or
- It is placed in a designated and secured area.

WESTON recognizes that all laboratory data has the potential to be used as evidence for litigation, and that an evidentiary trail is necessary to prove that the samples collected in the field was the sample actually analyzed. An appropriately documented chain-of-custody form provides the essential evidentiary trail that maintains sample integrity. Chain-ofcustody procedures are described within this section. Listed below are typical laboratory and field records used to establish chain-of-custody and sample identification.

• Field chain-of-custody forms, field sampling forms or other information which arrives with the sample.



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- The laboratory's "Custody Transfer Record/Lab Work Request" form. The sample custodians document relinquish and return-receipt of samples to the analyst for analysis or preparation/extraction procedures. Final custody is transferred upon sample disposal.
- Sample labels or tags that may be attached to sample containers may contain such information as: sample date/time of collection; sample description; sample matrix; filtration, preservation and other known hazards; sample management (disposal information); and parameter groupings. Any sample labels/tags are verified for accuracy against the associated information received with the samples. The signed chain-of-custody form serves as documentation of this information verification. If directed by the client or program requirements, sample tags are removed and placed in the sample/project file.
- Custody seals may be attached to sample containers and/or the transport coolers. Custody seals also prevent the containers or coolers from being unsuspectedly opened without authorization.
- Sample preparation logs, e.g., organic extraction and metals digestion, or separate chain-of-custody logs, e.g., electronic chain of custodies, document the custody transfer of the sample extracts/digestates from the preparation group to the analyst. These preparation logbooks are hard-bound laboratory books that are documented in legible hand-writing, and signed and dated by the analyst. The sample extracts remain in refrigerated storage while in the custody of the analytical section. Custody is transferred upon sample disposal.
- Sample storage log (same as the laboratory's "Custody Transfer Record/Lab Work Request" chain-of-custody form).
- Sample disposition log, which documents sample disposal by a contracted waste disposal company.
- Errors in all documentation are deleted with one line through it, the appropriate correction made, initialed and dated by the person making the correction. All documentation/logbooks are signed/initialed by the appropriate personnel.

#### 6.4 <u>Sample Identification</u>

The sample custodian organizes the sample containers, chain-of-custody records, and all pertinent information associated with the sample. The sample custodian then verifies the sample identity against all associated sample information. Any inconsistencies are resolved



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and corrective action is taken prior to assigning an internal identification number to the sample. This unique identification number (RFW #) is an eleven digit number in the following format: YYMMLBBB-XXX,

where YYMMLBBB is the RFW batch #, and

YYMM = year/month, e.g., 9401.

- L = Laboratory identifier, e.g., L = Lionville; S = Stockton; G = Gulf Coast.
- BBB = a computer assigned consecutive batch number which typically rolls over after 999 to 001. It may roll over sooner to assure project continuity or as otherwise documented.
- XXX = a consecutively assigned sample number unique to a specified field sampling point. Because of preservation and volume requirements for requested analytes, a sample from one field sampling point may arrive in more than one containers. In this case, each bottle from the same sampling point is assigned the same number.

Samples are maintained in refrigerated storage coolers (maintained at  $4 \pm 2$ °C) prior to analysis.

### 6.5 <u>Sample Storage</u>

The standard operating practices for sample storage are described in detail in OPs 21-08L-002, 21-08S-002 and 21-08G-0002. A summary of these procedures is described below.

Refrigerated storage coolers are maintained at  $4 \pm 2$ °C. The temperature is recorded twice daily during business days in a hard-bound logbook. Additionally, some storage systems are monitored by the laboratory security system. Quality assurance personnel or sample custodians monitor these temperatures. If equipment failure occurs during working hours (e.g., compressor failure, door left open, circuit breaker) and temperatures exceed the upper or lower control limits, the appropriate personnel are notified. If the temperature can not be returned to an in-control status, the samples are moved to a suitable storage cooler until the problem is corrected.



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Cooler storage is designed to segregate samples in such a way as to minimize the possibility of cross-contamination. This includes the storage of volatile samples separate from semi-volatiles and inorganic samples. Within each cooler, samples are organized by RFW batch # for easy retrieval.

Refrigerators and freezers are used for storing analytical standards. Within the refrigerators or freezers, standards are stored by an internal identification number for easy retrieval. Standards are stored separately from samples.

Access to laboratory facilities is restricted to laboratory personnel or escorted guests, as described in Section 6.8. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel.

#### 6.6 <u>Sample Tracking</u>

Sample Tracking Procedures are summarized in the following sections.

# 6.6.1 Organic Extraction/Analysis

The organic sample preparation section receives samples for extraction prior to analysis by gas chromatography, GC/MS, or liquid chromatography. A sample preparation batch number is assigned and all pertinent data are recorded in a bound laboratory notebook. The first two characters of the extraction/preparation batch number are the last two digits of the current year and followed by an unique laboratory code.

The extraction information is transferred to LIMS and a hard-copy Sample Extraction Record is generated. Original records are used for internal laboratory custody transfer when required. Copies provided to analysts are notification that extracts are ready for analysis. Extracts are maintained in refrigerated storage until needed for analysis.

# 6.6.2 Metals Digestion/Analysis

The metals sample preparation section receives samples for digestion prior to elemental analysis by Atomic Absorption Spectroscopy, Inductively Coupled Plasma Spectroscopy or Mercury autoanalyzer. Before samples are prepared, a sample preparation batch number is assigned and all pertinent data are recorded in a bound laboratory notebook. The first two characters of the digestion batch number are the last two digits of the current year and followed by an unique laboratory code.



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Digestion information is transferred to LIMS and a hard-copy Sample Digestion Record is generated. The Metals Analysis Section is notified and documentation of the transfer of digestates is maintained.

#### 6.7 <u>Record Keeping</u>

All data related to sample preparation, analysis, and general observations are recorded in appropriate hard-bound laboratory notebooks. These logbooks are pre-formatted notebooks which are issued and controlled by the laboratory's Quality Assurance Section. Laboratory notebook pages are reviewed, signed and dated by laboratory analysts and receive a secondary review by a trained data reviewer or supervisor who signs/initials and dates the data pages. After these first two reviews are completed, the data is entered into the Laboratory Information Management System (LIMS).

Corrections to notebook entries are made by drawing a single line through the erroneous entry and writing the correct entry next to the one crossed out. A reason for the correction is noted, as appropriate. All corrections are initialed and dated by the individual performing the correction.

All analytical data is either entered directly into LIMS or is transferred via spreadsheet. Using the latter form, the spreadsheet is reviewed by a supervisor or trained reviewer for transcription errors and acceptability of quality control measurements. Once approved, the data is transferred to LIMS.

A separate LIMS analytical batch number is established for each set of 20 or fewer samples for each type of analysis. This batch number is used to name spreadsheet files. All approved spreadsheets and laboratory data books are maintained as a historical record.

6.8 <u>Building Security</u>

The laboratories maintain controlled building access at all times. All non-WESTON laboratory personnel and service representatives are required to sign the visitors logbook in the reception area and are accompanied by laboratory personnel while in the building. The laboratory is locked at all times, unless a receptionist is present to monitor building access (e.g., between the hours of 8:00 a.m. and 5:00 p.m. Monday through Friday). Laboratory employees may be granted access to the facility during non-routine working hours by the use of access cards. Security systems are installed to monitor building access.



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#### 6.9 Electronic Data Records

WESTON utilizes a Laboratory Information Management System, or LIMS, for the management of sample tracking, data storage, data reduction, and data reporting functions. The LIMS acronym actually refers to the WESTON-customized programs or software. Personal computers and/or a local area network are utilized to access data from LIMS, transfer data to LIMS, and utilize the various LIMS features for data management. File transfers comprise the predominant analyst use of LIMS.

#### 6.10 LIMS Security System

All users log onto the system via the Multi-Terminal Monitor, or MTM. Security is controlled through individual user passwords which allow or deny access into specific accounts (e.g., Metals, Wetlab, Reports, etc.). Each account may further allow or deny access to specific programs or tasks.

While entry or modification of information in the data base is controlled by these security measures, the data base itself has some built in safeguards. When a sample is logged in for specific analyses, the analysis for that sample is assigned a status code by LIMS. The following is a list of typical status codes used by LIMS. Other codes may be established to assist project tracking.

#### Status Meaning

- Sample and Test are logged into LIMS
- 1234567 Sample is on hold
- Sample extraction is in process
- Sample has been subbed out
- Extraction/Digestion/Prep is complete
- Analyzed but not yet reviewed
- Completed (results reviewed by analyst)
- 89 Released to Data Management (results/deliverables in data reporting)
- Canceled
- 88 Released to Client

These and additional codes are automatically updated via programs used by the analysts when reporting data to LIMS. When an analytical result has been entered into the data base and the status set to 8 (released), the result cannot be accidentally overwritten if a new result is submitted. The result can only be changed by person(s) with the proper security clearance to change the status to a lower level. For example, a request from a Section Manager and full written documentation of the change is required to change a LIMS status code. Once the status has been reset, the results for that analysis can be updated.

The status of a result does not reach 8 (Released) until the result and its related Quality Control (QC) has been reviewed and approved by the Section Manager (or designee, e.g.,



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Unit Leader) of the section generating the result. A data summary report can be used by the supervisor to review results and associated QC. If the supervisor approves the data, the status code is then set to 8.

# 6.11 System Preventative Maintenance

The System Operator makes daily, weekly, and monthly checks on the system performance. A daily check is made of the computer's system log which records all activity regarding the computers OS/32 operating system. This log reveals problems with the basic system tasks that keep the system working. Some programs, aside from the Operating System, create their own logs which are also checked to verify they are performing properly.

A weekly on-line backup is made of the entire database, "on-line" meaning the system does not have to be stopped in order to backup all of the data base records to tape. In addition to backing up the database, all data files submitted by instruments or analysts used to upload analytical data into the database are also backed up to tape on a weekly basis.

A monthly Preventative Maintenance check from the computer manufacturer assures that the hardware is in good operating condition. This check requires that the system be brought down while the technician runs various diagnostic checks.

All system maintenance and problems are recorded in a bound system logbooks which are kept in the LIMS Section.

# 6.12 Software Updates and Revisions

WESTON has three laboratories, each with similar LIMS hardware and software. The Division Information Systems Manager works with Section Systems Managers to keep the three systems compatible.

Updates in WESTON software are done by distributing a Programming Completion Form to each laboratory by the laboratory preparing the modifications. This document is kept on file in the LIMS office along with the date the new software was loaded and implemented. New software is tested and verified for accuracy with dummy data before being implemented on the system. Software is approved for use by the Systems Operator and all documentation that it works correctly are maintained on a verification file. After implementation, all users are advised of the changes and requested to report any problems with the software to the System Operator.

When updates in software are received from manufacturers, this is noted in the bound LIMS logbook.



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### **FIGURE 6-1** Example: Internal Chain-of-Custody

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#### 7.0 <u>ANALYTICAL PROCEDURES</u>

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices.

Choice of method is determined by the type of samples and the client/agency program represented. These programs include, but are not limited to the following:

- Drinking Water.
- Wastewater.
- Hazardous Waste.
- Air.

For non-routine analytical services (e.g., special matrices, research projects, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology.

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the laboratory Project Manager in a Technical Profile for the project. The Technical Profile is distributed to appropriate laboratory management, such as the Laboratory Director, Section Managers, Unit Leaders, and QA Manager, to ensure that the proper analytical methods are applied when the samples arrive.

#### 7.1 <u>Method References</u>

The most commonly used method references for the analytical procedures used in the laboratory are listed below. These references are applicable to the analytical test methods used on a daily basis in the laboratory.

- ASTM = <u>Annual Book of ASTM Standards</u>, American Society for Testing and Materials, updated yearly.
- CLP-O = EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis, Multi-Media, Multi-Concentration: Document Number OLM01.8 and as updated.



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- CLP-I = EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Inorganics Analysis, Multi-Media, Multi-Concentration: Document Number ILM02.1 and as updated.
- E = EPA 600/4-79-020, <u>Methods for Chemical Analysis of Water and Wastes</u>, March 1989; EPA 600/4-88-039, <u>Methods for Determination of Organic</u> <u>Compounds in Drinking Water</u>, August 1993.
- 40CFR = 40 CFR Part 136, <u>Guidelines for Establishing Test Procedures for the</u> <u>Analysis of Pollutants</u>. Appendix A to Part 136 - Methods for Organics Chemical Analysis of Municipal and Industrial Wastewater.

Appendix C to Part 136 - Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes Methods 200.7.

- NIOSH = NIOSH <u>Manual of Analytical Methods</u>, 3rd Edition, February 1984, updated through Supplement 4, August 1990.
- SW = EPA SW846, <u>Test Methods for Evaluating Solid Waste</u>, 3rd Edition, promulgated update I, dated July 1992 and proposed Update II, dated November 1992.
- SM = <u>Standard Methods for the Examination of Water and Wastewater</u>: 15th Edition, 1980, 16th Edition, 1985, 17th Edition, 1989, 18th Edition, 1992 (Note method numbers changed format with 17th Edition)
- THAMA = USATHAMA PAM 11-41, <u>United States Army Toxic and Hazardous</u> <u>Materials Agency (USATHAMA) Quality Assurance Program</u>, Revision 0, January 1990. Methods certified per requirements set forth in Section 5.

Methods performed routinely are delineated in Operating Practices (OPs). The OPs applicable to any set of preparatory or analytical procedures are in area-specific manuals that are immediately available to analysts (Lionville and Stockton laboratories) or are furnished in individual manuals to the analysts (Gulf Coast laboratory). Methods that are performed only rarely for special requests are photocopied and furnished directly to the analyst. In no case is an analyst to perform a preparatory or analytical procedure, whether for quality control or on client samples, without a copy of the client or laboratory-approved method readily available.



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#### 7.2 **Document** Control

Each laboratory maintains a document control system that tracks the distribution of both administrative and laboratory Operating Practices (OP). This system, maintained in a data base. tracks the OP Number, copy number, distributee, document revision number, date of distribution, and the reason for distribution. Upon revision to a document, this database is used to identify previous distributees to ensure that all are provided appropriate updates and the database is then updated accordingly.

Document distribution is processed as a controlled or uncontrolled status. Controlled status is defined as the continuous distribution of document updates. Uncontrolled status is defined as the single distribution of the current OP. Document updates are not distributed to uncontrolled status holders. For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written in red ink to easily identify the OP as a controlled copy.

Each laboratory maintains original historical copies of OPs specific to the originating laboratory. Original-signature Divisional OPs are maintained by the Division QA Manager, currently located at the Gulf Coast Facility. For local laboratory distribution, a master copy of the Divisional OP is control-copied to each laboratory and becomes that laboratory's historical copy for subsequent distribution and archiving is conformance with the document control system procedures. By use of a prefix in the assigned document copy number, WESTON is able to track the origin of the OP's distribution. The following prefixes are used for OP distribution:

- Analytics Division (distributed by the Division QA Manager) Α G
  - Gulf Coast
  - Lionvillle =

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Stockton =

#### Material Procurement and Control 7.3

In conformance with the Corporate policy of Roy F. Weston, Inc. (WESTON<sub>®</sub>), it is the policy and practice of the Analytics Division to conduct its procurement activities with the highest integrity, under the most favorable terms to WESTON and its clients, and in full compliance with applicable Government Laws and Regulations.

This will be accomplished through the effective utilization and implementation of the system of procedures contained in WESTON's Procurement Procedures Manual. These procedures are applicable to all WESTON purchasing operations, including Corporate and Regional



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Offices, that place procurements under Government Contracts and Federal Acquisition Regulation (FAR).

Procurement activities are designed to assure a systematic approach to the procurement process. Suppliers are selected based on their capabilities to provide items or services in accordance with the laboratory requirements. The measurements for evaluation and selection of suppliers include the following:

- Evaluation of supplier's history of providing an identical or similar product.
- Supplier's current quality records supported by documented qualitative and quantitative information that can be objectively evaluated.
- Supplier's technical and quality capabilities as determined by a direct evaluation of his facility, personnel and implementation of his quality assurance program.
- 7.3.1 Acceptance of Items and Services

Items used by WESTON Analytics Division laboratories undergo inspection upon receipt, e.g., verify conformance with the requirements of the purchase order and freedom from damage during shipment. Analytical standards and critical-specification solvents, acids, and reagents are

supported by a certificate of quality and/or on-site analytical verification, as applicable. Items not meeting these requirements are returned to the supplier.

7.3.1.1 <u>Certificate of Conformance</u>

For items such as standards, solvents and acids a certificate of conformance may be acceptable. The certificate must at a minimum contain the following information: identity of the item, the pertinent information required (e.g., concentration, log number, expiration date), and signature and date.

# 7.3.1.2 Solvent Lot Verification

Pre-purchase approval is performed for all solvents purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, acetonitrile, ethyl ether, freon, hexane, iso-octane, methanol, methylene chloride, toluene, bottled deionized water, and bottled HPLC water. Prior to purchase, a sample case of the solvent is provided by the vendor to the laboratory for testing. If the solvent passes acceptance criteria, the vendor is notified and holds the respective lot in reserve for laboratory use.



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The approved lot is shipped to the laboratory in increments until the entire lot has been received. Prior to exhaustion of the reserve lot, the process will be repeated with a new lot to ensure a constant supply of approved solvent.

Supplier-provided samples of each solvent lot requiring pre-purchased approval are submitted to sample log-in and tracked through LIMS. Acceptance criteria and parameter lists are found in each laboratory's solvent approval procedures. A copy of the results and raw data will be filed in the Quality Assurance Section.

Each lot of incoming supplies of solvents requiring pre-approval is checked against an approved lot number list. If the lot number is not on the approved list, the lot is refused. If the case of solvent is an approved lot number, it is accepted and documented.

Near depletion of the lot, a sample from a new lot is requested for pre-approval testing, received and analyzed prior to the solvent use in the laboratory. The entire process is then repeated. With review and approval of the managers of the service group using the solvent, and the laboratory QA Section, vendor-performed assays may be used.

# 7.3.2 <u>Control of Materials</u>

All supplies are purchased through the laboratory purchasing agent and received and distributed through the shipping/receiving unit of the laboratory.

# 7.4 <u>Laboratory Glassware</u>

All glassware must be thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware. The laboratory maintains detailed Operating Practices for cleaning glassware documented in Gulf Coast Laboratory OP 21-06G-0007, Lionville Laboratory OP 21-15-0001 (inorganics) and OP 21-16-0001 (organics), and Stockton Laboratory OP 21-15S-0001 (inorganics) and OP 21-16S-0001 (organics). These OPs shall be posted in the appropriate glassware preparation areas.

For difficult to clean glassware, supervisory assistance is recommended. An attempt will be made to determine the composition of material so that appropriate cleaning procedures and safety measures can be taken. The following may be tried with proper precautions: NOCHROMIX, chloroform, acetone, strong base (50% NaOH), hot phosphorus, or other acids. Safety must be a primary concern. A summary of general cleaning procedures follows:



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•	General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.
•	Volumetric flasks and pipettes used for inorganics, test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.
•	BOD glassware cleaning includes a nitric acid and/or a NOCHROMIX- washing step.
•	Microbiological containers must be sterilized prior to use.
•	Organic glassware includes a solvent-wash.
•	Non-volumetric organic glassware may optionally be kiln dried at 450°C.
7.5	Reagent Storage

All laboratory chemicals are segregated according to group. For example, strong acids are never stored with strong bases. Upon receipt in the laboratory, all reagents are marked with the date of receipt, and are marked upon the date of opening.

Prudent safety practices are followed for possible accidental spills of laboratory reagents (e.g., sodium bicarbonate solutions for acid/base spills, or oil-drill/Xsorb for oil spills, etc.). Table 7-1 summarizes the general storage protocols for reagents.

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### TABLE 7-1

# **Reagent Storage**

Reagent	Method of Storage						
Acids	Acid storage lockers, segregated according to acid type.						
Bases	Base storage cabinet.						
Solvents	Solvent storage lockers, segregated by group: Extractions, VOAs, Semi-VOAs, and Pesticides/Herbicides.						
Dry Granular or Powder Reagents	Reagents are stored in each respective laboratory						

Note: All Reagents are documented with the date received, date opened, date expired (as applicable), and the analysts initials.

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#### 8.0 <u>QA TARGETS FOR PRECISION AND ACCURACY</u>

#### 8.1 <u>Precision</u>

Precision is the level of agreement among repeated independent measurements of the same characteristic, usually under a prescribed set of conditions (e.g., under the same analytical protocol). The most commonly used estimates of precision are the relative percent difference (RPD) for cases in which only two measurements are available, and the percent relative standard deviation (%RSD) when three or more measurements are available. In both cases, the quantitative measure of the variability of the group of measurements is compared with their average value. This is especially useful in normalizing environmental measurements to determine acceptability ranges for precision, since it effectively corrects for the wide variability in sample analyte concentration.

Precision control limits are established by duplicate analysis of Laboratory Control Samples (LCS). These LCSs may be purchased commercially or prepared at the laboratory. For organic analysis, they are typically referred to as blank spikes (BS). For multi-analyte methods, including preparation methods such as metals digestions, LCSs may only contain a representative number of target analytes rather than the full list. For organic analyses, the LCS pair may be surrogate compounds in the blank and the blank spike and/or a select number of target analytes in duplicate fortified blanks (blank spike/blank spike duplicate: BS/BSD). The duplicate LCSs are subjected to all sample preparation steps. The RPD of the duplicate analysis is recorded and evaluated by statistically generated warning and control limits or established program requirements that precision be  $\leq 20$  RPD. The calculations of the limits are defined in Section 11.0.

The RPD for duplicate investigative sample analysis provides a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to further assess a reported result within the context of the project Data Quality Objectives (DQOs). For results outside control limits provided as requirements in the QAPP, corrective action appropriate to the project will be taken and the deviation will be noted in the case narrative accompanying the sample results.



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# 8.2 <u>Accuracy and Bias</u>

Accuracy is the degree of agreement of an analytical measurement with the true or expected concentration. When applied to a set of observed values, accuracy will be a measure of both random error and systematic error (bias).

Bias is systematic error inherent in an analysis caused by some artifact of the measurement system or deviation from protocol. Temperature effects and extraction inefficiencies are examples of the first kind; contamination, mechanical losses, and calibration errors are examples of the latter kind.

Accuracy control limits are established and controlled by the analysis of Laboratory Control Samples (LCS), which are of water and/or solid/waste matrices. These LCSs may be purchased commercially or prepared at the laboratory, and are identified as blank spikes (BS) for organic analysis. For multi-analyte methods, including preparation methods such as metals digestions, the LCS may only contain a representative number of target analytes rather than the full list. For organic analyses, in particular for multi-analyte methods, the LCS may be surrogate compounds in the blank or a select number of target analytes in fortified blanks (blank spike: BS). The LCSs are subjected to all sample preparation steps. Additionally, an LCS in each matrix will be analyzed to demonstrate control of the analysis for water and soil samples. The amount of each analyte recovered in LCS analysis is recorded and entered into a method- and matrix-specific database to generate statistical control limits for percent recovery of that LCS. These empirical data are compared with available method reference criteria and available databases to establish control criteria.

The % R for fortified (spiked) investigative sample analysis provides a tool for evaluating how well the method worked for the respective matrix. These values are used by the client to assess a reported result within the context of the project DQO's. For results outside control limits provided as requirements in the QAPP, corrective action appropriate to the project will be taken and the deviation will be noted in the case narrative accompanying the sample results.

Accuracy for some procedures is evaluated as the degree of agreement between a new set of results and a historical database or a table of acceptable criteria for a given parameter. This is measured as percent difference (%D) from the reference value, and is primarily used by the laboratory as a means for documenting acceptability of continuing calibration or of standards.



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The quality assurance objectives for organic and inorganic analyses are tailored to the analytical technique used and are discussed in the ensuing subsections. The number and types of quality control samples for precision and accuracy and their associated acceptance criteria will be specified by the individual methods or project specific DQO's.

#### 8.2.1 <u>Metals/Inorganics Analysis</u>

For metals and inorganics analysis, analytical accuracy is obtained from the analyte recovery measured in a laboratory control standard, QC check sample, and/or a field sample fortified with the element of interest. See also Section 9.0 for further information on matrix QC indicators.

Laboratory-derived control limits will be developed after at least 20 points have accumulated, but only if the data set appears to be random. If there are insufficient points to develop such control limits, or if charts of the data show a pattern or trend that places their credibility as a source of control limits in question, the control limits below will be used. Out-of-control situations and the actions taken to return the system to a control status will be documented in a Corrective Action Report (CAR) or Sample Discrepancy Report (SDR).

The temporary default values will apply to routinely analyzed metals (e.g., metals included in the following lists: drinking water, priority pollutant, RCRA, Appendix IX, and EPA's Contract Laboratory Program target analyte list for hazardous waste) and inorganics. In the case of unusual analytes or infrequently used methods, control limits may be based on fewer than 20 data points if the data exhibit a random graphic pattern.

Laboratory Control Standard (LCS) Fortified Field Sample (matrix spike)

80-120% Recovery 75-125% Recovery

# 8.2.2 Organic Analysis (GC and GC/MS)

For organic analysis, analytical accuracy is based on a select set of analytes measured in a laboratory control standard, or a QC check sample. These recovery measurements comprise both target compounds and surrogate compounds, and are representative of compound lists analyzed routinely (e.g., compounds from the following lists: drinking water, priority pollutant, RCRA, Appendix IX, and EPA's Contract Laboratory Program target analyte list for hazardous waste). Refer to Tables 8-1 and 8-2 for accuracy objectives. See Section 9.0 for further information on matrix QC indicators.

Laboratory-derived control limits will be developed after at least 20 points have accumulated, but only if the data set appears to be random. If there are insufficient points



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to develop such control limits, or if charts of the data show a pattern or trend that places their credibility as a source of control limits in question, the published control limits for the respective CLP, EPA, or SW-846 methods will be utilized. Out-of-control situations and the actions taken to return the system to a control status will be documented in a Corrective Action Report (CAR) or Sample Discrepancy Report (SDR).

Fortification of the sample with target analytes prior to extraction (matrix spike) provides recovery data for the actual target compound as affected by the respective sample matrix. and requires analysis of a second sample, unspiked, to allow correction for any of the compound indigenous to the sample when evaluating recoveries.

## 8.3 <u>Representativeness and Comparability</u>

For laboratory procedures, an attempt will be made to ensure that all data are representative of the matrix and conditions of the sample being measured. The data will be calculated and reported in units consistent with standard reporting conventions to enable comparability to existing data, standards, and/or regulatory action limits.

A measure of inter-laboratory comparability is obtained through the laboratory's participation in performance evaluation programs established with Round Robin suppliers and the USEPA Water Supply (WS) and Water Pollution (WP) programs. In addition, the laboratories employ the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent on the sampling plan on a project specific basis, and therefore are not covered in this laboratory plan. Assessment of site and collection representativeness and comparability is performed by the field engineer.

## 8.4 Method Detection Limits

The method detection limit is the lowest concentration that can be seen for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDL's reflect a calculated value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.



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#### 8.5 <u>Reporting Limits</u>

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error. Because of the high level of quantitative error associated with determinations at the level of the MDL, WESTON endeavors to keep reporting limits significantly higher than the MDL, although client requirements may necessitate reporting at such levels. Wherever possible, reporting is limited to values approximately 3x the respective MDL to ensure confidence in the value reported.

Method detection level studies are performed annually, and PQLs are calculated. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optional performance or appropriate action is taken.

#### 8.6 <u>Completeness</u>

Completeness is a measure of the relative number of analytical data points which meet all the acceptance criteria for accuracy, precision, and any other criteria required by the specific analytical methods. Project specific completeness goals account for all aspects of sample handling, from collection through data reporting. The level of completeness can be affected by loss or breakage of samples during transport, as well as external problems which prohibit collection of the sample. The ability to meet or exceed completeness objectives is also dependent on the nature of samples submitted for analysis. For example, if the analytical methods proposed for use (particularly for organics analyses) are intended for analysis of environmental samples of low and medium hazard, the applicability of these methods to non-routine matrices such as drum samples, wipes, air samples, etc. may result in poor method performance and therefore adversely impact on achievement of the data completeness goal. Completeness is variable and a project-specific requirement.

## Completeness is calculated as follows:

Completeness = <u>Number of acceptable reported QC data</u> X 100% Total number of reported QC data

Criteria for evaluating completeness will be in accordance with the specific data quality objectives for a given project, as defined by the relevant Project Quality Assurance Plan.



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## TABLE 8-1

# Quality Assurance Objectives for Accuracy for Organic Surrogate Analyses\*

Fraction		Percent Recovery		
	Surrogate Compound	Water	Soil/Sediment	
VOA	Toluene-d.	88-110	84.120	
VOA	4-Bromofluorobenzene	86-115	84-138	
VOA	l,2-Dichloroethane-d,	76-114	59-113 70-121	
BNA	Nitrobenzene-de	35-114	23-120	
BNA	2-Fluorobiphenyl	43-116	30-115	
BNA	p-Terphenyl-d	33-141	·	
BNA	Phenol-d.	10-110	18-137	
BNA	2-Fluorophenol	21-110	24-113	
BNA	2,4,6-Tribromophenol	10-123	25-121	
BNA	2-Chlorophenol-d	33-110 (advisory)	19-122	
BNA	1,2-Dichlorobenzene-d,	16-110 (advisory)	20-130 (advisory 20-130 (advisory	
PEST	2,4,5,6-Tetrachloro-m-xylene	60-150 (advisory)	60 180 (	
PEST	Decachlorobiphenyl	60-150 (advisory) 60-150 (advisory)	60-150 (advisory)	
PEST	Di-n-butylchlorendate		60-150 (advisory	
		24-154 (advisory)	20-150 (advisory)	

This list includes selected compounds used for QA/QC accuracy and precision control in the groups (fractions) of analytes shown. Selected compounds are consistent with guidance presented in U.S.EPA SW-846, 3rd edition or the U.S. EPA Contract Laboratory Program (CLP) Statement of Work, Document No. OLM01.8. Stated control limits are based on performance and have been adopted from the cited SOW.

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#### TABLE 8-2

#### QA Objectives for Accuracy and Precision for Organic Target Compound Analyses\*

		% Recovery Limits		RPD Limits <sup>b</sup>	
Fraction	Matrix Spike Compound	Water	Soil/ Sediment	Water	Soil/ Sediment
VOA	l,1-Dichloroethene	61-145	59-172	14	22
VOA	Trichloroethene	71-120	62-137	14	24
VOA	Chlorobenzene	75-130	60-133	13	21
VOA	Toluene	76-125	59-139	13	21
VOA	Benzene	76-127	66-142	11	21
BN	1,2,4-Trichlorobenzene	39- 98	38-107	28	23
BN	Acenaphthene	46-118	31-137	31	19
BN	2,4-Dinitrotoluene	24-96	28-89	38	47
BN	Pyrene	26-127	35-142	31	36
BN	N-nitroso-di-N-propylamine	41-116	41-126	38	38
BN	1,4-Dichlorobenzene	36- 97	28-104	28	27
ACID	Pentachlorophenol	9-103	17-109	50	47
ACID	Phenol	12-110	26-90	42	35
ACID	2-Chlorophenol	27-123	25-102	40	50
ACID	4-Chloro-3-methylphenol	23- 97	26-103	42	33
ACID	4-Nitrophenol	10- 80	11-114	50	50
PEST	Lindane	56-123	46-127	15	50
PEST	Heptachlor	40-131	35-130	20	31
PEST	Aldrin	40-120	34-132	22	43
PEST	Dieldrin	52-126	31-134	18	38
PEST	Endrin	56-121	42-139	21	45
PEST	4,4-DDT	<b>38-127</b> ·	23-134	27	50
РСВ	Aroclor 1254	50-150	50-150	30	50

This list includes selected compounds used for QA/QC accuracy and precision control in the groups (fractions) of analytes shown. Selected compounds are consistent with guidance presented in U.S.EPA SW-846, 3rd edition and/or the U.S. EPA Contract Laboratory Program (CLP) Statement of Work, Document No. OLM01.8. Stated control limits are based on performance and have been adopted from the cited SOW, with the exception of PCB. PCB limits are based on laboratory performance data.

<sup>b</sup>RPD = Relative Percent Difference



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## 9.0 <u>QUALITY CONTROL CHECKS AND ROUTINES TO ASSESS</u> <u>PRECISION AND ACCURACY AND CALCULATION OF METHOD</u> <u>DETECTION LIMITS</u>

#### 9.1 <u>Quality Control Checks</u>

The quality of analytical data generated at WESTON's laboratories is controlled through management systems, processes and procedures described in the Quality Assurance Program Plan. This section describes the minimal internal quality control checks used in each laboratory operating section. If method Quality Control (QC) is more stringent than these laboratory guidelines, the method QC will be followed except when a method modification or variance is authorized by the client or a project specific QAPjP. The Project Manager must obtain such exceptions in writing from the client.

#### 9.2 <u>Quality Control Indicators and Analysis Frequency</u>

Quality control (QC) indicators, introduced in various phases of the analytical process, are tools used to evaluate method performance and to assist in the validation of analytical results. There are two basic types of QC indicators: (1) indicators to evaluate method performance at both the preparation and the measurement steps and (2) QC indicators to evaluate matrix effects. Refer to Section 13.0, Corrective Actions, for procedures to follow when QC indicators show deviation from acceptance criteria.

## 9.2.1 <u>Method Performance OC Indicators: Preparation Batch</u>

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Each prep batch has a maximum of 20 investigative samples.

QC indicators are added to each prep batch to monitor method performance. All QC indicators such as blanks, matrix spikes, matrix duplicates, blank spikes, control samples, or duplicates of these controls are processed through the entire analytical procedure with samples.

## 9.2.1.1 <u>Preparation Blanks</u>

The preparation blank (PB), also referenced as a method blank (MB) or reagent blank (RB), is used to monitor for potential contamination introduced during the sample



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preparation and analytical processes. For organics, preparation (prep) blanks are prepared by processing laboratory pure water for water samples or a purified solid matrix for soil, sediment or solid samples (when available or when requested). The solid matrix for nonvolatiles is generally sodium sulfate. The preparation blank volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation.

For metals, the prep blank consists of laboratory pure water for both water (PBW) and soil or sediment (PBS) samples. Final results are calculated as  $\mu g/L$  for the PBW in metals (exception: RCRA metals reported as mg/L) and mg/L for the PBW in wet chemistry. To facilitate comparison to actual field samples, final results for the PBS are calculated as mg/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding investigative samples.

Field blanks and trip blanks, when received, will be analyzed in the same manner as other investigative samples. However, a field blank should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the investigative samples. Usually, the client sample ID will provide a information to identify the field blanks with labels such as "FB", "TB", "Rinse Blank".

## 9.2.1.2 Laboratory Control Samples and Blank Spikes

Laboratory control sample (LCS) used by the inorganics sections and blank spike (BS) used by the organics sections have the same connotation. The LCS and BS are prepared from a reference source of known concentration and processed through the entire preparation and analysis steps. These QC indicators are processed concurrently with investigative samples and are used to assess method performance independent of potential investigative sample matrix affects. LCSs are performed in duplicate for each preparation batch of 20 or fewer samples, except in some organic analyses when surrogates in the method blank and blank spike are compared to assess method precision. Blanks spikes and blank spike duplicates are not required by the method but are performed based on WESTON and client specific requirements.

For solid matrices, an aqueous LCS may be processed, unless a solid LCS is requested, through the soil/solids preparation method (e.g., digestion for metals or distillation for cyanide). To facilitate comparison with the actual field samples, final results for the LCSs are calculated as mg/kg or  $\mu g/kg$ , assuming 100% solids and a weight equivalent to the aliquot used for the corresponding investigative samples. For organic analyses, the BS may consist of a representative selection of target analytes. The solid matrix used for non-volatiles is generally sodium sulfate.



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#### 9.2.1.3 Known QC Reference Samples

QC reference samples containing known analytes or compounds are obtained from outside suppliers or agencies. QC reference samples are obtained from the National Institute of Standards and Technology (NIST), state agencies, or commercial suppliers. These QC reference samples generally require preparation from concentrated materials by dilution into a standard matrix as instructed by the supplier. However, several suppliers provide fully constituted samples ready to analyze as received. Control limits are provided by the vendor, extrapolated from other in-house control data, or determined from control charts or method reference limits.

QC reference samples may be used to comply with regulatory requirements; to check the accuracy of an analytical procedure; to troubleshoot method performance problems; to verify an analyst in training's ability to accurately perform a method; and to verify the return-to-control after method performance problems. It is particularly applicable when a minor revision or adjustment has been made to an analytical procedure or instrument. It may also be used as an LCS.

## 9.2.2 <u>Matrix OC Indicators</u>

Matrix QC indicators include sample duplicates (DUP), sample matrix spikes (MS), and sample surrogate spikes. Matrix QC indicators help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix QC indicators measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratories avoid performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

## 9.2.2.1 <u>Matrix Spike (MS)</u>

A matrix spike (MS) is an aliquot of an investigative sample which is spiked with the analytes or compounds of interest. When requested by the client or the analytical method, an MS is analyzed for each associated sample type (e.g., soil, water, oils, etc.) and is used to monitor the effects of the investigative sample matrix on the accuracy of the selected analytical method. The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc.). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining percent recovery (% R).



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## 9.2.2.2 Duplicates

Laboratory duplicate samples (DUP) are performed by analyzing two aliquots of the same field sample independently and then performing an independent analysis. A DUP, when requested by the client or the analytical method, is analyzed for each associated sample type (e.g., soil, water, soil, etc.) and is used to monitor the matrix affects on the precision of the selected analytical method. Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of nonaqueous samples or aqueous samples with particulates. The DUP also provides a measure of the reproducibility of laboratory preparation and measurement techniques, but these steps are controlled by method QC indicators.

# 9.2.2.3 <u>Matrix Spike Duplicates</u>

A matrix spike duplicate (MSD) is an alternative to sample duplicates since it provides precision information. The MSD is preferable to a DUP if no target compounds are present in the sample. Generally, inorganic protocols specify an MS/DUP and organic protocols specify an MS/MSD. As with other matrix QC indicators, an MSD is analyzed when requested by the client or the analytical method.

## 9.2.2.4 <u>Surrogate Spikes</u>

Where required by the method, surrogates, compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each blank, blank spike and duplicate, matrix spike and duplicate, and sample, prior to preparation (e.g., extraction). Surrogates measure performance of the analysis in relation to the sample matrix. If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and QC samples is examined to determine the presence of matrix interference or the need for corrective action.

## 9.2.2.5 Internal Standards

Internal standards monitor the qualitative aspect of organic and inorganic analytical measurements. All internal standards are assessed after acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance. Internal standards are used to correct for matrix effects and to help troubleshoot variability in analytical response.



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## 9.2.2.6 <u>Matrix OC Frequencies</u>

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

For the Safe Drinking Water Act (SDWA) methods, a DUP is performed at a 10% frequency (1 per 10 samples) or one per preparation batch of 10 samples or less, whichever is more frequent.

For the Clean Water Act (CWA) 600 Series, MD/MSD or MS/DUP is performed at a 10% frequency (1 per 10 samples) or one per preparation batch of 10 samples or less, whichever is more frequent.

For EPA SW-846 methods, MS/MSD or MS/DUP is performed at a rate of 5% (1 per 20 samples) per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another clients sample within the same prep batch unless the Technical Profile indicates a client requirement for matrix QC. Matrix QC will only be reported to the client who owns the data.

For USEPA CLP Sample Delivery Groups (SDGs), MS/MSD or MS/DUP is performed at a rate of 5% (1 set per 20 samples), or one set per SDG per matrix, independent of the prep batch.

Matrix spikes (MS), matrix spike duplicates (MSD), and duplicates (DUP) may not be applicable to some analytical protocols because of the nature of the sample or protocol.

## 9.2.3 <u>Method Performance Indicators: Instrument Measurement</u>

Quality control indicators are used to ensure that optimum instrument performance is achieved. These indicators help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument QC indicators appropriate to each analytical technique are described in laboratory operating procedures for each respective method. A brief description of these checks is included in this section.

## 9.2.3.1 Initial Calibration Verification (ICV) (Inorganics)

The initial calibration verification is a calibration standard of known concentration prepared from a source other than that used for the calibration standards. The ICV is analyzed after the standard curve to confirm calibration.



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# 9.2.3.2 Initial Calibration Blank (ICB) (Inorganics)

The ICB is composed of blank water or solvent that is analyzed immediately after the ICV to confirm the calibration and to assure that any potential contamination is less than the reporting limit.

# 9.2.3.3 ICP Interference Check Samples (ICSA/ICSAB) (Inorganics)

ICP Interference Check Samples (ICSA/ICSAB) will be analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/ICSAB will be analyzed with the analytical sequence, before the final CCV/CCB. The ICSA/ICSAB are analyzed to verify the absence of spectral interferences.

Results for the ICP Interference Check Samples shall be within limits of 80-120% of the established mean value. If results for the ICSA/ICSAB do not fall within the control limit, the analysis will be terminated, the problem will be corrected, and the instrument will be recalibrated. If more than one ICSA/ICSAB was analyzed in an analytical sequence, any samples not bracketed by acceptable ICSA/ICSB will be reanalyzed.

Interferant elements for spectral interferences are not limited to Al, Ca, Fe, and Mg. Other elements may be added as needed and will be documented. The mean concentration is established by initially analyzing each lot of ICS Solution at least five times for the analytes of interest. The mean determination is performed during an analytical batch that meets all ICP QC specifications. Alternately, the ICSA/ICSB may be obtained from EPA or a commercial vendor with established mean values provided with the solution.

# 9.2.3.4 Detection Limit Verification Standard (Inorganics)

For Furnace AA, Flame AA, and Cold Vapor AA analysis, a standard at or near the detection limit is analyzed after the ICB to verify linearity near the reporting limit. Specific acceptance range criteria for the CRA are undefined by the EPA.

For ICP analysis, a standard at two times the reported detection limit (CRI) will be analyzed after the ICB to verify linearity near the reporting limit. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB. The CRI is not required for non-TAL metals Al, Ba, Ca, Fe, Mg, Na, or K. There are no established control limits for the CRA and CRI. In house acceptance criteria will be clarified in the individual laboratory OP.



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## 9.2.3.5 <u>Continuing Calibration Verification (CCV) (Inorganics)</u>

A continuing calibration standard (CCV) is a standard of known concentration, which may be made from a source other than that used for the standard curve. The CCV is analyzed for wet chemistry and metals to confirm that instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. The CCV is analyzed at a rate of 10%. For CLP metals, the CCV is analyzed at 10% or every two hours, whichever is more frequent.

## 9.2.3.6 <u>Continuing Calibration Blank (CCB) (Inorganics)</u>

The continuing calibration blank (CCB) is a reagent water blank used to confirm that the baseline has not drifted and to confirm that the blank is still reading less than the reporting limit. The CCB is analyzed at a rate of 10% (1 per 10 readings) for inorganics and at a rate of 10% (1 per 10 readings/injections) or every two hours, whichever is more frequent, for CLP metals.

## 9.2.3.7 Linear Range Analysis Standard (LRS) (Metals)

For ICP analysis, calibration is performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement. This is used to verify linearity and document the upper limit of the calibration range for each element. At least one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient equal to or greater than 0.995 in order to consider the responses linear over that range. All samples found to be above the ICP linear range are diluted and reanalyzed until the concentration falls within the instruments linear range.

## 9.2.3.8 Inter-Element Correction (IEC) (Metals)

For ICP analysis, correction factors for spectral interference due to Al, Ca, Fe, and Mg will be determined at least annually for all wavelengths used for each analyte reported by ICP, or any time the ICP is adjusted in any way that may affect the IEC's. Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed and documented with the ICP instrument records.

## 9.2.3.9 <u>GC/MS Tuning and Performance</u>

For GC/MS analysis, mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC 5311) as required to ensure correct mass assignment. At the



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beginning of the daily work shift, the GC/MS system is tuned with decafluorotriphenylphosphine (DFTPP) for semi-volatiles analysis, and 4-bromofluorobenzene (BFB) for volatiles analysis. Performance is further monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs). Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.

# 9.2.3.10 GC and HPLC Instrument Performance

For GC and HPLC analysis, calibration to target compounds will be as described in Section 10.2.6. Continuing Calibration Verification throughout the analytical sequence is accomplished through analysis of calibration check standards.

Instrument performance for chromatographic methods is monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for endrin or DDT). Throughout the analysis, blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.

# 9.2.4 <u>Method Performance OC Indicators: Analysis Batch</u>

Matrix specific QC indicators are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance QC indicators appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in this section.

These QC checks are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

## 9.2.4.1 <u>Serial Dilution</u>

For ICP metals, a dilution of an investigative sample is performed at the instrument to check for possible physical and/or chemical interferences. This sample is referred to as a serial dilution. For CLP, if the analyte concentration is at least fifty times greater than the IDL before dilution, the diluted value should agree within 10% of the original



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determination to demonstrate no interferences are present. For SW-846, if the analyte concentration is at least ten times or greater than the IDL before dilution, the diluted value should agree within 10% of the original determination to demonstrate no interferences are present. Field blanks may not be used for serial dilution analysis.

## 9.2.4.2 Analytical Bench Spike for Furnace AA

Analytical bench spikes are prepared at the instrument by fortifying the digestate with a known quantity of the analyte of interest. A bench spike is performed on each sample immediately following the unspiked original analysis, as required by the method. Specifics on preparation and acceptance criteria are provided in the applicable method protocols. Bench spikes are not required on samples that have been selected for matrix spikes.

## 9.2.4.3 <u>Method of Standard Additions</u>

Method of standard additions (MSA) is performed when specified by analytical protocol or by client request. The correlation coefficient of the MSA curve must be  $\geq 0.995$ . The sample concentration is defined as the x-intercept.

## 9.3 <u>Refrigerator Blanks</u>

Refrigerator storage blanks are placed in VOA sample storage coolers and are analyzed for full VOA TCL analytes by GC and/or GC/MS. The reporting limits are consistent with the GC and GC/MS sections. These blanks monitor VOA refrigerators to assure the absence of sample cross-contamination.

## 9.4 <u>Reagent Water Approval</u>

The laboratory's on-tap deionized water supply is similarly tested on a routine basis for pH and specific conductivity. Additionally, samples of the water supply are routinely collected and analyzed for metals.

## 9.5 Balances, Refrigerators

All analytical balances and reagent/standard storage refrigerators and freezers are monitored on routine working days. Sample storage areas, including walk-in coolers, are monitored twice daily on routine working days and once on weekends and holidays. These checks are recorded in the respective balance, refrigerator, or freezer log books.



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## 9.6 Instrument Time Check Verifications

An independent check of the GC/MS instrument time clocks is performed randomly. These checks are performed to assure that the instrument date and times reflect actual dates and times of analysis.

## 9.7 Blind QC Check Samples

WESTON participates in many blind QC Performance Evaluation Studies (PES). The laboratory participates in studies to monitor performance on a quarterly basis. All PES are analyzed by several methods to check performance for all analytical capabilities. Listed in Table 9-2 are the routine PES Programs and frequency in which the laboratories participate. The laboratorys' participation in many other agency- and client-specific programs and the results are available for review at each facility, unless client confidentiality was requested.

## 9.8 Routine Methods to Assess Precision and Accuracy

The QA objectives for precision and accuracy were provided and discussed in Section 8.0. This section details the necessary formulas for performing these calculations. All analytical data are reviewed relative to these criteria and specific project requirements to assess the quality of the analytical data. Where all criteria are met, data are deemed acceptable without qualification. Where precision and accuracy goals are not met, corrective action as described in Section 13.0 is taken. There are several factors which may influence the corrective action steps:

- Project-specific QA/QC requirements.
- Availability of sufficient sample for re-analysis.
- Holding time considerations.
- Regulatory action limits.
- Data quality objectives.

Refer to Table 9-1 for Precision and Accuracy Methods.

## 9.8.1 Precision

Precision is measured through analyses of replicate QC controls and investigative samples. Results from these measurements are calculated as relative percent difference (RPD) for inorganics or percent relative standard deviation (% RSD) for organics and evaluated according to the criteria set forth in Section 8.0. Laboratory QC control samples are used to demonstrate acceptable method performance and are used to trigger corrective action when control limits are exceeded.



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Precision is represented as the relative percent difference (RPD) between measurement of an analyte in duplicate samples or in duplicate spikes. RPD is defined as follows:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:

 $C_1$  = first measurement  $C_2$  = second measurement

The % RSD is calculated, expressing as a percentage, the standard deviation of the analytical results of the replicate determinations relative to the average of those results for a given analyte. This method of precision measurement can be expressed by the formula:

% RSD = <u>Standard Deviation</u> x 100 Mean

Where:

% RSD = percent relative standard deviation Std. Dev. =  $\int \sum (x_i - x(mean)^2 - n-1)^2$ 

Where:

x(mean)	=	concentration of analyte in the sample, and $(x + x + x)$ represents the sum of the concentration
		$(x_1 + x_2 +x_n)$ represents the sum of the concentration of each replicate
n	=	number of replicate analyses

9.8.2 <u>Accuracy</u>

Accuracy of analysis is represented by the percent recovery (%R) of various analytes added to samples and blanks. Percent recovery is defined as follows:

$$\%R = (\underline{A_{T} - A_{0}}) \times 100$$
$$\underline{A_{F}}$$



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Where:

 $A_{T}$  = Total amount recovered in fortified sample

- $A_0$  = Amount recovered in unfortified sample
- $A_F$  = Amount added to sample

Analytical accuracy is measured through the analysis of LCS/BS and MS/MSD samples. Results from these measurements are calculated as percent recovery. For metals and inorganic analysis, laboratory control samples are used to demonstrate acceptable method performance, and are used to trigger corrective action when control limits are exceeded. The accuracy of surrogate compound analysis is used similarly in organic analysis.

Spike recoveries on samples (MS/MSD) give an indication of sample's physical or chemical properties which may interfere with the identification and quantitation of analytes or compounds of interest. Sample homogeneity also becomes a factor in recovery determinations, as variable unspiked analytical results can affect the apparent analyte recovery.

Typically, the concentration of a spike is specified to meet requirements in laboratory operating practices or to meet project requirements. It may also be determined relative to background concentrations observed in the unfortified sample. In the latter case, the spiked concentration should be different enough (e.g., 2 to 5 times above the background concentration) to permit a reliable recovery calculation.

When the LCS % R exceeds the established acceptance limits, appropriate corrective action is taken (refer to Section 13.0). After the problem has been identified, corrected and system control has been re-established, sample analysis may continue. All data associated with the out-of control situation are evaluated with respect to project DQO's for useability, sample availability for re-analysis, etc. For rejected results, the samples will be re-prepared and/or re-analyzed after control has been re-established or action will be taken in conformance to client specifications. If data are used without re-analysis, the case narrative will address the deviation.

The % D is calculated by expressing as a percentage, the difference between the original value and new value relative to the original value. This method for precision measurement can be expressed by the formula:



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Percent Difference (% D) = 
$$\frac{|C_1 - C_2|}{C_1} \times 100$$

Where:

 $C_1 =$  concentration of analyte in the initial aliquot of the sample  $C_2 =$  concentration of analyte in replicate

## 9.8.3 <u>Representativeness and Comparability</u>

Sample handling procedures and analytical techniques are developed and implemented to help ensure that analytical results are representative of the matrix and conditions of the sample being measured. The data are calculated and reported in units consistent with standard reporting conventions to allow comparability with existing data, standards, and regulatory action limits. Project representativeness and comparability are dependent on site conditions and the sampling plan which addresses these conditions. Site conditions and sampling plans are generally the engineering/field contractor's responsibility and are usually prepared on a project specific basis. Therefore, representativeness and comparability are not addressed in this laboratory plan. Assessment of site and collection representativeness and comparability is performed by the field engineer.

#### 9.9 <u>Quality Control Limits and Charts</u>

Control limits and control charts are used in the laboratory to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory's results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

#### 9.9.1 Establishment of Limits

The purpose of using control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate QC indicators in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:



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## 9.9.1.1 <u>Accuracy</u>

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery  $(\bar{x})$  is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. In other words, only 3 results in 1,000 should fall outside the region  $\bar{x} \pm 3s$ . Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and is thus out of statistical control.

Likewise, roughly 95% of points should fall within 2s of the mean. The values  $\bar{x}+2s$  and  $\bar{x}-2s$  are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

## 9.9.1.2 Precision

Because important legal, financial, and environmental decisions may be based on any single analytical result, replicates are analyzed to provide reassurance that repeated measurements of a particular sample would produce the same result -- or at least results that don't vary significantly from one another. Duplicates would also indicate matrix variability so that appropriate decisions can be made when repeated analyses do vary significantly. Duplicate analyses of QC control samples, such as LCS and BS, are evaluated for consistency by tabulating the RPD over time. This establishes a range of expected precision between duplicate measurements.

Again, assuming that variation is random, and that an acceptable mean range  $(1.128 \times standard deviation)$  has been established for a measurement (e.g., duplicate LCS pairs for Pb in water by GFAA) the Control Limit for precision is 3.267 x mean range and the Warning Limit is 2.512 x mean range. The acceptable range is 0 to 3.267 (mean range), as "0" would indicate perfect agreement between measurements.

## 9.9.2 LIMS Programs

A custom LIMS QC charting system has been developed for the laboratory to automate the QC control limit and charting processes. This unique system blends the use of database and graphics functions.



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Laboratory control charts on LIMS are produced by four major application programs, which are built around two databases. The two databases that hold the information needed for the laboratory control charts are LM (Lab Method), and LQ (Lab QC Data). Each method constitutes a record in the LM database that will lead to the generation of one chart. The QC data points are extracted from the Analysis and Result data sets and are loaded into the LQ data set. This is done so that each record in LQ is associated with a certain method in LM. Both the LQ and LM data sets are then used by the various control chart programs. The details of producing QC charts using this LIMS system are detailed in WESTON OP 21-11-108 for routine laboratory charts.

9.9.3 <u>Control Limits</u>

## 9.9.3.1 Source of Limits

Analysts enter data for QC samples in LIMS along with sample data for each analytical batch via direct download from the instrument or LOTUS spreadsheet. The following identifying data are entered for each QC sample:

- RFW (sample) number
- Preparation date, e.g. extraction/digestion
- Preparation batch number if applicable, e.g. extraction/digestion
- Analytical date
- Analytical batch identifier (as applicable)
- QC qualifier (specifying MS, LCS, etc.)
- Replicate number (if applicable)
- LIMS Test Code
- Matrix
- Spike level (constant for method/matrix/QC analyte unless changed by a Supervisor)
- Instrument ID
- Analyst

## 9.9.3.2 <u>Accuracy</u>

Calculation of updated limits for a given accuracy QC parameter involves a search by LIMS through the LQ database for all points in the date range set by the requestor. Currently, LIMS is set to override the query range if it contains <40 data points and to add successively earlier data points until a data set of 40 points, if available, is obtained.



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LIMS automatically performs a Dixon Outlier Test (DOT) to identify statistical outliers in the data set. These are excluded by LIMS from the data set and from calculations of control limits.

The LIMS system calculates the mean of the data set, the sample standard deviation  $(s_{n-1})$ , and the values corresponding to +3s, +2s, -2s, and -3s about the mean.

## 9.9.3.3 <u>Precision</u>

LIMS performs a similar compilation of data points in response to a date-range request for calculation of RPD data. In this case, each of the 40-plus data points is the RPD of a pair of duplicate analyses, e.g., for duplicate LCS's for Ba in water by ICP. The Dixon Outlier Test is performed on the RPD data set before calculation of limits.

The LIMS system calculates the mean range, standard deviation and the values corresponding to 3.267 x mean range (control limit) and 2.512 x mean range (warning limit).

## 9.9.4 <u>Output</u>

With the calculation of new limits, LIMS produces a method/matrix-specific spreadsheet that provides the Upper and Lower Control and Warning Limits for each analyte. The summary spreadsheets are distributed to Unit Leaders and Section Managers and provide a ready reference for implementing control and warning limits. Additionally, LIMS produces, for each analyte, a complete tabulation of the data found in the aforementioned search, including the data used for calculation of the limits and those rejected by the Dixon Outlier Test.

## 9.9.5 <u>Update Control Limit Cycle</u>

QC control limits are updated annually and are effective within three months to allow sufficient implementation time, e.g., update of LIMS databases, calculation of new limits, review of the limits for reasonableness, and distribution and training on the new limits.

More frequent update of limits may occur on a case-by-case basis, with the approval of the QA Section Manager. Such updating may be required to accommodate a new method, to address a shift in method performance, or to allow for the availability of additional data for a specific method. At the beginning of the following year, these limits will be brought into the annual calculation schedule.



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#### 9.9.6 <u>Evaluation of Limits</u>

Summary sheets of updated limits are circulated to the QA Section and Technical Managers, the Laboratory Manager, and the appropriate Section Manager by the QA Technician or Specialist who performs the update. The limits are reviewed with corresponding method-specified limits. Summary spreadsheets are also circulated to Project Managers after technical review and correction for their use in complying with project specifications.

Empirical limits should be "better than" method-specified limits, but such evaluation often requires judgement. For example, SW846 Method 8270 acceptance limits for the surrogate Phenol- $d_5$  in water samples are 10-94%, demonstrating a range of 84%. If the laboratory's range from empirical limits is the same but the acceptance limits are distributed around a mean value closer to the true value (e.g., 25-109%), the laboratory may use its own empirical limits.

Trends or patterns should be identified as close to real time as possible. However, actual trends may not be identified real-time since the time between sample preparation and analysis is not immediate (e.g., 40 days for extractable organics). Also, samples may not be analyzed in the order of extraction, creating additional complexity for evaluating QC charting trends. With this complexity understood, the laboratories review for trends as described further in this section.

## 9.9.7 <u>Unscheduled Updates of Control Limits</u>

It is the responsibility of the Section Managers or designee to review control charts for the analyses they supervise each quarter and to evaluate the charts for the presence of trends, cycles, changes in accuracy (consistent above- or below-mean recoveries), or changes in precision. The charts should also be reviewed by the respective QA Specialist, QA Section, and Technical Managers. Trends and cycles should be investigated by the Section Managers for their sources, and a Corrective Action Report should be filed if a problem is found, e.g., declining LCS recovery is linked with a particular instrument, so that resolution can be documented. If consistently high or low recoveries are noted, or there seems to be tighter or looser precision than indicated by the annual control limits, the Section Manager is to notify the QA Section Manager, who will determine whether there is a statistical basis for changing the respective control limits.

It is important to retain an accessible record of what control limits were used to evaluate a given set of data. Each time control limits for a QC parameter are changed, it will be documented. This applies to annual updates of limits and to unscheduled updates.



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## TABLE 9-1

# **Precision and Accuracy Methods**

QC Sample	Purpose	Conc. Level	Method Reference
Matrix Duplicates	Laboratory Precision Sample Homogeneity	Low/Medium	Soil/Water methods for Metals/Wet Chemistry
Matrix Spikes	Laboratory Accuracy Sample Homogeneity Matrix Effects	Low/Medium	All soil and water matrices
Matrix Spike Duplicates	Accuracy/Precision Sample Homogeneity Matrix Effects	Low/Medium	Soil/water methods for Organics
Laboratory Control Samples	Accuracy/Precision	Low/Medium	Soil/water methods for Metals and Wet Chemistry
Blank Spikes and Blank Spike Duplicates	Accuracy/Precision	Low/Medium	Soil/water methods for Organics
Surrogates	Matrix Effects	Low/Medium	Soil/water methods for Organics



## ANALYTICS DIVISION STANDARD PRACTICES MANUAL COMPANY CONFIDENTIAL AND PROPRIETARY

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#### TABLE 9-2

## Routine Performance Evaluation Sample Programs Analytics Division

Program	Frequency	
USEPA Water Pollution (WP)	Semi-Annually	
USEPA Water Supply (WS)	Semi-Annually	
New York DOH Non-Potable Water	Semi-Annually	
New York DOH Potable Water	Semi-Annually	1
Analytical Products Group (APG)	Quarterly	



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## 10.0 CALIBRATION PROCEDURES AND FREQUENCY

## 10.1 <u>Standard Receipt and Traceability</u>

All standards and reagents used throughout the laboratory must have sufficient information to allow traceability of any solution or standard back to the parent lot number from which the solution came. Records are maintained that document the dates of preparation and expiration of all quality control chemicals and solutions. All standards are traceable to National Institute of Standards and Technology (NIST), American Association for Laboratory Accreditation (A2LA), Standard Analytical Reference Material (SARM), or other equivalent certified material sources as available.

The documentation for standards traceability is achieved through the use of Standard Preparation Logbooks. Documentation contained within the logbooks is detailed in each laboratories Operating Practices (OP).

## 10.1.1 <u>Inorganic Solutions</u>

The preparation of all inorganic stock solutions and intermediate solutions is documented in the Stock Standard Solution Preparation Log. The standard name, final volume, matrix, preservative, final concentration, analyst initials, prep date and expiration date are recorded. The prepared stock solution is given a new Source ID number based on the logbook number, page number, and line number that the solution is recorded in the Stock Standard Prep Log. Source ID numbers may also be documented in the standard traceability section of the formatted laboratory data book.

The initial calibration verification stock solution is from an alternate stock solution than the stock calibration standard solution. The continuing calibration verification stock solution may be either the same or alternate stock from the stock calibration solution. The two stock solutions, whether purchased or prepared, are from different lot numbered chemicals, preferably from different manufacturers. In rare cases where different lot numbers are not commercially available, the two stock solutions are prepared by different analysts.

## 10.1.2 Organic Solutions

The preparation of organic standards is similar to the inorganic format with the additional documentation of the compound/kit, % purity and weight/volume. Daily solutions are dilutions of the stock or the intermediate solutions. Procedural OPs provide instructions for daily solutions.

Each laboratory maintains detailed Operating Practices for Standards Traceability.



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## 10.2 Instrument Calibration

All instruments must be calibrated prior to use as a measurement device to establish the instrumental response to known reference materials. The manner in which various instruments are calibrated is dependent on the particular type of instrument, the method of analysis, and the intended use of the data. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted analysis logbooks. For GC/MS, the instrumental conditions and calibration are located in the method file in the computer database.

The pre-formatted logbooks provide a template to ensure appropriate data are documented in a retrievable format. Preparation of all reference materials used for calibration will be documented in a standards preparation notebook. The calibration standards are given a unique identification number based on the standards preparation book number, page number, and line number. This unique ID number is documented in the analytical raw data and/or run logs to trace the standard to a given calibration.

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

Continuing calibration usually includes measurement of the instrument response to fewer calibration standards and requires instrument response to compare within certain limits (e.g.  $\pm 10\%$ ) of the initial measured instrument response. Continuing calibration may be used within an analytical sequence to verify stable calibration throughout the sequence, and/or to demonstrate that instrument response did not drift during a period of non-use of the instrument.

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Further details are in the respective analytical methods and OPs.



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#### 10.2.1 <u>Metals by Atomic Absorption (AA) Spectroscopy: Graphite Furnace</u> (GFAA), Flame AA, and Cold Vapor AA

Atomic absorption spectrophotometers will be calibrated prior to each day of use. Calibration standards will be from appropriate reference materials, and working calibration standards will be prepared fresh daily. Calibration data are recorded and archived with the respective sample data. Lamps and flame/furnace conditions are optimized for maximum response prior to any analytical measurements.

## 10.2.1.1 <u>AA: Initial Calibration</u>

Initial calibration will include analysis of a calibration blank and a minimum of four (4) calibration standards covering the anticipated range of measurement. Duplicate injections are made for each concentration. Response readings, e.g., absorbance, are recorded and the resultant standard calibration curve calculated. If the OP-specified criteria are not met, the instrument is recalibrated prior to analysis of samples.

An initial calibration verification (ICV) standard will be analyzed immediately after standardization. The ICV must be within OP-specified (e.g., ten percent of the true value except for mercury within twenty percent of the true value), or the initial calibration must be repeated. The ICV must be from a source other than that used for initial calibration.

An initial calibration blank (ICB) will be analyzed after the ICV. The ICB must be free of target analytes at and above the reporting limit, or the initial calibration must be repeated.

A standard at the reported detection limit (CRA) will be analyzed after the ICB to verify linearity near the reporting limit for AA analysis. The CRA must indicate a positive recovery for the metal of interest. The CRA is not applicable to mercury.

## 10.2.1.2 AA: Continuing Calibration

The initial calibration is verified during the analysis sequence by evaluation of a continuing calibration verification (CCV) standard and a continuing calibration verification blank (CCB) after every ten (10) samples are analyzed. The CCV value must be within OP-specified criteria (e.g., ten percent recovery of the true value except for mercury within  $\pm$  twenty percent recovery of the true value). The CCB must be free of target analytes at and above the reported detection limit.



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If any initial/continuing calibration verifications or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed. During auto-sampler runs, sample results must be bracketed by acceptable calibration QC.

# 10.2.2 Metals by Inductively Coupled Argon Plasma (ICP)

Following a period of time sufficient to warm up the instrument, the ICP will be calibrated prior to each analytical run or minimally every 24 hours. Calibration standards will be prepared from reliable reference materials and contain all metals for which analyses are being conducted. Triplicate readings will be made for each QC and investigative sample, and the average value used. If the standard deviation of the three readings is greater than 20%, and one of the three burns appears to be in error (e.g., from poor sample injection), duplicate readings may be used. All reported results are based on at least two readings. If the standard deviation criteria still do not meet, the analysis must be repeated. Working calibration standards are prepared fresh daily. Calibration data is recorded and archived with the respective sample data.

## 10.2.2.1 ICP: Initial Calibration

Quarterly, multi-concentration calibration is performed as described in Section 9.2.3 to document linearity. On a day-to-day basis, a single standard is analyzed for initial calibration as described below.

Prior to an analytical run, the instrument is calibrated using a standard near the high end of the calibration range and a blank. An initial calibration verification (ICV) standard will be analyzed immediately after standardization, followed by an initial calibration blank (ICB). The ICV must be from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the reporting limit, or the initial calibration must be repeated.

A standard at two times the reporting limit (CRI) will be analyzed as described in Section 9.2.3.4 to verify linearity near the reporting limit for ICP analysis.

ICP Interference Check Samples (ICSA/ICSAB) will be analyzed at the frequency described in Section 9.2.3.3.

## 10.2.2.2 ICP: Continuing Calibration

The initial calibration is verified during the analysis sequence by analysis of a continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) as



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described in Sections 9.2.3.5 and 9.2.3.6. The response of the continuing calibration verification standard must be within OP-specified criteria (e.g.,  $\pm$  ten percent recovery of the true value). The continuing calibration blank must be free of target analytes at and above the reporting limit.

If any initial/continuing calibration verifications or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed.

- 10.2.3 Inorganic Colorimetric Methods
- 10.2.3.1 Colorimetric: Initial Calibration

A full initial standard calibration curve will be prepared for all colorimetric analyses on a daily basis. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards is at the desired reporting limit. The requirement for an acceptable initial calibration will be described in the related OP. If the criteria are not met, the instrument is recalibrated prior to analysis of samples. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook with the sample data to maintain a permanent record of instrument calibrations.

On a daily basis, verification of the full initial curve can be accomplished by analysis of a mid-range and high range standard. Results must be within OP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed.

For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within OP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment.

An initial calibration verification (ICV) standard will be analyzed immediately after the standardization, followed by an initial calibration blank (ICB). The ICV must be from a source other than that used for initial calibration. The ICV must be within OP-specified criteria and the ICB must be free of target analytes at and above the reporting limit, or the initial calibration must be repeated.



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# 10.2.3.2 <u>Colorimetric: Continuing Calibration</u>

The initial calibration is verified during the analysis sequence by analysis of a continuing calibration blank (CCB) and a continuing calibration verification standard (CCV) after every ten readings are analyzed. The response of the continuing calibration verification standard must be within OP-specified. The continuing calibration blank must be free of target analytes at and above the reported detection limit.

If any initial/continuing calibration verifications or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed.

10.2.4 <u>Total Organic Carbon (TOC)</u>

# 10.2.4.1 <u>TOC: Initial Calibration</u>

The total organic carbon analyzer will be calibrated prior to each day of use. Calibration will include a blank and a minimum of five (5) concentrations to cover the anticipated range of measurement. At least one of the calibration standards will be at the desired reporting limit. If the OP-specified acceptance criteria cannot be achieved, the instrument is recalibrated prior to analysis of samples. Calibration data, e.g., correlation coefficient, is entered into laboratory notebooks to maintain a permanent record of instrument calibrations.

An initial calibration verification (ICV) standard is analyzed immediately after standardization, followed by an initial calibration blank (ICB). The ICV must be from a source other than that used for initial calibration. The ICV must be within OP-specified criteria or the initial calibration must be repeated. The ICB must be free of target analytes at and above the reporting limit, or the initial calibration must be repeated.

# 10.2.4.2 <u>TOC: Continuing Calibration</u>

The initial calibration is verified during the analysis sequence by analysis of a continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) after every ten readings samples are analyzed. The response of the continuing calibration verification standard must be within OP-specified criteria. The continuing calibration blank must be free of target analytes at and above the reported detection limit.

If any initial/continuing calibration verifications or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed.



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#### 10.2.5 Gas Chromatography/Mass Spectrometry (GC/MS)

All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.

## 10.2.5.1 <u>Tuning and GC/MS Mass Calibration</u>

Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluoro-triphenylphosphine (DFTPP) for semi-volatiles analysis and 4bromofluorobenzene (BFB) for volatiles analysis.

The majority of WESTON's work utilizes EPA-CLP or SW-846 protocols, which define the work shift as a 12-hour period initiated by the injection of DFTPP, BFB, or the dioxin/furan window mix. For drinking water programs (500 series methods), an 8-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift. Ion abundances will be within the windows dictated by the specific program requirements.

## 10.2.5.2 <u>GC/MS: Initial Calibration</u>

After an instrument has been tuned, initial calibration curves (generally 3-5 points) are generated for the compounds of interest. Instrument response to these target compounds are evaluated against OP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against OP-specified criteria.

Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multipoint calibration if the OP-specified criteria are met. Otherwise, a multi-point curve must be re-established.

The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PP). For add-on compounds not on the current TCL or PP lists, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration.



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Calibration data, to include linearity verification, will be maintained in the laboratory's permanent records of instrument calibrations.

## 10.2.5.3 GC/MS: Continuing Calibration

During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific OPs. If criteria cannot be met, an acceptable multi-point initial calibration must be re-established.

## 10.2.6 <u>Gas Chromatography (GC) and High Performance Liquid Chromatography</u> (HPLC)

Gas chromatographs and high performance liquid chromatographs will be calibrated prior to each day of use. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis.

## 10.2.6.1 <u>GC & HPLC: Initial Calibration</u>

Initial calibration will include at least five calibration standards covering the anticipated range of measurement. The low level standard must be at or near a concentration which is equivalent to the reporting limits for the method. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet OP-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards.

## 10.2.6.2 <u>GC & HPLC: Continuing Calibration</u>

The response of the instrument will be verified for each analysis sequence by evaluation of a mid-range calibration check standard. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within OP-specified acceptance criteria for the compounds of interest or the instrument must be recalibrated. For multianalyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration (e.g., the full range of concentration levels) can be performed at the beginning of the analysis sequence.

Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every ten samples, including external QC. If the OP-specified calibration criteria are not met for the compounds of interest, appropriate corrective actions will be taken to restore confidence in the instrumental measurements.



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## 10.2.6.3 Ion Chromatography (IC)

#### 10.2.6.3.1 <u>IC Initial Calibration</u>

The ion chromatograph will be calibrated prior to each day of use. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at or below the desired instrument reporting limit. If OP-specified calibration criteria cannot be achieved, the instrument will be recalibrated prior to analysis of samples.

Calibration data, e.g., correlation coefficient, will be archived with sample raw data to maintain a permanent record of instrument calibrations.

## 10.2.6.3.2 IC: Continuing Calibration

A continuing calibration standard and blank will be analyzed at a frequency of ten percent (10%) and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet OP-specified criteria. The response of the blank must be less than the reporting limit.

## 10.2.7 <u>Total Organic Halogen (TOX)</u>

## 10.2.7.1 TOX: Initial Calibration

Duplicate instrument calibration standards (ICS) and blank standards are pyrolyzed each day before beginning sample analysis. Quantitation must meet OP-specified acceptance criteria prior to sample analysis.

## 10.2.7.2 TOX: Continuing Calibration

Calibration is verified by analyzing an ICS and a blank after every eight pyrolysis determinations. If OP-specified calibration criteria are not met, appropriate corrective actions will be taken to restore confidence in the instrumental measurements.

#### 10.3 Balances

Laboratory balances are calibrated and serviced at least annually by a factory representative. In addition, the balances are checked daily (each workday in which the balance is utilized) with a minimum of two masses: both in the gram range for top loading balances, and one



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each in the gram and milligram range for analytical balances. A record of calibrations and daily checks will be kept in the balance log.

Weights used by the analysts for daily balance checks are Class S weights or Class P weights calibrated against Class S reference weights. All S weights are certified and calibrated annually. All S and P weights that are located throughout the laboratory are calibrated annually against the certified S weights.

## 10.4 <u>Thermometers</u>

Oven and refrigerator/freezer thermometers are calibrated against a reference thermometer that is traceable to a National Institute of Standards and Technology (NIST) certified thermometer. The thermometer is graduated in at least 0.1°C increments. The specific procedures used for calibrating thermometers for the particular use is detailed in Gulf Coast Laboratory OP 21-06G-0003, Lionville Laboratory OP 21-06L-113, and Stockton Laboratory OP 21-06S-101.

The thermometers are labelled with an ID code, correction factor, and calibration date. This same information is documented and stored in the QA office in a Temperature Calibration Logbook. Mercury thermometers are recalibrated once a year. Digital or dial thermometers which are used to monitor temperature control are calibrated quarterly.

Extra calibrated thermometers are kept on hand in case of breakage or general laboratory use.

New thermometers are calibrated in the range in which they are to be used.

## 10.5 <u>Temperature Assurance</u>

A system for routine monitoring of refrigerators, freezers, and ovens is documented in Gulf Coast Laboratory OP 21-06G-0025, Lionville Laboratory OPs 21-06L-100, and Stockton Laboratory OP 21-06S-100.

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## 11.0 DATA REDUCTION AND REPORTING

Analytical data are recorded on pre-formatted bench sheets or analysis run logs in bound laboratory notebooks. These bound notebooks are issued and controlled by the laboratory's Quality Assurance Section. A unique document control code is assigned each book to assure that chronological record keeping is maintained.

Analytical data is referenced to a unique sample identification number (RFW #) for internal tracking and reporting. Notebook pages contain the following information, as applicable: analytical method, analyst, date, logbook number, sequential page number, associated RFW sample numbers, standard concentrations, instrument settings, and raw data. Entries for instrument logs are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The laboratory analysts sign and date all analytical book entries daily. The notebook pages are reviewed by a trained data reviewer. Copies of instrument outputs (chromatograms, strip charts, etc.) are maintained on file with the analyst's signature/initials and date.

## 11.1 Data Reduction

Data reduction is performed by the analyst and consists of calculating concentrations in samples from the raw data. The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values. All computer programs used to calculate results are validated by the IS Group. Copies of all raw data and the calculations used to generate the final results, such as bound laboratory notebooks, strip-charts, chromatograms, LOTUS spreadsheets and LIMS record files, are retained on file for a minimum of 6 years.

## 11.1.1 Significant Figures

All reported analytical values should contain only figures (or digits) which are known to be reasonably reliable, e.g., only significant figures. Significant figures consist of all digits that are definitely known and one last digit which is estimated. For example, if a value is reported as 18.8 mg/L, the 18 must be certain while the 0.8 is somewhat uncertain, but presumably a better estimate than a value of 0.7 or 0.9.

The number zero (0) may or may not be a significant figure depending on the situation. If it represents a measured quantity, it is a significant figure. If it merely locates the



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decimal point, it is not a significant figure. The following examples are used by the laboratory for CLP protocol.

- Zero within a number. A zero between two non-zero digits is always significant.
- Zero at the beginning of a number. With no preceding non-zero digits, a zero before the decimal point is not significant. If there are no non-zero digits preceding a decimal point, the zeros after the decimal point, but preceding other non-zero digits are not significant. These zeros only indicate the position of the decimal point.
- Zero at the end of a number. Final zeros to the right of a decimal point, if any nonzero digit precedes the decimal point, are always meant to be significant figures. Final zeros in a whole number may or may not be significant.

Significant figures reflect the limits in accuracy (reliability) of the particular method of analysis. At the project planning stage, method selection should include an evaluation of the number of significant digits of reported results. The resulting values for the analytical technique should be verified as sufficient for interpretation purposes before samples are received. If more significant figures are needed, a further improvement in method or selection of another method may be required. Once the number of significant figures is established, data resulting from such analyses are reduced, and reported according to scientific rounding rules.

Reporting to appropriate significant figures may be waived in instances where client report formats (e.g., CLP forms, some electronic data deliverables) define the number of digits to be reported. These data should be properly rounded when reported in WESTON format.

## 11.1.2 <u>Rounding Off Numbers</u>

Rounding off numbers is a necessary operation to account for the limits of measure of instrumentation and associated preparation steps. Results are rounded by dropping digits that are not significant, using scientific rounding rules:

- If the figures following those to be retained is less than 5, the figure is dropped and the retained figures are kept unchanged.
- If the figure following those to be retained is greater than 5, the figure is dropped and the last retained figure is raised by 1.



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#### ANALYTICS DIVISION STANDARD PRACTICES MANUAL COMPANY CONFIDENTIAL AND PROPRIETARY

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- If the figures following those to be retained is 5 and if there are no figures other than zeros beyond the 5, the figure 5 is dropped and the last place figure retained is increased by 1 if it is an odd number, and it is kept unchanged if it is an even number. Laboratory MDLs and CLP IDLs are always rounded up.
- When an arithmetic operation with a series of numbers is performed, the result should be rounded off to the same number of decimal places as the figure with the smallest number of places after the operation is completed with all decimal places intact or with the exceptions noted above.

#### 11.1.3 <u>Gravimetric Procedures</u>

Data reduction procedures for gravimetric analyses are detailed in the respective analytical OPs. These methods include solids, residue, oil and grease determinations. In general, the following calculation is used.

 $\frac{A (mg) - B (mg)}{C (mg \text{ or } mL)} \times 1000 \text{ mg/kg or } mg/L = R (mg/kg \text{ or } mg/L)$ 

Where:

- A = Gross weight of sample with bottle weight
- B = Tare weight of bottle
- C = Size of sample being analyzed
- R = Result

Note: All analytical calculations are based on samples weighed at room temperature.

#### 11.1.4 <u>Colorimetric Procedures</u>

Data reduction procedures for colorimetric analyses are detailed in the respective analytical methods. In general, a standard calibration curve is used to correlate the concentration vs. absorbance readings of known standards. The calibration curve is derived from a least squares fit (e.g., linear regression) from which the slope, y-intercept, and correlation coefficient are determined. The sample response (e.g., absorbance or % transmittance) is compared to the standard curve to obtain an initial raw concentration. This concentration is then factored into the final equation to quantitate the concentration in the original sample. Standards are generally expressed as weight per volume of the parameter of interest. The final calculation is as follows:



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$$\frac{A \times B \times D}{C} = R (mg/kg \text{ or } mg/L)$$

Where:

Α	8	Concentration obtained from the instrument response calculated by least squares regression.
B	_	Final volume of sample preparation
С	=	Initial weight or volume of sample
D	=	Dilution (equals 1 if no dilution)
R	=	Result

## 11.1.5 <u>Titrimetric Procedures</u>

Data reduction procedures for titrimetric analyses are detailed in the respective analytical methods. In general, the following calculation is used.

<u>mL Titrant x Normality of Titrant x Factor</u> = R Sample Size (mL or g)

Where:

R = Result in mg/kg or mg/L

The factor is dependent upon the type of analysis and will generally incorporate gram molecular weight, equivalent, or other formula related information. Refer directly to referenced procedures for specific factors. The following are examples of factors typical used in titrimetric analyses, as noted in laboratory OPs:

Alkalinity	50,000
Total Kjeldahl Nitrogen	14,010
Nitrogen Ammonia	14,010
Chloride	35,450
Sulfite	40,000

11.1.6 Direct Reading Instruments: e.g., ICP, GFAA, Cold Vapor Hg, TOC, TOX, Specific Conductance

ICP, GFAA, Cold Vapor Hg, TOC and TOX results are results obtained from a direct instrument reading. Standards of a known concentration are analyzed and an instrument

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establishes a calibration curve. The instruments typically allow any dilution factor to be entered into the instruments program prior to analysis.

If sample preparation was required prior to analysis (e.g., digestion), the reading is then related to the original sample matrix using the following calculation:

$$\frac{A \times B}{C} = R (mg/L \text{ or } mg/kg)$$

Where:

- A = Direct instrument reading (includes dilution factor)
- B = Final volume of sample being analyzed
- C = Initial weight or volume of samples
- R = Result

Specific Conductance results are directly read from an instrument which automatically corrects for temperature compensation. A cell constant is determined by rinsing the conductivity cell with at least three portions of 0.01 N KCl Solution, then adjusting the temperature of the fourth portion to  $25.0 \pm 0.1^{\circ}$ C. The resistance of the fourth portion is measured and temperature is noted. The cell constant is calculated as follows:

 $C = (0.001413)(R_{KC}) [1 + 0.019(t - 25)]$ 

Where:

 $R_{KCI}$  = measured resistance, Ohms t = observed temperature, °C

11.1.7 Instruments with Strip Chart Output: e.g., Flame AA, Cold Vapor Hg, Auto Analyzer Methods, etc.

A standard calibration curve is set up for each analysis, and strip chart measurements by peak height are recorded. The peak heights of the samples are compared to the standard curve and a value is calculated utilizing a least squares regression. The sample results are then corrected for dilution. Standards may be expressed on a weight basis (e.g.,  $\mu$ g), or may be expressed on a weight/volume basis (e.g., mg/L). Therefore, strip chart measurements by peak height are calculated by one of two methods:



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11.1.7.1 Method used when Standards are expressed on a Weight basis (e.g., µg)

 $\frac{A \times D}{\text{Sample Size (g or mL)}} = R (mg/L \text{ or mg/kg})$ 

Where:

- A = Value in  $\mu g$  obtained from Least Squares Fit calculation from computer/calculator
- D = Dilution Factor

R = Result

## 11.1.7.2 Method used when Standards are expressed on a Weight/Volume basis (mg/L)

 $(A \times B \times D)/C = R (mg/L \text{ or } mg/kg)$ 

Where:

- A = Value in mg/L obtained from the Least Squares Fit calculation from computer/calculator.
- B = Final volume of sample preparation.
- C = Initial weight or volume of sample
- D = Dilution Factor
- R = Result

## 11.1.8 HPLC and Gas Chromatography

Chromatography protocols specify data reduction by either a single-level calibration procedure, or a multi-level calibration procedure. The method selected is determined in the laboratory operating practice. However, client specific protocols may be developed to meet the clients data quality objectives and project needs.

## 11.1.8.1 Single-Level Calibration

For protocols requiring a single-level calibration procedure, instrument/detector linearity is first demonstrated in accordance with the OP. The concentration in the sample can be calculated using the following equations for external standards. Response can be measure by the manual peak height technique or by automated peak height or peak area measurements from a data system.



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Water Samples

Concentration 
$$(\mu g/L) = \frac{(A_z)(L)(V_z)(F)}{(A_z)(V_z)(V_z)(1 L/1000 mL)}$$

Where:

 $A_x$  = Response for the parameter to be measured.

 $A_{r}$  = Response for the external standard.

 $V_t = Volume of total extract (mL)$ 

 $I_{a}$  = Amount of standard injected in nanograms (ng)

 $V_i$  = Volume of extract injected (mL)

 $V_s = Volume of water extracted (mL)$ 

F = Dilution Factor

## Sediment/Soil Samples

Concentration  $(\mu g/kg) = (A_*)(I_*)(V_*)(F)(S)$ (Dry weight basis)  $(A_*)(V_i)(W_*)(D)(1 \text{ kg/1000 g})$ 

Where:  $A_{p}$ ,  $I_{p}$ ,  $A_{p}$ ,  $V_{i}$ , V = same as given above for water  $V_{t} =$  Volume of total extract (mL)  $W_{s} =$  Weight of sample extracted (g) S = Split multiplier (S=1 if no GPC cleanup, S=2 if GPC cleanup employed).  $D = \frac{100 - \% \text{ moisture}}{100}$ 

## 11.1.8.2 Multi-Level Calibration

For chromatography methods requiring a multi-level calibration procedure, the calculation of the final concentration of identified parameters can be performed using the following equations:

Water Samples

Concentration 
$$(\mu g/L) = (C_*)(V_*)(F)$$
  
 $(V_*)(1 L/1000 mL)$ 



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Where:

 $C_r = Extract$  concentration of parameter x ( $\mu g/mL$ ) determined by linear regression calculation from the multi-point calibration curve.

 $V_t = Volume of total extract (mL)$ 

F = Dilution factor

 $V_s = Volume of water extracted (mL)$ 

Sediment/Soil Samples

Concentration 
$$(\mu g/kg) = \frac{(C_*)(V_*)(F)(S)}{(W_*)(D)(1 \text{ kg}/1000 \text{ g})}$$

Where:

 $C_{p} V_{p} F$  = Same as given above for water S = Split multiplier (S = 1 if no GPC cleanup, S = 2 if GPC cleanup is employed)  $W_{s}$  = Weight of sample extracted (g) D = 100 - (% moisture content of sample)/100

## 11.1.9 GC/MS. Internal Standard Method

The GC/MS data system and the LIMS data system perform quantitation of target compounds as well as tentatively identified compounds (TICs) for water and soils. In general, up to as many as 10 TICs are identified for semi-volatiles, and up to as many as 10 TICs are identified for volatiles. However, for routine analysis up to 5 TIC's per fraction are searched.

GC/MS data reduction and review also involves operator professional judgements concerning the correctness of the identification of the target compounds and tentatively identified compounds. The final report will reflect these judgements.

11.1.9.1 <u>Target Compounds for Water</u>  $\frac{(A_{\bullet})C_{\pm})(V_{\bullet})(F)}{(A_{\pm})(RF)(V_{\bullet})(V_{i})} = R (\mu g/L)$ Where:  $A_{x} = Area of characteristic ion for compound of interest$   $C_{is} = Amount of internal standard injected (ng)$   $V_{t} = Volume of total extract (\mu L).$  This includes a measured final volume.



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- $A_{i}$  = Area of characteristic ion of internal standard
- RF = Response Factor<sup>•</sup> for compound being measured
- $V_{o}$  = Volume of water extracted (mL)
- $V_i$  = Volume of extract injected ( $\mu$ L)
- F = Dilution Factor
- $RF = (Area_r) \times (C_s injected)$  where: C = ng(Area\_s) (C<sub>r</sub>)

11.1.9.2 Target Compounds for Soil

 $\frac{(A_{x})(C_{z})(V_{i})(F)}{(A_{z})(RF)(V_{i})(W_{i})(D)} = R (\mu g/kg)$ 

All terms are the same as water calculation except:

Where:

 $W_{1} =$  Weight of sample extracted or diluted (g) D = 100 - (% moisture content of sample)/100

11.1.9.3 TICs for Water and Soils

Quantitation of TICs (Tentatively Identified Compounds) is performed by the instrument data system, the operator, and the LIMS data system. The formulas above for waters and soils can be used with the following exceptions:

 $A_r$  and  $A_i$  should be taken from the total ion current areas listing accompanying the TIC report. The nearest <u>non-interfered</u> internal standard should be used. The RF is assumed to be 1. The concentration is therefore estimated and is flagged generally with a "J" when reported.

The total ng of the TIC is calculated as follows:

$$\frac{(\text{Area of TIC}^{**})(C_{\pm})}{\text{Area}_{is}} = \text{ng of TIC}$$

This number can be entered into a LIMS spreadsheet that will calculate the final concentration of the TIC.



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## 11.2 Data Review

System reviews are performed at all levels. The individual analyst continually reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis. The analyst uses a data review checklist to verify that all analytical criteria have been met. This checklist provides a list of items to verify that all analytical specifications have been achieved. Any out-of-control items are documented on the checklist and verbally communicated to the Unit Leader or Section Manager for review and response.

A secondary review of the data is performed by the supervisor or a peer reviewer. The peer reviewer is trained by the QA Section, Section Manager or Unit Leader to perform the data review. This data review specialist is an analyst who is assigned data review responsibilities in addition to their normal duties. After these first two reviews are completed, the data is entered into the laboratory information management system (LIMS). For GC/MS, the data is processed by LIMS and then reviewed.

For all Inorganic analysis, LIMS prints out a data summary called a "Mapper" which is then reviewed by the Section Manager or designee. The Section Manager or designee reviews the data for precision and accuracy to assure that it meets all specifications. After the Section Manager's or designee's approval, analytical report is assembled.

The Section Manager and/or the Laboratory Project Manager review data to ensure consistency with laboratory QC requirements, to verify reasonableness with other generated data, and to determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. The final report is signed by the Section Manager, Project Manager and/or Laboratory Manager or designee.

Unusual or unexpected results will be reviewed and a resolution of the problem will be documented in a sample discrepancy report (SDR) or corrective action report (CAR). If suspect data is reported, the out-of-control events will be addressed in a case narrative. Copies of the SDR's and/or CAR's may be included in a data package as needed.

Prior to final review/sign off by the Laboratory Manager or designee, the Data Reporting Section will verify that the report is compiled in the proper format. The Laboratory Manager or designee provide the final laboratory review prior to reporting the results to the client. The PM will do a final completeness check before submitting the data report to the client.



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

Data audits are also performed by regulatory agencies or client representatives. The frequency, level of detail, and the areas of concern during these reviews are dependent on the specific program requirements.

## 11.3 Data Reporting

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit volume (e.g., milligrams of weight per unit weight of sample (e.g., micrograms per gram,  $\mu g/g$ ). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g., USATHAMA IRDMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a Case Narrative by the Section Manager. The Case Narrative is signed and dated by the Section Manager or Unit Leader and submitted to the Data Management Section to insert in the final report.

The Data Management Section prints the final report forms (unless provided from the respective unit), organizes the data package, adds the glossary of flags and acronyms, and paginates the report. The Project Manager and/or the Laboratory Manager or designee will review and sign the report prior to delivery.

## 11.4 Data Storage

All raw data, such as bound laboratory notebooks and logbooks, strip charts and instrument printouts, LOTUS spreadsheets, and magnetic tapes, as well as final reports, are retained for a minimum period of 6 years. These raw data and reports are documented and stored



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in a manner which are easily retrievable. Raw data records are maintained in the Laboratory:

- Instrument print-outs for conventional inorganic parameters are filed by parameter group and batch number. Generally, current year and previous year documents are kept on file in the laboratory.
- Instrument print-outs and strip charts for the Metals, GC, HPLC, and GC/MS groups are maintained in storage by the laboratory or transferred to corporate archives.
- Final sample reports are filed for easy retrieval. When an archived report is pulled a record of removal is maintained. This provides documentation of any report removed from the archival system.
- Due to the bulk of full data documentation packages, they are generally filed separately from the client files by RFW batch for future reference.
- The LIMS computer information is backed up on tape daily, and stored in a temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Specific criteria is presented in OP 21-11A-003, LIMS Backup Procedures.

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## 12.0 PREVENTATIVE MAINTENANCE

### 12.1 Introduction

The ability to generate valid analytical data requires that all analytical instrumentation be properly and regularly maintained. The responsibility of routine care lies with the analysts using the instruments. Guidance on required routine maintenance, as well as troubleshooting information, is provided in the respective instrument manuals and laboratory OPs. For more extensive preventative maintenance or emergency repair service, each analytical laboratory maintains full service contracts on all major instruments or has accounts established with vendors. The elements of the maintenance program are discussed in the following sections.

## 12.2 Instrument Maintenance Log Books

Each analytical instrument is assigned an instrument maintenance log book. All maintenance activities are recorded in the maintenance log. The information entered in the maintenance log includes:

- Date of service or maintenance.
- Person performing service or maintenance.
- Type of service performed and reason for service.
- Replacement parts installed (if appropriate).
- Documentation of the re-establishment of working order.
- Miscellaneous information.

If service is performed by the manufacturer, a copy of the service record (when available) is affixed to the notebook page, or cross-referenced in the notebook to a separate maintenance file. The service record should include sufficient detail to describe the service performed (e.g., not just "service call," but "replaced pump motor gear"). If the service record does not spell out this information, it must be written separately into the maintenance log. There must also be a reference to the file number, notebook page, etc., that substantiates re-establishment of working order.

## 12.3 Instrument Maintenance and Repair

Preventative maintenance and repairs that cannot be performed by laboratory staff are contracted to the manufacturer's service section or to an authorized maintenance vendor. Laboratory service agreements provide for preventative maintenance, emergency service, and emergency shipping of spare parts. Annual service of the laboratory balances is an example of contracted preventative maintenance. For emergency response, service contracts



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on the Gas Chromatographs, GC/MS instruments and AA-ICP require rapid response. (Typically, service representatives are on site within 24 hours of a service call.) The service contracts also provide for 24-hour delivery of critical spare parts in response to a service request.

Examples of maintenance procedures and suggested frequencies for major analytical instrumentation are summarized in Table 12-1. Actual procedures may vary significantly for specific instruments, depending on the manufacturer, peripherals, data system, etc.. However, this table provides an indication of the level of detail required for maintenance. The frequency of maintenance suggested here assumes daily operation of the instrumentation. Laboratory instrumentation specifies information is provided in the respective instrument manuals, and in the laboratory OPs.

## 12.4 Spare Parts

The laboratory maintains a supply of routinely required spare parts (for example, spare sources, vacuum pumps and filaments for GC/MS, spare torches, burner heads for AA-ICP).

The instrument operators have the responsibility, with the appropriate Section Manager, to ensure that an acceptable supply of spare parts is maintained.

## 12.5 <u>Contingency Plans</u>

Properly maintained equipment will provide dependable service; however, emergencies cannot be totally avoided. Supervisory and building maintenance personnel are notified of power failures during non-working hours, and can be on site or remain on stand-by alert until the emergency is passed or further action is necessary. Additionally, some laboratory personnel from night shift will often already be on site. Service is generally restored within an hour. For prolonged power outages, laboratory personnel on stand-by alert will prepare for an organized, systematic shut-down of major equipment. A decision on the need for auxiliary back-up generators to run storage refrigerators will be made.

With respect to instrument-related downtime, an attempt is made to maintain adequate redundancy in instrumentation to cover short term losses due to repairs. For long-term downtime, arrangements can be made to rent appropriate equipment until necessary repairs can be completed. In either case, with client approval, arrangements may be made to offload work to another laboratory to enable analysis within prescribed holding times.



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#### TABLE 12-1 Example: Equipment and Maintenance

Instrument	Procedure	Frequency
AA	Clean lens and furnace head	Daily
(Graphite Furnace)	Replace windows	As required
	Check or change cuvette	Daily
	Check & drain compressor drain	Daily
	Clean atomizer cell/furnace hood	Daily
	Nebulizer cleaned/dried	Weekly or as required
	Check/change marble stones	Weekly
	Clean filters	Weekly
	Change graphite tube/platform	As required
	Empty waste container	Daily
	Remove carbon tube and check wear	Daily
	Check sample introduction probe	Daily
AA (Flame)	Check gas pressure	Daily
	Check water level in drain receptacle	Daily
	Check nebulizer and tubing	Daily
	Check/clean lamps and windows	Daily
	Clean burner head in sonic bath	Weekly
	Clean mixing chamber, flow spoiler, and impact bead	Weekly
	Check/replace O-rings in mixing chamber	Monthly
	Check drainage hose	Monthly
Leeman Mercury	Check tubing for wear	Daily
Analyzer	Fill rinse tank with 10% HCl	Daily
·	Insert clean drying tube filled with Magnesium Perchlorate	Daily
	Fill reductant bottle with 10% Stannous Chloride	Daily



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# TABLE 12-1Example:Equipment and Maintenance

Instrument	Procedure	Frequency	
ICP	Check pump tubing	Daily	
	Check liquid argon supply	Daily	
	Check fluid level in waste container	Daily	
	Check filters	Weekly	
	Clean or replace filters	As required	
	Check torch	Daily	
	Check sample spray chamber for debris	Monthly	
	Clean and align nebulizer	Monthly	
	Check entrance slit for debris	Monthly	
	Change printer ribbon	As required	
	Replace pump tubing	As required	
			•
UV-Vis	Clean ambient flow cell	As required	
Spectrophotometer	Precision check/alignment of flow cell	As required	
	Wavelength verification check	Semi-annually	
		Sem-anitariy	
Auto Analyzers	Clean sampler	Daily	
-	Check all tubing	Daily	
	Clean inside of colorimeter	Daily	
	Record manifold temperature	Daily	
	Clean pump well and pump rollers	Quarterly	
	Clean wash fluid receptacle	2	
·	Oil rollers/chains/side rails	Weekiy	
	Clean optics and cells	Weekly	
R		Daily	
	Clean cell	Daily	
Spectrophotometer	Check/adjust cell alignment	As required	
ЮС	Check comblem access 11		
	Check scrubbers, reactor cell	Daily	
	Check infrared zero	Daily	
<u>~</u> .	Check carrier gas	Daily	
•	Check all tubing	Daily	
	Check/clean injector valve	Daily	
	Check infrared span	Quarterly	
	Check printer head	Quarterly	
	Check battery charge	Yearly	
	Check permeation dryer	As Needed	



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## TABLE 12-1Example:Equipment and Maintenance

Instrument	Procedure	Frequency	
ΤΟΧ	Clean sample boat/titration cell/electrodes	Daily	
	Check baseline and furnace temperature	Daily	
	Clean inlet/exit tubes	Weekly	
	Check or replace O-rings	Weekly	
	Check 3 gases	Weekiy	
	Check absorption rate	Monthly	
	Check electrode septa	Monthly	
	Recondition cell	Semi-annually	
	Re-coat electrodes	Semi-annually	
	Clean pyrolysis tube	As required	



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# TABLE 12-1Example:Equipment and Maintenance

Instrument	Procedure	Frequency
Finnigan	Clean ionizer source	Quarterly or as required
GC/MS	Change filament	Quarterly or as required
	Clean quadrupole rods	Quarterly or as required
	Change electron multiplier	As required
	CARD CAGE MAINTENANCE:	•
	Change air filter	As required
	<ul> <li>Clean cooling fans</li> </ul>	As required
	<ul> <li>All PCBA's: reseat boards connectors</li> </ul>	As required
	and check all voltages on PCBA's to see	•
	if within specifications. Adjust if necessary.	
	POWER CONTROLLER MAINTENANCE	
	Clean cooling fans	As required
	<ul> <li>All PCBA's:reseat all connections.</li> </ul>	As required
	VACUUM SYSTEM	
	<ul> <li>Mechanical pumps: change oil</li> </ul>	Quarterly or as required
	<ul> <li>Diffusion pump: change oil</li> </ul>	Annually or as required
	• Turbo pump: change oil, cooling fan,	Quarterly or as required
	check water level in recirculator, change	
	50/50 mixture water/ethylene glycol.	
	COMPUTER SYSTEM:	
	<ul> <li>Clean or replace cooling fans</li> </ul>	As required
	<ul> <li>All PCBA's: reseat boards, cables</li> </ul>	As required
	• Disk drive (CDC):	
	change filter	As required
	change pre-filter	As required
	• Disk drive (Priam/Winchester): clean	As required
	cooling fans	
	• Tape streamer: clean tape head, clean	As required
	capstan surface	
	<ul> <li>Printronix printers (MVP, P300): check</li> </ul>	As required
	print quality	

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#### TABLE 12-1 Example: Equipment and Maintenance

Instrument	Procedure	Frequency
Hewiett Packard	Ion gauge tube degassing	As required
GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Semi-annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
,	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required'
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	Monthly or w/cylinder change
	Check for loose/fray wires and insulation	Monthly
	Column temperature verification	Daily
	Visually check for shifting of column packing material resulting in forward movement beyond the bottom of the column exit or settling in excess of 1/2 <sup>n</sup> from the glass wool plug at the column inlet	Weekly or as needed
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
<u>~</u> .	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
	Q(-)	. n voden og
Electron Capture	Detector wipe test (Ni-63)	Semi-annually
Detector (ECD)	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required

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## TABLE 12-1 Example: Equipment and Maintenance

Instrument	Procedure	Frequency
Hall 700A Detector	Electrolyte change	As required by noise
Hall 1000 Detector	Reactor tube/teflon connecting tube change	As required
	Clean detector cell	As required
Photoionization	Change O-rings	As required
Detector (PID)	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
		····
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually
Conductivity Meter	0.01 M KCl calibration	<b>T</b> . <b>H</b>
	Conductivity cell cleaning	Daily
	Conductivity cen deaming	As required
<b>Turbidimeter</b>	Check light bulb	Daily, when used
		••
Deionized/Distilled	Check conductivity	Daily
Water	Check deionizer light	Daily
	Monitor for VOA's	Daily
	System cleaning	As required
~.	Replace cartridge & large mixed bed resins	As required
Orying Ovens	Temperature monitoring	
	Temperature monitoring	Daily
		As required
lefrigerators/	Temperature monitoring	Daily
reezers	Warning system checked	Monthly
	Temperature adjustment	As required
	Defrosting/cleaning	As required



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## TABLE 12-1Example:Equipment and Maintenance

Instrument	Procedure	Frequency
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed



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## 13.0 CORRECTIVE ACTION

To be of high technical quality and regulatory conformance, the data produced by an analytical laboratory must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Mechanisms used in WESTON Analytics laboratories to ensure problem definition include Operating Practices that set forth analytical QC criteria; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could affect the quality of services provided, corrective action is initiated. Procedures for corrective action, which are defined in WESTON OP 21-06-105, are summarized in this section. Corrective action may be immediate or long-term.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action system:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign, and obtain commitment to, responsibility for implementing the corrective action.
- Implement the correction.
- Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

## 13.1 Immediate Corrective Action

Immediate corrective action to correct or repair non-conforming equipment and systems is generally initiated as the result of quality control (QC) procedures. The individual has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. Quality indicators are evaluated against laboratory- established or against method- or client-specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must



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immediately assess the analytical system to correct the problem. Analysts are instructed to be circumspect in their assessments, as an out-of-control situation may be caused by more than one variable. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control" or the measures required to put the system "in control" have been identified and scheduled, the problem and resolution or planned action is documented in the appropriate notebook. Data generated by an analytical system that is determined to be out of control must never be released without approval of the Supervisor, the QA Manager, or the Laboratory Manager.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify the appropriate supervisor and initiate a Sample Discrepancy Report (SDR) (see Figure 13-1). If an SDR is required, it is routed for proper authorizations and direction.

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

If a large number of SDR's are received by the QA Section or further action is required, a Corrective Action Documentation (CAD) Form (Figure 13-2) is initiated. This CA Form may also be initiated by any laboratory employee for problems not suited to, or broader in scope than, the SDR's.

The Quality Assurance Manager has the authority to stop the analysis and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

## 13.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to quality assurance (QA) issues, which are most often identified during internal and external audits (see Section 14.0). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LIMS reprogramming, are examples of long-term corrective action.



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### 13.3 <u>Responses to External On-Site Audits and Performance Samples</u>

When the results from an external on-site audit or performance evaluation study are received by the laboratory, a summary of the results is distributed to appropriate laboratory personnel.

If deficiencies exist, the QA Manager will issue a memo addressing the findings and assigning responsibility for correcting each deficiency. Upon receipt of all corrective action responses, the QA Manager will forward the information to the respective outside client or agency.

#### 13.4 <u>Responsibility and Closure</u>

The Section Manager, with the respective Unit Leaders and Supervisors, is responsible for correcting out-of-control situations, placing highest priority on this endeavor.

Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to Corporate Quality Assurance Management by the Quality Assurance Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure QA policies and procedures are enforced.



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## FIGURE 13-1 Example: Sample Discrepancy Report (SDR)

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## FIGURE 13-2 Example: Corrective Action Report (CAR)

WESTON-Gulf Coast, Inc. Analytical Corrective Action Report - Wet Chemistry

Date Originated:	_ Dets Corrected:
Test/Mathod:	
Lab Book # / Documentation Source:	Page #:
Problem:         (Circle One)           1.         LCS / Prop Blank exceeds the control limit           2.         Duplicate Analysis RPD exceeds the control           3.         Matrix Spike Recovery exceeds the control           4.         Other:	its. rol limits. 4 limits.
Batch #:	RFW #
Matrix: a. Water b. Soil c. Waste	d. Other:
Problem 1: Prop Blank LCS Resul MDL: LCS Known	
S Recover	y:
Problem 2:	Corrective Action:
Original Rasult: Duplicates Rasult: RPD:	Replicate Spiker
Problem 3:	Corrective Action:
Original Result:	Replicate Spike:
Spiked Result:	Spike Level:
Spite Level:	% Recovery:
\$ Recovery:	
Yes - Redigestics Required	No - Redigestion Not Required
New Prop Batch ID: Samples to be re-digested:	
Problem 4:	
Corrective Action:	
Cimetana and a second	
Signeture:	
Return to Control Documentation:	
Reviewed By:	Data:



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#### 14.0 <u>Performance and Systems Audits</u>

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements. They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts on laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

Performance audits test the laboratory's ability to assay an unknown sample correctly. They may be single blind or double blind. In a single blind study, the analyst is not provided with the acceptable result for the unknown sample until after the experimental result is reported; however, it is known that the sample is a performance test. In a double blind performance test, the analyst not only has no knowledge of the acceptable result, but the sample is disguised in such a manner as to maintain anonymity as a performance test sample. In some double-blind studies, even laboratory management is unaware that PEs are not actual samples.

Systems audits and surveillances are used to evaluate the operational details of the QA program. An audit is a systematic assessment of an area of laboratory operation. It may comprise evaluation of an operational unit, e.g., GC/MS Analysis or Data Reporting; or it may have a more narrow focus, such as an assessment of adherence to chain-of-custody procedures. Audits are conducted by persons other than those who performed or directly supervised the work being inspected. A surveillance consists of inspection or monitoring of a specific targeted area for compliance with requirements, such as an evaluation of a single analytical method to ensure conformance with the written OP.

## 14.1 <u>External Audits</u>

Performance audits, as well as on-site systems audits, by non-Analytics Division agencies and clients are an on-going, continual occurrence. Whether the audit is scheduled or a surprise, full cooperation with the audit team will be provided by laboratory staff during the on-site visit. A full response to any deficiencies cited as a result of a performance audit or on-site visit will be addressed within the schedule determined by the client. The QA Manager is responsible for scheduling and coordinating all external audits and any resultant corrective actions.

Audits conducted by government agencies are summarized in Table 14-1.



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## 14.1.1 <u>Performance Audits</u>

Single blind performance audits (e.g., Performance Evaluation Studies: PES) are routinely analyzed at least four times a year: twice as U.S. EPA WS and WP studies, and twice as New York State DOH Potable, Non-Potable, Solid and Hazardous Waste, and Air and Emissions. Additionally, air matrix NIOSH PAT samples are analyzed quarterly, and  $NO_x$ and SO<sub>x</sub> are analyzed annually. Other water/wastewater PES are received annually from Illinois EPA and NC DNR & CD, quarterly from confidential clients for full TCL/TAL plus selected wastewater parameters, and semi-annually from the Oklahoma Water Resources Board (OWRB).

PES samples are additionally analyzed as double- and single-blinds on a project-specific basis for selected clients, including the U.S. Army Corps of Engineers, Federal and State Agencies, and non-government clients.

## 14.1.2 Systems Audits

On-site evaluation by agencies, clients, or designated third-party auditors (both government and non-government) are routinely conducted to assure program compliance. These audits are preferentially pre-scheduled with the client to ensure that appropriate personnel are available to answer questions. However, audits may also be unannounced. In either instance, full cooperation with audit team the laboratory staff during the on-site visit will be provided.

Program compliance may be defined as adherence to requirements for a specific certification or licensing program, e.g., state certifications, or adherence to specific project requirements. Government program clients include State Regulatory Agencies, U.S. EPA, the Nuclear Regulatory Commission (NRC), the Department of Defense, and the Department of Energy. Non-government clients include industrial clients and engineering firms utilizing our laboratory services for drinking water, groundwater, wastewater, hazardous waste, and mixed waste water, soil, air, tissue, and miscellaneous waste matrices.

## 14.2 Internal Audits

The laboratory QA Manager has overall responsibility for monitoring the internal Quality Assurance/Quality Control program. The QA Section Manager has a staff to provide inhouse audits and to review and validate analytical data packages.

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#### 14.2.1 <u>Performance Audits</u>

Internal performance audits conducted at the bench level provide the analyst with a tool to evaluate the acceptability of a specific data set. This is accomplished through analysis of laboratory control samples or spiked blanks of known concentration to the analyst which must meet minimum performance standards. When these QC checks are performed in duplicate, method accuracy and precision information can be generated to demonstrate the proper functioning of the total measurement system.

As an additional feature of the laboratory's internal QA Program, couble blind performance evaluation samples are submitted to the laboratory periodically for analysis. These samples originate both internally and externally, and are scheduled through the laboratory's project management system to ensure anonymity. Over the course of a year, samples are submitted to cover all routinely analyzed methods.

Externally originated double blinds are analyzed quarterly by the Lionville Laboratory for full organic and inorganic target compound list parameters in both soi<sup>1</sup> and water. Externally originated samples are purchased from a commercial vendor (currently ERA) in a constituted form. WESTON initiates these external double-blind samples using the same procedures utilized for routine clients through a designated project manager, to include, for example, assigning of work order numbers, forward scheduling the analyses (using a "fake" client name, which changes quarterly), generation of bottles orders so that samples arrive in standard containers, etc. This system effectively gets samples into the laboratory for unbiased analysis. Results are compiled by the project manager and submitted to the QA Section for review and evaluation. Any deficiencies noted are addressed with the appropriate laboratory service group and a corrective action plan is implemented.

Internally generated samples are handled in the same manner as the externally purchased double blinds, except that they are prepared by the laboratory, unknown to the analysts, using NIST or commercially available reference materials.

#### 14.2.2 Systems Audits and Surveillances

Internal laboratory systems audits and surveillances will be conducted and documented such that all laboratory sections receive a QA audit at least annually. Unique client audit procedures and data requirements will be complied with as specified contractually. The internal audit consists of a review of laboratory systems, procedures, and documentation. Internal systems audits include specific attention to issues noted in external audits. Any deficiencies and/or deviations are documented and a summary report is prepared.

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The following items may be included for focus in routine laboratory audits and surveillances:

- Life of reagents
- Holding times
- Interferences (it any)
- Maintenance logs
- Standards traceability
- Preparation of glassware
- sample preservation
- Equipment/instrumentation

- Computer spreadsheets
- Calculations
- Standard deliverables
- Lab book documentation
- Safety
- Method detection limits
- Current operating practice (OP)
- A system audit report is prepared and distributed to the responsible party, including the appropriate supervisor. A maximum of one calendar month is given to address any recommended corrective actions. The original copy of the completed responses is kept on file in the QA Section. QA also conducts follow-up audits to ensure clocure of items noted in the respective original systems audits.

#### 14.2.3 <u>Raw Data Audits and Surveillances</u>

All laboratory notebooks are routinely reviewed by the analyst and a second reviewer to assure correctness of sample and quality control calculations. In addition, all active laboratory data books and QC files are subject to periodic audits/surveillances by QA personnel and/or Unit Leaders.

Raw data evaluations will be based on the following completed information, as applicable:

- Parameter and n.ethod
- Instrument ID and settings
- Date and full signature of analyst
- Valid standard curve
- Frequency of QC
- QC calculations and recoveries
- Sample calculations
- Neatness and ease of data interpretation

Reviewed data will be documented as "reviewed by" and signed (initials or full signature) and dated by the reviewer.



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## **TABLE 14-1**

## **External Agency Performance and Systems Audits**

Адевсу	Parameters	Audit Type/Frequency	Required For
California State Dept. of Health	Water Supply Water Pollution	Performance/Semiannually; and System/Biennially	Water/Wastewater Certification
Colorado State Dept. of Health	Water Supply	Performance/Semiannually; and System Biennially	Water Certification
Florida Dept. of Health and Rehab Services	Water Supply	Performance/Semiannually; and System/Biennially	Water/Wastewater Certification
Illinois EPA	Water Supply	Performance/Semiannually; and System/Biennially	Water/Wastewater Certification
Louisiana Dept. of Health and Hospitals	Water Supply	Performance/Semiannually; and System/Annually	Drinking Water Certification
New Hampshire Department of Environmental Services	Water Supply Water Pollution	Performance/Semiannually; and System/Triennially	Water/Wastewater Certification
lew Jersey Department of Environmental	Water Supply Water Pollution	Performance/Annually; System/Biennially	Water/Wastewater Certification
rotection	Hazardous Waste	Performance and System/ Per Project Requirements	Hazardous Waste Approval, RI/FS
lew York State Dept. of Health	Water Supply Water Pollution	Performance/Quarterly; System/Annually	Water/Wastewater Certification
forth Caroling	Water Supply	Performance/Semiannually; System/Biennially	Drinking Water Certification
orth Carolina ept of Natural esources and community evelopment	Water Pollution	Performance/Semiannually; System/Biennially	Wastewater Certification



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## TABLE 14-1 (continued) External Agency Performance and Systems Audits

Agency	Parameters	Andit Type/Frequency	Required For
Pennsylvania Department of Environmental Resources	Water Supply	Performance/Annually; System/Biennially	Drinking Water Certification
U.S. EPA	Water Supply Water Pollution Hazardous Waste	Performance and system CLP-type audits/ Per Contract Requirement	Superfund Related Analytical Work
U.S. Air Force	Water Supply Water Pollution Hazardous Waste	Performance and system CLP-type audits/ Per Contract Requirement	Water/Wastewater, Superfund Related Analytical Work
U.S. Army Corps of Engineers	Water Supply Water Pollution Hazardous Waste	Performance and system CLP-type audits/ Per Contract Requirement	Water/Wastewater, Superfund Related Analytical Work
U.S. Army Corps of Engineers	Hazardous Waste	Performance and system CLP-type audits/ Per Contract Requirement	Superfund Related Analytical Work
U.S. Navy	Water Supply Water Pollution Hazardous Waste	Performance and system CLP-type audits/ Per Contract Requirement	Water/Wastewater, Superfund Related Analytical Work
Utah State Health Laboratory	Water Supply Water Pollution	Performance/Semiannually; System/Biennially	Water/Wastewater Certification
Washington State Dept. of Ecology	Water Pollution	Performance/Biennially; System/Triennially	Wastewater Certification
Wisconsin Dept. of Natural Resources	Water Supply Water Pollution	Performance/Semiannually; System/Triennially	Water/Wastewater Certification

 Water Supply:
 Drinking Water

 Water Pollution:
 Wastewater

 Hazardous Waste:
 "Target Compound List" Superfund Analyses

 CLP:
 U.S. EPA and/or State-Lead Contract Laboratory Program

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## 15.0 <u>QUALITY ASSURANCE REPORTS</u>

Quality assurance reports communicate quality status and needs to upper management. By providing summaries of quality data and analysis of relevant situations, these reports provide management a feedback loop to monitor the effectiveness of laboratory improvement activities. Quality assurance reports distinguish between major and minor issues, while minimizing false alarms. The reports emphasize objective quality measurements, but when subjective elements are reviewed, these are reported without blame.

For day-to-day problems, a Sample Discrepancy Report (SDR) and/or Corrective Action Report (CAR) are used. These forms are typically used for process related circumstances requiring immediate attention (see Section 13.0). Distribution of these corrective action documents includes the appropriate Project Manager and/or Section Manager that must acknowledge and approve the corrective actions in order to remedy project related out-ofcontrol situations.

Regular and periodic quality reports are prepared for Section Management, Division Management and the Corporate Quality Assurance Officer (e.g., monthly laboratory QA reports, quarterly Division QA reports, quality improvement project reports). These reports are less uniform by nature, and therefore result in more subjective measurements. In a cumulative sense, where applicable, these reports comprise or address the following issues:

- Identifies the "vital few" quality problems to help focus resources where they will be most beneficial.
- Reports on the status of quality improvement initiatives; results achieved; work in progress; next steps and by whom; action needed by upper management; and lessons learned.
- Summarizes audit or assessment findings and observations, whether intrasection, Division or of subcontractors and suppliers.
- Discusses major quality developments occurring in the environmental industry, government agencies, clients, competitors or other macro-environmental forces.
  - Proposes initiatives for upper-management consideration.

QA reports to laboratory management summarize a wide range of QA activities. These activities may include, but are not limited to:



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## 15.1 <u>Performance Evaluation Studies (PES)</u>

Results of both external and internal performance audits are distributed to laboratory line management for review and action, as appropriate. Any required response to deficiencies must be submitted to QA for review. After acceptable corrective action responses are received for all noted deficiencies, a summary of the results with attached responses is distributed.

## 15.2 Agency and Client On-Site Audits

On-site evaluations of laboratory facilities and procedures are conducted by external auditors to ensure conformance to the requirements of their respective programs. These are scheduled and coordinated through the QA Section. The summary evaluation received from the client reporting findings and/or recommendations and observations are distributed through the QA Section to laboratory line management for review and action, as appropriate. Any required response to deficiencies must be submitted to QA for review. After acceptable corrective action responses are received for all noted deficiencies, a summary of the results with attached responses is distributed.

## 15.3 <u>Management Systems Reviews</u>

Thorough and efficiently documented laboratory operational procedures and systems facilitate the generation of a quality analytical product. Audits and surveillances of established procedures are conducted on a continuing basis to verify conformance. Based on these procedural reviews, a report is issued summarizing any findings and recommendations for revising laboratory procedures to improve the analytical systems.

## 15.4 <u>Employee Orientation and Training Activities</u>

Orientation to laboratory policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. The Quality Assurance Section is responsible for the documentation of these activities. A summary is provided to laboratory management in the monthly QA report.



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## 16.0 WASTE MANAGEMENT

Wastes generated in WESTON laboratories can be divided into four types: hazardous, nonhazardous, radioactive, and mixed wastes. The EPA defines wastes as hazardous if they exhibit the characteristics of ignitability, corrosivity, reactivity, or toxicity as identified in 40 CFR 261, Subpart C, or are listed in 40CFR 261, Subpart D. For the intents and purposes of laboratory waste management procedures, substances to be disposed of as hazardous wastes include, but are not limited to:

- All environmental samples. (Disposed 30 days after the data report is submitted to the client unless longer specific contract/program requirements were established. Disposal may include return of the samples to the client.
- By-products of sample preparation or analysis.
- Standards or reagent solutions.
- End products of sample preparation or analysis.
- Used chemicals, or chemicals that have exceeded their shelf life.
- Waste products generated from spill cleanup.

Since each WESTON laboratory may use different categories for waste segregation and storage as approved by their disposal company, the laboratories maintain detailed Operating Practices for Waste Management in the Standard Practices Manual, Chapter 21-12. Detailed procedures are documented in the Gulf Coast, Lionville, and Stockton Laboratory OPs 21-12G-0001, 21-12L-001, and 21-12S-003 Hazardous Waste Segregation; and 21-12G-0001, 21-12L-001, and 21-12S-0012 Hazardous Waste Management.

## 16.1 Waste Management Procedures

The following sections outline general guidelines for managing hazardous wastes in the laboratory. Managing hazardous wastes will be consistent with the requirements of the Resource Conservation and Recovery Act (RCRA). RCRA is intended to provide cradle-to-grave control of hazardous waste, and is the federal regulation governing waste management programs. Waste management procedures will vary depending on the facility, waste hauler requirements, and final disposal options.



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### 16.1.1 Managing Acid Wastes

In order to determine a material's acidity, litmus paper or a pH meter can be used. If a material has a pH of 4.0 or below, it should be treated as an acid, and must be placed into temporary containers (e.g., poly drums or poly lined metal drums) marked "ACID WASTES".

## 16.1.2 Managing Alkaline Wastes

In order to determine a material's alkalinity, litmus paper or a pH meter can be used. If a material has a pH of 10.0 or above, it should be treated as a base and must be placed into containers labeled "ALKALINE WASTES" or "CAUSTIC WASTES".

16.1.3 Managing Neutral Wastes

All materials that have a pH of 4.0 - 10.0 are treated as neutral wastes, and must be placed into containers labeled "NEUTRAL WASTES".

## 16.1.4 Managing Flammable Liquid Wastes

Drums containing flammable liquid wastes should be properly grounded and the transfer container bonded to the drum to avoid the build up of static electricity when pouring the waste.

## 16.1.5 <u>Radioactive Waste Management</u>

Guidance for radioactive waste management is provided in Section 9.5.4 of Lionville's OP 21-12L-001.

### 16.1.6 Sample Waste Management

Used environmental samples will be routinely discarded to minimize the back log of samples in storage areas. Packaging of used samples will be performed by the Laboratory Waste Technician, or an individual designated by the Laboratory Manager. This procedure will be completed in a properly ventilated room using an air purifying respirator.

Mixed waste or radioactive samples will typically be returned to their point of origin.



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## 16.1.7 Empty Sample Containers

Empty sample containers must not be disposed of as municipal waste. Sample containers must either be crushed/shredded or recycled. Crushed/shredded sample container must be periodically analyzed and disposed appropriately to verify no regulated contaminates are present. Sample container that are recycled must be rinsed prior to offering to the recycler. The recycler must be informed of the previous contents of the containers.

## 16.1.8 Waste Accumulation Area

Each laboratory facility shall have a secured waste accumulation area, where 55 gallon drums of the laboratory waste materials will be stored until removed by a licensed waste management firm. Drums will be marked with the current date and applicable information about their contents when transferred into the accumulation area. This area shall be properly ventilated, and drums shall be stored no more than 90 days before they are removed off-site by WESTON's contracted hazardous waste disposal firm.

The waste accumulation area shall be secured against unauthorized personnel by having a locked door and appropriate warning signs posted outside the area. Only authorized personnel shall have access to the waste accumulation area. The area will be locked whenever it is unoccupied.

## 16.2 Waste Manifesting

The uniform hazardous waste manifest (UHWM) is completed prior to loading waste on a vehicle for shipment to a transporter, storage, or disposal (TSD) facility. All applicable spaces are completed.

Once the UHWM is completed (with the exception of signatures), the waste is loaded on the vehicle and the UHWM is signed by the Hazardous Waste Manager. The UHWM is then separated by giving the transporter the "TSD to generator copy", the "TSD to EPA copy", the "TSD" copy, and all transporter copies.

The laboratory maintains the generator copy for three (3) years, but it is suggested that the document remain on file for the life of facility. The copy marked "generator send to EPA" is sent to the generator's state EPA unless otherwise stated within the time period on the UHWM (each state may vary).



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## 16.3 <u>Waste Minimization</u>

WESTON laboratories perform various analyses in strict compliance with EPA protocols. EPA protocols restrict substitution of chemicals and specify the amounts of solvents and chemicals to be used. Therefore, waste minimization through elimination, substitution, or volume reduction is not a viable option. Segregation of waste streams as identified in previous section is the primary means of waste minimization.

No radioactive samples or waste may be placed into any of the hazardous waste streams.

Extracts, digestates, and residues from radioactive samples are filtered, if appropriate, then screened for gross alpha/gross beta activity at a detection limit of 5 picocuries per gram. If this screening demonstrates no radioactivity, the respective extract, digestate or residue will be disposed of as hazardous waste rather than mixed waste.

WESTON will continue to explore additional waste minimization options as they become available.



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## 17.0 <u>HEALTH AND SAFETY</u>

It is the policy of WESTON to assure that its employees are provided a work environment in which they are secure from recognized health and safety hazards. In addition, WESTON is committed to protecting the public and the environment from potential hazards that may result from laboratory activities. To accomplish these objectives, all reasonable precautions are taken to ensure that exposures to potentially hazardous materials are maintained as low as possible for employees and members of the public. Employees conduct work activities in accordance with all applicable health and safety regulations, and corporate guidelines. In addition, a program has been adopted for a pro-active approach to minimizing exposures and accidents, and for identifying potential deficiencies in facilities and work practices which may impact personnel protection and health and safety.

The laboratories follow all corporate, as well as divisional health and safety policies. Corporate policies are outlined in Operating Practice 11-01-001 and provide the basis for the Corporate Health and Safety Program. Division policies are presented in OP 21-09A-005, "Chemical Hygiene Plan" and related supplements, as well as in Chapter 21-09 of the Standard Practices Manual.

The laboratories are also responsible for complying with additional corporate policies relating to health and safety, such as the Hazard Communication Program and Radiological Health Program.

## 17.1 Laboratory Safety Organization

It is the responsibility of the Division Manager to implement the Health and Safety Program and to select a Division Safety Officer (DSO). The Corporate Health and Safety Director reviews and approves the designation of Division Safety Officers.

Laboratory Managers are responsible for the Health and Safety Program in each laboratory. They will appoint a Laboratory Health and Safety Officer (LHSO) to administer the Health and Safety Program in their laboratory.

WESTON employees are required to carry out their assignments with the health and safety of those involved as the primary concern, following the provisions of the Corporate Health and Safety Program and the Analytics Division Health and Safety Program.

The following are the Health and Safety responsibilities of all laboratory personnel:

Report accidents, hazards, or unsafe conditions to the Area Supervisor and LHSO immediately.



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•	Wear safety glasses, laboratory coats, gloves, and other protective equipment as required for the procedures they are performing.
•	Identify the deficient work practices of other laboratory employees that are not in full compliance with corporate, division, or laboratory safety policies and politely inform co-workers of the deficiency. (Employees, once informed, must immediately correct the deficiencies.)
•	Know the location and availability of MSDS, and maintain an awareness of the toxicity and hazard data for chemicals which they encounter on a daily basis.
•	Know the location and use of all emergency equipment within the laboratory.
•	Know and comply with emergency evacuation and rescue procedures.
•	Participate in the WESTON Medical Monitoring Program.
•	Attend Laboratory Health and Safety training meetings.
•	Note potentially unsafe conditions, and present suggestions for improving safety procedures and practices to supervisors or to members of the Safety Committee.
•	Keep work areas clean and neat.
)	Make sure all visitors comply with safety regulations.
7.2	Laboratory Safety Equipment
afety emine	nent including fire extinguishers another that the second
Clear, unobst	nent, including fire extinguishers, eyewash stations, safety showers and first-aid tained in easily accessible locations in the operational areas of the laboratory. ructed access are maintained to safety equipment. Safety equipment locations

# 17.3 Employee Information and Training

The OSHA Standard For Occupational Exposure To Hazardous Chemicals In Laboratories (29 CFR 1910.1450) requires that information concerning the hazards associated with a chemical be transmitted to the employees who handle that chemical. This is accomplished by proper container labeling, material safety data sheets, and employee training.

will be clearly marked and made apparent to laboratory employees and visitors.



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Eff. Date: 09/01/94 Initiated By: R. J. Frederici Approved By: E. M. Hansen Authorized By: A. M. Henry SP No. 21-06A-001

## 17.4 Labeling

All chemical containers shall be labeled. Chemical manufacturers, importers, or distributors are responsible for labeling containers with the identity of the hazardous chemical, appropriate hazard warnings, and the name and address of the chemical manufacturer, importer, or distributor. In the event that chemicals are received which do not meet OSHA labeling requirements, the supplier will be contacted and informed that labeling must meet OSHA requirements or another supplier will be sought. Employees shall not remove or deface existing container labels. Temporary containers such as squeeze bottles shall be labeled with the identity of the hazardous chemical and appropriate hazard warnings.

## 17.5 <u>Material Safety Data Sheets</u>

Material safety data sheets (MSDS) are prepared by chemical manufacturers for distribution to the end user of a hazardous chemical. MSDS contain information concerning physical and chemical characteristics, physical and health hazards, signs of exposure, primary routes of entry, exposure limits, carcinogen information, handling precautions, control measures, emergency and first aid procedures, and the name, address and telephone number of the manufacturer. MSDS shall be obtained for each hazardous chemical used in the laboratory. MSDS must be readily accessible to employees on all shifts. The Laboratory Health and Safety Officer shall review each MSDS received for missing information and obvious errors. Additional information or corrections will be requested when appropriate.

## 17.6 <u>Training</u>

Each employee who works in the laboratory shall attend an Introductory Laboratory Health and Safety Course. This course includes information concerning detection and monitoring of hazardous chemicals, physical and health hazards, measures employees can take to protect themselves from chemical hazards, contents of 29 CFR 1910.1450, review of the Analytics' Chemical Hygiene Plan, exposure limits, symptoms associated with chemical exposures, location of health and safety references, materials handling, reviewing MSDS, and understanding labeling systems. This course will also include a review of fundamental laboratory safety practices. Additional details of the program may be found in the laboratory Right-to-Know Training OP.

# SECTION 4 HEALTH AND SAFETY PLAN

### 4.1 INTRODUCTION

The purpose of this Health and Safety Plan (HASP) is to define specific procedures and protocols that will be implemented to ensure the health and safety of all Roy F. Weston, Inc. (Weston<sub>®</sub>) personnel and their subcontractors during field activities related to the Supplemental Remedial Work Plan at the Black & Decker facility in Hampstead, Maryland.

A copy of the HASP will be provided to each subcontractor, and a copy will be available at each work location. The information contained in this document is proprietary and cannot be released or duplicated without the written permission of Weston. As stated previously, this HASP applies to all subcontractors of Weston and all subcontractors to Weston subcontractors. In addition, visitors to Weston work locations will also be required to follow Weston health and safety protocols. Any deviations from the Weston HASP or program will be noted in Weston's Health and Safety Log.

#### 4.2 HEALTH AND SAFETY RESPONSIBILITIES

#### 4.2.1 Weston and Weston Subcontractors

Weston will assign one individual to serve as Site Health and Safety Coordinator (SHSC) during each portion of scheduled field activities. That individual will be responsible for ensuring that all personnel and activities are in conformance with the protocols defined in this document. The SHSC will have complete control over health and safety matters for the field activity. He or she may at any time stop a field activity if health and safety procedures are being compromised or are not sufficient. The SHSC will maintain direct contact with Weston's Corporate Health and Safety Director.

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If more than one field crew is required, one member from each crew will be assigned as the Field Safety Officer (FSO). The FSOs will have responsibility for health and safety compliance at each work location and will maintain contact with the SHSC.

Weston's Corporate Health and Safety Director is ultimately responsible for ensuring that corporate health and safety programs are adhered to by all Weston employees and subcontractors. In regard to work at Black & Decker, the Corporate Health and Safety Director will review and approve this document. The Project Health and Safety Coordinator, under the jurisdiction of the Director, will serve an audit function to ensure that the defined protocols are being implemented during field activities.

Other individuals responsible for the project's HASP include the Program Manager and the Project Director. The ultimate responsibility for project health and safety lies with the Project Director. In fulfillment of this responsibility, the Program Manager and the Project Director lend their support to health and safety programs. Their support will be manifested by approving this HASP and by emphasizing the successful and safe completion of the project.

## 4.3 WESTON'S HEALTH AND SAFETY PROGRAM

#### 4.3.1 Medical Monitoring

In compliance with OSHA standards, all Weston personnel will be enrolled in a medical monitoring program. The medical status of all Weston personnel is monitored through an annual physical examination. Medical results and monitoring data for Weston personnel are reviewed by an independent oversight group, Washington Occupational Health Associates, Inc., Washington, DC. All subcontractor personnel will be required to have a medical monitoring program in place and must be certified by a physician to be medically fit to wear respiratory protection and to work at hazardous waste sites. The specific test parameters of the Weston medical exam are as follows:

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- Medical and exposure history questionnaire.
- Physical examination by physician.
- Audiometric test and questionnaire (0.5K, 1K, 2K, 3K, 4K, 6K, and 8K Hz levels).
- Pulmonary function test (FVC and FEV1).
- Visual acuity test.

- Resting electrocardiogram (12 lead).
- Laboratory analyses:
  - Blood chemistry profile.
  - BC with differential.
  - Routine urinalysis.
  - Blood lead level.
  - Zinc protoporphyrin determination.
- Heavy metal testing.
- Urinary arsenic, mercury, cadmium.
- PA chest x-ray (one view).
- Other specific tests are performed on an individual basis.

#### 4.3.2 Personnel Training

All Weston personnel are required to attend the Roy F. Weston, Inc. "Hazardous Incidents Response Operations Course." This is currently a 40-hour training course. Individuals who, prior to 1987, have attended Weston's 24- or 32-hour training courses meet the 40-hour requirement based on "grandfathering" of previous experience. These courses certify Weston personnel to perform various activities in potentially hazardous locations in EPA-designated levels of protection B and C.

To serve as an SHSC, an individual must have additional training (8 hours), 24 hours of work experience in the prescribed level of protection, and final approval by Weston's Corporate Health and Safety Director.

Prior to commencement of intrusive activities on site, all personnel and subcontractors will attend a task-specific Health and Safety Orientation. The purpose of this training will be to familiarize project personnel with project-specific hazards, to ensure compliance with the HASP, and to fulfill "right-to-know" regulations. The contents of this training will include the following:

- Potential chemical hazards.
- Potential physical hazards.
- Levels of protection.
- Decontamination procedures.
- Emergency procedures/telephone numbers.
- Directions to the nearest medical facilities.
- Health and safety chain of command.
- Respiratory protection check-out procedures.

#### 4.3.3 <u>Subcontractors</u>

All subcontractors to Weston will be required to comply with Weston standards as a minimum. In addition, they must adhere to all pertinent federal, state, and local health and safety standards.

The following information must be supplied by each subcontractor to Weston:

• A general statement indicating that the subcontractor's health and safety program is in compliance with applicable sections of 29 CFR 1910 and 1926. Specifically, the statement must identify that the subcontractor's employees are aware of and that the subcontractor is in compliance with the intent of OSHA standard 1910.120, "Hazardous Waste Operations and Emergency Response."

A statement indicating that all employees who take part in activities are enrolled in and current with respect to a medical monitoring program that complies with OSHA standards.

#### 4.4 SITE DESCRIPTION

The Black & Decker facility is located in Hampstead, Maryland, in northeastern Carroll County, approximately 35 miles north of Baltimore (Figure 1-1). The plant is situated on 185 acres of property in a predominantly rural setting. Two separate parcels of farmland containing 138 and 173 acres of property are to the north and west of the site, respectively. The population center of Hampstead is approximately 0.8 mile north of the plant along Hanover Road, State Route 30.

#### 4.4.1 Site History

An environmental site investigation was initiated in 1987 at the request of Black & Decker for its Hampstead facility. A series of phases have been completed at the site and a groundwater recovery system has been installed. A series of 10 extraction wells have been installed, 5 on the western boundary and 5 on the eastern boundary. In addition:

- A PCE plume is present primarily on the western half of the facility, while TCE is present in groundwater primarily in the northeastern part of the facility.
- Lagoon samples taken in 1987 contained low levels of TCE and PCE in the surface water and sediment.

A list of the primary chemicals of concern is shown on Table 4-1.

Table 4-1

## Primary Chemicals of Potential Concern, Exposure Limits, and Potential Acute Health Effects Black & Decker Facility Hampstead, Maryland

Chemical of Potential Concern	Standards/ Criteria*	IDLH	Maximum Concentrations	Exposure Route	Symptoms of Acute Exposure	Physical Properties	Reactivity/ Incompatible With	Relevant Instrumental Information**
Acetone	TLV 750 ppm PEL 750 ppm	20,000 ppm	Equipment Decon.	I G C	Irritated eyes, nose, and throat; headache; dizziness; dermatitis	FP = 1.4°F LEL = 2.6% UEL = 12.8% State = Liquid VP = 266 mm at 77°F VD = 2.00	Oxidizing material; acids	IP = 9.69 eV HNu (10.2 eV) = 63% OVA = 60%
Trichloroethene (TCE)	TLV 50 ppm PEL 50 ppm	Ca	GW: RFW-10, 16 EW-1, 2, 4, 5	I G C	Irritated eyes; headache; vertigo; visual distortion; tremors; nausea; vomiting	FP = NA LEL = 11% UEL = 41% State = liquid VP = 100 mm at 90°F VD = 4.35	Strong caustics; chemically active metals	IP = 9.47 eV OVA = 70%

TLV	= Threshold limit value	FP	= Flash point
PEL	= Permissible exposure limit	LEL	= Lower explosive limit
IDLH	= Immediately dangerous to life and health	UEL	= Upper explosive limit
GW	= Groundwater	VP	= Vapor density
Ι	= Inhalation	IP	= Ionization potential
G	= Ingestion	HNu	= PI-101 photoionization detector equipped with 11.7 eV probe
С	= Contact	OVA	= Organic vapor analyzer
S	= Skin absorption	Ca	= Carcinogen
		NA	= Not applicable
C S	= Skin absorption	Ca	= Organic vapor analyzer = Carcinogen

\* OSHA PELs are standards; ACGIH TLVs are criteria.

\*\* HNu and OVA response factors reported as percentage of total compound concentration that can be detected by instrument.

Table 4-1 **Primary Chemicals of Potential Concern** (Continued)

Chemical of Potential Concern	Standards/ Criteria*	IDLH	Maximum Concentrations	Exposure Route	Symptoms of Acute Exposure	Physical Properties	Reactivity/ Incompatible With	Relevant Instrumental Information**
I,I,i-Trichloroethane (TCA)	TLV 350 ppm		GW: RFW-10	I S C	Causes CNS effects; moderate skin irritant, severe eye irritation; also cardiovascular and respiratory effects	FP - NA VP = 100 mm (20°F)	Reacts violently with nitric oxide, oxygen, liquid oxygen, Na, sodium hydroxide, and chemically active metals such as: aluminum, magnesium powder, sodium, potassium; upon contact with hot metal or exposure to ultra-violet radiation, it will decompose to form hydrochloric acid, phosgene and dichloroacetylene gases	OVA = 105% HNu (11.7eV) = 90%
Tetrachloroethylene (PCE)	TLV 25 ppm	Ca 550 ppm	GW: RFW 4A, 4B EW 9, 10	I G C S	Nausea, abdominal pain, jaundice	BP = 296° VP = 9 mm	Chemically-active metals, strong caustics, fuming sulfuric acid	OVA
 I/2-Dichloroethene (DEC)	TLV 200 ppm	4,000 ppm	GW: EW-1, 8	I G C	Irritated eyes, respiratory system, central nervous system	BP = 140°F VP = 180-264 mm	Strong oxidizers, strong alkalide potassium hydroxide copper	HNu (10.2) OVA - IP = 9.65 V

TLV = Threshold limit value

PEL = Permissible exposure limit

- IDLH = Immediately dangerous to life and health
- GW = Groundwater
- I = Inhalation G
- = Ingestion С = Contact
- S
- = Skin absorption

= Flash point LEL

FP

UEL

VP

IP

HNu

OVA

NA

- = Lower explosive limit
- = Upper explosive limit
- = Vapor density
- = Ionization potential
- = PI-101 photoionization detector equipped with 11.7 eV probe
- = Organic vapor analyzer
- Ca = Carcinogen
  - = Not applicable

\* OSHA PELs are standards; ACGIH TLVs are criteria.

\*\* HNu and OVA response factors reported as percentage of total compound concentration that can be detected by instrument.

#### 4.4.2 Task Analysis

All physical and mechanical activities pose some level of potential health and safety risks. These risks typically are characterized as low, medium, and high. To minimize potential on site risks, Weston has outlined operational procedures that outline the risks and designate protective measures to help eliminate the risks. Operational Procedures are attached for all risks that are expected to be encountered.

#### Level of Protection for Tasks Level

Task 1	Groundwater Sampling/Water Levels Previous sampling has indicated no readings in the breathing zone	D
Task 2	Lagoon Surface Water and Sediment Sampling No risk of airborne particulates and skin contact is minimal	D
Task 3	Test Pits Air will be monitored in the breathing zone if above 5 ppm in the breathing zone, shut down of work and upgrade to Level C	D
Task 4	Soil Sampling No risk of airborne particulates and skin contact is minimal	D

#### 4.4.3 Task-By-Task Risk Assessment

## Task 1: Groundwater Sampling/Water Level Measurements

<u>Physical Risks:</u> (Low)	Lifting hazards; associated with the lifting of pumps
	and generators (proper lifting techniques will be
	used).
	Slip, trip, and fall; some work areas have uneven
	footing (good housekeeping will be used).
	Hand tools; used during sampling create pinch
	points.

Chemical Risks: (Low)	Exposure to groundwater during the pumping and the sampling of the wells poses the risk of skin contact or ingestion. Body cover (i.e., coveralls or tyvek) will be used when appropriate.
Biological Risks: (Low)	Exposure to rodents, snakes, ticks and insects create a slight risk in this phase.

# Task 2: Lagoon Sampling - Sediment and surface water sampling

Physical Risks: (Low)	Since sampling will occur from a rowboat on a shallow lagoon, a risk of drowning is present. Life preservers will be used.
Chemical Risks: (Low)	Very low VOC levels are in the surface water of lagoon. It has already been treated prior to entering lagoon.
Biological Risks: (Low)	N/A

## Task 3: Test Pits - Excavating of shallow test pits

Physical Risk: (Medium)	Risks associated with the use of heavy machinery, collapse of test pits, underground utilities.
Chemical Risk: (Low)	Skin contact will be minimal. Areas of test pitting will be monitored using air monitoring equipment during the excavations.
Biological Risk: (Low)	Heavy machines will clear areas of brush prior to the beginning of test pitting. Exposure to rodents, snakes and insects should be minimal. Gloves and body cover will be used to shield workers from poison ivy.

## Task 4: Soil Sampling - Surface soil samples

<u>Physical Risk:</u> (Low) No heavy machinery or power tools used. Just slip, trip and fall.

<u>Chemical Risk:</u> (Low)	Skin contact will be minimized by the use of gloves. Should be no airborne particles.
Biological Risk: (Low)	Exposure to rodents, snakes and insects should be minimal. Gloves and body cover will be used to shield workers from poison ivy.

## 4.4.4 Description of Level of Protection

#### Level D

#### Level C

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- Head
   hard hat when test pitting
- Protective Goggles
   when risk of splash
- Hand
   gloves
- Foot - steel-toe safety boots

- Head - hard hat
- Eye and face - full face respirator with GMCH cartridge
- Hand
  - inner surgical gloves
  - outer nitrile gloves
  - Foot
    - inner steel-toe safety boots
    - latex overboots
  - Body - tyvek oversuit

## 4.5 EMERGENCY CONTACTS

## 4.5.1 Emergency Information

In case of an emergency, the following information should be utilized. Contacts have been provided to cover health emergencies, fire emergencies, and chemical emergencies.