

REQUIREMENTS FOR QUALITY CONTROL OF ANALYTICAL DATA

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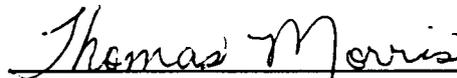

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LIST OF ACRONYMS

AA	atomic absorption
AESG	Analytical Environmental Support Group
AQCS	Analytical Quality Control Specialist
ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing and Materials
CCC	calibration check compounds
CCV	calibration check verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain of custody
CRP	Community Relations Plan
CRQL	contract required quantitation limits
CVAA	cold-vapor atomic absorption
%D	percent difference
DBC	dibutylchloroendate
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenylethane
DDT	dichlorodiphenyltrichloroethane
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
GFAA	graphite furnace atomic absorption
HASP	Health and Safety Plan
HAZWRAP	Hazardous Waste Remedial Actions Program
HPLC	high-pressure liquid chromatography
ICP	inductively coupled plasma
ICV	initial calibration verification
IDL	instrument detection limit
IS	internal standard
LCS	laboratory control sample
LQAC	Laboratory Quality Assurance Coordinator
LQAP	Laboratory Quality Assurance Plan
MPR	Monthly Progress Report
MS/MSD	matrix spike/matrix spike duplicate

LIST OF ACRONYMS (continued)

NCP	National Contingency Plan
NPDES	National Pollution Discharge Elimination System
NPL	National Priorities List
OSWER	Office of Solid Waste and Emergency Response
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PCB	polychlorinated biphenyl
PE	Performance Evaluation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
%R	percent recovery
RAP	Remedial Actions Planning
RRF	relative response factor
RRF	average relative response factor
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SCO	Support Contractor Office
SOP	standard operating procedure
SOW	statement of work
SPCC	system performance calibration compounds
TCL	target compound list
TIC	tentatively identified compounds
VTSR	verified time of sample receipt
XRF	X-ray fluorescence

1. INTRODUCTION AND PURPOSE

The National Contingency Plan (NCP) of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) provides procedures for the identification, evaluation, and remediation of past hazardous waste disposal sites. The Hazardous Materials Response section of the NCP consists of several phases: Preliminary Assessment, Site Inspection, Remedial Investigation, Feasibility Study, Remedial Design, and Remedial Action. During any of these phases, analysis of soil, water, and waste samples may be performed. The Hazardous Waste Remedial Actions Program (HAZWRAP) is involved in performing field investigations and sample analyses pursuant to the NCP for the U.S. Department of Energy and other federal agencies.

The purpose of this document is to specify the requirements of Martin Marietta Energy Systems, Inc., for the control of accuracy, precision, and completeness of samples and data from the point of collection through analysis. Requirements include data reduction and reporting of resulting environmentally related data. Because every instance and concern may not be addressed in this document, HAZWRAP subcontractors are encouraged to discuss any questions with the Analytical Quality Control Specialist (AQCS) and the HAZWRAP Project Manager. This revision supercedes all other versions of this document.

1.1 SCOPE

The requirements of this document apply to HAZWRAP subcontractors and their selected analytical laboratory(s) in conduct of the remedial response actions process.

Laboratories performing studies in support of HAZWRAP are required to pass HAZWRAP review before beginning field studies or analyses of samples and to maintain active status throughout duration of the studies. This document provides the requirements that laboratories must follow to pass review and maintain active status. Should more than one laboratory be involved in the analysis of samples from a single site, each laboratory performing analysis must undergo review and must comply with the quality control (QC) requirements specified in this document. These objectives and requirements conform, in general, with "Toxic Substances Control; Good Laboratory Practice Standards; Final Rule," U.S. Environmental Protection Agency, *Federal Register*, Vol. 48, November 29, 1983; "Nonclinical Laboratory Studies; Good Laboratory Practice Regulations," the Food and Drug Administration, *Federal Register*, Vol. 43, December 22, 1978; and "Quality Assurance Program Requirements for Nuclear Facilities," American National Standards Institute/American Society of Mechanical Engineers NQA-1, 1986 ed. Individual projects shall also comply with the "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," U.S. Environmental

Protection Agency, EPA-600/4-83-004, QAMS-005/80, February 1983 ed.; and *Quality Control Requirements for Field Methods*, DOE/HWP-69/R1, July 1990.

Each laboratory is required to submit a Laboratory Quality Assurance Plan (LQAP) through the HAZWRAP subcontractor. The LQAP is emphasized because review of, and adherence to, its contents are essential for obtaining and maintaining HAZWRAP active status. Certain basic requirements stressed are a Laboratory Quality Assurance Coordinator (LQAC); the use of accepted analytical methods; careful documentation of chain-of-custody (COC) forms; a corrective action policy; submission of Monthly Progress Reports (MPRs), and use of control charts. The laboratory review process and subsequent laboratory reporting requirements provide the mechanism for verifying that a laboratory is adhering to the LQAP.

1.2 APPROACH

The approach reflected in this document is one of outlining requirements and allowing the laboratories, principally through their LQAP, to detail their approach to meeting these requirements. For example, with the exception of the Laboratory Control Sample (LCS) program (see Sect. 5.1.9), the discussion of QC procedures includes a requirement that warning and control limits be set but allows each laboratory to describe its procedures for establishing such limits. The specific organization and presentation of the LQAP are left largely to the discretion of the laboratory, although certain areas must be addressed.

For this approach to work, emphasis will be placed on effective communication between the laboratory, the HAZWRAP Project Manager, the AQCS, and the subcontractor. All documents will be concise, well organized, and free of jargon that might hinder constructive review and evaluation.

2. ROLES AND RESPONSIBILITIES

As indicated in Fig. 2.1, organizations involved in QC of analytical data are the sponsor, HAZWRAP, and the subcontractors. Each organization has multiple tasks and groups that support the project. Fig. 2.1 includes the structure of the HAZWRAP organization relative to the Remedial Actions Planning (RAP) process. A brief description of key roles and responsibilities is listed below.

2.1 SPONSOR

The sponsor is responsible for project funding; providing the site information, history, and logistical assistance; specifying the site(s) that requires investigation; and reviewing results and making recommendations.

2.2 HAZWRAP PROJECT MANAGER

The HAZWRAP Project Manager is responsible for managing (organizing, coordinating, directing, and controlling) all activities concerned with planning and executing the project to meet project cost, schedule, and technical and quality objectives.

Specific responsibilities include:

- o Identifying project team members by requesting personnel support from the respective Support Contractor Office (SCO) program functions.
- o Planning and directing the collective actions of assigned team members and the SCO subcontractor to meet project objectives.
- o Identifying project requirements and developing project work plans to meet requirements.
- o Defining work, assigning responsibilities, and holding functional elements responsible for specific tasks or objectives.
- o Implementing project requirements and integrating project technical and programmatic activities with the project team.

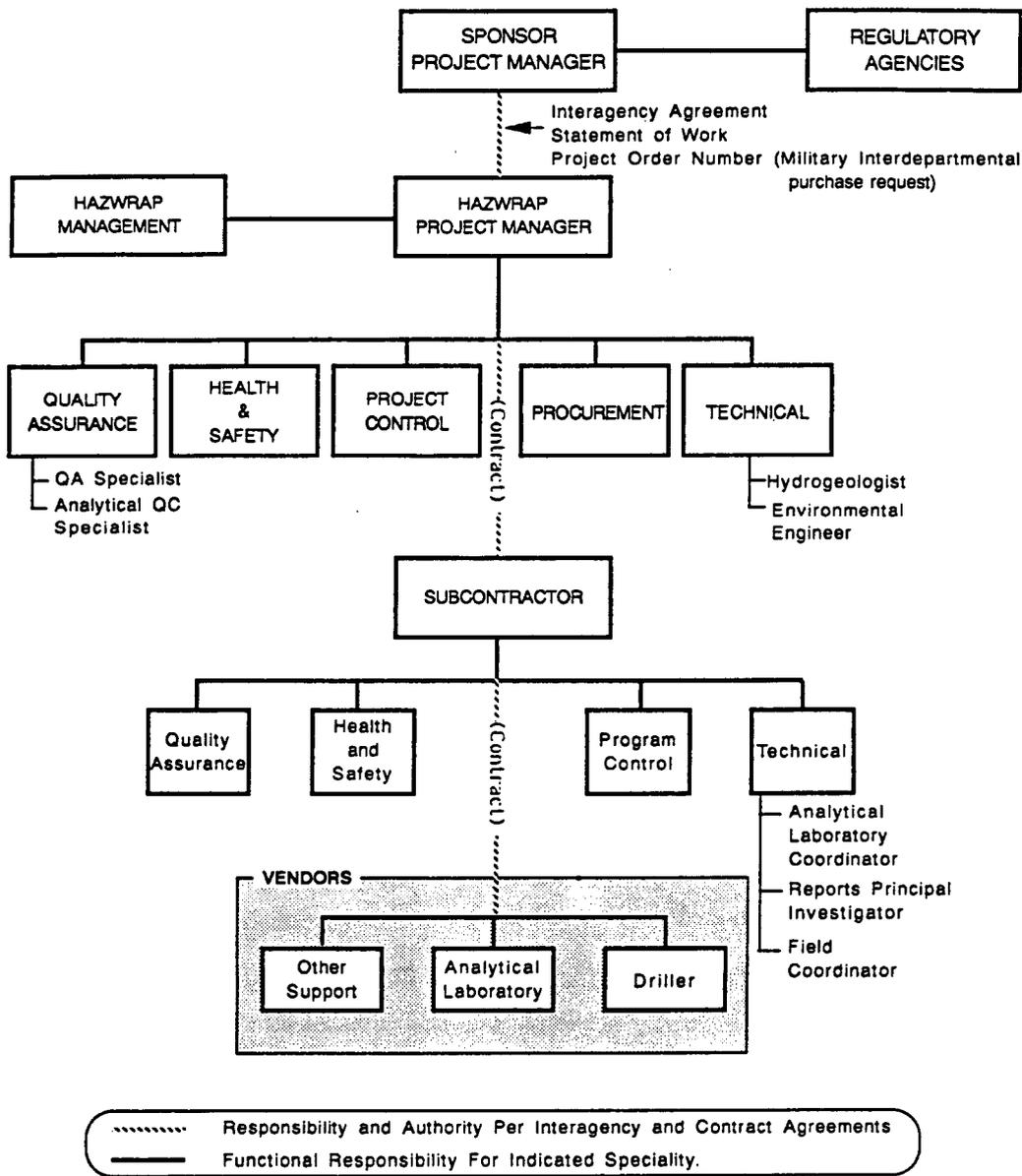


Fig. 2.1. Hazardous Waste Remedial Actions Program (HAZWRAP) Remedial Actions Planning Project team functional organization. (QA = quality assurance, QC = quality control.)

- o Developing a procurement strategy and the statements of work (SOWs) in concert with the sponsor, defining contracts, and monitoring contract negotiations in concert with the responsible procurement contracting officer (buyer).
- o Controlling the project to ensure successful achievement of objectives.
- o Conducting project reviews and preparing monthly status reports.
- o Evaluating quality performance data from quality investigations, audits, and reviews related to the project on a periodic basis. Tracking reports on conditions adverse to quality, reviewing corrective actions, and tracking completion.
- o Coordinating project activities and interfacing with the sponsor.
- o Ensuring that project team comments to project documents are addressed and mutually resolved.

2.3 HAZWRAP QUALITY ASSURANCE SPECIALIST

The HAZWRAP Quality Assurance Specialist (QA Specialist) works with the HAZWRAP Project Manager to ensure that project plans and necessary actions are taken to provide confidence that project objectives are met. The QA Specialist is responsible for ensuring that items and services are defined and executed in accordance with applicable policies and directives.

Specific responsibilities include:

- o Advising the HAZWRAP Project Manager and project team members on QA matters.
- o Ensuring that requirements delineated in *HAZWRAP SCO Implementation Plan, Quality Assurance Requirements*, DOE/HWP-38, November 1987, are effectively implemented.
- o Ensuring, through the subcontractor, that the Quality Assurance Project Plan (QAPP) is adequately developed and effectively implemented.
- o Identifying project QA requirements, preparing QA procedures, and assisting in the development of other implementing instructions, as required.
- o Participating in the development, review, and approval of quality requirements contained in program procurement documents and other program documentation, as required.

- o Assisting in the identification of problems concerning the project or for unique project actions/events. Taking actions, as assigned, to eliminate or minimize potential problems (risk management).
- o Reviewing and providing comments on program documentation, such as SOWs, work plans, subcontractor proposals, and other project deliverables.
- o Reviewing and commenting on subcontractor work plans, LQAPs, standard operating procedures (SOPs), and other related documents and reports.
- o Evaluating quality performance data from quality investigations, audits, and reviews related to the project on a periodic basis. Tracking reports on conditions adverse to quality, reviewing corrective actions, and tracking completion.
- o Conducting surveillances (audits/reviews) of subcontractor activities to determine compliance to QA requirements and associated procedures.
- o Preparing the project audit schedule in concert with the AQCS, obtaining approval from the HAZWRAP Project Manager, and assisting in planning, conducting, and reporting QA reviews/audits and follow-up activities, as required.
- o Conducting quality investigations and participating in the preparation of the corrective action plans accordingly.
- o Providing QA training for project personnel.
- o Defining project QA documents/records in concert with the HAZWRAP Project Manager and maintaining project QA files.
- o Coordinating all project QA activities and interfacing with sponsor and regulatory agency counterparts.

2.4 ANALYTICAL QUALITY CONTROL SPECIALIST

The AQCS is responsible to the HAZWRAP Project Manager for ensuring that appropriate project QC requirements for data quality are identified and that data quality meets contractual requirements. This function is being filled by the Analytical Environmental Support Group (AESG), Sampling and Environmental Support Department, Oak Ridge K-25 Plant.

Specific responsibilities include:

- o Advising the HAZWRAP Project Manager and project team members on QC matters concerning quality of environmentally related measurement data.
- o Developing QC requirements for sample collection, sample analysis, and data reporting.
- o Preparing QC procedures and assisting in the development of other procedures, as required.
- o Participating in the development, review, and approval of QC requirements contained in program and project documents relative to the control of data quality. Reviewing and providing comments on program documentation, such as SOWs and subcontractor proposals. Reviewing and commenting on subcontractor work plans, LQAPs, associated procedures, and related documents and reports.
- o Evaluating quality performance data relative to the QC of environmentally related measurements, reporting trends, and ensuring that corrective action is reviewed and tracked to completion.
- o Planning QC reviews of subcontractor sampling, analysis, and data reporting activities and follow-up QC reviews, as required.
- o Conducting and reporting RAP site surveillance of subcontractor activities concerning sampling.
- o Auditing the subcontractor's selected analytical laboratory to determine compliance with QA requirements, including functional and programmatic procedures and instructions concerning quality of environmentally related measurement data.
- o Assisting in conducting SCO quality investigations, as assigned, and participating in the preparation of corrective action plans accordingly.
- o Providing information to the project QA Specialist for preparation of the project surveillance schedule.
- o Providing QC training for assigned project personnel.
- o Maintaining QC files, records, and documents.
- o Assisting in the identification of potential problems for the project or for unique project actions/events. Taking actions, as assigned, to eliminate or minimize potential problems.
- o Attesting that measurement data meet the project Data Quality Objectives (DQOs).

2.5 HAZWRAP TECHNICAL SUPPORT

HAZWRAP Technical Support consists of individuals responsible for providing technical direction and support to the Project Manager in specified areas such as hydrogeology, risk assessment, environmental engineering, and toxicology. This support is also responsible for managing the specified technical function to meet project objectives.

Specific responsibilities include:

- o Ensuring that technical objectives are identified and achieved.
- o Maintaining detailed knowledge of technical problems.
- o Developing technical specifications and defining technical requirements.
- o Reviewing and commenting on project technical documentation prepared by the subcontractor for accuracy and adequacy.
- o Assisting in development of the project work plan.
- o Developing technical sections of project documentation, such as SOWs and work plans.
- o Providing technical support to project audit teams, as requested.
- o Assisting in the identification of problems and taking actions, as assigned, to eliminate or minimize potential problems (risk management).

2.6 SUBCONTRACTOR

Subcontractors are responsible for providing specified technical support to the HAZWRAP Project Manager. Responsibilities will vary, based on the specific remedial actions process phase being addressed and the sponsor's project needs. The subcontractor's role in the project will be defined in the planning process.

Specific responsibilities include:

- o Identifying problems and initiating the implementation of corrective actions, if required (risk management).

- o Implementing work plan specifications.
- o Producing technical and project status reports.

2.7 ANALYTICAL LABORATORY

The analytical laboratory is employed by the subcontractor and must adhere to the laboratory requirements in this document.

Specific responsibilities include:

- o Preparing and submitting an LQAP.
- o Analyzing and submitting results for Performance Evaluation (PE) samples.
- o Submitting to on-site laboratory audits.
- o Correcting any deficiencies cited in the LQAP, PE sample review, and laboratory audit.
- o Identifying an LQAC responsible for overall QA. The LQAC position must fulfill the following requirements.
 - Provides reports to the laboratory director.
 - Is independent of project cost or profit responsibilities, schedules, or personnel, other than QA assistants.
 - Has the authority to stop work, if QC problems arise affecting the quality of data produced.
- o Submitting MPRs to the AQCS to maintain an active status in the program (see Sect. 5.4.1 for further information).
- o Adhering to specific project QA plan requirements. The laboratory should have input into the development of these plans.

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3. LEVELS OF QUALITY CONTROL

3.1 DATA QUALITY OBJECTIVES

DQOs are statements of the uncertainty level a decision maker is willing to accept in results derived from environmental data. As such, they are a management tool used to limit the chance of data leading to an incorrect conclusion. DQOs must strike a balance between time, money, and data quality. The DQO process must be initiated during project planning to produce work plans resulting in data that have a quantifiable degree of certainty. The end use of data to be collected and the cost to produce that data will determine the required DQOs.

DQOs are specified in documents, such as the Sampling and Analysis Plan (SAP) and the QAPP. Five general levels of analytical options to support data collection are identified by CERCLA and have been adopted by HAZWRAP to define QC requirements. HAZWRAP QC Levels are A, B, C, D, and E, which correlate with Levels 1, 2, 3, 4, and 5 as described by the U.S. Environmental Protection Agency (EPA) document, "Data Quality Objectives for Remedial Response Activities Development Process," March 1987, Office of Solid Waste and Emergency Response (OSWER) Directive 9355.0-7B. These levels are based on the type of site to be investigated, the level of accuracy and precision required, and the intended use of the data. The level of QC required at the site will be decided by the HAZWRAP Project Manager in concert with the sponsor. Table 3.1 outlines DQO levels, along with HAZWRAP QC levels. Table 3.2 outlines the basic HAZWRAP QC requirements for each level. Laboratory method requirements for each level of QC are outlined in Sect. 5.1.2. QC requirements regarding performance sample analysis, laboratory audits, LQAP approval, and work plan review do not change with the level. NOTE: Levels of QC are for individual measurement activities, and more than one level may be used at a given site.

3.2 LEVELS A AND B QUALITY CONTROL

QC Levels A and B have been specified as the criteria to be used with field instruments. The level of QC required for field analysis will be decided by the HAZWRAP Project Manager, the sponsor, the subcontractor, the hydrogeologist, and the AQCS.

Applications of Levels A and B are based on the intended use of the data as stated in the site-specific project work plan. Data usability is most often restricted by instrument limitations. There are two basic types of field instruments: (1) nonquantitative and semiquantitative screening instruments (e.g., total organic vapor meters, colorimetric indicator tubes, pH indicator paper, etc.) and (2) quantitative instruments that measure specific analytes, but often with less sensitivity than conventional laboratory units [e.g., portable gas chromatographs and portable

Table 3.1. Summary of analytical levels appropriate to data uses

Examples of data uses	DQO level	HAZWRAP QC level	Examples for use by HAZWRAP
Site characterization Monitoring during implementation Field screening	I	A	Qualitative or semiquantitative analysis Indicator parameters Immediate response in the field
Site characterization Evaluation of alternatives Engineering design Monitoring during implementation Field screening	II	B	Semiquantitative or quantitative analysis Compound specific Rapid turnaround in the field
Risk assessment Site characterization Evaluation of alternatives Engineering design Monitoring during implementation	III	C	Quantitative analysis Technically defensible data Sites near populated area Major sites
Risk assessment Site characterization Evaluation of alternatives Engineering design	IV	D	Quantitative analysis Legally defensible data National Priorities List sites
Risk assessment Evaluation of alternatives Engineering design	V	E	Qualitative to quantitative analysis Method specific Unique matrixes (i.e., pure waste, biota, explosives, etc.)

Note: DQO = Data Quality Objectives, HAZWRAP = Hazardous Waste Remedial Actions Program, QC = Quality Control.

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Table 3.2. Quality control level vs requirement summary

QC level	<u>Laboratory review requirements</u>			Methods	Deliverables	Review/validation
	QAP	PE	Audit			
A	No	No	HAZWRAP optional	Refer to DOE/HWP-69/R1 Sect. 8.1	Refer to this report Sect. 5.4.2	Refer to this report Sect. 6.1
B	Yes	HAZWRAP optional	HAZWRAP optional	Refer to DOE/HWP-69/R1 Sect. 8.2	Refer to this report Sect. 5.4.2	Refer to this report Sect. 6.1
C	Yes	Yes	Yes	Refer to this report Sect. 3.3	Refer to this report Table 5.1	Refer to this report Sect. 6.2
D	Yes	Yes	Yes	Refer to this report Sect. 3.4	Refer to this report Sect. 5.4.2	Refer to this report Sect. 6.3
E	Yes	HAZWRAP optional	HAZWRAP optional	Refer to this report Sect. 3.5	Refer to this report Sect. 5.4.2	Refer to this report Sect. 6.4

Note: QAP = Quality Assurance Plan, PE = Performance Evaluation Sample.

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X-ray fluorescence units (XRF)]. Nonquantitative and semiquantitative equipment will always be governed by Level A criteria. Quantitative field instruments are usually governed by Level B criteria; however, Level A criteria may be sufficient. The level will be determined by the end use of the data.

Neither Level A or B data alone can be used to dismiss a site. A representative percentage of all field sample results must be confirmed by sample analyses at Level C or D and must be supported by risk assessment. The number of sample analyses required by a nonfield laboratory will vary by site. The project work plan must define the number or percentage of samples to be submitted for confirmation analysis. This is mandatory; confirmation is required regardless of whether field results are positive or negative. Applicable or Relevant and Appropriate Requirements (ARARs) are often below the lower detection limits of field instruments; confirmation, therefore, is required to ensure that negative results are below the ARARs. Levels A and B QC are more fully explained in the HAZWRAP document, *Quality Control Requirements for Field Methods*, DOE/HWP-69/R1, July 1990.

3.2.1 Level A Quality Control

Data meeting Level A criteria are qualitative or semiquantitative in nature and are used as indicator parameters. Data are obtained by use of approved field equipment, such as total organic vapor analyzers, colorimetric indicator kits, dissolved oxygen meters, and geophysical survey instruments. Other instruments and methods may be used, if approved by the HAZWRAP Project Manager.

Equipment capability, or the analytical QC implemented, will limit data obtained to qualitative, or at best, semiquantitative. Quantitative data are not obtained on an analyte-specific basis. Level A data may be used for the following: (1) delineation of contaminated zones, (2) gross determination of analytes in samples, or (3) health and safety screening. Level A data can provide information to the in-house laboratory regarding expected concentration ranges. This information can assist the laboratory in determining applicable analytical ranges. For more information on Level A QC, see DOE/HWP-69/R1, Sect. 8.1.

3.2.2 Level B Quality Control

Level B is also used in field screening QC. It is, however, more quantitative than Level A. Level B QC will generally apply to on-site field laboratories conducting quantitative analyses for rapid turnaround. Level B field instruments are more compact and rugged than traditional laboratory units. Most field instruments, however, are less sensitive than traditional laboratory instruments. Quantitative field instruments, which are designed for in situ measurements and do not require field laboratory support, are also governed by Level B protocols.

NOTE: Field laboratories can be designed to obtain Level C or D data. When generating

level C or D quality data, laboratories must meet all requirements as defined in this document, including undergoing the laboratory review process as defined in Sect. 5.2. Similarly, data obtained from instruments with quantitative capabilities may be employed for Level A, depending on the proposed use.

The QC level required must be determined before sampling and analysis begin. All analyses must meet requirements defined by the applicable QC levels.

Data from Level B are used for site characterization, evaluation of alternatives, engineering design, and monitoring during implementation or sampling. For more information on Level B QC, see DOE/HWP-69/R1, Sect. 8.2.

3.3 LEVEL C QUALITY CONTROL

Level C QC would be required at a site near a populated area, not on the National Priorities List (NPL), and not likely to be undergoing litigation. Level C QC includes review of the LQAP and project work plan - including the SAP and QAPP. The laboratory shall successfully analyze a PE sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QC.

It is suggested that most laboratory soil and water analyses be performed using Level C. Level D should only be used at NPL sites, as required by regulators, or at sites where legal action is pending. At many sites, Level C laboratory data confirming the field screening data will be sufficient.

Level C provides low detection limits, a wide range of calibrated analytes, matrix recovery information, laboratory process control information, and known precision and accuracy. EPA-accepted methods, such as those in SW-846, the National Pollution Discharge Elimination System (NPDES), and the Contract Laboratory Program (CLP), are utilized under Level C. Reference to the CLP forms later in this document is provided as an example of the type of information required from the laboratory. Since these forms are commonly used, are computerized, and present information essential to review data quality, they are referenced. If the laboratory chooses to present the same information in a different format, with non-CLP methods, such as SW-846 or NPDES, this is acceptable. Level C is not exclusively CLP. CLP methods are allowed but not required. **VOLATILE AND SEMIVOLATILE ORGANIC ANALYSES BY GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS), HOWEVER, MUST BE PERFORMED BY THE MOST CURRENT CLP METHODS.**

Advantages of Level C QC are (1) greater precision and accuracy than Levels A and B and (2) more established and documented QC. Level C can be used for risk assessment, while

Levels A and B cannot. A disadvantage is the time required to obtain data (typically 20 to 30 days).

These data may be used for risk assessment, site characterization, evaluation of alternatives, engineering design, and monitoring during implementation.

3.4 LEVEL D QUALITY CONTROL

Level D QC is used when comprehensive data quality documentation is required. Typically, this level is needed for select samples at NPL sites. These sites are typically near populated areas and are likely to undergo litigation.

Level D QC includes review of the LQAP and project work plan, including the SAP and QAPP. The laboratory shall successfully analyze a PE sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QC.

For Level D, CLP methods and full data package deliverables are required for analytes covered by these methods. Methods not included in the CLP will be elevated to Level D by including appropriate QC samples and submitting all raw sample and calibration data.

An advantage of Level D QC is that methods are accepted by all EPA states, regions, and courts. The methods provide the most documented information on matrix effects and on precision and accuracy of all environmental methods. Methods are detailed; therefore, more consistency between laboratories is observed. Because raw sample data, calibration, and QC documentation are presented, the reviewer can fully assess data quality. Disadvantages are 30- to 40-d turnaround, large quantities of data for storage and review, and higher costs.

These data may be used for risk assessment, site characterization, evaluation of alternatives, engineering design, and monitoring implementation.

3.5 LEVEL E QUALITY CONTROL

Level E QC is used for analysis of nonstandard sample matrixes, such as air, biota, and pure waste. It may also be employed for nonstandard methods, such as explosives. Level E QC is also appropriate for analysis of the contents of underground storage tanks, where samples are primarily pure product or waste. This document defines the minimum QC requirements for

Level E; however, specific QC requirements may vary between sites. Specific QC requirements must be clearly and completely identified in the project work plan when Level E is employed.

Level E QC includes review of the LQAP and project work plan, including the SAP and QAPP. If requested by HAZWRAP, the laboratory shall successfully analyze a PE sample, undergo an audit, and correct deficiencies found during the audit. The laboratory shall provide MPRs on QC. Because few methods are available for nonstandard matrixes, the methods to be used for Level E analysis must be submitted to the HAZWRAP Project Manager for review and approval before the initiation of work.

The major disadvantage of Level E QC is that frequently methods must be developed along with precision and accuracy information. Method development is often time consuming and costly.

These data may be used for risk assessment, site characterization, evaluation for alternatives, engineering design, and monitoring implementation.

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4. PROJECT REQUIREMENTS

Once the project team (as described in Sect. 2) is assembled, the project moves through an orderly series of events toward successful completion. Some of these events, related to analytical QC requirements, are addressed in this section.

4.1 IDENTIFICATION OF DATA QUALITY OBJECTIVES

One of the first and most important decisions is the identification of DQOs. Directions for this decision can be found in "Data Quality Objectives for Remedial Response Activities, Development Process," March 1987, OWSER Directive 9355.0-7B.

DQOs are developed through a three-stage, interactive, continuous-thought process. The first stage is to identify suspected sources, contaminant pathways, and potential receptors and to use this information to specify decisions. The second stage is to identify data uses/needs and to use this information in the selection of sampling approaches and analytical options for the site. This decision is weighed against the cost and time of data collection evaluation. The third stage is to design the data collection program so that data of acceptable quantity and quality will be generated from which decisions can be made.

There are many considerations to make during this development process. Some of these considerations are as follows:

- o data uses/needs,
- o risk assessment needs,
- o data quality needs,
- o cost,
- o time,
- o statistical considerations,
- o analytical methods,
- o sampling considerations,
- o enforcement concerns, and
- o potential ARARs.

After DQOs are defined and the QC level is selected, work on the project can proceed.

4.2 DEVELOPMENT AND APPROVAL OF THE PROJECT WORK PLAN

The project work plan documents decisions and evaluations made during the scoping process and provides a general framework for addressing identification and subsequent actions for installations suspected of having environmental contamination problems. The objective of the plan is to provide a detailed technological structure for addressing identified sites and for accumulating data of sufficient quantity to at least support completion of a final report. Data collected must be of sufficient quality to support future project planning and to support the necessary activities associated with the chosen approach, including a risk assessment and/or decision document. Guidance for preparing work plans may be found in the document, "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA," Interim Final, October 1988, OSWER Directive 9355.3-01. Work plans usually consist of the following:

- o Work Plan - Provides an overall technical strategy and management approach for completing investigation of the sites.
- o SAP - Consists of two parts: (1) the Field Sampling Plan (FSP) that provides guidance for all fieldwork by defining, in detail, the sampling and data-gathering methods to be used and (2) the QAPP that describes the policy, organization, functional activities, and QA/QC protocols necessary to achieve the DQOs dictated by the intended use of data.
- o Health and Safety Plan (HASP) - Identifies site-specific measures for ensuring worker health and safety. This document must conform to the subcontractor's health and safety program, which must be in compliance with the Occupational Safety and Health Act.
- o Community Relations Plan (CRP) - Documents the community relations history and the issues of community concern.

These plans may be submitted as a single document, although they are more easily used in the field if bound separately.

Requirements for each plan are described herein. Other requirements may be imposed by the HAZWRAP Project Manager, based on site needs.

4.2.1 Suggested Work Plan Format

Executive Summary -- A synopsis or summary of the project scope, allowing the reader to obtain a broad perspective of major document content attributes.

Introduction -- A general explanation of the reason for the plan and the expected results or goals for the entire process.

Site Background and Setting -- A description of the current understanding of the physical setting of the site, including surrounding land and water uses, site history/background, and existing information on the site condition. This document also identifies the sites and includes maps, photographs, and/or drawings.

Initial Evaluation -- The conceptual site model, developed during the scoping process, including:

- o information from previous record searches and/or sampling rounds,
- o types and volumes of wastes present,
- o a rationale for analytical methods selected,
- o intended use of data collected,
- o preliminary identification of ARARs,
- o preliminary risk assessment, and
- o site geology and hydrogeology.

Rationale -- A description and presentation of the work plan approach, identification of data needs and DQOs with QC levels, and an explanation of how the samples selected for analysis will be determined.

Tasks -- A definition of the scope and objectives of various tasks, to the extent possible.

Costs and Key Assumptions -- A detailed summary of projected labor and expense costs and a description of the key assumptions required to make such a cost estimate. In the HAZWRAP program, this section is included in the Business Proposal.

Schedule -- An anticipated schedule formulated on the basis of the project scope, including identification of key activities and deliverable dates.

Project Management -- A description of relationships and responsibilities for selected task and project management items. The following considerations should be discussed:

- o staffing,
- o coordination,
- o interfaces,
- o potential problem identification, and
- o training.

References -- Any published materials used to support the information in the report.

Appendixes -- Additional supporting information, as required.

4.2.2 Suggested Sampling and Analysis Plan Format

The format for Part 1, the FSP, of the SAP follows.

Site Background -- A summary of existing data, a description of the site and surrounding areas, a discussion of known and suspected contaminant sources, and a listing of probable transport pathways.

Sampling Objectives -- A description of intended data uses.

Sampling Location and Frequency -- Identification of each sample matrix to be collected, identification of the constituents to be analyzed, maps and/or drawings identifying the location of sampling points, and summary tables showing numbers of samples by matrixes and sites.

Sample Designation -- A description of the sample numbering system.

Sampling Equipment and Procedures -- A description of sampling procedures, including equipment to be used and material composition of the equipment; a detailed description of decontamination procedures; a discussion of mobilization and demobilization; a detailed description of, and procedures for, field screening methods, including preventive maintenance; a discussion of surveying wells and sampling points; a detailed description of water-level measurement procedures; a detailed description of borehole and well drilling methods; a detailed description of piezometer and monitoring well installation procedures, construction design, and materials; and a detailed discussion of well development and purging methods.

Sample Handling and Analysis -- Identification of sample holding times, preservation methods, types of sample containers, and volumes of samples to be collected; shipping requirements and procedures; chain-of-custody procedures; disposal of wastes generated; and a discussion of field logbooks/forms/notebooks, including how to complete them and how they are controlled.

The format for Part 2, the QAPP, of the SAP follows.

Title Page -- Page for signatures of approval personnel, including the subcontractor project manager and QA manager.

Table of Contents -- Outline of report.

Project Description -- A general site history, objectives of the investigation, and the site description.

Project Organization and Responsibilities -- Organizational chart, identifying key personnel and organizations and responsibilities of key personnel.

Quality Assurance Objectives for Measurement -- Intended data use; a listing of method detection limits; a table of QC samples (duplicates, trip blanks, field blanks, and equipment rinseates) vs the number of samples by method and matrix (include extra sample volumes for QC samples); a detailed discussion of DQOs, including how they will be implemented; and a table, broken down by site, showing the analysis method, method number, sample media, DQO level, and number of samples.

Sampling Procedures -- A description of sampling procedures; a discussion of the cleaning/preparation of sample containers; a description of sample preservation techniques and holding times; a discussion of field logbooks/forms/notebooks; and a discussion of material blanks, materials certification, and readiness review.

Sample Custody -- Chain-of-custody procedures.

Calibration Procedures -- Written field calibration procedures, including frequency of calibration, source of calibration standards, and calibration acceptance criteria; a detailed discussion of accuracy and precision of field instruments; and a detailed discussion of the field data evaluation process.

Analytical Procedures -- Tables of analyses method numbers and numbers of analyses per matrix for each site and the name of the analyte list and a list of analytes for multianalyte methods.

Data Reduction, Validation, and Reporting (these are subcontractor responsibilities) -- The principal criteria used to validate data, a detailed discussion of data handling and reduction procedures, methods for evaluation of blanks, and QC acceptance criteria.

Internal Quality Control -- Discussion of matrix spike/matrix spike duplicates (MS/MSD); field duplicates, field blanks, trip blanks, equipment rinseates, surrogates; and identification of ways in which the QC information will be used to qualify data.

Performance and Systems Audits -- A discussion of performance and system audits to be performed.

Preventive Maintenance -- Discussion of preventive maintenance, including critical spare parts.

Data Assessment Procedures -- Discussion of precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters and statistical applications of data (subcontractor responsibility).

Correction Actions -- A discussion of corrective action procedures, including field changes and responsibilities for corrective actions, and a discussion of out-of-control conditions reporting and follow-up procedures.

Quality Assurance Reports -- Results of audits, significant QA problems encountered, and recommended solutions; a discussion of project deliverables, including laboratory deliverables, MPRs, and the final report and its contents; a summary of final data quality; and summary tables of the data. (See Table 4.1 for the format to be followed.)

4.2.3 Suggested Health and Safety Plan Format

Specific requirements for a site HASP are listed in 29 CFR 1910.120, "Hazardous Waste Operations and Emergency Response," *Federal Register*, Vol. 54, No. 42, March 6, 1989. Each site HASP must include, at a minimum, the following 11 elements:

1. The name of a site health and safety officer and the names of key personnel and alternates responsible for site safety and health.
2. A health and safety risk analysis for existing site conditions and for each site task and operation.
3. Employee training assignments.
4. A description of personal protective equipment to be used by employees for each of the site tasks and operations being conducted.
5. Medical surveillance requirements.
6. A description of the frequency and types of air and personnel monitoring and environmental sampling techniques and instrumentation to be used.
7. Site control measures.
8. Decontamination procedures.
9. SOPs for the site.
10. A contingency plan that meets the requirements of 29 CFR 1910.120(I)(1) and (I)(2).
11. Entry procedures for confined spaces.

Table 4.1. Example of data summary tables

Sample No.:	S3-MW1	S3-SS6-6.0'
Lab Sample No.:	890321-11	890321-17
Matrix:	Water	Soil
Associated Samples:	TB-3	TB-7
	FB-2	FB-4
	ER-c	ER-9

<u>Volatile organics</u>	<u>µg/L</u>	<u>µg/Lg</u>
Chloromethane	10 U	10 UJ
Bromomethane	10 U	10 UJ
Vinyl chloride	10 U	10 UJ
Chloroethane	10 U	10 UJ
Methylene chloride	13 J	5 UJ
Acetone	36	26 J
Carbon disulfide	5 U	5 UJ
1,1-Dichloroethene	5 U	5 UJ
1,1-Dichloroethane	5 U	5 UJ
1,2-Dichloroethene (Total)	5 U	5 UJ
Chloroform	5 U	5 UJ
1,2-Dichloroethane	5 U	5 UJ
2-Butanone	6 J	10 UJ
1,1,1-Trichloroethane	5 U	5 UJ
Carbon tetrachloride	5 U	5 UJ
Vinyl acetate	10 U	10 UJ
Bromodichloromethane	5 U	5 UJ
1,2-Dichloropropane	5 U	5 UJ
cis-1,3-Dichloropropene	5 U	5 UJ
Trichloroethene	7	14 J
Dibromochloromethane	5 U	5 UJ
1,1,2-Trichloroethane	5 U	5 UJ
Benzene	5 U	5 UJ
trans-1,3-Dichloropropene	5 U	5 UJ
Bromoform	5 U	5 UJ
4-Methyl-2-pentanone	10 U	10 UJ
2-Hexanone	10 U	10 UJ
Tetrachloroethene	5 U	5 UJ
1,1,2,2-Tetrachloroethane	5 U	5 UJ
Toluene	5 U	5 UJ
Chlorobenzene	5 U	5 UJ
Ethylbenzene	5 U	5 UJ
Styrene	5 U	5 UJ
Xylene (Total)	5 U	5 UJ

Table 4.1 (continued)

Sample No.:	S3-MW1	S3-SS6-6.0'
Lab Sample No.:	890321-11	890321-17
Matrix:	Water	Soil
Associated Samples:	TB-3	TB-7
	FB-2	FB-4
	ER-6	ER-9

<u>Semivolatile organics</u>	($\mu\text{g/L}$)	($\mu\text{g/kg}$)
Phenol	10 U	330 U
bis(2-Chloroethyl)ether	10 U	330 U
2-Chlorophenol	10 U	330 U
1,3-Dichlorobenzene	10 U	330 U
1,4-Dichlorobenzene	10 U	330 U
Benzyl alcohol	10 U	330 U
1,2-Dichlorobenzene	10 U	330 U
2-Methylphenol	10 U	330 U
bis-(2-Chloroisopropyl)ether	10 U	330 U
4-Methylphenol	10 U	330 U
N-Nitroso-di-n-propylamine	10 U	330 U
Hexachloroethane	10 U	330 U
Nitrobenzene	10 U	330 U
Isophorone	10 U	330 U
2-Nitrophenol	10 U	330 U
2,4-Dimethylphenol	10 U	330 U
Benzoic acid	50 U	1600 U
bis(2-Chloroethoxy)methane	10 U	330 U
2,4-Dichlorophenol	10 U	330 U
1,2,4-Trichlorobenzene	10 U	330 U
Naphthalene	10 U	330 U
4-Chloroaniline	10 U	330 U
Hexachlorobutadiene	10 U	330 U
4-Chloro-3-methylphenol	10 U	330 U
2-Methylnaphthalene	10 U	330 U
Hexachlorocyclopentadiene	10 U	330 U
2,4,6-Trichlorophenol	10 U	330 U
2,4,5-Trichlorophenol	50 U	1600 U
2-Chloronaphthalene	10 U	330 U
2-Nitroaniline	50 U	1600 U
Dimethylphthalate	10 U	330 U
Acenaphthylene	10 U	330 U
2,6-Dinitrotoluene	10 U	330 U
3-Nitroaniline	50 U	1600 U
Acenaphthene	10 U	330 U

Table 4.1 (continued)

Sample No.:	S3-MW1	S3-SS6-6.0'
Lab Sample No.:	890321-11	890321-17
Matrix:	Water	Soil
Associated Samples:	TB-3	TB-7
	FB-2	FB-4
	ER-6	ER-9

<u>Semivolatile organics</u> (continued)	($\mu\text{g/L}$)	($\mu\text{g/kg}$)
2,4-Dinitrophenol	50 U	1600 U
4-Nitrophenol	50 U	1600 U
Dibenzofuran	10 U	330 U
2,4-Dinitrotoluene	10 U	330 U
Diethylphthalate	10 U	330 U
4-Chlorophenyl-phenylether	10 U	330 U
Fluorene	10 U	330 U
4-Nitroaniline	50 U	1600 U
4,6-Dinitro-2-methylphenol	50 U	1600 U
N-Nitrosodiphenylamine	10 U	330 U
4-Bromophenyl-phenylether	10 U	330 U
Hexachlorobenzene	10 U	330 U
Pentachlorophenol	50 U	1600 U
Phenanthrene	10 U	330 U
Anthracene	10 U	330 U
Di-n-butylphthalate	75 J	330 U
Fluoranthene	10 U	330 U
Pyrene	10 U	330 U
Butylbenzylphthalate	10 U	330 U
3,3'-Dichlorobenzidine	20 U	660 U
Benzo(a)anthracene	10 U	330 U
Chrysene	10 U	330 U
bis(2-Ethylhexyl)phthalate	70 J	330 U
Di-n-octylphthalate	10 U	330 U
Benzo(b)fluoranthene	10 U	330 U
Benzo(k)fluoranthene	10 U	330 U
Benzo(a)pyrene	10 U	330 U
Indeno(1,2,3-cd)pyrene	10 U	330 U
Dibenz(a,h)anthracene	10 U	330 U
Benzo(g,h,i)perylene	10 U	330 U

Table 4.1 (continued)

Sample No.:	S3-MW1	S3-SS6-6.0'
Lab Sample No.:	890321-11	890321-17
Matrix:	Water	Soil
Associated Samples:	TB-3	TB-7
	FB-2	FB-4
	ER-6	ER-9

<u>Pesticides/polychlorinated biphenyls</u>	($\mu\text{g/L}$)	($\mu\text{g/kg}$)
alpha-BHC ^c	0.5 U	8 U
beta-BHC	0.5 U	8 U
delta-BHC	0.5 U	8 U
gamma-BHC	0.5 U	8 U
Heptachlor	0.5 U	8 U
Aldrin	0.5 U	8 U
Heptachlor epoxide	0.5 U	8 U
Endosulfan I	0.5 U	8 U
Dieldrin	1 U	16 U
4,4'-DDE ^c	1 U	16 U
Endrin	1 U	16 U
Endosulfan II	1 U	16 U
4,4'-DDD ^c	1 U	16 U
Endosulfan sulfate	1 U	16 U
4,4'-DDT ^c	1 U	16 U
Methoxychlor	5 U	80 U
Endrin ketone	1 U	16 U
alpha-Chlordane	5 U	80 U
gamma-Chlordane	5 U	80 U
Toxaphene	10 U	160 U
Arochlor-1016	5 U	80 U
Arochlor-1221	5 U	80 U
Arochlor-1232	5 U	80 U
Arochlor-1242	5 U	80 U
Arochlor-1248	5 U	80 U
Arochlor-1254	10 U	160 U
Arochlor-1260	10 U	160 U
<u>Metals</u>	(mg/L)	(mg/kg)
Antimony	2.5 U	2.5 U
Arsenic	0.5 U	0.5 U
Beryllium	0.3	0.5
Cadmium	0.5 U	0.5 U
Chromium	12	23

Table 4.1 (continued)

Sample No.:	S3-MW1	S3-SS6-6.0'
Lab Sample No.:	890321-11	890321-17
Matrix:	Water	Soil
Associated Samples:	TB-3	TB-7
	FB-2	FB-4
	ER-6	ER-9

<u>Metals</u> (continued)	(mg/L)	(mg/kg)
Copper	14	17
Lead	6.3	14.6
Mercury	0.1 U	0.1 U
Nickel	27	29
Selenium	0.5 U	0.5 U
Silver	0.6 U	0.6 U
Thallium	0.5 U	0.5 U
Zinc	52	47

Note: U = quantitation limit, J = estimated value, UJ = estimated quantitation limit, TR = trip blank, FB = field blank, ER = equipment rinsate, BHC = benzene hexachloride, DDE = dichlorodiphenyldichloroethylene, DDD = dichlorodiphenyldichloroethane, DDT = dichlorodiphenyltrichloroethane.

4.2.4 Suggested Community Relations Plan Format

Overview of Community Relations Plan -- A general introduction briefly stating the purpose of the CRP and the distinctive or central features of the community relations program planned for this specific site. Also, any special circumstances that the CRP has been designed to address shall be noted.

Site Description -- For readers unfamiliar with the site, the basic historical, geographical, and technical details necessary to demonstrate why the site is about to be, or is already, on the NPL, if available. Topics to be covered include:

- o site location and proximity to other landmarks,
- o history of site use and ownership,
- o date and type of release,
- o nature of threat to public health and environment, and
- o responsibility for site (e.g., state- or federal-lead).

Community Background -- A description of the community and its involvement with the site. It covers the following three topics:

- o Community profile: The economic and political structure of the community and key community issues and interests.
- o Chronology of community involvement: How the community has reacted to the site in the past, actions taken by citizens, attitudes toward government, and roles and responsibilities. Discussion of actions taken by any government agencies or officials, such as public meetings or news releases.
- o Key Community Concerns: How the community regards the risks posed by the site or the remedial process used to address those risks. One approach would be to break down the analysis by community group or segment (i.e., public environmental interest groups, nearby residents, and elected officials).

In all three topics, but particularly the last, the focus should be on the community perceptions of the events and problems at the site and not on the technical history of the site.

Highlights of Program -- Details on community relations approaches to be taken, which would follow directly and logically from the discussion of the community and its perception of the problems posed by the site. Development of a strategy for communicating with the specific community. Suggested topics include:

- o Resources to be used in the community relations program (e.g., local organizations and meeting places).
- o Key individuals or organizations that will play a role in community relations activities.
- o Areas of sensitivity that must be considered in community relations.

Techniques and Timing -- Description of community relations activities that will be conducted at the site and when. Additional techniques that might be used at the site as the response action proceeds and when they are likely to be most effective should be suggested.

Appendixes --

- o Mailing list of interested parties and key contacts.
- o Suggested locations of meetings and information repositories.

4.3 DEFINITION OF DATA VALIDATION FOR THE PROJECT

As listed in the aforementioned requirements, the subcontractor shall indicate in the site-specific QAPP the systematic process to be used to validate project data. Data must be validated against a set of accepted criteria to provide assurance that data are adequate for their intended use. The process shall consist of data editing, screening, checking, auditing, verification, flagging, certification, and review. The subcontractor, or designated representative, shall perform data validation. The laboratory shall NOT perform data validation; validation is independent of laboratory data review. The subcontractor shall certify in writing that data have been validated and flagged in accordance with the defined process. Specific guidelines per QC level are presented in Sect. 6.

4.4 PROJECT FINAL REPORT

A draft of the final report shall be sent to the HAZWRAP Project Manager and forwarded to the AQCS for review before its release. This report is the final deliverable from the subcontractor. It shall be designated as a HAZWRAP QA record. An outline for a typical report includes the following:

- o Project name and HAZWRAP contract number.
- o Foreword signed by those with major responsibilities for the QA program and by project management.
- o Executive Summary, presenting a brief review of the report and a site description.
- o Table of Contents with approximately the same level of specificity as the Table of Contents in this HAZWRAP document.

- o Introduction that summarizes the HAZWRAP project (sites of interest, dates of sampling, and dates of analyses), including objectives of the QAPP as they relate to the study.
- o Data Summary that provides a synopsis of results on a site-by-site basis (see Table 4.1).
- o Additional Information, including presentation of other requested information from the SOW, such as risk assessment, recommendation for continued site characterization, or recommend site closure. This information was specified before beginning work and is directed by the HAZWRAP Project Manager.
- o Findings from the analytical data. As stated previously, blank subtraction is not allowed. Data will be flagged as per data validation guidelines; all data validation flags will be included with results of the final data.
- o A QC summary that will include a discussion of all flagged data. Validation notes, including flagged data (defined as data for which trip, field, or laboratory blanks were contaminated, matrix spike/spike duplicates exceeded limits, surrogate recovery criteria were exceeded, calibration criteria were not met, and LCS recoveries exceeded acceptable limits) will be included. The QC summary will discuss results of laboratory blanks, matrix spikes/spike duplicates, duplicates, control charts, surrogate recoveries, holding times, field blanks, trip blanks, equipment rinseates, and field duplicates. This section will also discuss PARCC parameters, QC frequency, audits, corrective actions, and holding times.
- o Appendices, including all field and analytical data. One appendix shall contain field logs and forms.

A second appendix shall contain the laboratory data for each sample. All trip, field, and laboratory blanks shall be marked allowing for sample and blank association. For Level C QC, deliverables as discussed in Sect. 5 shall be presented. For Level D QC, the subcontractor shall submit full CLP or CLP-type data packages. This will consist of a minimum of 20% each of water and soil samples. Control charts for LCS data will be submitted. For Level E QC, sample results, initial and continuing calibration forms, method blanks, and LCS charts are required. Exact deliverables will have been stated and approved in the project work plan.

A third appendix shall include LCS control charts, surrogate recoveries, matrix spike and duplicate analysis, field and laboratory duplicates for all spike samples, and any additional QC analyses associated with the project.

A fourth appendix shall include all validation notes, as appropriate, to the QC level defined for the project.

- o Investigation activities shall include (where applicable) a discussion of drilling methods; decontamination waste disposal; well installation, development, and purging; sampling methods; water-level measurements; geophysical testing; field screening; surveying; and

custody and shipping of samples. These items must be addressed when they differ from those in the work plan.

- o The report shall indicate the duration and location of storage for data. Stored data will consist of all raw data, QC charts, corrective actions, logs, sample lists, COC information, notebooks, work sheets, automated data processing system output, calibration information, and validation notes.

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5. ANALYTICAL LABORATORY REQUIREMENTS

Before beginning any field studies or analyses of samples, contract laboratories must fulfill HAZWRAP requirements. This section describes laboratory responsibilities in terms of activities and documentation required of participants in the process.

5.1 HAZWRAP-SPECIFIC REQUIREMENTS

The following items outline minimum requirements established by HAZWRAP for sample handling and analysis. The laboratory must address how it intends to meet each of these requirements in the LQAP (see Sect. 5.2.1) and the QAPP. It is permissible to address these items in a HAZWRAP-specific LQAP addendum or in SOPs provided by the laboratory with the LQAP.

5.1.1 Approved Analytical Methods

The current CLP methods and documentation shall be followed for Level D QC activities. For methods not available under CLP, the latest edition of SW-846 or other EPA methods may be used. For Levels C and E activities, CLP, SW-846, or other EPA methods shall be used. **THE EXCEPTION TO LEVELS C AND E METHOD REQUIREMENTS IS VOLATILE AND SEMIVOLATILE ORGANICS ANALYZED BY GC/MS, WHICH MUST EMPLOY THE CURRENT CLP METHODS.**

In the case of munitions-related substances, the appropriate method(s) developed by the U.S. Army Toxic and Hazardous Materials Agency shall be used. The AQCS will make a copy available upon request.

For biota and air samples, the methods shall be evaluated individually by the AQCS to determine applicability and acceptability for the work in question. Nonstandard methods must receive review and approval before implementation by the laboratory. The method approval process is outlined in Fig. 5.1.

5.1.2 Quality Control Requirements for the Laboratory

Following are the minimum QC requirements for the laboratory. In Levels C, D, and E, a blank/spike control or an LCS shall be analyzed with each batch and recoveries plotted on control charts. The method for pesticides/polychlorinated biphenyls (PCBs) is an exception, due to recovery problems of the dibutylchloroendate (DBC) surrogate. For pesticide/PCB analysis, a method blank and an LCS containing two pesticides and one PCB spiking compound shall be

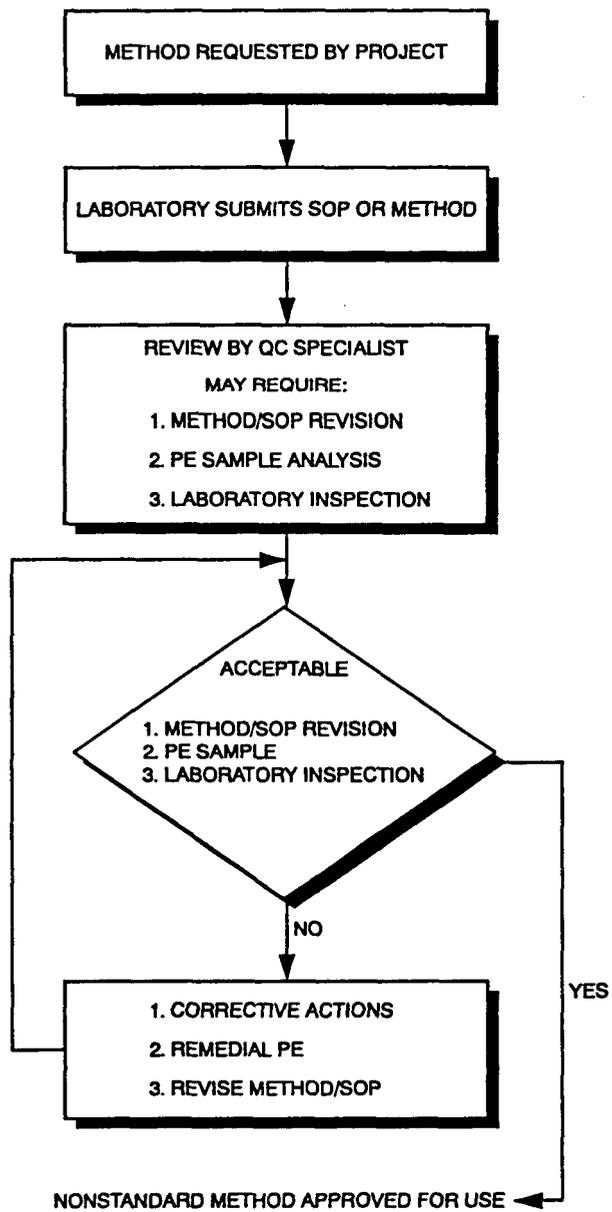


Fig. 5.1. Nonstandard analytical method review. (SOP = standard operating procedure, QC = quality control, PE = performance evaluation.)

analyzed as separate samples with each batch. In methods not using surrogates, such as metals, anions, and wet chemical analyses, a method blank and an LCS shall be analyzed.

When performing PCB analysis alone (no pesticide analytes required), the CLP method need not be employed. Most QC in the CLP method is in the pesticide fraction. The SW-846 Method 8080 is preferred in this case.

For all GC methods used in Levels C and D, second-column confirmation shall be required on all positive responses for the analytes of interest. In Level E, second-column confirmation may be requested in the project work plan, based on the needs of the site.

For Level D, current CLP QC requirements are specified. When analytes requiring methods other than CLP are used in Level D, CLP-type QC requirements are specified.

In Levels C and E, optimum batch size is determined by the number of samples of similar matrix with the ability to be processed simultaneously through the entire preparation and analytical process within a normal work shift. For example, if 5 samples can be extracted but 20 can be analyzed by the instrument during a normal shift, the batch size is 5.

In Level C, when performing petroleum hydrocarbons, oil and grease, anions (such as nitrates, sulfates, and chloride), and other wet chemical methods, a matrix spike and duplicate are required for every 20 samples of similar matrix. Similar matrix is defined as either soil or water from the same site.

All specified methods require calibration. In keeping with the calibration requirements of these methods, the following requirements are presented. For all semivolatile and volatile analysis by GC/MS, the current CLP calibration method shall be used. The current CLP criteria shall be used for frequency of calibration and for checking the system performance calibration compounds (SPCCs) and calibration check compounds (CCCs).

For other methods, a minimum of three different concentration standards for each analyte shall be analyzed for initial calibration. Calibration shall be checked every 12 h of operation and before analysis. The laboratory shall use the calibration check acceptance criteria specified by the methods. The daily calibration acceptance criteria to be used for each method shall be documented in the LQAP or in the site-specific QAPP. The initial calibration curve shall be plotted and the correlation coefficient and response factors evaluated. The laboratory shall indicate in the LQAP, or in the site-specific QAPP, the acceptance criteria to be used for the initial calibration curve. Calibration shall include one standard of a concentration at method detection limits. If samples are not within calibration range, appropriate dilution shall be performed to bring samples into range. The aforementioned calibration requirements shall be used for Levels C and E.

In Level C, a matrix spike and matrix spike duplicate are required for volatiles, semivolatiles, and all GC analyses for every 20 samples of similar matrix. For metals analysis, a duplicate and a matrix spike are required for every 20 samples of similar matrix.

It must be clearly understood that three sets of regulations pertain to holding times. The SW-846 and NPDES regulations require that holding times begin at the time of sample collection. The contract requirements from the CLP require that holding times begin in the laboratory from Verified Time of Sample Receipt (VTSR). When data validation is performed, the holding times for all methods (SW-846, NPDES, and CLP) require that the holding time begin at time of sample collection. It is the policy of HAZWRAP that the holding time begin at the time of sample collection for all methods, and samples must be shipped by overnight delivery on the day of collection. If this is not done, the HAZWRAP Project Manager and the AQCS must be notified in writing, or by telephone, to obtain permission for delayed delivery. Holding times to be used shall be so noted in the work plan and shall be listed by analysis method, along with the type and volume of bottle used and storage conditions.

The HAZWRAP policy regarding holding times is as follows:

- o Holding times are met when sample extraction or digestion is initiated.
- o The time between completion of extraction and the beginning of concentration shall not exceed 24 h.
- o For organics, storage between the time of extraction and concentration shall be at 4°C, and storage for metals shall be at room temperature.
- o Medium- or high-concentration volatile organics shall not be extracted and held. The analysis must take place immediately after extraction. In cases where an autosampler is used for volatile analysis, samples may be loaded in the autosampler and held until analysis without being kept at 4°C.

Volatile organics are to be analyzed by the low-level method unless the concentration criteria listed for medium- or high-concentration analysis in the requested method are met.

5.1.3 HAZWRAP Specifications for Sample Receipt

- o The laboratory must sign air bills upon receipt and keep copies in the project file.
- o Shipping container custody seals must be inspected and the condition documented.
- o Integrity of the coolant must be determined and documented.
- o Condition of the samples must be documented in a signed, dated, and bound logbook and on the COC form with signature and date of person checking samples.
- o The pH of preserved samples (except volatile organics) must be checked upon receipt and documented.
- o Any breakage, discrepancy, or improper preservation will be noted by the laboratory as an out-of-control event and must be documented on an out-of-control form with the

corrective action taken. The out-of-control form must be signed and dated by the custodian and any other person responsible for corrective action. The sample custodian must notify the engineering subcontractor of discrepancies in shipments.

5.1.4 HAZWRAP Specifications for Error Corrections

Any changes in entries in field or laboratory notebooks or on computer-printed data must be corrected by drawing a single line through the error and initialing and dating the new entry. The use of correction tape or fluid is not acceptable.

5.1.5 HAZWRAP Specifications for Sample Container Cleaning Procedures

In general, glass bottles with Teflon lids are used for organic samples, while polypropylene or polyethylene bottles are used for metals and other inorganics. The following specifies required bottle cleaning. If bottles are cleaned in the laboratory, bottle blanks must be performed on each cleaned lot of bottles and verification of bottle cleanliness provided.

If precleaned bottles are purchased, this must be noted in the work or field QAPP and approved by the AQCS and certificates of cleanliness kept on file.

All bottles should be capped, labeled, and packed in a cooler or box. Bottles should be stored in a contaminant-free area.

5.1.5.1 Cleaning Procedure for Glass Bottles (Except Volatile Organics)

- o Wash glass bottles, Teflon liners, and caps in hot tap water with laboratory-grade nonphosphate detergent.
- o Rinse three times with tap water.
- o Rinse with 1:1 nitric acid (metals-grade), prepared with American Society for Testing and Materials (ASTM) Type II deionized water.
- o Rinse three times with ASTM Type II deionized water.
- o Rinse with pesticide-grade methylene chloride using 20 mL for 0.5-gal container and 5 mL for 4- and 8-oz containers.
- o Oven dry at 125°C. Allow to cool to room temperature in an enclosed, contaminant-free area.

5.1.5.2 Cleaning Procedure for Bottles Used for Volatile Organics

- o Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot tap water, using laboratory-grade nonphosphate detergent.
- o Rinse three times with tap water.
- o Rinse three times with ASTM Type II deionized water.

- o Oven dry vials, septa, and caps at a minimum of 125°C.
- o Allow vials, septa, and liners to cool to room temperature in an enclosed, contaminant-free environment.
- o Seal 40-mL vials with septa (Teflon side down) and cap.
- o Store in a contaminant-free area.

5.1.5.3 Cleaning Procedure for Plastic Bottles

- o Wash plastic bottles and caps in hot tap water with laboratory-grade nonphosphate detergent.
- o Rinse with 1:1 nitric acid (metals-grade), prepared with ASTM Type II deionized water.
- o Rinse three times with ASTM Type II deionized water.
- o Invert and air dry in a contaminant-free environment.

5.1.6 HAZWRAP-Specific Requirements for Reporting Out-of-Control Events

HAZWRAP requires that the AQCS be notified as soon as possible when any out-of-control event occurs. The AQCS must also be informed as soon as the problem is solved and the corrective action completed. An example of this type of event would be the breakdown of a GC/MS system used for volatiles that could not be repaired for several days. If the laboratory cannot use another instrument in its facility, provisions shall be made for another HAZWRAP-reviewed laboratory to analyze the samples.

All out-of-control events and subsequent corrective actions shall be submitted, in report form, to the AQCS in the MPR. This corrective action report shall be signed by the laboratory director and the LQAC and shall discuss the following topics:

- o When and where the out-of-control incident occurred (laboratory name, address, telephone number, and section name).
- o Who discovered the out-of-control incident, any witnesses, and who took corrective action.
- o What analyses were being conducted. This must include a list of all samples affected. Sample problems and possible effects must be discussed.
- o Disposition of the test or control and/or instrument. Corrective actions must be described, along with any measures enacted to prevent a recurrence of the problem.
- o Any scientific explanation for the out-of-control event. A copy of subject control charts or other data describing the out-of-control conditions shall be included in the corrective action report.

All out-of-control incident documentation and copies of the corrective action reports sent to the AQCS shall be

- o placed in the laboratory archive record for the sample(s) in question,
- o placed in the LQAC's file of incident's documentation, and
- o included in the MPR.

5.1.7 HAZWRAP-Specific Requirements for Document Control

HAZWRAP requires that the laboratory maintain copies of all data packages, calibration records, and other QA-related records until the HAZWRAP Project Manager either asks for the records or writes a letter requesting destruction of same. The laboratory must develop an SOP providing instructions for all QC-related paperwork and instructions for recording storage for document control to include tracking and retrieval.

5.1.8 HAZWRAP-Specific Requirements for Control Samples

Control samples monitor performance of the analytical system. These samples contain known concentrations of analytes and are introduced into the normal environmental sample run sequence. Control samples, including duplicates, blanks, analytical standards, reference materials, and spikes, can be employed in different phases of the overall analysis. This may include sampling, storage, transportation, preparation, and the analytical method itself. The choice of control relates to the phase(s) to be controlled and the information (e.g., precision, accuracy, interferences, and contamination) to be developed.

HAZWRAP requires a description of how and where such control mechanisms are used by the laboratory. Control materials may be purchased from commercial sources, the National Institute of Standards and Testing, or the EPA. A brief description of each control sample (or set of samples) used will be provided in the MPR, subsequent to its introduction, and will cover the following items:

- o Where and how control samples are made.
- o How many control samples are made and with what frequency.
- o How control samples are used.
 - Physically (e.g., placed in the sample tray along with 14 environmental samples just before the samples enter the processing stream).
 - Analytically (e.g., used to determine the procedural recovery factor used to check for interferences).
- o Frequency of control sample analysis.

5.1.9 Laboratory Control Sample Program

LCSs are required for only those methods and analytes pertinent to the program. The laboratory will employ a measurement-control program that, as a minimum, consists of monitoring the results or control samples for laboratory preparation and analysis. Statistically based control charts will be employed for documentation. The purpose of this program is to demonstrate that the laboratory process for sample preparation and analysis is in control.

Analytes selected for spiking should be representative of the compound class. It is suggested that surrogates used for volatiles and base/neutral/acids analyses be used as control analytes for the GC/MS methods. At least two pesticides should be used when pesticide methods are performed and one PCB when PCBs are analyzed. For wet chemical methods, a single spike of an appropriate control for each method may be used (i.e., cyanide, a control standard of sodium cyanide from a source other than that used for calibration may be spiked into water and analyzed with the water samples). For metals, at least three metals typically analyzed by inductively coupled plasma (ICP) must be monitored, and each element analyzed by graphite furnace atomic absorption (GFAA) and cold-vapor atomic absorption (CVAA) shall be monitored.

Two matrix types must be employed. One type of control material is the spiked laboratory blank water. The second type of control material is a spiked laboratory soil or blank sand. This soil can be pulverized and homogenized. If the soil used is known to contain some analytes of interest, no spiking may be required. Additional spiking may be done to an aliquot of control soil just before sample preparation. The LCS matrix should be comparable to the sample matrix (i.e., analyze water control samples when water samples are analyzed).

This minimum program consists of using the laboratory's distilled and/or deionized water and spiking it with known concentrations of specific compounds or elements. By plotting results of the LCS on control charts, a true picture of the actual laboratory analytical process control is obtained. Few problems will be encountered from matrix effects and sample nonhomogeneity. This information, used in conjunction with sample matrix spike recoveries, can aid in determining whether an out-of-control condition is due to laboratory problems or matrix problems.

5.1.9.1 Laboratory Control Sample Quality

The laboratory will describe steps taken to ensure and verify quality of the two types of control samples. The following concerns pertaining to the control sample must be addressed.

- o How the LCS will be selected.
- o Shelf life of the LCS.
- o Under what condition the LCS will be stored.

- o How the LCS will be homogenized.
- o How and when individual samples will be taken.
- o How and when the sample will be spiked.
- o How the LCS will be replaced as it is depleted.
- o How control charts will be affected by changes in the LCS.

The LQAP must address the following concerns pertaining to spikes.

- o What compound/element will be used for spiking.
- o How the spike material will be selected.
- o Target concentration of spiking compound/element.
- o How long the spike is expected to last.
- o Under what conditions the spike will be stored.
- o How the spike will be homogenized.
- o How and when individual samples will be taken.
- o How the spike will be replaced as it is depleted.
- o How control charts will be affected by changes in spikes.

5.1.9.2 Control Charts

Control charts provide a useful tool in assessing QC conformance through the graphic display of a parameter's variability over time. The parameter plotted on the chart is usually related to control sample testing, either directly in terms of concentration or indirectly in terms of derived information (i.e., means, ranges, percent recoveries, relative percent differences, or slopes of least-squares data fits).

The laboratory must provide a brief description of the basic methodology for control chart use. Considerations covered include:

- o Verification that methods are valid and working properly before beginning control charts.
- o Establishing the number of LCSs per run sequence.
- o Determining parameters to be plotted against time and general formulas for developing these parameters.
- o Defining statistical/mathematical basis for assigning warning and action limits.
- o Identifying shifts and trends that may typically be revealed by these charts.

Administration of the control charts

The LQAP will address the following aspects of administering control charts.

- o What types of laboratory activities the control charts will monitor.
- o How often the LCSs will be run.

- o How soon after results are obtained the charts will be monitored.
- o Who is responsible for reading the charts.
- o How changes in personnel, equipment, or processes will affect the charts.
- o How often, and under what circumstances, limits will be updated.

Statistical quality of the control charts

Formulas used for the calculation of control chart limits must be provided. They are based on normally distributed measurements and short-term variation. When these formulas are properly fitted, the charts will perform as desired. Otherwise, the charts will either falsely signal out-of-control warnings more frequently than usual, fail to detect existing out-of-control conditions as often as they ordinarily would, or both (for different types of out-of-control situations). To correct any problems caused by improperly fitting control charts, the laboratory may propose alternate methods for setting control chart limits. All such proposals must include data and supportive statistical evidence. Possible alternate statistical approaches can include using nonparametric techniques, using medians instead of averages for the centerlines, identifying sources of variation, using long-term variation instead of short-term variation in setting limits, and transforming the data.

Minimum statistical control charting

At a minimum, the laboratory must create an LCS control chart for each method of analysis and sample matrix. These charts will monitor laboratory measurements obtained from the LCS.

Each control chart must consist of a centerline, two warning limits, and two control limits. Control chart parameters should be calculated by moving range or by standard deviation.

A minimum of 20 points per chart will be obtained before the initial attempt to establish control chart parameters. If the laboratory does not have 20 points to establish control chart limits, recommended EPA recovery limits for the method must be used until the necessary 20 points are attained.

HAZWRAP encourages the use of control charts as a normal routine procedure in the laboratory, regardless of the source of samples. To help encourage the use of control charts at all times, HAZWRAP does not limit control charts to only HAZWRAP samples. Control charts showing every LCS analyzed would be preferred, with control limits calculated on the first 20 points. Once control limits are established, they would remain in effect until a change in the process warranted recalculation of control limits on a new set of 20 points. The laboratory would attempt to identify reasons for the process change and submit a corrective actions report, explaining the reason for changing control limits. It is acceptable if a program is used that recalculates control limits each time a point is entered or recalculates control limits

every 20 points, but this must be noted in the MPRs and the LQAP. Control charts must be updated daily.

Criteria for an Out-of-Control Condition

In the LQAP, the contract laboratory must specify its criteria for defining an out-of-control condition related to control chart limits and patterns [e.g., data beyond rejection limits, data in zone(s) between the rejection and warning limits, data inside warning limits, the number of consecutive data points on one side of the mean, the number of consecutive data points in the middle zone, the number of monotonically changing data points, and obvious repetitive patterns (Garfield, 1984)].

A laboratory process for a particular analyte will be considered out-of-statistical-control whenever, at a minimum, any one of the following conditions is demonstrated by control chart monitoring of that analyte.

- o Any one point is outside control limits.
- o Any three consecutive points are outside warning limits.
- o Any eight consecutive points are on the same side of the centerline.
- o Any six consecutive points are such that each point is larger (smaller) than its immediate predecessor.
- o Any obvious cyclic pattern is seen in the points.

If a software program is used that is not capable of flagging data that are outside these criteria, it is the responsibility of laboratory personnel to flag these out-of-statistical control conditions manually.

Reactions to Out-of Statistical-Control Conditions on Control Samples

The LQAP must describe steps that will be taken in the event of an out-of-statistical-control condition. The steps will be similar to those requested under "Out-of-Control Events and Corrective Action" in Sect. 5.2.1.2, but will include those actions related to the quality and stability of control samples, sampling, spiking, and handling of control samples.

The laboratory must identify what action will be taken when warning and/or control limits are exceeded. Warning conditions may only require more frequent observations of a piece of equipment, while rejection conditions require shutting down an instrument and implementing corrective action.

5.1.10 HAZWRAP-Specific Requirements for Standard Operating Procedures

The laboratory must have written SOPs detailing each facet of work performed. These SOPs shall be reviewed and signed by the LQAC and must be available to personnel at the work station. These SOPs must be kept as controlled documents.

5.2 LABORATORY REVIEW

Laboratory review is necessary to ensure that contract laboratories meet minimum requirements for a QC program that facilitates the generation of data of defensible accuracy and precision. Specific objectives of the approval process follow:

- o To communicate HAZWRAP's QC requirements to the laboratories.
- o To ensure that proper communication and planning between the subcontractor and the laboratory has occurred before the laboratory receives samples.
- o To verify that such requirements are being met by each laboratory before analysis of HAZWRAP field samples.
- o To establish a plan for maintaining the QC program while work is being performed for HAZWRAP.

These objectives will be met through a review process that includes four major elements:

- o review of the LQAP (Sect. 5.2.1),
- o proficiency testing through analysis of a PE sample (Sect. 5.2.2),
- o laboratory inspection and audits (Sect. 5.2.3), and
- o review of laboratory submissions to assess capability to perform site-specific QAPP requirements.

All laboratories considered for HAZWRAP review must be identified by a subcontractor for a specific HAZWRAP project. HAZWRAP will issue a written request to the AESG to initiate the review process.

The review process is described in the remainder of this section and on Fig. 5.2.

5.2.1 Laboratory Quality Assurance Plan Requirements

The general LQAP will be forwarded to the HAZWRAP Project Manager for review by the AQCS. The LQAP is submitted immediately after the request for review by HAZWRAP. The AQCS will respond with comments on the LQAP within ten working days of receipt. The laboratory will respond to these comments, with changes in the LQAP or with a plan of action,

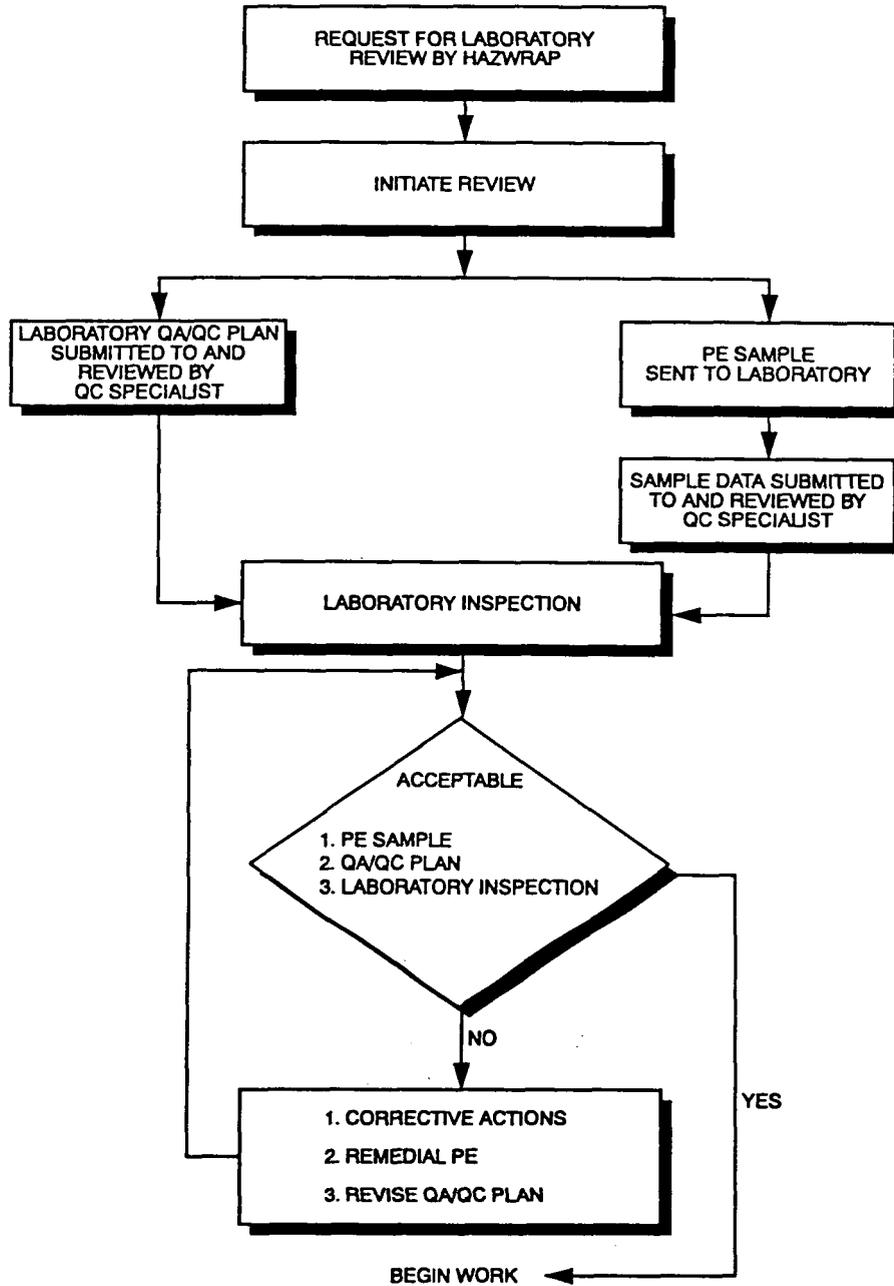


Fig. 5.2. Initial laboratory review process. (HAZWRAP = Hazardous Waste Remedial Actions Program, QA/QC = quality assurance/quality control, PE = performance evaluation.)

within ten working days of receipt of comments. All changes and/or plans of action must be in effect before laboratory review.

5.2.1.1 Purpose and Scope

The LQAP is a statement of the laboratory's approach to ensuring that quality data are generated from the analysis of HAZWRAP samples. In the context of laboratory review, the plan provides a basis for evaluating a laboratory's QC procedures. This evaluation includes a critical review of the LQAP and verification of the laboratory's adherence to the LQAP through inspection.

5.2.1.2 Organization and Contents of the Laboratory Quality Assurance Plan

The following items are required in the LQAP; however, they may be presented in any order that the laboratory desires.

- o Title Page with Provision for Signatures - A title page with provisions for approval signatures and dates of revision will be provided.
- o Table of Contents - A detailed Table of Contents will be provided.
- o Laboratory Organization and Personnel - This section provides an overview of the laboratory organization as it relates to implementation of the QC program. The roles, responsibilities, and authorities of key laboratory personnel are described, with emphasis on the authority given the LQAC regarding QC monitoring, reporting, and corrective action.

An appendix will contain a list of all personnel, their assignments and responsibilities, degrees of education, and the years of applicable experience. This information may be supplied in the form of resumes. All management personnel responsible for performing analytical work will be listed, along with their job assignments and years of experience in performing applicable work. Any education and training related to tasks performed for this project will also be listed.

- o Personnel Training - The plan will address how personnel are trained in laboratory analytical methods, QC procedures, and safety policies. Frequency of training and training records will be addressed.
- o Sample Management Practices and the COC - This section will include procedures for tracking samples through the laboratory, receipt of samples, verification of preservation, log-in of samples, and COC documentation. Sample storage and disposal will also be included, along with preparation of bottles and glassware cleaning.
- o Material Procurement and Control - This section will include a description of procedures for purchasing materials, quality inspection before use in sample analysis, chemical standard inventory procedures, solvent storage policies, and laboratory waste disposal.

- o Facilities and Equipment - This section will include a list of basic types of equipment, year of purchase, and general description of the facility to ensure that the laboratory is large enough to handle the sample load expected and that the equipment is capable of performing the required analyses.
- o Equipment Maintenance - This section will include general information as to who performs major, preventive, and day-to-day equipment maintenance and how it is documented.
- o Analytical Procedures - This section will contain a list of procedures the laboratory offers (by method number and matrix). If future work requires analyses not specified in the current SOW, this information may prove useful.

Any method variances shall be documented and reported. Documentation for EPA method variance approvals will be presented to ensure that the approvals are known before sample analysis.

The laboratory policy and its implementation will ensure that controlled copies of analytical procedures and SOPs are available to the analysts.

- o Calibration - This section will include calibration procedures by instrument type. Calibration frequency, reference standards, calibration acceptance criteria, and calibration documentation procedures must be addressed. Calibration applies to both instruments and procedures, such as gas and liquid chromatography, GC/MS, ICP, atomic absorption (AA), infrared and ultraviolet spectroscopy, and wet chemical methods.

Procedures must be defined for ensuring that balances, refrigerators, and ovens are accurate and that their performance is monitored and documented. Balances and ovens must be checked before use. Balances must also be calibrated annually by an independent company. Refrigerator temperatures must be checked daily.

- o Limits of Detection - The laboratory will indicate typical method-detection limits achieved for water, soil, and other matrixes commonly analyzed by the laboratory. It is understood that these may vary with individual samples. Procedures for determining limits of detection and the frequency of detection-limit verification will be outlined.
- o Analysis of QC Samples and Documentation - This section will summarize QC procedures and documentation to be employed in the day-to-day operation of the laboratory. The discussion will emphasize the following:
 - Analysis of field, method, and reagent blanks.
 - Analysis of duplicates, spiked samples, spiked laboratory blanks, and reference or control standards, such as EPA check standards.

- The criteria used to establish warning and action limits for the above types of QC samples.
 - Documentation and examples of control data and control charts (see Sect. 5.1.9.2 for an explanation of control charts and their usage).
 - The frequency of blanks and other QC samples including LCSs.
 - How data from QC samples are reported and reviewed.
 - Who reviews and makes decisions relative to QC data.
 - How requirements of the minimum control program will be met.
 - Verification of calibration.
- o Out-of-Control Events and Corrective Action - This section will define types of out-of-control occurrences, how these occurrences are documented, and who is responsible for correction and documentation. It is recognized that several types of out-of-control events may occur. Four examples follow:
- Corrective actions at the receiving level - A sample is broken during transport. The sample custodian observes the problem. The occurrence is documented on the COC form and on an out-of-control form. Corrective actions include notification of project management, who will determine the need for resampling.
 - Observations corrected at the bench - Calibration of an instrument is not linear. The analyst finds this and corrects the problem before continuing sample analysis. The laboratory must document this and note that the corrective action was to recalibrate and that no samples were affected, because none was analyzed before calibration.
 - Corrective actions taken by supervisor - A matrix spike recovery is out of control and the laboratory supervisor discovers this after samples have been analyzed. The supervisor must document the occurrence and the corrective actions taken.
 - Statistical out-of-control events - A control chart is being monitored, and the measured parameter exceeds control limits. The occurrence is documented on an out-of-control form, the root cause is established, affected samples are identified, and corrective actions are defined.

The laboratory must specify protocols for reporting any incident that delays sample processing for a period of time, affects holding times, or delays work.

Examples of forms used to document out-of-control events are to be provided in the LQAP.

- Corrective Action Reports - For out-of-control incidents, it is essential to document the nature of the incident and corrective actions taken to set the system back "in control." A copy of an out-of-control corrective action report must be supplied in the LQAP.
- o Document Control - The LQAP will outline document flow from the COC to the final data. The LQAP will explain how documents are reviewed, signed, and filed.
- o Data Evaluation - A discussion of data evaluation procedures for each analytical method, as well as for an entire data set, will be included. The process for data review and approval will be outlined. Data qualification and flagging procedures will be implemented.
- o Holding Times and Preservatives - The document will include the holding time policy for ensuring sample analyses procedures are met. Sample storage, holding times, and preservatives specified by the methods are minimal criteria for HAZWRAP approval.
- o Internal Laboratory Audits and Approvals from Other Agencies - The document will include a listing of approvals from other agencies and states. This provides an indication of the organization's general quality and type of laboratory experience. When the laboratory performs self-audits, frequency and method of documentation will be outlined.
- o QA Reports to Management - The plan will include the frequency and information (general contents) of QA reports to management.
- o Accuracy, Precision, and Completeness - The plan will include the laboratory's definition of accuracy, precision, and completeness. The method for evaluating measured parameters and data sets for accuracy, precision, and completeness will be incorporated.

5.2.2 Proficiency Testing Requirements

Before beginning analysis of field samples, each laboratory must analyze PE samples for chemical substances representative of those anticipated in environmental samples. The purpose of PE sample analysis is to gage each laboratory's proficiency by providing samples designed to mimic field samples. A second benefit of PE samples is to provide a known material from a source outside the laboratory that can be used to evaluate performance. A third benefit is to be able to review reporting format contents and compliance to specifications.

5.2.2.1 Submission of Performance Evaluation Samples

PE samples will be provided to the LQAC after receipt of the request for approval from HAZWRAP. Samples may be soil, water, or vials of concentrate. The laboratory will be supplied with directions necessary for sample reconstitution and preparation and with analytes to be determined. If analyses are to be subcontracted to a second laboratory, appropriate proficiency samples will be provided to that laboratory as well.

5.2.2.2 Performance Evaluation Sample Deliverables

The laboratory shall utilize the EPA CLP methods and criteria to identify and quantitate concentrations of compounds (volatile, semivolatile, and pesticides/PCBs) on the Target Compound List (TCL) of the July 1988 or February 1988 CLP revision. For metals, the laboratory shall also use the EPA CLP methods and criteria to identify and quantitate concentrations of metals. The July 1988 CLP revisions shall be used for inorganics. Such criteria shall include, but not be limited to, the use of approved instruments, digestion and analysis methods, QC requirements, and documentation.

Test results will be reported in a full CLP data package and format. The data package will include clear explanations of all calculations performed to acquire the values reported in the corresponding data sheets.

5.2.2.3 Evaluation of Performance Evaluation Sample Results

The AQCS will compare the laboratory's evaluation of PE sample results with peer group PE sample results. Performance will be acceptable if laboratory results are within the 95% confidence interval established by the peer group, the data package deliverables conform to CLP-type criteria, and no procedural problems are found during laboratory inspection. Nonacceptable results will initiate a review of records to determine the cause of nonconformance. If results are outside the 95% confidence interval, proficiency testing may have to be repeated using a remedial PE sample.

The remedial PE sample will be analyzed in the same manner as the initial and must be accompanied by all specified deliverables.

The laboratory must pass proficiency testing before approval for HAZWRAP work. The AQCS will respond to the laboratory with results of the proficiency test within ten working days of receipt of the data.

5.2.3 Laboratory Audit

A laboratory audit will be conducted after the following events take place:

- o The LQAP is reviewed.
- o The laboratory has satisfactorily responded to LQAP review comments.
- o The laboratory has satisfactorily performed required proficiency testing.

The audit will be performed by the AQCS. The HAZWRAP Project Manager and/or sponsor representative may also be a part of the audit team.

5.2.3.1 Purpose of Laboratory Audit

The purpose of laboratory audit is to verify that HAZWRAP QC requirements are being implemented, as reflected by the laboratory's daily operations in adherence to the LQAP.

5.2.3.2 Laboratory Inspection Process

The laboratory inspection involves three phases.

Preaudit Meeting (Overview and Orientation) - The audit team meets with laboratory management, including the laboratory director, the LQAC, and others, as the director deems appropriate. Objectives of the visit are reviewed, and a schedule is established. The audit team discusses comments on the LQAP and proficiency samples and resolves any outstanding issues on these items. Basic requirements, as outlined in this document, are discussed. Laboratory personnel provide information on training, the laboratory's history, and capabilities for performing work for HAZWRAP. Project- and program-specific requirements may be discussed at this time.

Observation, Examination, and Review (Laboratory Walk-Through) - According to the schedule, the audit team performs the following activities.

- o Reviews sample receiving, handling, and storage procedures. The audit team will follow the trail of the PE sample through the laboratory.
- o Witnesses performance of specified analytical procedures in each section of the laboratory.
- o Examines QC records, including manuals, instrument calibration and maintenance records, control charts, instrument run logs, sample preparation logs, notebooks used to document analyses, corrective action reports for out-of-control events, and performance data generated for other programs, such as Superfund CLP and state drinking water. SOPs for all activities performed may be examined. Other items examined are waste disposal procedures, water sources, bottle preparation activities, records for balances, refrigerators and ovens, data review procedures, and audit procedures.

Close-out Meeting (Exit Interview) - The audit team will conduct an exit interview with the laboratory director, LQAC, and any other laboratory personnel the director deems appropriate. The audit team will summarize findings of the visit, detail specific deficiencies to be addressed by corrective actions, and make recommendations regarding corrective actions. A written report summarizing audit findings is provided to the LQAC within ten working days of the inspection.

5.2.3.3 Corrective Action (if Required)

Within ten working days of receipt of audit findings, the laboratory must submit a plan to the HAZWRAP Project Manager to correct deficiencies identified. The plan will include, for each deficiency, a description of the corrective action and a date within 45 working days indicating when the corrective action is to be implemented or completed.

The laboratory will send a follow-up report that supplies information indicating proof that the plan has been carried out. For example, if no control charts exist, the plan would state that these would be in place by a specific date, and the follow-up report would contain copies of the control charts.

5.2.3.4 Follow-Up Audits

A repeat audit may be required in instances where deficiencies requiring corrective action are complex. Repeat audits will be scheduled for the earliest possible date after the last corrective action plan is received by the AQCS.

5.2.4 Notification of Successful Performance

Upon acceptance of the LQAP and PE sample package and upon satisfactory dispensation of all audit findings, the laboratory will be allowed to work on HAZWRAP projects. The laboratory will receive a written notification of successful performance.

5.3 LABORATORY FOLLOW-UP REVIEWS

5.3.1 Scheduled Reviews

All HAZWRAP-cleared laboratories will be automatically slated to undergo follow-up reviews every 15 to 18 months. Two conditions are necessary for follow-up reviews:

- o The laboratory must be currently performing analyses for HAZWRAP or the laboratory must still be needed for future work on a project.
- o The follow-up review must be requested by HAZWRAP program management.

Conditions for follow-up reviews: After a follow-up review is requested by HAZWRAP program management, the laboratory will be reviewed in four areas:

- o Review of the current general LQAP per Sect. 5.2.1.
- o Successful completion of a set of PE samples per Sect. 5.2.2.
- o Successfully passing of a laboratory audit per Sect. 5.2.3.
- o No major problems found in MPRs and/or final data reports in the last year. Any problems found must have been satisfactorily addressed during the past year.

The follow-up review process is outlined in Fig. 5.3.

5.3.2 Remedial Reviews

Several incidents can occur that may require a remedial review for all or part of a laboratory's operation. Conditions that may precipitate an additional laboratory review follow:

- o Problems found in MPRs and/or in Final Data Reports on projects (see Sects. 5.4.1 and 5.4.2).
- o New methods used in the laboratory.
- o Suspension of the laboratory by another review agency.
- o When considered necessary by the HAZWRAP Project Manager or the AQCS.

5.4 LABORATORY DELIVERABLES

All laboratories receiving samples associated with HAZWRAP projects are required to provide deliverables as specified below. Deliverables must be presented to the HAZWRAP Project Manager through the subcontractor or the AQCS. Approved laboratory forms shall be used when reporting data in MPRs and in submitting the final data package before its inclusion in the appendix and summary tables of the Project Final Report.

Failure to provide these deliverables will result in suspension of HAZWRAP work.

5.4.1 Laboratory Monthly Progress Reports

The primary means of communication between laboratories and the AQCS is by MPRs. These reports are to be submitted by the laboratories to the AQCS by the 15th of each month. This report is due each month, regardless of whether HAZWRAP samples are being analyzed during the month. The following information is to be included in the MPR:

- o Project name and contract number.
- o Lists of numbers, types, and locations of samples collected and analyzed for the Fig. 5.3

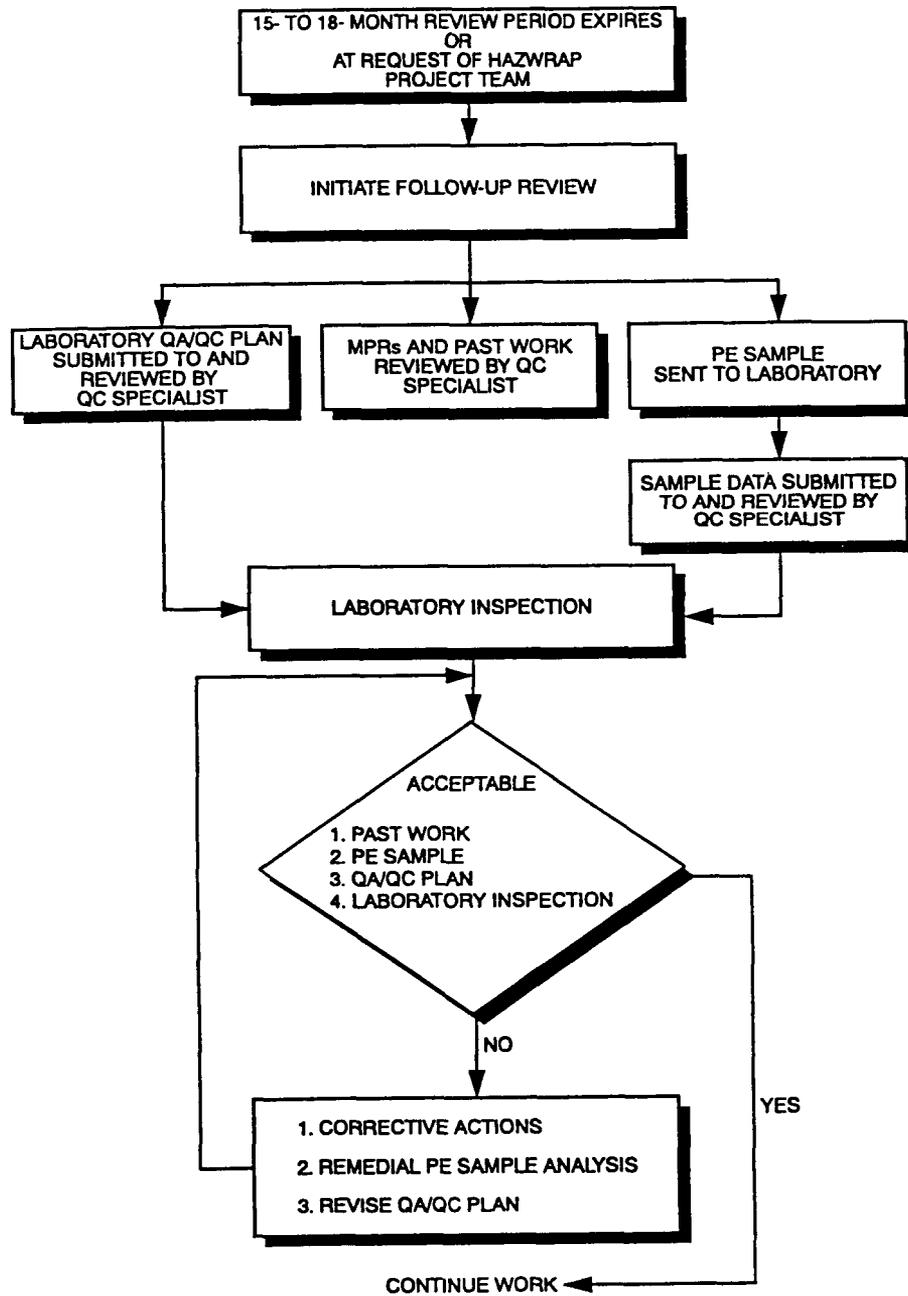


Fig. 5.3. Laboratory follow-up review process. (QA/QC = quality assurance/quality control, MPR = monthly progress report, PE = performance evaluation, HAZWRAP = Hazardous Waste Remedial Actions Program.)

- HAZWRAP project only and the disposition of those samples.
- o New methods used for analysis and changes in old methods.
 - o Copies of all control charts from the LCS program that are pertinent to HAZWRAP samples and to which results have been added over the reporting period.
 - o Summaries of out-of-control incidents during the reporting period, including references to documentation and corrective action reports. Include a list of any samples/analyses that might have been affected by these incidents.
 - o Descriptions of, and justifications for, significant changes in the QAPP.
 - o Changes in laboratory QA personnel and other key technical personnel (resumes of new personnel must be submitted).
 - o Copies of signed COC forms.
 - o Changes in certification status with any regulatory or certifying agencies and unacceptable results obtained on any external proficiency testing programs. Include laboratory responses to these results and corrective action plans.

Much of the information presented in an MPR is incremental in nature and relates to changes and findings since the previous MPR. Typical changes that could be pertinent, even when samples are not being processed, include:

- o Any additional control charts from monitoring matrix spikes, duplicates, or other QC parameters.
- o Key changes in technical and QA personnel.
- o Method changes (e.g., a minor modification with an attached EPA variance).
- o Procedural changes in establishing control limits and/or the preparation and use of control charts.

Because the first such report for each laboratory has no precedent, more explanation and detail may be necessary. Subsequent MPRs will likely not require as much detail in some areas.

NOTE: On some larger projects, the work plan may specify periodic submission of data reports with the MPR. These data reports will consist of all routine and quality control data associated with samples analyzed for that particular month. Review of the data report will permit identification of possible systematic errors associated with the generation of data before receipt of the final data report. The HAZWRAP Project Manager always has the right to request submission of data reports at his discretion.

5.4.2 Laboratory Final Report

The final report submitted by the laboratory is basically a package of the deliverables as listed below. Format of the package will follow the basic outline as stated by the CLP. Contents of the final package will be determined by the deliverables required for the QC level

performed. Final data deliverables will be presented to the HAZWRAP Project Manager at least 3 weeks before issuing the draft of the final report.

Level A - A formal final report is not required; the only deliverables are sample results. The daily single point calibration must be kept on file. More information on Level A deliverables can be found in DOE/HWP-69/R1, Sect. 8.1.

Level B - Deliverables include sample results, method blanks, three-point calibration, and continuing calibration checks. More information on Level B deliverables can be found in DOE/HWP-69/R1, Sect. 8.2.

Level C - See Table 5.1 for deliverables. The forms referred to are from the current CLP guidelines. These, or similar forms, are required. If forms other than CLP-type are used, the laboratory must include a copy of those forms in the LQAP or send them to the HAZWRAP Project Manager for approval before initiating work.

Level D - A CLP data package is required for Level D. When CLP methods are performed or when methods other than CLP are performed, CLP-type forms must be provided. Deliverables include the summary package and remainder of the data package, including initial and continuing calibration, matrix spikes, matrix spike duplicates, blanks, duplicates, surrogate recoveries, chromatograms, mass spectra, and absorbance data. For methods not defined by the CLP, calibration information, method blanks, blank/spikes, chromatograms, absorbance, matrix spikes, and matrix spike duplicates will be reported. Plotted control charts associated with the LCS will be presented with the data. Other information required includes copies of signed COC forms and laboratory case narratives.

Level E - The minimum information to be submitted must include sample results, method blank data, initial and continuing calibration data, and control charts from the LCS data. Exact deliverables will be stated in the work plan and approved by the HAZWRAP Project Manager before initiating work. Other information required includes copies of signed COC forms and laboratory case narratives.

Table 5.1. Data set deliverables for Level C quality assurance

Method requirements	Deliverables
Requirements for all methods:	
- Holding time information and methods requested	Signed chain-of-custody forms
- Discussion of laboratory problems	Case narratives
- LCS with results on control charts. Run with each batch of samples processed	Control charts
Organics:	
- Sample results	CLP Form 1 or equivalent
- Surrogate recoveries. Surrogates to be used in volatiles, semivolatiles, pesticides/PCBs. For volatiles by GC, surrogate names should reflect the appropriate surrogate used	CLP Form 2 or equivalent
- Matrix spike/spike duplicate. One spike and spike duplicate per 20 samples of similar matrix	CLP Form 3 or equivalent
- Method blank data	CLP Form 4 or equivalent
- GC/MS tuning for volatiles/semivolatiles	CLP Form 5 or equivalent
- GC/MS initial calibration data for volatiles/semivolatiles	CLP Form 6 or equivalent
- Pesticide/PCB calibration data	CLP Form 9 or equivalent
- For volatiles by GC; initial calibration data	
If calibration factors are used	CLP Form 8D or equivalent, with five columns for multilevel calibration factors
If a calibration curve is used	A plot of the calibration curve is required, and a linear regression determination, with flagged correlation coefficient, if it is less than 0.995

Table 5.1 (continued)

Method requirements	Deliverables.
Organics (cont'd):	
<ul style="list-style-type: none"> - For volatiles by GC; continuing calibration data If calibration factors are used, calibration factors and their percent differences from the initial calibration must be reported. Retention Time (RT) windows and analyte RTs for the analytes must be included in this form 	CLP Form 9 or equivalent
<ul style="list-style-type: none"> - GC/MS continuing calibration data No chromatograms or mass spectra are presented for calibration. These data should be filed in the laboratory and available if problems arise in reviewing/validating the data. The calibration information should be available for checking during on-site audits 	CLP Form 7 or equivalent
<ul style="list-style-type: none"> - GC/MS internal standard area data - Second column confirmation shall be done for all GC work when compounds are detected above reporting limits. Chromatograms of confirmation must be provided 	CLP Form 8 or equivalent Chromatograms for all samples and CLP Form 10 or equivalent for all positive hits
Metals:	
<ul style="list-style-type: none"> - Sample results 	CLP Form 1 or equivalent
<ul style="list-style-type: none"> - Initial and continuing calibration 	CLP Form 2 or equivalent
<ul style="list-style-type: none"> - Method blank taken through digestion (one per 20 samples of same matrix) 	CLP Form 3 or equivalent
<ul style="list-style-type: none"> - ICP interference check sample 	CLP Form 4 or equivalent
<ul style="list-style-type: none"> - Spike sample recovery (one per 20 samples of similar matrix) 	CLP Form 5A or equivalent

Table 5.1 (continued)

Method requirements	Deliverables
Metals (cont'd):	
- Postdigestion spike sample recovery for ICP metals. Only done if predigest spike recovery exceeds CLP limits	CLP Form 5B or equivalent
- Postdigestion spike for GFAA	Recovery will be noted on raw data
- Duplicates (one per 20 samples will be split and digested as separate samples)	CLP Form 6 or equivalent
- LCS	CLP Form 7 or equivalent
- Standard addition. The decision process outlined in CLP Page E-3 will be used to determine when standard additions are required	CLP Form 8 or equivalent
- Holding times	CLP Form 10 or equivalent
Wet Chemistry:	
- LCS (one/batch)	Control chart
- Method blank (one/batch)	Report result - no format
- Sample results	Report result - no format
- Spike/spike duplicate or calibration information	Report result, if applicable
- Calibration check; report percent Relative Standard Deviation or percent difference from calibration	Report percent or percent difference - no format

Note: LCS = laboratory control standard, CLP = contract laboratory program, PCB = polychlorinated biphenyls, GC = gas chromatograph, MS = mass spectrometry, ICP = inductively-coupled plasma.

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6. DATA VALIDATION GUIDELINES

As listed in project requirements, the subcontractor must indicate in the site-specific QAPP, the systematic process to be used to validate project data. Data must be validated against a set of accepted criteria to provide assurance that data are adequate for their intended use. The process will consist of data editing, screening, checking, auditing, verification, flagging, certification, and review. The subcontractor, or a designated representative, will perform data validation. The laboratory will NOT perform data validation; validation is independent of laboratory data review. The subcontractor will certify in writing that data have been validated and flagged in accordance with the defined process.

Tables 6.1-6.5 outline holding times, containers, and preservatives for some methods applicable to the HAZWRAP program. Methods not included in these tables will follow the holding times and preservation techniques stated by the method. In the HAZWRAP program, CLP holding times are consistent with CLP validation guidelines, and water samples for volatile organics must be preserved.

It must be clearly understood that three sets of regulations pertain to holding times. The SW-846 and NPDES regulations require that holding times begin at the time of sample collection. The contract requirements from the CLP require that holding times begin in the laboratory from VTSR. When data validation is performed, the holding times for all methods (SW-846, NPDES, and CLP) require that the holding time begin at time of sample collection. It is the policy of HAZWRAP that the holding time begin at the time of sample collection for all methods, and samples must be shipped by overnight delivery on the day of collection. If this is not done, the HAZWRAP Project Manager and the AQCS must be notified in writing, or by telephone, to obtain permission for delayed delivery. Holding times to be used shall be so noted in the work plan and shall be listed by analysis method, along with the type of bottle used and storage conditions.

The HAZWRAP policy regarding holding times is as follows:

- o Holding times are met when sample extraction or digestion is initiated.
- o The time between completion of extraction and the beginning of concentration will not exceed 24 h.
- o For organics, storage between the time of extraction and concentration will be at 4°C, and storage for metals can be at room temperature.
- o Medium or high concentration volatile organics will not be extracted and held. The analysis must take place immediately after extraction. In cases where an autosampler is used for volatile analysis, samples may be loaded in the autosampler and held until analysis without being kept at 4°C.

Table 6.1. Hazardous Waste Remedial Actions Program requirements summary for Contract Laboratory Program methods

Parameter	Matrix	Holding time ^a (from time of collection)	Container	Preservative	Minimum ^b sample size
Volatile organics	Water	14 d	Two 40-mL vials with Teflon-lined caps.	4 drops conc. HCl 4°C	40 mL
	Soil	14 d	Brass or Teflon core tube sealed on both ends	4°C	10 g
Extractable organics	Water	7 d extn. ^c 40 d anal. ^c	1-L glass with Teflon liner.	4°C	1000 mL
	Soil	14 d extn. 40 d anal.	Glass jar with Teflon liner or core tube	4°C	50 g
Metals (other than mercury)	Water	180 d	P,G ^d	HNO ₃ to pH <2 ^e	100 mL
	Soil	180 d	P,G	4°C	10 g
Mercury	Water	28 d	P,G	HNO ₃ to pH <2	100 mL
	Soil	28 d	P,G	4°C	10 g
Cyanide	Water	14 d	P,G	0.6 g ascorbic ^f acid, NaOH to pH >12, 4°C	100 mL
	Soil	14 d	P,G	4°C	10 g

^aHolding times are consistent with CLP validation guidelines.

^bAdditional sample must be collected for matrix spike/matrix spike duplicate samples or matrix spike/duplicate.

^cExtn. = extraction; anal. = analysis.

^dPolyethylene (P) or glass (G).

^eDissolved metals require filtration before pH adjustment.

^fOnly used in the presence of residual chlorine.

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Table 6.2. Hazardous Waste Remedial Actions Program volatile organic requirements

Matrix	Holding time (from time of collection)	Container	Preservative	Minimum ^a sample size
Water samples				
No residual chlorine present	14 d	Two 40-mL vials with Teflon-lined septum caps	4 drops conc. HCl, 4°C	40 mL
Residual chlorine present	14 d	Two 40-mL vials with Teflon-lined septum caps	4 drops of 10% sodium thiosulfate, 4 drops conc. HCl, 4°C	40 mL
Acrolein and acrylonitrile	14 d	Two 40-mL vials with Teflon-lined septum caps	Adjust to pH 4-5, 4°C	40 mL
Soil/sediments and sludges	14 d	Brass or Teflon core tube sealed on both ends	4°C	10 g

^aAdditional sample must be collected for matrix spike/matrix spike duplicate samples or matrix spike/duplicate.

NOTE: The above information applies to the following parameters and methods:

<u>Parameter</u>	<u>Method (gas chromatography)</u>
Volatile halocarbons	601/8010
Volatile aromatics	602/8020
Volatile organics	8015
Acrolein/acrylonitrile	603/8030

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Table 6.3. Hazardous Waste Remedial Actions Program extractable organic requirements

Matrix	Holding time (from time of collection)	Container	Preservative	Minimum ^a sample size
Water samples				
No residual chlorine present	Samples must be extracted within 7 d and analyzed within 40 d of extraction	1-L glass with Teflon liner	4°C	1 L
Residual chlorine present	Samples must be extracted within 7 d and analyzed within 40 d of extraction	1-L glass with Teflon liner	Add 1 mL 10% sodium thiosulfate per liter, 4°C	1 L
Soil/sediments and sludges	Samples must be extracted within 14 d and analyzed within 40 d of extraction	Glass jar with Teflon liner or core tube	4°C	50 g

Note: The above information applies to the following parameters and methods:

Parameter	Method
Phenols	604/8040 gas chromatography (GC)
Phthalate esters	606/8060 (GC)
Organochlorine pesticides/Polychlorinated Biphenyls	608/8080 (GC)
Polyaromatic hydrocarbons	610/8310 (HPLC) ^b
Organophosphate pesticides	614/8140 (GC)
Phenoxy acid herbicides	615/8150 (GC)
Carbamate and urea pesticides	632 (HPLC)

^aAdditional sample must be collected for matrix spike/matrix spike duplicate samples or matrix spike/duplicate.

^bHigh-Pressure Liquid Chromatography.

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Table 6.4. Hazardous Waste Remedial Actions Program requirements for other organics

Parameter	Method No.	Matrix	Holding time (from time of collection)	Container	Preservative	Minimum ^a sample size
Dioxins/ furans	8280	Water	30 d extn. ^b 45 d anal. ^b	1-L glass	4°C	1000 mL
		Soil/waste	30 d extn. 45 d anal.	Core tube	4°C	50 g
Petroleum hydrocarbons as gasoline	TPH-gasoline Purge and Trap (LUFT manual)	Water	14 d	Two 40-mL vials w/Teflon liners	4°C, HCl to pH <2	40 mL
		Soil/waste	14 d	Core tube	4°C	50 g
Petroleum hydrocarbons as gasoline	TPH-gasoline extractable (LUFT manual)	Water	14 d extn. 40 d anal.	1-L glass	4°C, HCl to pH <2	500 mL
		Soil/waste	14 d extn. 40 d anal.	Core tube	4°C	50 g
Petroleum hydrocarbons as diesel	TPH-diesel extractable (LUFT manual)	Water	14 d extn. 40 d anal.	1-L glass	4°C	500 mL
		Soil/waste	14 d extn. 40 d anal.	Core tube	4°C	50 g
Petroleum hydrocarbons (TPH)	TPH-IR (418.1)	Water	28 d	1-L glass	4°C, HCl to pH <2	1000 mL
		Soil	28 d	Glass jar with Teflon liner or core tube	4°C	50 g

^aAdditional sample must be collected for matrix spike/matrix spike duplicate samples or matrix spike/duplicate.

^bExtn. = extraction; anal. = analysis.

Note: TPH = Total Petroleum Hydrocarbons, LUFT = Leaking Underground Fuel Tanks, TPH-IR = Total Petroleum Hydrocarbons-Infrared.

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Table 6.5. Hazardous Waste Remedial Actions Program metals requirements

Parameter	Method No.	Matrix	Holding time (from time of collection)	Container	Preservative ^a	Minimum ^b sample size
Metals (ICP)	200.7/6010	Water	6 months	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	6 months	Core tube/glass jar	4°C	10 g
Arsenic (GFAA)	206.2/7060	Water	6 months	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	6 months	Core tube/glass jar	4°C	10 g
Mercury (CVAA)	245.1/7470 7471	Water	28 d	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	28 d	Core tube/glass jar	4°C	10 g
Selenium (GFAA)	270.2/7740	Water	6 months	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	6 months	Core tube/glass jar	4°C	10 g
Thallium (GFAA)	279.2/7841	Water	6 months	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	6 months	Core tube/glass jar	4°C	10 g
Lead (GFAA)	239.2/7421	Water	6 months	Poly	HNO ₃ to pH <2.0	100 mL
		Soil/waste	6 months	Core tube/glass jar	4°C	10 g
Chromium (VI)	218.4/218.5 7196/7197	Water	24 h	Poly	4°C	100 mL
		Soil/waste	24 h	Core tube/glass jar	4°C	10 g

^aDissolved metals require filtration before pH adjustment.

^bAdditional sample must be collected for matrix spike/matrix spike duplicate samples or matrix spike/duplicate.

Note: ICP = Inductively Coupled Plasma, GFAA = Graphite Furnace Atomic Absorption, CVAA = Cold Vapor Atomic Absorption.

Volatile organics are to be analyzed by the low-level method unless the concentration criteria listed for medium- or high-concentration analysis in the requested method are met.

6.1 LEVELS A AND B DATA VALIDATION GUIDELINES

Level A data require no data validation because only sample results are presented.

Level B data do not undergo a validation process, but must undergo a formal review process. The subcontractor, in concert with field laboratory analysts, will indicate in the site-specific QAPP the systematic process to be used to review data. Set criteria for data evaluation must be defined before sample analysis. The process will address data editing, screening, and verification. Data verification must include checking calibration and blanks to ensure criteria have been met. Review procedures must include instructions for flagging samples associated with blanks or calibrations that are out of criteria. The subcontractor shall use matrix spike data to evaluate and flag routine data accordingly.

The subcontractor must certify in writing that data have been reviewed in accordance with the defined process.

Specific information on Level A and Level B data review is available in DOE/HWP-69/R1, Sects. 8.1 and 8.2.

6.2 LEVEL C DATA VALIDATION GUIDELINES

Listed below are validation criteria that will be used in evaluating analytical data for a Level C QC analysis. For methods not listed here, a similar procedure, outlining validation of holding times, initial calibration, continuing calibration, and blank vs sample results, shall be submitted by the prime contractor and laboratory. The validation procedure must be approved by the AQCS.

6.2.1 Petroleum Hydrocarbons (EPA Method 418.1)

Holding Times - Holding times are 28 d from the day collected for water samples that are preserved and refrigerated. No holding times are cited for soils.

If the holding time is exceeded:

- o Flag all associated positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).
- o Document that holding times were exceeded.

NOTE: These holding times apply only to petroleum hydrocarbons analyzed by EPA Method 418.1. Holding times for other petroleum hydrocarbon analytical methods (including California Modified 8015) can be found in Table 6.4.

Calibration - Ensure that a three- to five-point standard curve bracketing sample concentration is performed daily. The correlation coefficient must meet or exceed 0.995 before the analysis of samples.

If the minimum number of standards was not used for initial calibration:

- o Qualify data as unusable (R).

If the instrument was not calibrated daily before sample analysis:

- o Qualify data as unusable (R).

If the correlation coefficient is less than 0.995:

- o Qualify sample results greater than the Instrument Detection Limit (IDL) as estimated (J).
- o Qualify sample results less than the IDLs as estimated (UJ).

Blanks - A blank must be prepared and analyzed with each batch of samples.

If the concentration in the sample is less than five times the concentration found in the blank:

- o The result is considered as a nondetect and flagged as such (U).

If the concentration in the sample is greater than five times the concentration found in the blank:

- o The result is considered positive and no flag is required.

BLANK RESULTS ARE NOT TO BE SUBTRACTED FROM SAMPLE VALUES FOR ANY REASON.

LCSs and Duplicates - Ensure that each sample is analyzed in a batch in which an LCS and a duplicate have been performed. Flagging is not required; however, the LCS (see

Sect. 5.1.9) should also be examined to evaluate whether the laboratory is in control. Any problems in the LCS or duplicate shall be noted in the case narrative, and informed professional judgment shall be used for interpretation of results.

6.2.2 Gas Chromatograph/Mass Spectrometer Volatile Organics

Validation for GC/MS volatile organics will essentially follow the CLP functional validation guidelines.

Holding Times - Samples must be analyzed within 14 d from date collected for water samples that are preserved and refrigerated. The same holding times are applied to soil samples.

If water samples are unpreserved, the holding time is 7 d from date collected. If there is no indication of preservation, assume samples are unpreserved.

If the holding time is exceeded:

- o Flag all associated positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).
- o Document that holding times were exceeded.

GC/MS Tuning - Make certain that a bromofluorobenzene (BFB) tune, meeting the CLP criteria, is completed every 12 h of sample analysis and that each sample is associated with a tune.

If tunes do not meet the expanded criteria as listed in the latest CLP functional guidelines:

- o Flag associated data as unusable (R).

Initial Calibration - The Average Relative Response Factor (\overline{RRF}) for all compounds must be greater than or equal to 0.05, and all Percent Relative Standard Deviations (%RSD) must be less than or equal to 30%.

If any compound has an \overline{RRF} of less than 0.05:

- o Flag all positive results for that compound as estimated (J).
- o Flag nondetects for that compound as unusable (R).

If any compound has a %RSD of greater than 30%:

- o Flag positive results for that compound as estimated (J).
- o Qualify nondetects using professional judgment.

Continuing Calibration - The Relative Response Factor (RRF) for all compounds must be greater than or equal to 0.05, and all percent difference (%D) must be less than or equal to 25%.

If any compound has an RRF of less than 0.05:

- o Flag positive results for that compound as estimated (J).
- o Flag nondetects for that compound as unusable (R).

If any compound has a %D greater than 25%:

- o Flag positive results for that compound as estimated (J).
- o Flag nondetects using professional judgment.

Blanks - Blank criteria apply to all blanks, including method, trip, and field blanks. Verify that all blanks have been analyzed at the frequency indicated in the project work plan.

RESULTS MAY NOT BE CORRECTED BY SUBTRACTING ANY BLANK VALUES.

If a compound is found in a blank but not found in the associated sample, no action is taken.

In instances where more than one type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Differences in weights, volumes, and/or dilution factors between blanks and associated samples must be taken into consideration.

The following two rules (5x and 10x) apply:

- o The 10x Rule applies to the four common laboratory contaminants listed below.

methylene chloride
acetone
toluene
2-butanone (methyl ethyl ketone)

- When the concentration of that compound is greater than the Contract Required Quantitation Limit (CRQL) but less than 10x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).

- When the concentration of that compound is less than the CRQL and less than 10x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
- When the concentration of the compound is greater than 10x the highest concentration found in any blank, the result is considered as positive, and no flag is required.
- o The 5x Rule applies to all compounds other than the four common laboratory contaminants listed previously.
 - When the concentration of that compound is greater than the CRQL but less than 5x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).
 - When the concentration of that compound is less than the CRQL and less than 5x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
 - When the concentration of the compound is greater than 5x the highest concentration found in any blank, consider the result as positive, and no flag is required.
- o Sample analytes not detected or detected at levels less than CRQL are reported as the CRQL with a (U) flag added.

If gross contamination exists (saturated peaks):

- o Flag all compounds affected as unusable (R).

If inordinate amounts of other compounds and/or Tentatively Identified Compounds (TICs) are found in any blank:

- o Note this in the validation comments.

LCSs - Any LCS exceeding internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected.

If no analytical problems are found:

- o Data analyzed with the out-of-control point shall be discussed in the QC section of the MPR and final report.

If problems are found in the analytical data:

- o Samples associated with the batch shall be reanalyzed and data from the reanalysis reported.

If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the LCS results are outside internal laboratory limits and if matrix spike results are outside the CLP limits, the laboratory shall either reanalyze samples within holding times or data shall be flagged as unusable (R).

Surrogates - If surrogates exceed the CLP limits, data shall be flagged to indicate the violation.

If at least one surrogate recovery is out of specification but greater than 10% recovery:

- o Flag positive results as estimated (J).
- o Flag negative results with the CRQL as estimated (UJ).

If any surrogate shows less than 10% recovery:

- o Flag positive results as estimated (J).
- o Flag negative results as unusable (R).

If any blank has surrogates out of specification:

- o It may be an isolated occurrence, and no qualification is required.
- o There may be a fundamental problem with the analytical process that must be corrected by the laboratory. All associated data would require reanalysis.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) - Ensure that 1 out of 20 samples has been spiked in duplicate. Recoveries shall meet the CLP criteria. If the recoveries do not meet the criteria, examine the LCS data.

If the LCS data exceed the upper or lower limits defined by the laboratory and the matrix spikes exceed the upper or lower limits defined by the method:

- o Flag data as unusable (R).

If the LCS data from the batch are satisfactory, data may be usable. The low matrix spike recovery and its implications, however, shall be discussed in the final report.

Internal Standard (IS) Area Performance - IS area counts must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

Retention time of the IS must not vary more than plus or minus 30 s from the associated calibration standard.

If an IS area count is outside -50% or +100% of the associated calibration standard:

- o Flag positive results for compounds quantitated using that IS as estimated (J).
- o Flag nondetects for compounds quantitated using that IS with the CRQL as estimated (UJ).
- o If extremely low area counts are reported or if performance exhibits a major abrupt drop-off, a severe loss of sensitivity is indicated, and nondetects should be flagged as unusable (R). A discussion must be included in the case narrative describing the problem.

If an IS retention time varies by more than plus or minus 30 s, the chromatographic profile for that sample must be examined to determine if false positives or negatives exist. If such shifts occur, the reviewer may consider partial or total rejection of the data.

6.2.3 Gas Chromatograph Volatile Organics

Holding Times - Holding time is measured from the time of sample collection to the time of analysis. Water samples that are preserved and refrigerated must be analyzed within 14 d. If water samples are unpreserved, the holding time is 7 d. If there is no indication of preservation, assume samples are unpreserved. Soil samples must be analyzed with 14 d.

If holding time is exceeded:

- o Flag all positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).
- o Document that holding times were exceeded.
- o If the holding time is grossly exceeded, use best professional judgment as to data reliability. The reviewer may flag all associated nondetect data as unusable (R).

Calibration - An External Calibration Procedure is used for quantitation by the laboratory.

If the Calibration Factor is used for sample quantitation:

- o For initial calibration, all %RSD must be less than or equal to 20%.
- o For continuing calibration, all %D must be less than 15%.

If the Linear Regression Method is used for sample quantitation:

- o Verification of the calibration curve is required, and the correlation coefficient must be greater than or equal to 0.995.

In the primary analysis, all standards are analyzed at the beginning of the 12-h period, followed by the proper sample/standard sequence. Confirmation analysis requires a mid-level standard at the beginning of the 12-h period. The midlevel standard must be repeated after every five samples.

If the criteria for initial calibration are not met:

- o Flag all associated quantitative results as estimated (J).

If the criteria for continuing calibration are not met:

- o In the primary analysis, flag all associated quantitative results as estimated (J).
- o In the confirmation analysis, use professional judgment as to data reliability.

If proper standards have not been analyzed:

- o Use professional judgment as to data reliability.

Blanks - Blank criteria apply to all blanks, including method, trip, and field blanks. Verify that all blanks have been analyzed at the frequency indicated in the project work plan.

RESULTS MAY NOT BE CORRECTED BY SUBTRACTING ANY BLANK VALUES.

If a compound is found in a blank but not found in the associated sample, no action is taken. In instances where more than one type blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Differences in weights, volumes, and/or dilution factors between blanks and associated samples must be taken into consideration.

The following two rules (5x and 10x) apply:

- o The 10x Rule applies to the four common laboratory contaminants listed below.

methylene chloride
acetone
toluene
2-butanone (methyl ethyl ketone)

- When the concentration of that compound is greater than the CRQL but less than 10x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).

- When the concentration of that compound is less than the CRQL and less than 10x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
 - When the concentration of the compound is greater than 10x the highest concentration found in any blank, consider the result as positive, and no flag is required.
- o The 5x Rule applies to all compounds other than the four common laboratory contaminants listed previously.
- When the concentration of that compound is greater than the CRQL but less than 5x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).
 - When the concentration of that compound is less than the CRQL and less than 5x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
 - When the concentration of the compound is greater than 5x the highest concentration found in any blank, consider the result as positive, and no flag is required.
 - Sample analytes not detected or detected at levels less than CRQL are reported as the CRQL with a (U) flag added.

Surrogates - All samples are spiked with the surrogate compounds stated in the specific volatile method. Control limits must be established by the laboratory for each method.

If low recoveries are obtained:

- o Flag associated positive results and quantitation limits as estimated (J).

If high recoveries are obtained:

- o Professional judgment should be used to determine appropriate action.

If zero recovery is reported:

- o The reviewer should examine the sample chromatogram to determine if the surrogate may be present but slightly outside its retention time window. If this is the case, in addition to assessing surrogate recovery for quantitative bias, the overriding consideration is to investigate qualitative validity of the analysis.

If the surrogate is not present:

- o Flag all negative results as unusable (R).

MS/MSD - Control limits must be established by the laboratory for each method.

These criteria cannot be used alone to evaluate precision and accuracy. Flagging is not required.

If the LCS is within limits:

- o The laboratory is in control, and MS/MSDs outside limits on the sample could be due to matrix effects.

If the LCS is outside control limits:

- o The laboratory may be out of control, and associated samples may have to be reanalyzed. This requires informed professional judgment for interpretation of results.

Compound Identification - Retention times of reported compounds must fall within the calculated window for two chromatographic columns. Second column confirmation is mandatory.

If the qualitative criteria for two-column confirmation were not met:

- o All reported positive detects should be considered nondetects. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:
 - If the misidentified peak was sufficiently outside the target compound retention time window, the CRQL can be reported.
 - If the misidentified peak poses an interference with potential detection of a target peak, the reported value should be considered and flagged as the estimated quantitation limit (UJ).

LCSs - Any LCS exceeding internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected. If no analytical problems are found, data analyzed with the out-of-control point shall be discussed in the QC section of the MPR and final report. If problems are found in the analytical data, samples associated with the batch shall be reanalyzed and data from the reanalysis reported. If holding times are exceeded during reanalysis, both sets of data shall be presented.

If the LCS results are outside internal laboratory limits and if matrix spike results are outside method limits:

- o The laboratory will either reanalyze the sample within holding times or data shall be flagged unusable (R).

6.2.4 Semivolatile Organics

Validation for semivolatile organics will essentially follow the CLP functional validation guidelines.

Holding Times - Holding time is measured from the time of collection to the time of extraction and analysis. Both samples and extracts must be preserved at 4°C. Water samples must be extracted within 7 d, and the extract must be analyzed within 40 d. Soil samples must be extracted within 14 d, and the extract must be analyzed within 40 d.

If holding time is exceeded:

- o Flag all associated positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).
- o Document that holding times were exceeded.

If holding time is grossly exceeded:

- o Use best professional judgment as to data reliability. All associated nondetect data may be flagged as unusable (R).

GC/MS Tune - Make certain that a decafluorotriphenylphosphine tune, meeting the CLP criteria, is completed every 12 h of sample analysis and that each sample is associated with a tune.

If tunes do not meet the expanded criteria, as listed in the latest CLP functional validation guidelines:

- o Flag associated data as unusable (R).

Initial Calibration - The \overline{RRF} for all compounds must be greater than or equal to 0.05, and all %RSD must be less than or equal to 30%.

If any compound has an \overline{RRF} of less than 0.05:

- o Flag positive results for that compound as estimated (J).
- o Flag nondetects for that compound as unusable (R).

If any compound has a %RSD of greater than 30%:

- o Flag positive results for that compound as estimated (J).
- o Nondetects may be qualified using professional judgment.

Continuing Calibration - The RRF for all compounds must be greater than or equal to 0.05, and all %D must be less than or equal to 25%.

If any compound has an RRF of less than 0.05:

- o Flag positive results for that compound as estimated (J).
- o Flag nondetects for that compound as unusable (R).

If any compound has a %D of greater than 25%:

- o Flag positive results for that compound as estimated (J).
- o Nondetects may be qualified using professional judgment.

Blanks - Blank criteria apply to all blanks, including method and field blanks. Verify that all blanks have been analyzed at the frequency indicated in the project work plan. Verify that an associated method blank has been performed with each sample per matrix.

RESULTS MAY NOT BE CORRECTED BY SUBTRACTING ANY BLANK VALUES.

If a compound is found in a blank but not found in the associated sample:

- o Take no action.

In instances where more than one type blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Differences in weights, volumes, and/or dilution factors between blanks and associated samples must be taken into consideration.

The following two rules (5x and 10x) apply.

- o The 10x Rule applies to those phthalate esters that are common laboratory contaminants.
 - When the concentration of that compound is greater than the CRQL but less than 10x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).
 - When the concentration of that compound is less than the CRQL and less than 10x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
 - When the concentration of the compound is greater than 10x the highest

concentration found in any blank, consider the result as positive, and no flag is required.

- o The 5x Rule applies to all compounds other than the common laboratory contaminants listed previously.
 - When the concentration of that compound is greater than the CRQL but less than 5x the highest concentration found in any blank, consider the result as a nondetect and flag it with a (U).
 - When the concentration of that compound is less than the CRQL and less than 5x the highest concentration found in any blank, report the result as the CRQL with a (U) qualifier.
 - When the concentration of the compound is greater than 5x the highest concentration found in any blank, consider the result as positive, and no flag is required.
 - Sample analytes not detected or detected below the CRQL are reported as the CRQL with a (U) flag added.

If gross contamination exists (saturated peaks):

- o Flag all compounds affected as unusable (R).

If inordinate amounts of other compounds and/or TICs are found in any blank:

- o Note this in the validation comments.

LCSs - Any LCS exceeding internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected.

If no analytical problems are found:

- o Data analyzed with the out-of-control point shall be discussed in the QC section of the MPR and final report.

If problems are found in the analytical data:

- o Samples associated with the batch shall be reanalyzed and data from reanalysis reported.

If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the LCS results are outside internal laboratory limits and if matrix spike results are outside the CLP limits, the laboratory will either reanalyze samples within holding times or the data will be flagged as unusable (R).

Surrogates - If any two surrogate recoveries are outside the limits in any one fraction or any one surrogate in any fraction is below 10% recovery, there should be a reanalysis of the sample by the laboratory. (If reanalysis shows unsuccessful surrogate recoveries, report both analyses. If reanalysis shows successful recoveries, report only the successful run.)

If two or more surrogate recoveries in one fraction are out of specification but greater than 10% recovery:

- o Flag positive results as estimated (J).
- o Flag negative results with CRQL as estimated (UJ).

If any surrogate shows less than 10% recovery:

- o Flag positive results as estimated (J).
- o Flag negative results as unusable (R).

If any blank has surrogates out of specification:

- o It may be an isolated occurrence, and no qualification is required.
- o There may be a fundamental problem with the analytical process that must be corrected by the laboratory. All associated data would require reanalysis.

MS/MSD - Ensure that 1 out of 20 samples has been spiked in duplicate. Recoveries shall meet the CLP criteria.

If recoveries do not meet the criteria, examine the LCS data.

If LCS data and matrix spikes exceed limits:

- o Flag data as unusable (R).

If LCS data from the batch are satisfactory:

- o Data are usable, and the low recovery shall be discussed in the final report.

IS Area Performance - IS area counts must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

Retention time of the IS must not vary more than plus or minus 30 s from the associated calibration standard.

If an IS area count is outside -50% or +100% of the associated calibration standard:

- o Flag positive results for compounds quantitated using that IS as estimated (J).
- o Flag nondetects for compounds quantitated using that IS with the CRQL as estimated (UJ).

If extremely low area counts are reported or if performance exhibits a major abrupt drop-off, a severe loss of sensitivity is indicated and nondetects should be flagged as unusable (R). A discussion must be included in the case narrative describing the problem.

If an IS retention time varies by more than plus or minus 30 s, the chromatographic profile for that sample must be examined to determine if false positives or negatives exist. If such shifts occur, the reviewer may consider partial or total rejection of the data.

6.2.5 Pesticides/Polychlorinated Biphenyls

Validation for pesticides/PCBs will essentially follow the CLP functional validation guidelines.

Holding Times - Holding time is measured from the time of sample collection to the time of sample extraction and analysis. Both samples and extracts must be preserved at 4°C. Water and soil samples must be extracted within 7 d of collection, and the extract must be analyzed within 40 d.

If holding time is exceeded:

- o Flag all positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).
- o Document that holding times were exceeded.

If holding time is grossly exceeded:

- o Use best professional judgment as to data reliability. Reviewer may flag all associated nondetect data as unusable (R).

Instrument Performance - If the retention time of dichlorodiphenyl-trichloroethane (DDT) is less than 12 min (with the exception of OV-1 and OV-101), a close examination of the chromatogram is necessary to ensure $\geq 25\%$ resolution between individual components is achieved.

If $\geq 25\%$ resolution between individual components is not achieved:

- o Flag all affected data as unusable (R).

If standards do not fall within the retention time windows:

- o Evaluate associated sample results carefully. All samples injected after the last in-control standard are potentially affected.

If no TCL peaks are present, either within or close to the retention time window:

- o There is usually no effect on the data. (Nondetected values can be considered valid.)

If the affected sample chromatograms contain peaks of concern, the following efforts may be taken to determine a usable retention time window for affected samples:

- o The reviewer should examine the data package for the presence of three or more standards containing the pesticide of interest. These standards must be run within a 72-h period during which the sample was analyzed.
- o If three or more such standards are present, the mean and standard deviation of the retention time window can be reevaluated.
- o If all standards and matrix spikes fall within the revised window, valid positive or negative sample results can be determined using this window.
- o The narrative should identify additional efforts taken by the reviewer and the resultant impact on data usability. In addition, support documentation should contain all calculations and comparisons generated by the reviewer.

If DDT breakdown is greater than 20%, beginning with the sample following the last in-control standard:

- o Flag all quantitative results for DDT as estimated (J). If DDT was not detected, but results for dichlorodiphenyldichloroethane (DDD) and dichlorodiphenylethane (DDE) are positive, flag the quantitation limit for DDT as unusable (R).
- o Flag results for DDD and/or DDE as presumptively present at an estimated quantity (NJ).

If endrin breakdown is greater than 20%:

- o Flag all quantitative results for endrin as estimated (J).

If endrin was not detected, but endrin aldehyde and endrin ketone are positive:

- o Flag the quantitation limit for endrin as unusable (R).
- o Flag results for endrin ketone as presumptively present at an estimated quantity (NJ).

If the retention time shift for dibutylchlorodate (DBC) is greater than 2.0% for packed column, greater than 0.3% for narrow-bore capillary column, or greater than 1.5% for wide-bore capillary column:

- o Flag the analysis for that sample as unusable (R).

Calibration - The %RSD of calibration factors for aldrin, endrin, DDT, and DBC must not exceed 10%.

If toxaphene is identified and quantified, a three-point calibration is required. If the calibration factor for DDT or toxaphene is outside the 10% RSD window, calibration curves must be used for quantitation of DDT, DDE, DDD, or toxaphene.

At the beginning of each 72-h period, all standards must be analyzed.

- o Evaluation Standard Mixes A, B, and C are all required for the curve.
- o Only standards containing the compound to be confirmed are required. These standards must be repeated after every five samples.
- o Evaluation Mix B is required after every ten samples.

The calibration factor for each standard must be within 15% of the standard at the beginning of the analytical sequence on quantitation columns (20% on confirmation columns).

If criteria for the linearity of initial calibration are not met:

- o Flag all associated quantitative results as estimated (J).

If proper standards have not been analyzed:

- o Data may be affected. The reviewer must use professional judgment to determine the severity of the effect and to qualify data accordingly.

If the %D between calibration factors is greater than 15% for the compounds being quantitated (20% for compounds being confirmed):

- o Flag all associated positive quantitative results as estimated (J).

Blanks - Blank criteria apply to all blanks, including method and field blanks. Verify that field blanks and equipment rinseates have been collected at the frequency indicated by the project. Verify that associated method blanks have been performed with each sample and each matrix. If problems exist with any blank, all data associated with the case must be carefully evaluated.

RESULTS MUST NOT BE CORRECTED BY SUBTRACTING ANY BLANK VALUES.

If a compound is found in a blank but not in the associated samples, no action is taken.

In instances where more than one type of blank is associated with a given sample, qualification should be based on a comparison with the associated blank having the highest concentration of contaminant. Differences in weights, volumes, and/or dilution factors between blanks and associated samples must be taken into consideration.

Any compound detected in the sample and in any associated blank must be qualified when sample concentration is less than five times blank concentration.

When the sample result is greater than the CRQL but less than five times blank concentration, the sample is flagged as nondetect (U).

When the sample result is greater than the CRQL and greater than five times blank concentration, no flag is required.

Compound Identification - Retention times of reported compounds must fall within the calculated window for two chromatographic columns. Second column confirmation is mandatory. GC/MS confirmation is required if the concentration of a compound exceeds 10 ng/ μ L in the final sample extract.

If the qualitative criteria for two-column confirmation were not met:

- o All reported positive detects should be considered nondetects. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:
 - If the misidentified peak was sufficiently outside the target compound retention time window, the CRQL can be reported.
 - If the misidentified peak poses an interference with potential detection of a target peak, the reported value should be flagged as estimated quantitation limit (UJ).

If PCBs or multippeak pesticides exhibit marginal pattern-matching quality:

- o Professional judgment should be used to establish whether differences are attributable to environmental "weathering."

If the presence of a PCB/multipeak pesticide is strongly suggested:

- o Results should be reported as presumptively present (N).

If an observed pattern closely matches more than one Aroclor:

- o Professional judgment should be used to decide whether the neighboring Aroclor is a better match or if multiple Aroclors are present.

If GC/MS confirmation was required but not performed:

- o The reviewer should immediately notify the HAZWRAP Project Manager and the AQCS.

Compound Quantitation - Quantitation limits affected by large, off-scale peaks should be flagged as unusable (R).

If the interference is on-scale:

- o The reviewer can provide an estimated quantitation limit (UJ) for each affected compound.

The reviewer should use professional judgment to decide whether a much larger concentration obtained on one column vs the other indicates the presence of an interfering compound.

If an interfering compound is indicated:

- o The lower of the two values should be reported and qualified as presumptively present at an estimated quantity (NJ).

6.2.6 Metals and Cyanide

Validation for metals and cyanide will essentially follow the CLP function validation guidelines.

Holding Times - Most metal samples must be analyzed within 6 months of sample collection. The exceptions follow:

- o Mercury, which shall be analyzed within 28 d from sample collection.
- o Cyanide, which shall be analyzed within 14 d from sample collection.
- o Hexavalent chromium, which shall be analyzed within 24 h of sample collection.

All holding times listed above apply to preserved samples. Tables 6.1 and 6.5 outline applicable holding times and preservatives.

If holding time is exceeded:

- o Flag all associated positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).

Initial Calibration Verification (ICV) - Instruments are to be calibrated daily and each time they are set up.

For ICP analysis, a blank and at least one standard must be used in establishing the calibration curve.

For AA analysis, a blank and at least three standards, one of which must be at the Contract Required Detection Limit, shall be used in establishing the calibration curve, and the correlation coefficient must equal or exceed 0.995.

For mercury analysis, a blank and at least four standards must be used in establishing the analytical curve, and the correlation coefficient must equal to or exceed 0.995.

If the minimum number of standards is not used for initial calibration or if the instrument is not calibrated daily and each time the instrument is set up:

- o Qualify data as unusable (R).

If the correlation coefficient is less than 0.995:

- o Qualify results greater than the IDL as estimated (J).
- o Qualify results less than the IDL as estimated (UJ).

Continuing Calibration Verification (CCV) - Analysis results must fall within the control limits of 90 to 110% recovery (%R) of the true value for all analytes, except mercury and cyanide.

Analysis results for mercury must fall within control limits of 80 to 120% recovery.

Analysis results for cyanide must fall within control limits 85 to 115% recovery.

If the ICV or CCV %R falls outside criteria:

- o Qualify results greater than the IDL as estimated (J).
- o Qualify results less than the IDL as estimated (UJ).

Method Blanks - At least one preparation blank must be prepared with each batch of samples. Each blank shall contain less than the detection limit for all analytes.

If contaminant concentration in the associated blank is above the detection limit and if the lowest analyte concentration is <5 times the blank:

- o Perform reanalysis of the sample.

If reanalysis was not possible:

- o Report and flag data as estimated (J).

BLANK VALUES SHALL NEVER BE SUBTRACTED FROM THE SAMPLE.

Field and Equipment Blanks - If contaminant analytes are detected in samples at concentrations of <5 times the concentration found in the highest associated blank:

- o Consider results suspect and flag them as estimated (J).

LCSs - Any LCS exceeding internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected.

If no analytical problems are found:

- o The data and out-of-control point shall be discussed in the QC section of the report.

If problems are found in the analytical data:

- o Samples associated with the batch shall be reanalyzed and data from reanalysis reported.

If holding times are exceeded in the reanalysis, both sets of data shall be presented.

A discussion of data reported when the LCS is out of control must be presented in the QC section of both the MPR and final report.

If the LCS results are outside internal laboratory limits and if matrix spike results are outside the CLP limits:

- o The laboratory will either reanalyze the samples or data will be flagged as unusable (R).

Spike/Duplicate - Spike recovery (%R) must be within the limits of 75 to 125%.

If spike recovery is greater than 125% and reported sample results are less than the IDL:

- o Data are acceptable.

If spike recovery is greater than 125% or less than 75% and sample results are greater than the IDL:

- o Qualify data for these samples as estimated (J).

If spike recovery falls within the range of 30 to 74% and sample results are less than the IDL:

- o Qualify data for these samples as estimated (UJ).

If spike recovery results fall less than 30% and sample results are less than the IDL:

- o Qualify data for these samples as unusable (R).

6.2.7 Wet Chemistry

Holding Times - Samples must be analyzed and/or extracted within holding times specified by the method.

If the holding time is exceeded:

- o Flag all associated positive results as estimated (J).
- o Flag all associated sample quantitation limits as estimated (UJ).

Initial Calibration - A three- to five-point curve bracketing sample concentration, plus a blank, must be generated daily. The correlation coefficient of the curve must be equal to or exceed 0.995.

If the minimum number of standards is not used for initial calibration or if the instrument is not calibrated daily and each time the instrument is set up:

- o Qualify data as unusable (R).

If the correlation coefficient is less than 0.995:

- o Qualify results greater than the IDL as estimated (J)
- o Qualify results less than the IDL as estimated (UJ).

Continuing Calibration - The %R must be within the 90 to 110% control limit.

If the continuing calibration %R falls outside the criteria:

- o Qualify results greater than the IDL as estimated (J) and results less than the IDL as estimated (UJ).

Method Blanks - At least one preparation blank must be prepared with each batch of samples. Blanks shall contain less than the detection limit for all analytes.

If concentration of the associated blanks is above the detection limit and if the lowest analyte concentration is <5 times the blank:

- o Perform reanalysis of the sample.

If reanalysis was not possible:

- o Report and flag data as estimated (J).

BLANK VALUES SHALL NEVER BE SUBTRACTED FROM THE SAMPLE.

Field Blanks - If contaminant analytes are detected in samples at concentrations of <5 times the concentration found in the highest associated blank:

- o Consider results suspect and report them as estimated (J).

LCSs - Any LCS exceeding internal QC limits set by the laboratory for a given sample matrix shall require all data from the associated batch of samples to be closely inspected.

If no analytical problems are found:

- o The data and out-of-control point shall be discussed in the QC section of the report.

If problems are found in the analytical data:

- o Samples associated with the batch shall be reanalyzed and data from reanalysis reported.

If holding times are exceeded in the reanalysis, both sets of data shall be presented.

A discussion of data reported when the LCS is out of control must be presented in the QC section of both the MPR and final report.

If the LCS results are outside internal laboratory limits and if matrix spike results are outside the CLP limits:

- o The laboratory will either reanalyze the samples or data will be flagged not usable (R).

Spike/Duplicate - Spike recovery (%R) must be within the limits of 75 to 125%.

If spike recovery is greater than 125% and reported sample results are less than the IDL:

- o Data are acceptable.

If spike recovery is greater than 125% or less than 75% and sample results are greater than the IDL:

- o Qualify data for these samples as estimated (J).

If spike recovery falls within the range of 30 to 74% and sample results are less than the IDL:

- o Qualify data for these samples as estimated (UJ).

If spike recovery results fall less than 30% and sample results are less than the IDL:

- o Qualify data for these samples as unusable (R).

6.3 LEVEL D DATA VALIDATION GUIDELINES

At a minimum, data generated from Level D analyses shall be validated per the CLP criteria as outlined in the following documents.

- o EPA, Hazardous Site Control Division, "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses," latest edition.

- o EPA, Office of Emergency and Remedial Response, "Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses," latest edition.

For methods not listed in these documents, a similar procedure outlining validation of holding times, initial calibration, continuing calibration, spikes, blank/spikes, duplicates, and blank vs sample results will be submitted by the prime contractor and the laboratory. The validation procedure must be approved by the AQCS.

6.4 LEVEL E DATA VALIDATION GUIDELINES

Level E data review and validation guidelines are dependent upon the analyses requested. Review and validation guidelines must be defined in the project work plan before the initiation of sampling. At a minimum, criteria for evaluating holding times, initial calibration, LCSs, and blanks must be defined.

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