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SAMPLING AND CHEMICAL ANALYSIS QUALITY ASSURANCE REQUIREMENTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM

NEESA 20.2-047B

NAVAL ENERGY AND ENVIRONMENTAL SUPPORT ACTIVITY
Port Hueneme, California 93043

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CONTENTS

	Page No.
LIST OF FIGURES	v
LIST OF TABLES	vii
LIST OF ACRONYMS	ix
1. INTRODUCTION	1
1.1 SCOPE	1
1.2 APPROACH	2
1.3 LEVELS OF QC	2
1.3.1 Level D QC	4
1.3.2 Level C QC	4
1.3.3 Level E QC	4
1.4 ROLES AND RESPONSIBILITIES	5
2. APPROVAL PROCESS	9
2.1 OVERVIEW	9
2.1.1 The Laboratory Approval Process	9
2.1.2 Laboratory Reapproval	11
3. SITE-SPECIFIC QC REQUIREMENTS	13
3.1 CONTENTS OF SITE-SPECIFIC QC SECTION	13
3.2 STATE AND REGION REQUIREMENTS FOR SITE QC PLAN	13
3.3 SAMPLING DESIGN	14
3.4 PRESERVATIVES	14
3.5 SAMPLE CONTAINER CLEANING PROCEDURES	14
3.6 ORGANIZATION AND PERSONNEL	18
3.7 FIELD QC SAMPLES	18
3.8 CHAIN OF CUSTODY	20
3.9 SHIPPING REQUIREMENTS	21
3.10 SAMPLE RECEIPT	21
4. LABORATORY QA PLAN REQUIREMENTS	23
4.1 PURPOSE AND SCOPE	23
4.2 ORGANIZATION AND CONTENTS OF PLAN	23
4.3 CONTROL SAMPLES	27
4.4 CONTROL CHARTS	28

CONTENTS (continued)

	Page No.
4.4.1 Method Blank/Spike Control Program	29
4.4.2 Control Sample Quality	29
4.4.3 Minimum Statistical Control Charting	30
4.4.4 Minimum Criteria For An Out-Of-Control Condition	30
4.4.5 Reactions to Out-of-Statistical-Control Conditions on Control Samples	31
4.4.6 Administration of the Control Charts	31
4.4.7 Statistical Quality of the Control Charts	31
4.4.8 Example of Setting Control Limits	32
4.5 OUT-OF-CONTROL EVENTS	33
4.6 CORRECTIVE ACTION REPORT	37
5. PROFICIENCY TESTING	39
5.1 SUBMITTING THE PROFICIENCY SAMPLES	39
5.2 RESULTS OF PROFICIENCY SAMPLES	39
5.3 EVALUATION OF PROFICIENCY SAMPLE RESULTS	39
6. LABORATORY INSPECTION	41
6.1 PURPOSE OF INSPECTION	41
6.2 INSPECTION PROCEDURE	41
7. ANALYTICAL METHODS	43
7.1 QC REQUIREMENTS FOR THE LABORATORY	44
7.2 DELIVERABLES	60
7.3 DATA VALIDATION	60
7.3.1 Level D Validation	60
7.3.2 Level C Data Validation Guidelines	63
8. MAINTAINING LABORATORY APPROVAL	69
8.1 MONTHLY PROGRESS REPORT	69
8.2 FINAL REPORT	70
8.3 FINAL QC DATA REPORT	71
9. DATA FORMAT	73
GLOSSARY	75
BIBLIOGRAPHY	83
KEY CONTACTS FOR THE NAVY INSTALLATION RESTORATION PROGRAM	87

LIST OF FIGURES

Figure No.	Title	Page No.
1.1	QA Organizations	6
2.1	Approval and Review Process	10
2.2	Laboratory Reapproval Process	12
4.1	Control Chart Example Data and Control Value of Table 4.3	35
4.2	Control Chart Example Data and Second Iteration Control Values of Table 4.3	36
9.1	Example of Data Format for Final Report	74

LIST OF TABLES

Table No.	Title	Page No.
1.1	Overall Plan for QC Based on Type of Site	3
3.1	Required Containers, Preservation Techniques, and Holding Times	15
3.2	Preservative and Holding Times for the CLP	16
3.3	Preservative and Holding Times for EPA-Document SW846 (3rd Ed.)	17
3.4	Field QC Samples Per Sampling Event	19
4.1	Typical Methods to be Monitored Through Measurement Control Program	31
4.2	Control Charts Formulae for Water and Soil Control Batch Program	32
4.3	Data and Caculations for Control Chart Example	34
7.1	List of Approved Biological Test Procedures	45
7.2	List of Approved Inorganic Test Procedures	46
7.3	List of Approved Test Procedures for Nonpesticide Organic Compounds	54
7.4	List of Approved Test Procedures for Pesticides	56
7.5	List of Approved Radiological Test Procedures	58
7.6	Data Set Deliverables for Level C QA	61

LIST OF ACRONYMS

AA	Atomic Absorption
ASTM	American Society for Testing Materials
BNA	Base Neutral Acids
CCC	Calibration Check Compounds
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Protocol
COC	Chain of Custody
DQO	Data Quality Objective
EFD	Engineering Field Division (U.S. Navy)
EIC	Engineer in Charge (U.S. Navy)
FEL	Field Equipment Log
GC	Gas Chromatography
HSL	Hazardous Substances List
IRP	Installation Restoration Program
ICP	Inductively Coupled Plasma
LQAC	Laboratory Quality Assurance Coordinator
MPR	Monthly Progress Report
MS	Mass Spectrometry
NCR	NEESA Contract Representative (Martin Marietta Energy Systems, Inc.)
NEESA	Naval Energy and Environmental Support Activity
NPL	National Priorities List
PCB	Polychlorinated Biphenyl
PVC	Polyvinyl Chloride
QA	Quality Assurance
QC	Quality Control
RRF	Relative Response Factor
RSD	Relative Standard Deviation
SOW	Statement of Work
SPCC	System Performance Check Compounds
TCL	Toxic Contaminant List
VOA	Volatile Organic

FIELD REQUIREMENTS HIGHLIGHTED IN

1. INTRODUCTION

The Installation Restoration Program (IRP) identifies and evaluates past hazardous material disposal sites in order to control the migration of hazardous contaminants. The program also controls hazards that may result from these past disposal operations. The IRP has the following phases: Preliminary Assessment/Records Search, Site Inspection/Remedial Investigation, Technical Base Development, Feasibility Study, and implementation of selected alternatives for remediation. During any of these phases, analysis of soil, water, and waste samples may be performed. The Navy program for the IRP includes performing field investigations and analysis of samples. The purpose of this document is to specify the requirements for the control of the accuracy, precision, and completeness of the samples, and data from the point of collection through reporting. Because every instance and concern may not be addressed in this document, contractors are encouraged to discuss any questions with the Navy engineer in charge (EIC) or the appropriate Naval Energy and Environmental Support Activity (NEESA) contract representative (NCR).

1.1 SCOPE

Laboratories performing studies in support of the IRP are required to obtain Navy approval prior to beginning field studies or analyses of samples and to maintain that approved status throughout the site characterization. The laboratory approval is specific to a particular study for a given site and Statement of Work (SOW). The Navy Requirements document provides guidance to the laboratories on obtaining and maintaining approval. Should more than one laboratory be involved in the analysis of samples from a single site, each laboratory performing analysis must be approved and must comply with the quality control (QC) requirements. These objectives and requirements conform, in general, with the *U.S. Environmental Protection Agency Federal Register*, November 29, 1983 (p. 53937 or 40 CFR 792), the *Food and Drug Administration Federal Register*, December 22, 1978 (p. 59986 or 21 CFR 58), the *Quality Assurance Program Requirements for Nuclear Facilities*, ANSI/ASME NQA-1, 1986 ed., and the *Interim Guidelines and Preparing Quality Assurance Project Plans* (U.S. EPA, EPA-600/4-83-004, QAMS-005/80).

Each laboratory is required to submit a Laboratory Analysis Quality Assurance (QA) Plan. Each engineering contractor must submit a site work plan as part of the approval process. The laboratory's QA plan and the site work plan are emphasized, since the content of those plans and the laboratory's strict adherence to it are essential for obtaining and maintaining Navy approval. Certain basic requirements are stressed--a laboratory QA coordinator (LQAC), the use of accepted analytical methods, careful documentation of chain of custody (COC), corrective action policy, and use of control charts. The laboratory-approval process and subsequent laboratory reporting requirements provide the mechanism for verifying that a laboratory is adhering to its QA and work plan.

Currently, most IRP studies do not include analysis of air, plant, or tissue samples. Future revisions that will include more discussion as to available methods for biota and air are planned. If questions on these methods arise, the NCR may be consulted at the Martin Marietta Energy Systems, Inc., Analytical Chemistry Department at the Oak Ridge Gaseous Diffusion Plant. Where Environmental Protection Agency (EPA) methods are not available for biota, methods from other agencies and published methods which have undergone method validation by the laboratory requesting approval must be used. On occasion, when methods are required for biota and no EPA method is available, the proposed method must be submitted to the NCR for approval.

1.2 APPROACH

The approach reflected in this document is one of outlining requirements and allowing the laboratories, principally through their QA plans, to detail their approach to meeting these requirements. For example, with the exception of the laboratory control sample program, see Sect. 4.4, the discussion of QC procedures includes a requirement that warning and action limits be set but allows each laboratory to describe its procedures for establishing such limits. The specific organization and presentation of the laboratory plan are left largely to the discretion of the laboratory, although certain areas must be addressed.

In order for the above approach to work, emphasis must be placed on effective communication between the laboratory, the Navy EIC, the NCR, and the engineering subcontractor. All documents must be concise, well organized, and free of jargon that might hinder constructive review and evaluation.

1.3 LEVELS OF QC

Data quality objectives (DQOs) are requirements needed to support decisions relative to the various stages of remedial actions. Throughout the project planning process, DQOs are supplied through qualitative and quantitative statements. They are specified in such documents as sampling plans, work plans and QA plans. Five general levels of analytical options to support data collection are identified by Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). The Navy has adopted three of the analytical levels as QC requirements. They are D, C, and E, which correlate with Levels 3, 4, and 5 described in *Data Quality Objectives for Remedial Response Activities Development Process* by the EPA. 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 Navy EIC. Analytical requirements for the remaining two levels have not been defined. Table 1.1 outlines the basic QC requirements at each level. The laboratory method requirements for each level of QC are outlined in Sect. 7.

Table 1.1. Overall plan for QC based on type of site

DQO Levels[1]	Type of Site	QC Requirements						
3	NPL Level D	PE sample	Laboratory[2] audit	QA Plan review	Use CLP procedures	Monthly review	10% field duplicates report	CLP validation
4	Major Non-NPL Level C	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved method[3]	Monthly review	10% field duplicates	Review of final data
5	Non-NPL Level E	PE sample	Laboratory[2] audit	QA Plan review	Use EPA-approved methods.[3] Non-EPA methods for tissue and explosives.	Monthly review	5% Field duplicates	Review of final data

[1]QC criteria for DQO Levels 1 and 2 has not been defined.
[2]All laboratory audits will be performed by the NCR.
[3]Includes methods from SW 846, Amercian Society for Testing Materials, and Federal Register.

CLP = Contract laboratory protocol
PE = Performance evaluation samples
DQO = Data quality objective

1.3.1 Level D QC

Level D QC is to be used for sites that are on or about to be on the National Priorities List (NPL). These sites are typically near populated areas and are likely to undergo litigation. Level D QC includes review and approval of the laboratory QA plan, the site work plan, and the field QA plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide monthly progress reports (MPRs) on QA. These activities will be administered and evaluated by the NCR. This audit and the analysis performance sample are in addition to those related to the EPA Superfund Program. The laboratory that performs Level D QC must have passed the performance sample furnished through the Superfund Contract Laboratory Protocol (CLP) and must be able to generate the CLP deliverables. For a Level D site, the CLP methods are used and the CLP data package generated. The Navy audit and performance sample are required in addition to any specified by the EPA Superfund Program.

1.3.2 Level C QC

A site requiring Level C QC would be a site near a populated area, not on the NPL, and not likely to be undergoing litigation. The Level C QC includes review and approval of the laboratory QA and the site work plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. The laboratory that performs Level C QC must have passed the performance sample furnished by the Superfund CLP in the past year. The laboratory does not need to be receiving CLP bid lots of samples.

Level C allows the use of non-CLP methods but requires that the methods be accepted EPA methods listed in Tables 7.1 through 7.5. All methods used must be EPA methods or be equivalent to EPA methods. Further discussion about these methods is presented in Sect. 7. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. These audits will be administered and evaluated by the NCR. The Navy audit and performance sample are required in addition to any specified by the EPA Superfund Program.

1.3.3 Level E QC

A site requiring Level E QC will be located away from a populated area, will not be an NPL site, and will have a low probability of litigation. Level E QC includes review and approval of the laboratory QA plan and the site work plan. The laboratory must successfully analyze a performance sample, undergo an audit, correct deficiencies found during the audit, and provide MPRs on QA. For Level E, the laboratory is not required to have passed a CLP performance sample. Level E allows the use of non-CLP methods but requires that the methods be accepted EPA methods listed in Tables 7.1 through 7.5. All methods used must be EPA or equivalent. Further discussion about these methods is presented in

Sect. 7. Level E QC is also appropriate for analysis of the contents of underground storage tanks where the samples are primarily pure product or waste.

1.4 ROLES AND RESPONSIBILITIES

As indicated in Fig. 1.1, the organizations involved are NEESA, the Navy Engineering Field Division (EFD), and the subcontractors. Each organization has multiple tasks and groups that support the project. Fig. 1.1 includes the structure of the organization related to the IRP process. A brief description of the key roles and responsibilities is listed.

1. Navy Energy and Environmental Support Activity

   NEESA is responsible for ensuring that the quality of laboratory analyses performed during the various phases of the IRP is acceptable. NEESA is also responsible for managing the NCR.

2. Engineering Field Division

   The EIC at the EFD provides the site information and history, provides logistical assistance, specifies the site requires investigation and reviews results and recommendations.

3. Engineer in Charge

   The EIC is responsible for coordinating procurement, finance, and reporting; for ensuring that all documents are reviewed by the NCR; for communicating comments from the NCR and other technical reviewers to the subcontractors; and for ensuring that the subcontractors address all the comments submitted and take appropriate corrective actions.

4. NEESA Contract Representative

   The NCR is responsible for ensuring that each project has appropriate overall QA. The NCR reviews laboratory QA plans, work plans, submits performance sample data, provides field and laboratory audits, and reviews data from the site. The questions from subcontractors and the EIC regarding specific field and laboratory QC practices are directed to the NCR. The NCR also provides evaluation of referee samples.

5. Engineering Subcontractor

   Each project has an engineering subcontractor that specializes in setting up the sampling for IRP studies, evaluating the hydrology and geology of a site, assessing risks of contamination, and designing and implementing clean-up techniques. Each engineering firm is required to have a laboratory available to perform sample analysis.

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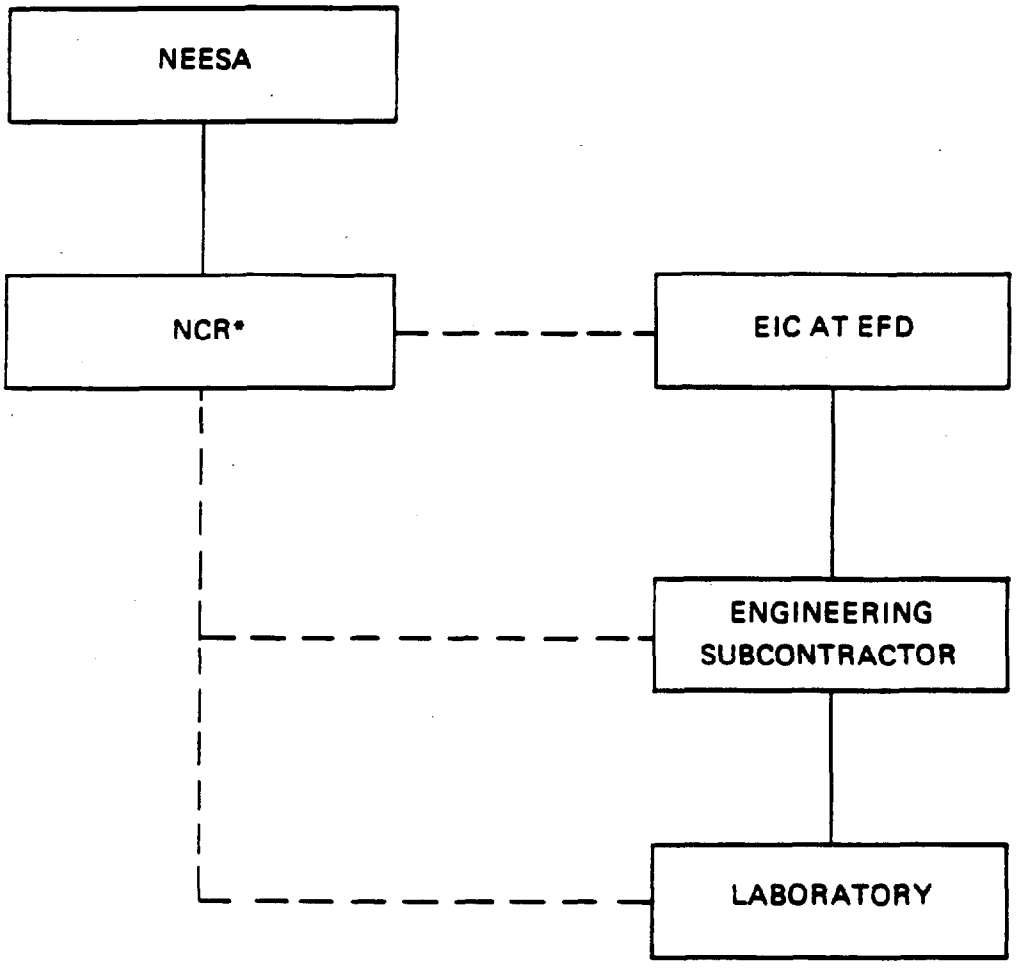

*NCR-MARTIN MARIETTA ENERGY SYSTEMS, INC.

Fig. 1.1. QA Organization

The engineering firm also employs drillers and other personnel to perform IRP tasks. The engineering firm submits a site-specific work plan.

6. Analytical Laboratory

The analytical laboratory is employed by the engineering firm and must adhere to the laboratory requirements in this document. The laboratory is required to prepare and submit a laboratory QA plan, to analyze and submit the results of proficiency testing, to submit to an on-site inspection, and to correct any deficiencies cited during inspection by the NCR. The laboratories are required to identify a LQAC responsible for overall QA. The LQAC must not be responsible for schedule, costs, or personnel other than QA assistants. It is preferred that the LQAC report to the laboratory director. The LQAC must have the authority to stop work on projects if QC problems arise which affect the quality of the data produced.

2. APPROVAL PROCESS

Prior to beginning any field studies or analysis of samples from the field, contract laboratories will be required to receive Navy approval. This section describes the laboratory approval process in terms of the activities and documentation required of participants in the process.

2.1 OVERVIEW

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

- to communicate Navy's QC requirements to the laboratories,
- to verify that such requirements are being met by each laboratory prior to analysis of Navy field samples,
- to establish plans for maintaining the QC program while work is being done for the Navy, and
- to ensure that proper communication and planning have been done between the engineering subcontractor and the laboratory prior to the laboratory receiving samples.

The above objectives will be met through an approval process that includes the following elements:

- proficiency testing through analysis of performance samples,
- laboratory inspection and audit,
- reviewing laboratory QA plans,
- reviewing site-specific QC plans, and
- reviewing of sampling plans including QC procedures.

The overall process and the above elements are described in detail in the remainder of this section.

2.1.1 The Laboratory Approval Process

The laboratory approval process, as depicted in Fig. 2.1, begins with the engineering subcontractor awarding a contract to the laboratory. The engineering subcontractor is responsible for supplying a site-specific work plan to the NCR. The laboratory and engineering contractors are required to prepare a site-specific work plan and laboratory QA plan.

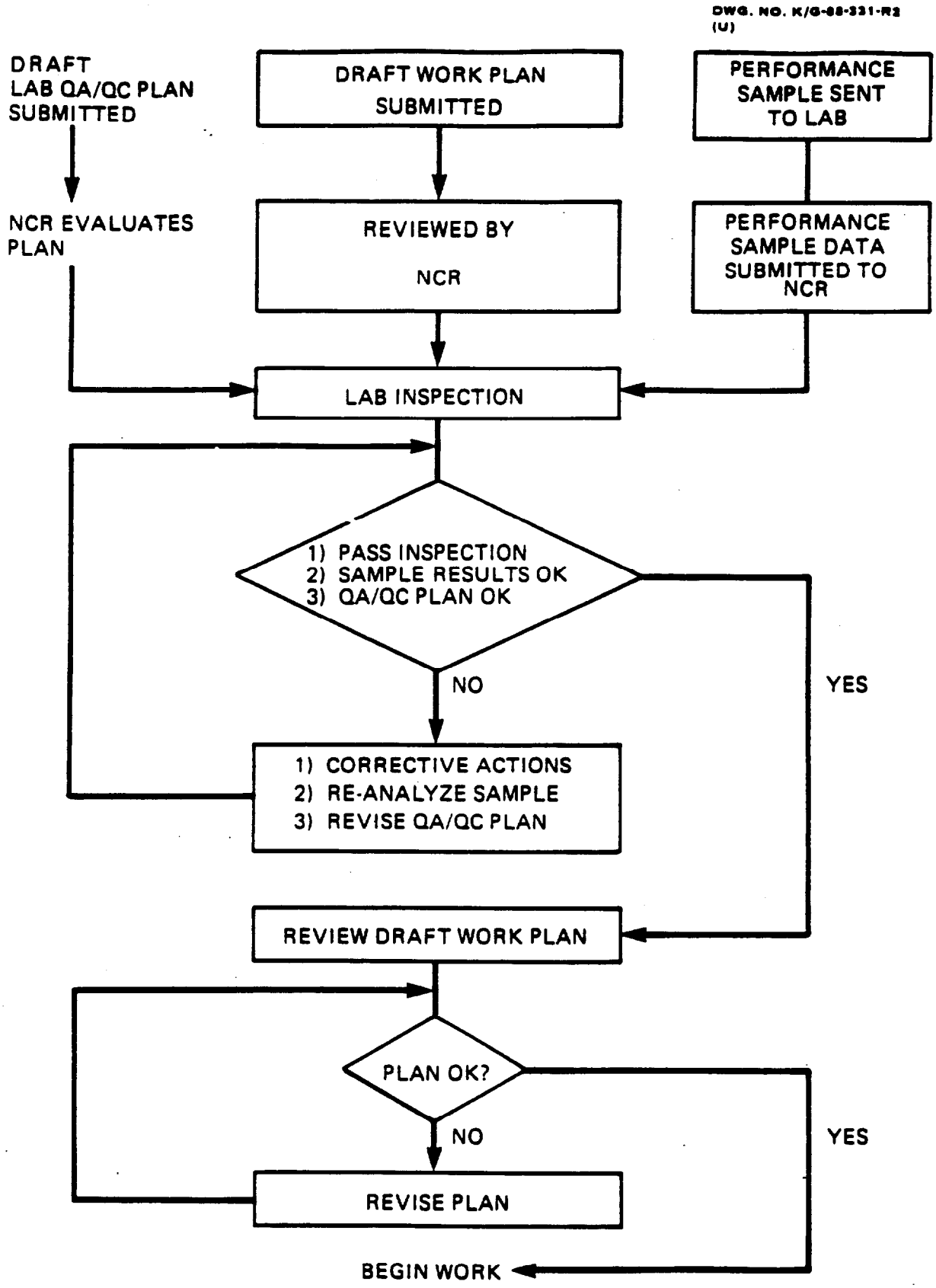

Fig. 2.1. Approval and Review Process

The site-specific work plan shall include a section on QA. This section shall either outline the field and laboratory QA or shall reference documents which outline the QA procedures. The laboratory shall successfully analyze proficiency samples. The site-specific work plan, QA plans, and the results of the proficiency test are submitted to the NCR who will evaluate this information. The QA plans, the proficiency test results, and a draft work plan shall be received and evaluated by the NCR prior to scheduling a laboratory inspection. Based on the results of these evaluations and the inspection, the laboratory and engineering firm may be required to revise their QA plans, to retest a proficiency sample(s), to revise the work plan, or to prepare and implement a corrective action plan addressing deficiencies cited during the inspection.

Approval to begin work on samples is based on a combination of satisfactory QA plans and a site-specific work plan, satisfactory results of proficiency testing, and acceptable laboratory inspection. Approval may be granted to perform all or part of the methods required for a study.

2.1.2 Laboratory Reapproval

If a laboratory is requested to analyze samples from a second site, the NCR will evaluate the similarity between the analysis from the first and the second sites. The past performance of the laboratory and the time elapsed from previous sample analysis determine the steps the laboratory must follow to be reapproved. If a laboratory has performed well in the past, if the methods in the first and second work plan are similar, and if it has been less than a year since the first approval, the engineering contractor may only need to submit the site work plan, and work may proceed. If the laboratory's past performance was satisfactory but it has been longer than a year since a performance sample was analyzed, the laboratory must successfully analyze a new performance sample.

Any changes in personnel or general laboratory QA must be submitted to the NCR prior to receiving approval to begin the next site.

Figure 2.2 shows a flow diagram of the reapproval process.

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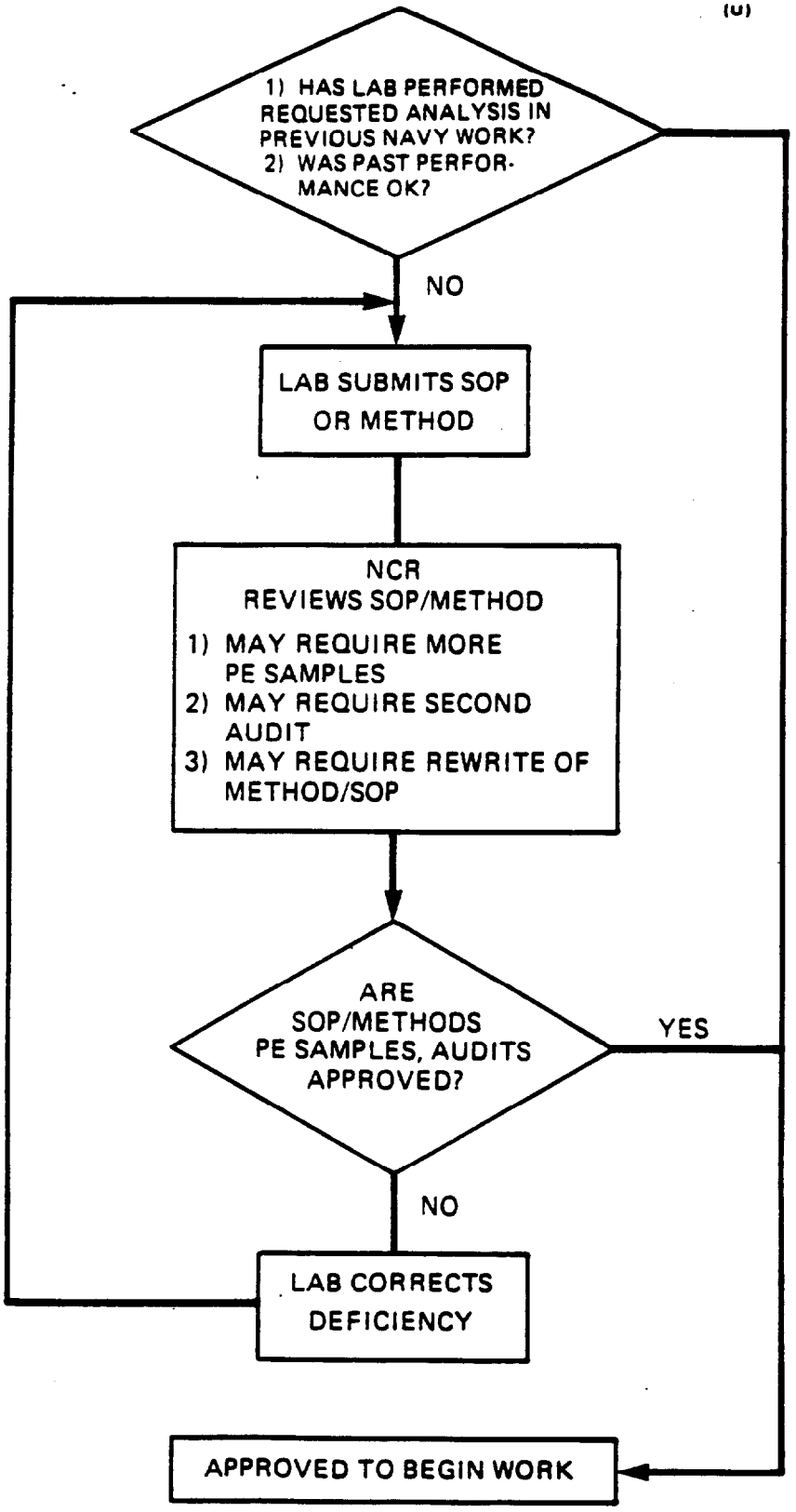

Fig. 2.2. Laboratory Reapproval Process

3. SITE-SPECIFIC QC REQUIREMENTS

The following are the requirements for the site-specific QC section to be included in the site-specific work plan or to be presented as a separate QC document.

3.1 CONTENTS OF SITE-SPECIFIC QC SECTION

1. The laboratory must be identified along with all other subcontractors.

2. Any pertinent state environmental or EPA federal/regional requirements shall be presented. This includes specific procedures or clean-up levels.

3. References must be made to the appropriate corporate or laboratory QA plans which contain pertinent information.

4. A discussion of COC and shipping practices must be provided.

5. Tables of the following shall be included.

   - Analytical methods and numbers of samples of each matrix to be collected at each site.

   - List of analytes to be identified and quantitated.

   - List of holding times, preservatives, amount of sample required, and container requirements.

   - List of the number, type, and matrix of field and laboratory QC samples by site. This includes trip blanks, equipment rinsates, field blanks, field duplicates, laboratory method blanks, laboratory matrix spikes and duplicates.

   - List of sample volume and bottles vs method.

6. All site-specific field sampling procedures which are not included in any corporate QA plan shall be presented.

7. Decontamination procedures for both drilling and sampling equipment shall be described.

8. Data quality objectives shall be discussed. This shall include precision, accuracy, and completeness required for acceptable data.

3.2 STATE AND REGION REQUIREMENTS FOR SITE QC PLAN

In addition to, or in place of, the requirements in this document, those requirements specific to the state or EPA region applicable to the

site shall be considered. Any state- or region-specific requests must be addressed in the site work plan.

3.3 SAMPLING DESIGN

Every site is unique in its own way. To this end, a sampling rationale shall be included with the work plan. The rationale should define and explain thoroughly the sampling statistics, the equipment involved, and anticipating data to be gained by this proposed methodology.

3.4 PRESERVATIVES

After samples have been taken, they shall be sent to the laboratory for analysis within 24 h after collection to ensure that the most reliable and accurate answers will be obtained as a result of the analysis. The holding time begins from the date of collection in the field. Preservatives shall be added in the field. Tables 3.1, 3.2, and 3.3 present the holding times, type of containers, and preservatives to be used. A table corresponding to each of the three different methods such as those from the *Federal Register; SW-846* 3rd ed.; and CLP is presented. The site-specific plan shall outline which preservatives will be used, and it shall be based on these tables. Freezing of samples shall not be permitted.

3.5 SAMPLE CONTAINER CLEANING PROCEDURES

In general, glass bottles with Teflon lids are used for organic samples, while polypropylene is used for metals and other inorganics. The following specifies the bottle cleaning required. If precleaned bottles are purchased, this must be noted in the work or field QA plan and approved by the NCR. If precleaned bottles are used, a certificate indicating that the bottles are analyte free must be provided.

3.5.1 Cleaning Procedure for Glass Bottles

1. Wash glass bottles, Teflon liners, and caps in hot tap water with laboratory-grade nonphosphate detergent.

2. Rinse three times with tap water.

3. Rinse with 1:1 nitric acid (metals-grade), American Society for Testing Materials (ASTM) Type I deionized water.

4. Rinse three times with ASTM Type I deionized water.

5. Rinse with pesticide-grade methylene chloride using 20 mL for 1/2-gal container and 5 mL for 4- and 8-oz containers.

6. Oven dry at 125°C. Allow to cool to room temperature in an enclosed contaminant-free environment.

Table 3.1 Required containers, preservation techniques, and holding times (40 CFR, Part 136, July 1, 1987)

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Table IA—Bacterial Tests:			
1–4 Coliform, fecal and total	P, G	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	6 hours.
5 Fecal streptococci	P, G	do	Do.
Table IB—Inorganic Tests:			
1 Acidity	P, G	Cool, 4°C	14 days.
2 Alkalinity	P, G	do	Do.
4 Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
9 Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
11 Bromide	P, G	None required	28 days.
14 Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15 Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
16 Chloride	P, G	None required	Do.
17 Chlorine, total residual	P, G	do	Analyze immediately.
21 Color	P, G	Cool, 4°C	48 hours.
23–24 Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH > 12, 0.6g ascorbic acid [6]	14 days [7]
25 Fluoride	P	None required	28 days.
27 Hardness	P, G	HNO_3 to pH < 2, H_2SO_4 to pH < 2	6 months
28 Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43 Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Metals: [1]			
18 Chromium VI	P, G	Cool, 4°C	24 hours.
35 Mercury	P, G	HNO_3 to pH < 2	28 days.
3, 5–8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75 Metals, except chromium VI and mercury	P, G	do	6 months.
38 Nitrate	P, G	Cool, 4°C	48 hours.
39 Nitrite-nitrate	P, G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
40 Nitrite	P, G	Cool, 4°C	48 hours.
41 Oil and grease	G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
42 Organic carbon	P, G	Cool, 4°C, HCl or H_2SO_4 to pH < 2	Do.
44 Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46 Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately
47 Winkler	G only	Fix on site and store in dark	8 hours.
48 Phenols	G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
49 Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50 Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH < 2	28 days.
53 Residue, total	P, G	Cool, 4°C	7 days.
54 Residue, Filterable	P, G	do	48 hours.
55 Residue, Nonfilterable (TSS)	P, G	do	7 days.
56 Residue, Settleable	P, G	do	48 hours.
57 Residue, volatile	P, G	do	7 days.
61 Silica	P	do	28 days.
64 Specific conductance	P, G	do	Do.
65 Sulfate	P, G	do	Do.
66 Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH > 9.	7 days.
67 Sulfite	P, G	None required	Analyze immediately
68 Surfactants	P, G	Cool, 4°C	48 hours
69 Temperature	P, G	None required	Analyze
73 Turbidity	P, G	Cool, 4°C	48 hours
Table IC—Organic Tests.[8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 66, 68, 69, 92–95, 97 Purgeable Halocarbons.	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	14 days.
6, 57, 90 Purgeable aromatic hydrocarbons	do	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5], HCl to pH2 [9]	Do.
3, 4, Acrolein and acrylonitrile	do	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5], Adjust pH to 4–5 [10]	Do.
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 98 Phenols [11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
7, 38 Benzidines [11]	do	do	7 days until extraction [12]
14, 17, 48, 50–52 Phthalate esters [11]	do	Cool, 4°C	7 days until extraction, 40 days after extraction
72–74 Nitrosamines [11,13]	do	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$ [5]	Do.
76–82 PCBs [11] acrylonitrile	do	Cool, 4°C	Do.
54, 55, 85, 86 Nitroaromatics and isophorone [11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5] store in dark	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 84, 88, 94, 96 Polynuclear aromatic hydrocarbons [11]	do	do	Do.
15, 16, 21, 31, 75 Haloethers [11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	Do.
29, 35–37, 60–63, 91 Chlorinated hydrocarbons [11]	do	Cool, 4°C	Do.
97 TCDD [11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$ [5]	Do.
Table ID—Pesticides Tests.			
1–70 Pesticides [11]	do	Cool, 4°C, pH 5–8 [14]	Do.
Table IE—Radiological Tests.			
1–5 Alpha, beta and radium	P, G	HNO_3 to pH < 2	6 months.

[1] Polyethylene (P) or Glass (G).

[2] Samples preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnotes 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidines).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxygen-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of diquat, add 0.008% $Na_2S_2O_3$.

Table 3.2 Preservative and holding times for the CLP

Parameter	Container	Preservative	Holding Time Soil	Holding Time Water
Volatiles by gas chromatography/mass spectroscopy (GC/MS)	Water - 40-mL glass vial with Teflon-lined septa	Cool, 4°C	10 days	10 days
	Soil-glass with Teflon-lined septa			
PCB/ pesticides	G, Teflon-lined-lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
Extractable organics	G, Teflon lined-lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
Metals	P, G	HNO_3 to pH<2	6 months	6 months
Mercury	P, G	HNO_3 to pH<2	30 days	30 days
Cyanide	P, G	NaOH to pH>12 Cool 4°C add 0.6 g ascorbic acid if residual chlorine present	14 days	14 days
Chromium VI	P, G	HNO_3 to pH<2	24 h	24 h

7. Place liners in lids and cap containers.

8. Store in contaminant-free area. (Amber glass containers shall not be exposed to sunlight).

3.5.2. Cleaning Procedure for Bottles Used for Volatile Organics (40-mL Glass)

1. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot tap water using laboratory-grade nonphosphate detergent.

Table 3.3 Preservatives and holding times for EPA document SW-846 (3rd ed.)

Parameter	Container	Preservative	Holding Time Soil	Holding Time Water
Volatiles by GC/MS, and GC	Water - 40-mL glass vial with Teflon-lined septa Soil-glass with Teflon-lined septa	Cool, 4°C	14 days	14 days
PCB/ pesticides	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Extractable organics	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Metals	P, G	HNO_3 to pH<2	6 months	6 months
Mercury	P, G	HNO_3 to pH<2	30 days	30 days
Cyanide	P, G	NaOH to pH>12 Cool 4°C add 0.6 g ascorbic acid if residual chlorine present	14 days	14 days
Chromium	P, G	HNO_3 to pH<2	24 h	24 h

2. Rinse three times with tap water.

3. Rinse three times with ASTM Type I deionized water.

4. Oven dry vials, septa, and liners at 125°C.

5. Allow vials, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

6. Seal 40-mL vials with septa (Teflon side down) and cap.

7. Store in contaminant-free area.

3.5.3. Cleaning Procedure for Polyethylene Bottles

1. Wash polyethylene bottles and caps in hot tap water with laboratory-grad nonphosphate detergent.

2. Rinse with 1:1 nitric acid (metals-grade), ASTM deionized water).

3. Rinse three times with ASTM Type I deionized water.

4. Invert and air dry in contaminant-free environment.

3.5.4. All Bottles Should Be

1. Capped and labeled with sample numbers and packed in cooler or box.

2. Stored in contaminant-free area.

3.6 ORGANIZATION AND PERSONNEL

All management personnel responsible for performing field sampling or analytical work shall be listed along with their job assignment and years of experience in performing this type of work. Any education and training related to the tasks performed for this project shall also be listed.

3.7 FIELD QC SAMPLES

Although the number of QC samples changes, the types of field QC samples remain the same regardless of the level of QC implemented. Table 3.4 lists the percentage of field QC samples per level per sample matrix. A sampling event is considered to be from the time the sampling personnel arrive at the site until these personnel leave for more than a day. An example of two events would occur if sampling personnel went to a site for three weeks, drilled borings, and put groundwater wells in place. During this visit, soil and water samples were collected. The sampling crew left the site for two months, thus concluding the first sampling event. The crew later returned to collect another set of groundwater samples over a three-day period. The second visit would constitute the second sampling event.

The following information defines and explains the blanks, duplicates, and referee samples.

1. Trip Blanks

Trip blanks are defined as samples which originate from analyte-free water taken from the laboratory to the sampling site and returned to the laboratory with the volatile organic (VOA) samples. One trip

Table 3.4. Field QC samples per sampling event

Type of Sample	Level C		Level D		Level E	
	Metal	Organic	Metal	Organic	Metal	Organic
Trip blank (for volatiles only)	NA[1]	1/cooler	NA[1]	1/cooler	NA[1]	1/cooler
Equipment rinsate[2]	1/day	1/day	1/day	1/day	1/day	1/day
Field blank	1/source/event for all levels and all analytes					
Field duplicates[3]	10%	10%	10%	10%	5%	5%
Referee duplicate[3]						

[1]NA - Not applicable.

[2]Samples are collected daily; however, only samples from every other day are analyzed. Other samples are held and analyzed only if evidence of contamination exists.

[3]The duplicate must be taken from the same sample which will become the laboratory matrix/spike duplicate for organics or for the sample used as a duplicate in inorganic analysis.

blank should accompany each cooler containing VOAs, should be stored at the laboratory with the samples, and analyzed by the laboratory. Trip blanks are only analyzed for VOAs.

2. Equipment Rinsates

Equipment rinsates are the final analyte-free water rinse from equipment cleaning collected daily during a sampling event. Initially, samples from every other day should be analyzed. If analytes pertinent to the project are found in the rinsate, the remaining samples must be analyzed. The results from the blanks will be used to flag or assess the levels of analytes in the samples. This comparison is made during data validation. The rinsates are analyzed for the same parameters as the related samples.

3. Field Blanks

Field blanks consist of the source water used in decontamination and steam cleaning. At a minimum, one field blank from each event and each source of water must be collected and analyzed for the same parameters as the related samples.

has been interpreted as [illegible] water used in steam [illegible]
and [illegible] water used in [illegible]

4. Field Duplicates/Splits

Duplicates or splits for soil samples are collected, homogenized, and split. All samples except VOAs are homogenized and split. Volatiles are not mixed, but select segments of soil are taken from the length of the core and placed in 40-mL glass vials. Cores may be sealed and shipped to the laboratory for subsampling if the project deems this appropriate. The duplicates for water samples should be collected simultaneously. Field duplicates should be collected at a frequency of 10% per sample matrix for Levels D and C. For Level E, the duplicates should be analyzed at a frequency of 5%. All the duplicates should be sent to the primary laboratory responsible for analysis. The same samples used for field duplicates shall be split by the laboratory and be used as the laboratory duplicate or matrix spike. This means that for the duplicate sample, there will be analyses of the normal sample, the field duplicate, and the laboratory matrix spike/duplicate.

5. Referee Duplicates

Duplicates/splits shall be sent to the referee QA laboratory if regulators (state or region) collect split samples or if a special problem occurs in sample analysis or collection. These duplicates/splits are collected and analyzed in addition to the field duplicates mentioned in the previous paragraph.

3.8 CHAIN OF CUSTODY

Samples, other than those collected for *in situ* field measurements or analyses, are identified by using a standard sample label which is attached to the sample container. The sample labels are sequentially numbered and are accountable. The following information shall be included on the sample label.

1. Site name.
2. Field identification or sample station number.
3. Date and time of sample collection.
4. Designation of the sample as a grab or composite.
5. Type of sample (matrix) and a brief description of the sampling location.
6. The signature of the sampler.
7. Sample preservation and preservative used.
8. The general types of analyses to be conducted.

If a sample is split with another party, sample labels with identical information shall be attached to each of the sample containers.

The COC record is used to record the custody of samples and shall accompany samples at all times. The following information shall be supplied to complete the COC record.

1. Project name.
2. Signature of samplers.
3. Sampling station number or sample number, date and time of collection, grab or composite sample designation, and a brief description of the type of sample and sampling location.
4. Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time that they received the samples on the form.
5. Matrix.

Sample analysis request sheets serve as official communication to the laboratory of the particular analyses required for each sample and provide further evidence that the COC is complete.

COC records initiated in the field shall be placed in a plastic cover and taped to the inside of the shipping container used for sample transport from the field to the laboratory.

3.9 SHIPPING REQUIREMENTS

Shipping containers shall be secured using nylon strapping tape and custody seals to ensure that samples have not been disturbed during transport. The custody seals shall be placed on the containers so they cannot be opened without breaking the seal.

Samples which must be kept at 4°C shall be shipped in insulated containers with either freezer forms or ice. If ice is used, it shall be placed in a container so that the water will not fill the cooler as the ice melts. The samples shall be shipped within 24 h of collection to allow the laboratory to meet holding times. The Department of Transportation regulations shall be used for packaging, quantities of shipment, and the way samples are sent. Each subcontractor responsible for sampling shall become familiar with the regulations.

Copies of the signed COC forms shall be delivered with the data packages. The originals shall remain on file with the contractor or with the laboratory.

3.10 SAMPLE RECEIPT

Upon receipt, the laboratory shall sign and keep copies of the air bill. The COC shall be signed. The temperature of the cooler shall be measured and documented. The condition of the samples shall be documented. If any breakage or discrepancy arises between COC, sample labels, and requested analysis, the sample custodian will notify the engineering subcontractor. The pH of incoming samples shall be checked and documented upon receipt. Any discrepancy or improper preservation shall be noted by the laboratory as an out-of-control event and shall be documented on

an out-of-control form with the corrective action taken. The out-of-control form shall be signed and dated by the custodian and any other person responsible for corrective action.

4. LABORATORY QA PLAN REQUIREMENTS

An essential step in the sequence of events leading to Navy approval of contract laboratories is the preparation and acceptance of a QA plan for each laboratory.

The contents and format of an acceptable plan are described below. If the laboratory has a general QA plan in place, this should be sent for review. In this case, a site-specific QA plan may not be needed. Any deviations or additions to the normal laboratory QA should be documented in the site-specific work plan.

4.1 PURPOSE AND SCOPE

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

4.2 ORGANIZATION AND CONTENTS OF PLAN

The items listed below may be presented in any order that the laboratory desires; however, the list includes the items that are required in the QA plan.

1. Title Page with Provision for Signatures
2. Table of Contents
3. Organization and Personnel
4. Personnel Training
5. Sample-Handling Practices and COC
6. Material Procurement and Control
7. Facilities and Equipment
8. Equipment Maintenance
9. Analytical Procedures
10. Calibration
11. Limits of Detection
12. Analysis of QC Samples and Documentation
13. Out-of-Control Events and Corrective Action
14. Data Evaluation and Data Reduction
15. Holding Times and Preservatives
16. Internal Laboratory Audits and Approvals from Other Agencies
17. Document Control
18. QA Reports to Management
19. Accuracy, Precision, and Completness

1. Title Page with Provisions for Signature

   A title page with provision of approval signatures and date of revision shall be provided.

2. Table of Contents

   A table of contents shall be provided.

3. 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 authority of key laboratory personnel are described with emphasis on the authority given the LQAC with regard to QC monitoring, reporting, and corrective action.

   An appendix shall contain a list of all the personnel, their assignments and responsibilities, degrees of education, and the years of applicable experience.

4. Personnel Training

   The plan shall address how personnel are trained in laboratory methods, in QC, and in safety policies.

5. Sample-Handling Practices and COC

   This section shall include tracking of samples through the laboratory, receipt of samples, verification of preservation, login of samples, and COC. Sample storage and disposal shall also be included. Preparation of bottles and glassware washing shall also be included.

6. Material Procurement and Control

   This section shall include a description of procedures for purchasing materials, quality inspection prior to use in sample analysis, chemical and standard inventory, solvent storage policies, and laboratory waste disposal.

7. Facilities and Equipment

   A list of basic types of equipment, year of purchase, and general description of the facility assures that the laboratory is large enough to handle the sample load expected and that the equipment is capable of performing the analysis.

8. Equipment Maintenance

   This section shall include general information as to who performs both major, preventive, and day-to-day maintenance and how it is documented.

9. Analytical Procedures

This section shall contain a list of all procedures that the laboratory offers (by method number and matrix) in the event that future work may require analyses not specified in the SOW.

Any method variances must be reported, and any documentation from EPA for approvals-of-method variances shall be presented to assure that they are known prior to sample analysis.

The laboratory policy and implementation procedures shall emphasize that methods are available to the analyst.

10. Calibration

This section shall include calibration procedures by instrument type, calibration frequency, reference standards used, calibration acceptance criteria, and calibration documentations procedures. Calibration applies to both instruments such as gas and liquid chromatography, GC/MS, inductively coupled plasma (ICP), atomic absorption (AA), infrared and ultraviolet spectroscopy, and wet chemical methods.

The method for assuring that balances, refrigerators, and ovens are accurate and how these pieces of equipment are checked must be outlined. Balances and ovens must be checked prior to use. Balances must also be checked by an outside company annually.

11. Limits of Detection

The laboratory shall indicate what the typical method-detection limits are for water, soil, and any other matrix commonly analyzed by the laboratory, with the understanding that this varies with the sample matrix. The procedures for determining the limits of detection for each type of method and the frequency of detection limit verification shall be outlined.

12. Analysis of QC Samples and Documentation

This section shall summarize the QC procedures and documentation to be used in the day-to-day operation of the laboratory. The discussion shall 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. 4.4) for explanation of control charts and their usage;
- the frequency of blanks and other QC samples and controls;
- how the data from the QC samples are reported and reviewed;
- who reviews and makes decisions from the QC data;
- details of how requirements of minimum control program in Sect. 8.2 will be met; and
- verification of calibration.

13. Out-of-Control Events and Corrective Action

This section shall contain a definition as to the types of out-of-control occurrences, how these occurrences are documented, and who is responsible for correction and documentation. It is recognized that several out-of-control events occur. Four examples are given.

- Observations Corrected at the Bench - If the calibration of an instrument is not linear, the analyst may find this and correct it prior to continuing to analyze samples. The laboratory may document this and note that the corrective action was to recalibrate and that no samples were affected, as none were analyzed prior to calibration.
- Corrective Actions Taken by Supervisor - A matrix spike recovery is out of control and the laboratory supervisor finds this after the samples for the day have been analyzed. The supervisor shall document that the laboratory blank spiked with surrogates or standards was in control and that other sample spikes were in control; therefore, no reanalysis of the sample is required.
- Corrective Actions at the Receiving Level - If the sample is broken, the analyst may note this and document whether or not more sample is available. If no more sample is available, the customer shall be notified and the decision documented.
- Statistical Out-of-Control Events - If a control chart is being monitored and the measured parameter exceeds the 99% confidence limit then explanation shall be documented as to when the parameter exceeded statistical limits.
    - Procedures shall be outlined as to what corrective action is taken if an out-of-control event occurs and how it is documented and used to improve laboratory performance. The documentation shall be easily used by all personnel and shall be part of routine laboratory procedure.

- Procedures for assuring that results for samples processed during out-of-control conditions are not reported shall be outlined.

- The conditions necessary to reestablish control and criteria for assuring the system is operating properly.

14. Data Evaluation and Data Reduction

A discussion of data evolution procedures for each analytical method as well as for an entire data set shall be included. The process of certification of reviewed data shall be outlined with an explanation of how suspect data are flagged if they are suspect but still reported.

15. Holding Times and Preservatives

The document shall include laboratory policy for meeting holding times for sample analysis and how it is assured that these are met. The sample storage requirements, holding times, and preservatives specified in Tables 3.1, 3.2 and 3.3 are minimal criteria for Navy approval.

16. Internal Laboratory Audits and Approvals from Other Agencies

A listing of approvals from other agencies and states gives an indication of the general quality and type of laboratory experience the organization has. If the laboratory performs self-audits, the frequency and method of documentation shall be outlined.

17. Document Control

The QA plan shall outline the flow of documents containing COC and data. The plan shall explain how documents are checked, signed, and filed.

18. QA Reports to Management

The plan shall include the frequency and information of management QA reports.

19. Accuracy, Precision, and Completeness

The plan shall include the laboratory's definition and method of evaluating the precision, accuracy, and completeness of measurement parameters and of evaluating data sets.

4.3 CONTROL SAMPLES

Control samples are those samples containing known concentrations of analytes that are introduced into a run of environmental samples to

monitor the performance of the analytical system. Control samples involving duplicates, blanks, analytical standards, reference materials, and spikes can be used in different phases of the overall analysis from sampling through storage, transportation, and preparation to the analytical method itself. The choice of types of controls relates to the analysis phase(s) to be controlled and the information (e.g., precision, accuracy, interferences, recovery) to be developed.

The QA plan describes generally how and where such control mechanisms are used by the laboratory. Control materials may be purchased from commercial sources, the National Bureau of Standards, or the EPA. A brief description of each control sample (or set of samples) used shall be provided in the MPR, subsequent to its introduction, and shall cover the following items.

1. Where the control samples are made.

2. How they are made.

3. How many are made and with what frequency.

4. How they 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 recovery factor of the procedures; used to check for interferences).

5. Frequency of analysis of control sample.

4.4 CONTROL CHARTS

Control charts provide a useful tool in assessing QC efforts and improving processes through graphic displays of a parameter(s) and its variability over time. The parameter plotted on the chart is usually related to control sample testing--either directly in terms of concentrations or indirectly in terms of derived information such as means of concentrations, ranges of concentrations, percent recovery of spikes, relative percent differences based on duplicate results, or slopes of least-squares data fits.

The laboratory should include in its QA plan, as required in Sect. 4.2, a brief description of the basic methodology used in control charting, covering such considerations as the following.

1. Verification that the methods are valid and working properly prior to beginning control charts.

2. Number of control samples per run.

3. Number of runs analyzed.

4. Parameters to be plotted against time and the general formulae for developing these parameters.

5. Statistical/mathematical basis for assigning warning and rejection limits on the charts in terms of, for example, standard deviation.

6. Types of shifts, trends, or biases that may typically be revealed by these charts.

4.4.1 Method Blank/Spike Control Program

Controls are required for only the methods and analytes pertinent to the program. The laboratory shall employ a measurement-control program which, as a minimum, consists of monitoring the results of laboratory preparation and analysis of control samples using statistical control charts. The basis of this program is to demonstrate that the laboratory method for sample preparation and analysis is working properly. This minimum program consists of using the laboratory's distilled and/or deionized water and spiking it with known compounds or elements. By plotting the results of the method blank spike on control charts, a true picture of the actual process of sample analysis is obtained with fewer problems from matrix effects and sample nonhomogeneity. This information, used in conjunction with matrix spike recoveries, can aid in determining whether an out-of-control condition is due to laboratory problems or matrix problems. Therefore, one batch of control material is the spiked laboratory blank water. The second batch of control material is a soil or sand. This soil can be pulverized and homogenized. If the soil used is known to contain some of the analytes of interest, then no spiking may be required. Additional spiking may be done to an aliquot of control soil just prior to sample preparation. The method blank/spike water (laboratory water) should be analyzed when water samples are analyzed and the method blank/spike soil analyzed alongside soil or waste samples.

The analytes selected for spiking should be representative of the compound class for the organics. It is suggested that the surrogates used for volatiles and base/neutral/acids (BNAs) 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 polychlorinated biphenyl (PCB) when PCBs are analyzed. For wet chemical methods, a single spike of an appropriate control for each method may be used. As an example for cyanide, a control of sodium cyanide from a source other than that used for calibration may be spiked into water and analyzed alongside the water samples. For the metals, it is suggested that at least three of the metals typically analyzed by ICP be monitored and that each element analyzed by furnace or flame atomic absorption be monitored.

4.4.2 Control Sample Quality

The laboratory QA plan shall describe the steps which will be taken to ensure and verify the quality of the two types of control samples of Sect. 4.4.1. The QA plan shall address the following concerns pertaining to the control batches.

1. How the batch will be selected.
2. Shelf life of control batch.
3. Under what conditions the batch will be stored.
4. How the batch will be homogenized.
5. How and when the individual samples will be taken.
6. How and when the sample will be spiked.
7. How the batch will be replaced as it is depleted.
8. How the control charts will be affected by changes in batches.

The QA plan shall address the following concerns pertaining to the spikes.

1. What compound/element will be used to spike.
2. How the spike material will be selected.
3. Target concentration of spiking compound/element.
4. How long the spike is expected to last.
5. Under what conditions the spike will be stored.
6. How the spike will be homogenized.
7. How and when the individual samples will be taken.
8. How the spike will be replaced as it is depleted.
9. How the control charts will be affected by changes in spikes.

4.4.3 Minimum Statistical Control Charting

As a minimum, the laboratory shall run two control charts for each analyte listed in Table 4.1. These charts shall monitor the laboratory measurements obtained from individually spiked water samples and individually spiked soil samples.

Each control chart shall consist of a center line, two warning limits, and two control limits. The control chart parameters should be calculated according to the formulae provided in Table 4.2. A minimum of 20 points/chart shall be obtained prior to the initial attempt to establish the control chart parameters.

If the laboratory does not have 20 points to use in setting control chart limits, the recommended EPA recoveries for the method will be used until such time as 20 points are attained.

4.4.4 Minimum Criteria for an Out-of-Control Condition

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

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

Table 4.1. Typical number of analyses to be monitored through measurement control program

Number	Analyses
10	Metals by AA and ICP
1	Mercury
3	Volatiles
1	Wet chemicals
1	PCB
2	Pesticides
3	Base neutrals
3	Acids

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

The laboratory QA plan shall describe the steps which will be taken in the occurrence of an out-of-statistical-control condition from the control charts of Sect. 4.2.3. The steps should be similar to those requested in Sect. 4.6 but shall include those actions related to the quality and stability of the control batches, sampling, spiking, and handling of the control samples.

4.4.6 Administration of the Control Charts

The laboratory QA plan shall address the following aspects of administering the control charts of Sect. 4.4.2.

1. What types of laboratory activities the control charts will monitor.
2. How often control samples will be run.
3. How soon after results are obtained will charts be monitored.
4. Who is responsible for reading the charts.
5. How will changes in people, equipment, processes affect the charts.
6. How often and under what circumstances will limits be updated.

4.4.7 Statistical Quality of the Control Charts

The formulae for the control chart parameters given by Table 4.2 are those commonly accepted and used. They are based on normally distributed measurements and short-term variation. If these bases are inappropriate, the charts will not perform as desired. 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 states). In order to correct any problems due to improperly fitting control charts, the laboratory may propose alternate methods for setting the control chart parameters for those analytes of Table 4.2. All such proposals

Table 4.2. Control chart formula for Water and Soil Control Batch Program

Definitions

Let X_1, X_2, X_3, ..., X_n ($n>=20$) represent the first n time-ordered determinations for an analyte of Table 4.2 from either the water or soil control batch program.

Then, define the following:

$\bar{X}$ = average = $(1/n)(X_1 + X_2 + \ldots + X_n)$,
$Ri = |X_i - X_{(i-1)}|$ $i = 2,3,\ldots,n$
R_2 = average moving range of two successive points,

$$= [1/(n-1)][\,|X_2 - X_1| + |X_3 - X_2| + \ldots + |X_n - X_{(n-1)}|\,] .$$

Control Chart Parameter Estimation

Parameter	Symbol	Formula
Centerline	CL	X
Upper control limit	UCL	$\bar{X} + 3R_2/d_2$
Lower control limit	LCL	$\bar{X} - 3R_2/d_2$
Upper warning limit	UWL	$\bar{X} + 2R_2/d_2$
Lower Warning limit	LWL	$\bar{X} - 2R_2/d_2$

(d_2 = 1.128, factor from tables for control charting within n = 2, see American Society for Quality Control)

should include data and supportive statistical evidence. Possible alternate statistical approaches can include using nonparametric techniques, medians instead of averages for the centerlines, identifying sources of variation, using long-term variation instead of short-term variation in setting limits, and transformations of the data.

4.4.8 Example of Setting Control Limits

As an example of setting control chart parameters and a very brief introduction to interpretation of the chart, consider the following:

A sample is obtained from the batch of control soil which has been thoroughly mixed and is stored in a special atmospherically controlled location. It is carefully spiked with known amounts of the constituents of Table 4.2 and sent to sample preparation to be processed with a customer's solid waste samples. It is analyzed along with the other samples. It is subjected to the same types of treatment as the other samples in the batch. This scenario is repeated until 20 control samples have been analyzed.

The data are listed in Table 4.3. Also shown are calculations according to the formulae in Table 4.2. Figure 4.1 displays the results of the initial attempt at sizing the data to the control chart parameters. The point falling above the upper control limit was investigated. It was determined that the sample had received a double spiking and, thus, was deleted from the second iteration calculation of the chart parameters. Figure 4.2 shows the second fitting. This fit appears adequate, and the chart is approved by the LQAC authority. Had no explanation for the high result been found, the first calculations would have been used. The chart would have been placed under a probationary condition and its performance monitored with guarded caution.

4.5 OUT-OF-CONTROL EVENTS

The interpretation of control charts can reveal shifts, trends, biases, and conditions where parts of the analytical system are out-of-control. The contract laboratory should specify in the QA plan its criteria of defining an out-of-control condition related to the different zones on a control chart [e.g., data beyond the rejection limits, data in the zone(s) between the rejection and warning limits, and data inside the warning limits] and different patterns within these zones [e.g., number of consecutive data points on one side of the mean, number of consecutive data points in the middle zone number of monotically changing data points, obviously repetitive patterns (Garfield, 1984)].

The laboratory shall identify what actions will be taken when the 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.

Any incident that delays sample processing for a period of time, affects holding times, or delays work by more than two days should immediately be reported by phone to the NCR. The NCR should be informed as soon as the problem is solved and an explanation given as to the corrective action taken. An example of this type of event would be the breakdown of a GC/MS system used for VOAs which could not be repaired for several days. If the laboratory could not use another instrument in its laboratory, then provisions for another approved laboratory to analyze the samples would need to be made.

Table 4.3. Data and calculations for control chart example

Order I	Result X	Moving range $\lvert X_I - X_{I-1} \rvert$
1	12.25	
2	7.52	4.73
3	12.29	4.78
4	10.04	2.25
5	8.48	1.56
6	10.89	2.40
7	9.57	1.32
8	11.40	1.83
9	9.28	2.12
10	11.66	2.39
11	12.06	0.40
12	8.52	3.54
13	11.14	2.62
14	19.56	8.42
15	10.48	9.08
16	9.12	1.35
17	12.79	3.66
18	10.30	2.49
19	5.54	4.76
20	8.93	3.39
Sum	211.82	63.0

First calculations (Fig. 4.1)

Average = 211.82/20 = 10.591
Average moving range = 63.09/19 = 3.321

Centerline = 10.591
Upper control limit = 10.591 + 3 × 3.321/1.128 = 19.423
Lower control limit = 10.591 - 3 × 3.321/1.128 = 1.758
Upper warning limit = 10.591 + 2 × 3.321/1.128 = 16.479
Lower warning limit = 10.591 - 2 × 3.321/1.128 = 4.703

Second Iteration after Removing Point #14 (Fig. 4.2)

Average = 192.26/19 = 10.119
Average moving range = 46.26/18 = 2.570

Centerline = 10.119
Upper control limit = 10.119 + 3 × 2.570/1.128 = 16.954
Lower control limit = 10.119 - 3 × 2.570/1.128 = 3.284
Upper warning limit = 10.119 + 2 × 2.570/1.128 = 14.676
Lower warning limit = 10.119 - 2 × 2.570/1.128 = 5.562

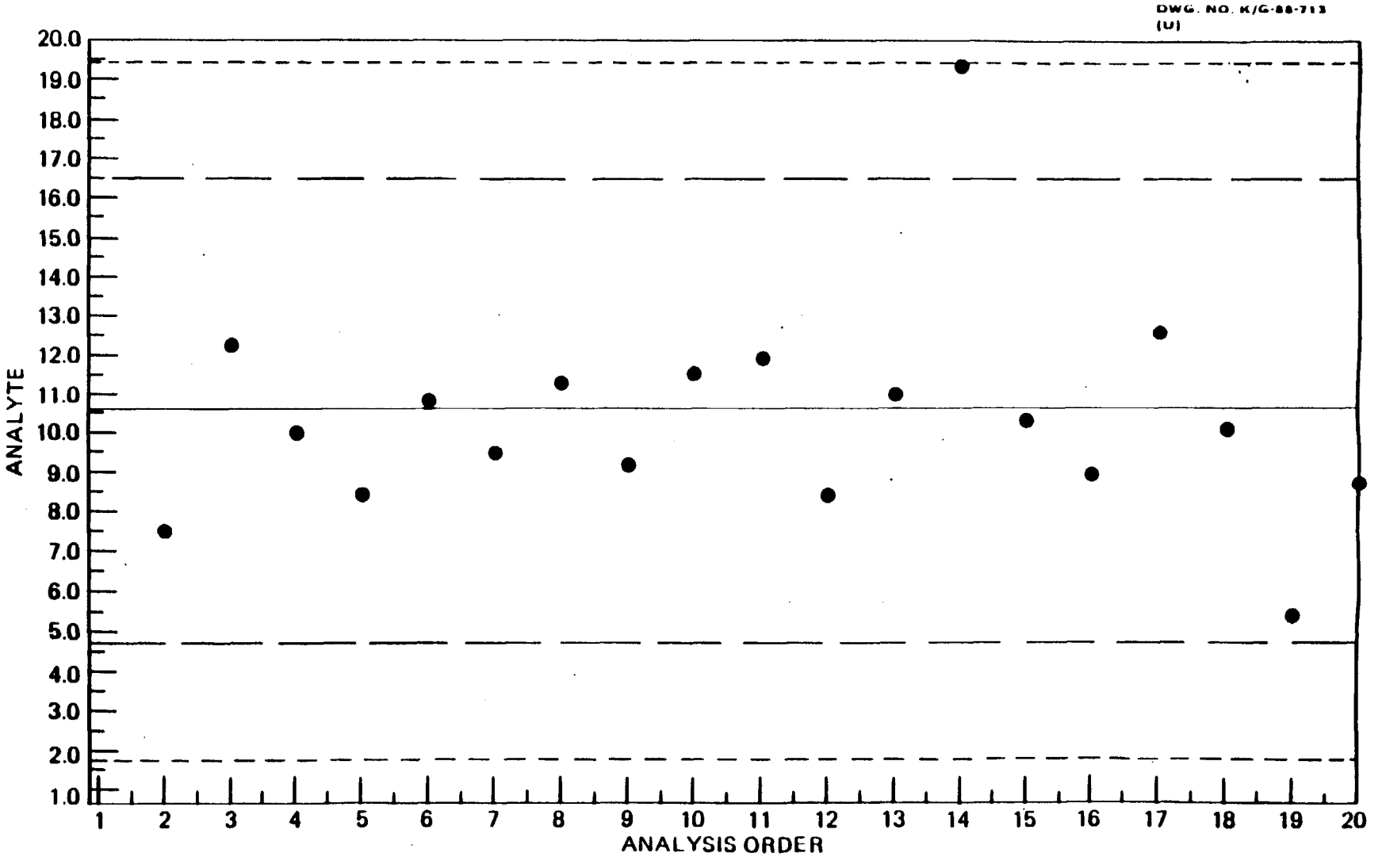

Fig. 4.1. Control Chart Example Data and Control Values of Table 4.3

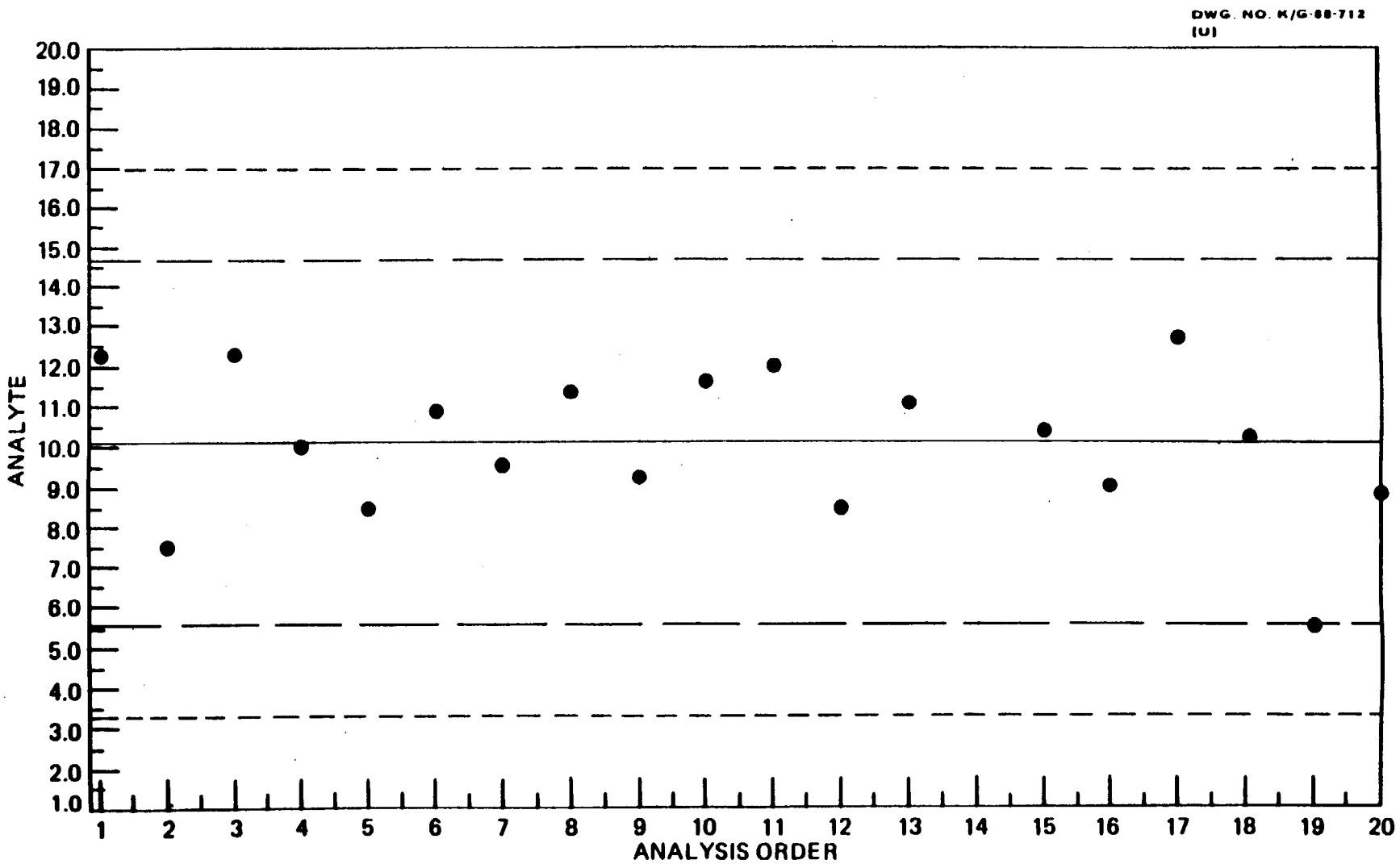

Fig. 4.2. Control Chart Example Data and Second Iteration Control Values of Table 4.3

Many laboratories use forms to aid in rapidly reporting out-of-control events and corrective actions. One way to expedite the more routine out-of-control occurrences, which are frequently corrected by the analyst prior to running samples, is to list these on a form. For instance, a form for the GC/MS laboratory might list events such as continuing check standard outside limits, tune not met, and peak areas for the internal standard outside criteria. The analyst would check the occurrence, note that the item was corrected prior to sample analysis, initial, and date the form. If forms for out-of-control events are made specific to the group using these, time can be saved in documenting events and corrective actions (see Sect. 4.2 for examples of out-of-control events).

4.6 CORRECTIVE ACTION REPORT

For out-of-control incidents, it is essential to document the nature of the incident and the corrective actions taken to set the system back "in control." A corrective action report, to be signed by the laboratory director and the LQAC, should be reported in the MPR to the NCR and discuss the following topics.

1. Where - did the out-of-control incident occur (laboratory name, address, telephone number, section name)?

2. When - did the incident occur?

   - was it corrected?

3. Who - discovered the out-of-control incident?

   - verified the incident?

   - corrected the problem?

4. What - was the name of the test?

   - was the disposition of the test or control and/or instrument?

   - was the nature of the corrective action?

   - will be done to prevent the reoccurrence of the problem?

5. Why - did the incident happen (if scientific explanation is available)?

A copy of the subject control charts and other data describing the out-of-control conditions should be included in the corrective action report.

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

1. placed in the laboratory archive record for the sample(s) in question,
2. placed in the LQAC's file of incidents documentation, and
3. referenced and briefly described in the MPR.

5. PROFICIENCY TESTING

Prior to beginning analysis of field samples, each laboratory must analyze proficiency samples for chemical substances representative of those anticipated in environmental samples. The purpose of proficiency testing is to gage each laboratory's proficiency with samples which are designed to mimic field samples. A second benefit of performance samples is to provide a known material from a source outside the laboratory which can be used to evaluate the laboratory's performance.

5.1 SUBMITTING THE PROFICIENCY SAMPLES

Proficiency samples will be provided to the LQAC within ten working days of receipt of the site work plan. The samples may be soil or water samples or vials of concentrate. The laboratory will be directed as to any required sample reconstitution and the analytes to be determined. If analyses are to be subcontracted to a second laboratory, appropriate proficiency samples must be sent by the NCR to that laboratory as well.

5.2 RESULTS OF PROFICIENCY SAMPLES

Results of proficiency sample analyses are to be received by the NCR within 20 working days after receipt of the samples. The NCR must have the data at least five working days prior to inspection in order to properly evaluate the data. If performance samples are to be subcontracted to a second laboratory, the data report should be sent directly to the primary laboratory by the subcontract laboratory. The entire performance data package is then submitted to the NCR. QC data such as blanks, spikes, EPA controls, daily calibration check standards, sample chromatograms, mass spectra of identified compounds and raw absorbance data for metals shall be provided.

5.3 EVALUATION OF PROFICIENCY SAMPLE RESULTS

The NCR will compare the laboratory's evaluation of proficiency sample results to peer group proficiency sample results.

Performance will always be acceptable if the laboratory results are not statistically different from the peer group results, at 95% confidence, and no procedural problems are found during the laboratory inspection. For nonacceptable results, the records will be reviewed to determine the cause for the nonconformance. The actual limits for a batch of performance samples will be provided only after the batch is discontinued.

6. LABORATORY INSPECTION

The laboratory inspection will occur within 45 days after the laboratory(s) have provided the first edition of the QA plan, the site-specific work plan, and the performance sample data. The inspection will be performed by an experienced chemist from the NCR. The chemist may be accompanied by the EIC.

6.1 PURPOSE OF INSPECTION

The purpose of laboratory inspection is to verify that the Navy QC requirements are being met as reflected by the laboratory's daily operations in adherence to the QC plan received by the NCR.

6.2 INSPECTION PROCEDURE

The laboratory inspection involves four phases.

1. Overview and Orientation

   The inspector meets with the laboratory management, including the laboratory director, the LQAC, and others as the director deems appropriate. The objectives of the visit are reviewed and a schedule established. The inspector discusses the review of the laboratory's QA/QC plan and the results of the proficiency testing.

2. Observation, Examination, and Review

   According to the schedule, the inspector does the following.

   - Witnesses performance of specified analytical procedures.
   - Reviews sample handling and storage procedures. The inspector will follow the trail of the performance samples through the laboratory.
   - Examines the QC records including QC manuals, instrument calibration and maintenance records, control charts, instrument run logs, sample preparation logs, notebooks used to document analysis, corrective action reports for out-of-control events, and performance data generated for other programs such as Superfund CLP and state drinking water.

3. Findings

   The inspector conducts an exit interview with the laboratory director, the LQAC, and any other laboratory personnel the director deems appropriate. The inspector summarizes the findings of the visit and details specific deficiencies to be addressed by corrective action.

Recommendations regarding corrective action may be provided. A written report summarizing the findings is provided to the LQAC, the Navy EIC and NCR, and engineering contractor within ten working days of the inspection.

4. Corrective Action (if required)

Within ten days of receipt of the findings, the laboratory submits to the NCR and the EIC a plan to correct the deficiencies identified in the inspection. The plan should include for each deficiency, a description of the corrective action and a date indicating when the action is to be implemented or completed.

A repeat inspection may be required in instances where the number of deficiencies requiring corrective actions are complex. Repeat inspections will be scheduled for the earliest possible date after the last corrective action plan is received by the NCR.

The laboratory shall send a follow-up report which supplies information indicating the proof that the plan has been carried out. For example, if no control charts exist, then 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.

7. ANALYTICAL METHODS

An analytical method is a series of steps or procedures that must be performed to determine the identity and quantity of analyte in a sample. The methods to be employed by the Navy-approved laboratory fall mainly into two categories--those which have been approved by the EPA and those which have been developed by the Army. The former refer primarily to the methods presented in the *Federal Register* of October 26, 1984 (49 FR 43234), where the EPA has listed ~250 pollutants (pp. 43251-43258) or pollutant categories and the method(s) by which each must (by virtue of final or final interim ruling status of the methods) be tested. The acceptance of methods not under either status will be handled on a case-by-case basis among the concerned parties. Non-standard methods shall be submitted to the NCR who will discuss the method with Navy and EPA personnel prior to use on Navy projects. Other applicable EPA methods include the *SW-846* methods which are applicable to Resource Conservation and Recovery Act sites and the Superfund CLPs which are applicable to the CERCLA sites.

Many of the EPA methods are found in the documentation of other organizations (e.g., U.S. Geological Services, ASTM) and are incorporated by reference into the regulations. Such incorporation involves listing the organization, the specific document and its date, the method number assigned by the other organization, and perhaps a page number in the document of the other organization. Technically speaking, to maintain the applicability of the regulation, no deviations from the given citations are allowed by the EPA, even in cases where an organization (ASTM, for example) may have an updated version of the method. However, there are instances where EPA regional offices have granted exceptions to different laboratories for the testing of various substances. If a laboratory has such a variance, in writing, from the EPA (either to use a different ASTM method, for example, than the one cited in the October 26, 1984 *Federal Register* or to use a somewhat modified method, for example, than the one cited in the October 26, 1984 *Federal Register*), a copy of the variance (sent to the NCR) may be used to seek Navy approval of the different or modified method. It must also be shown that the conditions for which the variance was issued by the EPA are similar to the expected conditions (sampling and handling techniques, environmental matrix, concentration range, interferences, etc.) in the IRP.

It is also recognized that the analyst may have some leeway resulting from the regulations themselves. For instance, in the October 26, 1984 *Federal Register*, several methods are listed involving GC. Typically, in paragraph 8.1.2, these methods allow the analyst "certain options," provided various subsequent QC requirements are met. For example, the EPA allows some flexibility in the procedures (and no written permission is needed from the EPA) once a sample has been extracted and placed into the instrument. On the other hand, changes in operations prior to this instrumental analysis (e.g., preparation, storage) would probably require written documentation.

The *Federal Register* of October 26, 1984 (49 FR 43437) also contains a proposed ruling where additional substances and methods are listed: specifically, some proposed modifications to Tables IC and ID (Tables 7.1 through 7.5 of this guide) of the previously mentioned final rule. In those cases where a substance/method does not appear on one of the earlier tables but does occur on one of the proposed listings, the method in the proposed listing is recommended by the EPA (Medz, 1985) but without any regulatory force.

For the analytical method to be used in the case of munition-related substances, the laboratory should consult the NCR who will forward a copy(s) of the appropriate method developed by the Army Toxic and Hazardous Materials Agency.

For biota and air samples, the methods must be evaluated individually by the NCR to determine whether they may be used for the work in question.

A list of references containing methods, statistics, and sampling information is supplied in the Bibliography of this document.

For Level D QC sites, the current CLP methods and documentation must be followed. For methods not covered by CLP and for sites requiring Level D, the latest edition of *SW-846* or other methods listed in Tables 7.1 through 7.5, may be used. For the Levels C and E sites, CLP methods, *SW-846* methods, or other methods listed in Tables 7.1 through 7.5 shall be used. The exception to the Levels C and E method requirement occurs in the volatile and semivolatile area. In any level of QC and for any site where volatiles and semivolatiles are analyzed by GC/MS, the current CLP methods shall be used.

7.1 QC REQUIREMENTS FOR THE LABORATORY

The following are the minimum QC requirements for the laboratory analyses. For Level D QC, the current CLP QC requirements are specified. For methods not defined in the CLP, the blank, blank/spike, matrix spike, and matrix spike duplicate shall be performed for every 20 samples of similar matrix. The batch size for Level D QC is 20 samples.

In Levels C and E, the optimum batch size is determined by the number of samples of similar matrix which can be processed simultaneously through the entire preparation and analysis process. For example, if 5 samples can be extracted and 20 analyzed by the instrument, the batch size is 5. Once this is determined, it is used with the blank/spike control program in the following manner.

In Levels C and E, a blank/spike control shall be analyzed with each batch and shall be plotted on control charts as described in Sect. 4.4. For metals, anions, and other wet chemical analysis, a method blank shall also be processed with each batch and shall contain less than the method detection limit for compounds of interest. In any method using surrogates spiked into the blank, the blank shall serve as both the method blank

Table 7.1. List of approved biological test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and units	Method [1]	EPA [2]	Reference (Method Number or Page)		
			Standard Methods 15th Ed.	ASTM	USGS
Bacteria:					
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF) [4], single step	p. 132	908C		
		p. 124	908C		B-0050-77
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution	p.132	908C		
3. Coliform (total, number per 100 ml	MPN, 5 tube, 3 dilution; or, MF [4] single step or two step	p. 114	908A		
		p. 108	908A		B-0025-77
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or MF [4] with enrichment	p. 114	908A		
		p. 111	908 (A+A.5c)		
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF [4]; or, plate count	p. 139	910A		
	p. 136	910B		B0055-77 [5]	
	p. 143	910C			

Table IA Notes

[1] The method must be specified when results are reported.

[2] "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978", EPA-600/8-78-017, U.S. Environmental Protection Agency.

[3] Greeson, P.E., *et al.*, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, "U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their grow development.

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B-0055-77) is made in a boiling water bath to avoid scorching of the med

Table 7.2. List of approved inorganic test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
1. Acidity, as $CaCO_3$ mg/L	Electrometric end point or phenolphthalein end point.	305.1	402(4.a)	D1067-82(E)	—	
2. Alkalinity, as $CaCO_3$, mg/L	Electrometric or colorimetric titration to pH 4.5, manual, or	310.1	403	D1067-82(B)	I-1030-84	33.014[2]
	Automated.	310.2	—	—	I-2030-84	
3. Aluminum—Total[3] mg/L	Digestion[3] followed by					
	AA direct aspiration,	202.1	303C	—	I-3051-84	
	AA furnace,	202.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Eriochrome cyanine R).	—	306B	—	—	
4. Ammonia (as N), mg/L	Manual distillation (at pH 9.5)[5], followed by	350.2	417A	—	—	33.057[2]
	Nesslerization,	350.2	417B	D1426-79(A)	I-3520-84	33.057[2]
	Titration,	350.2	417D	—	—	
	Electrode,	350.3	417E or F	D1426-79(D)	—	
	Automated phenate or	350.1	417G	D1426-79(C)	I-4523-84	
	Automated electrode.	—	—	—	—	Note 6
5. Antimony—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	204.1	303A	—	—	
	AA furnace, or	204.2	304	—	—	
	Inductively coupled plasma	—	—	—	—	200.7[4]
6. Arsenic—Total[3], mg/L	Digestion[3] followed by	206.5	—	—	—	
	AA gaseous hydride,	206.3	303E	D2972-84(B)	I-3062-84	
	AA furnace,	206.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (SDDC)	206.4	307B	D2972-84(A)	I-3060-84	
7. Barium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	208.1	303C	—	I-3084-84	
	AA furnace, or	208.2	304	—	—	
	Inductively coupled plasma	—	—	—	—	200.7[4]
8. Beryllium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	210.1	303C	D3654-84(A)	I-3095-84	
	AA furnace,	210.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (aluminon).	—	309B	—	—	
9. Biochemical oxygen demand (BOD_5), mg/L	Dissolved Oxygen Depletion.	405.1	507	—	I-1578-78[7]	33.019[2], p.17[8]
10. Boron—Total, mg/L	Colorimetric (curcumin), or	212.3	404A	—	I-3112-84	
	Inductively Coupled plasma.	—	—	—	—	200.7[4]
11. Bromide, mg/L	Titrimetric.	320.1	—	D1246-82(C)	I-1125-84	p.544[9]

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page) EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
12. Cadmium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	213.1	303A or B	D3557-84(A or B)	I-3135-84 or I-3136-84	33.089[3], p.37[9]
	AA furnace,	213.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Voltametry[10], or	—	—	D3557-84(C)	—	
	Colorimetric (Dithizone).	—	310B	—	—	
13. Calcium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	215.1	303A	D511-84(B)	I-3152-84	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Titrimetric (EDTA).	215.2	311C	D511-84(A)	—	
14. Carbonaceous biochemical oxygen demand ($CBOD_5$), mg/L[11]	Dissolved Oxygen Depletion with nitrification inhibitor.	—	507(5.e.6)	—	—	
15. Chemical oxygen demand (COD), mg/L	Titrimetric, or,	410.1, 410.2, or 410.3	508A	D1252-83	I-3560-84 or I-3562-84	33.034[3], p.17[9]
	Spectrophotometric, manual or automated.	410.4	—	—	I-3561-84	Notes 12 or 13
16. Chloride, mg/L	Titrimetric (silver nitrate)	—	407A	D512-81(B)	I-1183-84	
	or (Mercuric nitrate), or	325.3	407B	D512-81(A)	I-1184-84	33.067[3]
	Colorimetric, manual or	—	—	D512-81(C)	I-1187-84	
	Automated (ferricyanide).	325.1, or 325.2	407D	—	I-2187-84	
17. Chlorine—Total residual, mg/L	Titrimetric					
	Amperometric direct,	330.1	408C	D1253-76(A)	—	
	Starch end point direct,	330.3	408A	D1253-76(B) Part 18.3	—	
	Back titration either end point[14], or	330.2	408B	—		
	DPD-FAS;	330.4	408D	—	—	
	Spectrophotometric, DPD,	330.5	408E	—	—	
	or Electrode.	—	—	—	—	Note 15
18. Chromium VI dissolved, mg/L	0.45 micron filtration followed by					
	AA chelation-extraction,	218.4	303B	—	I-1232-84	
	or Colorimetric (Diphenylcarbazide).	—	—	—	I-1230-84	307B[16]
19. Chromium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	218.1	303A	D1687-84(D)	I-3236-84	33.089[3]
	AA chelation—extraction,	218.3	303B	—	—	
	AA furnace,	218.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Diphenylcarbazide).	—	312B	D1687-84(A)	—	
20. Cobalt—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	219.1	303A or B	D3558-84(A or B)	I-3239-84 or I-3240-84	p.37[9]
	AA furnace, or	219.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
21. Color, platinum cobalt units or dominant wavelength, hue, luminance, purity.	Colorimetric (ADMI), or	110.1	204D	—	—	Note 17
	(Platinum cobalt), or	110.2	204A	—	I-1250-84	
	Spectrophotometric.	110.3	204B	—	—	
22. Copper—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	220.1	303A or B	D1688-84(D or E)	I-3270-84 or I-3271-84	33.089[2], p.37[6]
	AA furnace,	220.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Colorimetric (Neocuproine), or	—	313B	D1688-84(A)	—	
	(Bicinchoninate).	—	—	—	—	Note 18
23. Cyanide—Total, mg/L	Manual distillation with $MgCl_2$ followed by	—	412B	—	—	
	Titrimetric, or	—	412C	—	—	p.22[6]
	Spectrophotometric,					
	manual or	335.2	412D	D2036-82(A)	I-3300-84	
	Automated.[10]	335.3	—	D2036-82(A)	—	
24. Cyanide amenable to chlorination, mg/L	Manual distillation with $MgCl_2$ followed by titrimetric or spectrophotometric	335.1	412F	D2036-82(B)	—	
25. Fluoride—Total, mg/L	Manual distillation[5] followed by	—	413A	—	—	
	Electrode, manual or	340.2	413B	D1179-80(B)	—	
	Automated,	—	—	—	I-4327-84	
	Colorimetric (SPADNS),	340.1	413C	D1179-80(A)	—	
	or Automated complexone.	340.3	413E	—	—	
26. Gold—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	231.1	303A	—	—	
	AA furnace.	231.2	304	—	—	
27. Hardness—Total, as $CaCO_3$, mg/L	Automated colorimetric,	130.1	—	—	—	
	Titrimetric (EDTA), or	130.2	314B	D1126-80	I-1338-84	33.082[2]
	Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33.)					
28. Hydrogen ion (pH), pH units	Electrometric, measurement, or	150.1	423	D1293-84(A or B)	I-1586-84	33.006[2]
	Automated electrode.	—	—	—	—	Note 20
29. Iridium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	235.1	303A	—	—	
	AA furnace.	235.2	304	—	—	
30. Iron—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	236.1	303A or B	D1068-84(C or D)	I-3381-84	33.089[2]
	AA furnace,	236.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Phenanthroline).	—	315B	D1068-84(A)	—	Note 21

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
31. Kjeldahl nitrogen—Total, (as N), mg/L	Digestion and distillation followed by	351.3	420A or B	D3590-84(A)	—	
	Titration,	351.3	417D	D3590-84(A)	—	33.051[2]
	Nesslerization,	351.3	417B	D3590-84(A)	—	
	Electrode,	351.3	417E or F	—	—	
	Automated phenate,	351.1	—	—	I-4551-78[7]	
	Semi-automated block digestor, or	351.2	—	D3590-84(A)	—	
	Potentiometric.	351.4	—	D3590-84(A)	—	
32. Lead—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	239.1	303A or B	D3559-85(A or B)	I-3399-84	33.089[2]
	AA furnace,	239.2	304	—	—	
	Inductively coupled plasma,	—	—	—	—	200.7[4]
	Voltametry[10], or	—	—	D3559-85(C)	—	
	Colorimetric (Dithizone).	—	316B	—	—	
33. Magnesium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	242.1	303A	D511-84(B)	I-3447-84	33.089[2]
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Gravimetric	—	318B	D511-77(A)	—	
34. Manganese—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	243.1	303A or B	D858-84(B or C)	I-3454-84	33.089[2]
	AA furnace,	243.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Persulfate), or	—	319B	D858-84(A)	—	33.126[2]
	(Periodate)	—	—	—	—	Note 22
35. Mercury—Total[3], mg/L	Cold vapor, manual or	245.1	303F	D3223-80	I-3462-84	33.095[2]
	Automated.	245.2	—	—	—	
36. Molybdenum—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	246.1	303C	—	I-3490-84	
	AA furnace, or	246.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
37. Nickel—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	249.1	303A or B	D1886-84(C or D)	I-3499-84	
	AA furnace,	249.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Heptoxime).	—	321B	—	—	
38. Nitrate (as N), mg/L	Colorimetric (Brucine sulfate), or	352.1	—	D992-71	—	33.063[2], 419D[16], p.28[6]
	Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).					
39. Nitrate-nitrite (as N), mg/L	Cadmium reduction,					
	Manual or	353.3	419C	D3867-85(B)	—	
	Automated, or	353.2	419F	D3867-85(A)	I-4545-84	
	Automated hydrazine.	353.1	—	—	—	

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
40. Nitrite (as N), mg/L	Spectrophotometric,					
	Manual or	354.1	419	D1254-67	—	Note 24
	Automated (Diazotization).	—	—	—	I-4540-84	
41. Oil and grease—Total recoverable, mg/L	Gravimetric (extraction).	413.1	503A	—	—	
42. Organic carbon—Total (TOC), mg/L	Combustion or oxidation.	415.1	505	D2579-85(A or B)	—	33.044[2], p.4[33]
43. Organic nitrogen (as N) mg/L	Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4.)					
44. Orthophosphate (as P), mg/L	Ascorbic acid method,					
	Automated or	365.1	424G	—	I-4601-84	33.116[2]
	Manual single reagent,	365.2	424F	D515-82(A)	—	33.111[2]
	or Manual two reagent.	365.3	—	—	—	
45. Osmium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	252.1	303C	—	—	
	AA furnace.	252.2	304	—	—	
46. Oxygen, dissolved, mg/L	Winkler (Azide modification), or	360.2	421B	D888-81(C)	I-1575-78[7]	33.028[2]
	Electrode.	360.1	421F	—	I-1576-78[7]	
47. Palladium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	253.1	—	—	—	p.S27[9]
	AA furnace.	253.2	—	—	—	p.S28[9]
48. Phenols, mg/L	Manual distillation[25] followed by	420.1	—	D1783-80(A or B)	—	Note 26
	Colorimetric (4AAP)					
	manual, or	420.1	—	—	—	Note 26
	Automated[19].	420.2	—	—	—	
49. Phosphorus (elemental) mg/L	Gas-liquid chromatography.	—	—	—	—	Note 27
50. Phosphorus—Total, mg/L	Persulfate digestion followed by	365.2	424C(III)	—	—	33.111[2]
	Manual or	365.2 or 365.3	424F	D515-82(A)	—	
	Automated ascorbic acid reduction, or	365.1	424G	—	I-4600-84	33.116[2]
	Semi-automated block digestor.	365.4	—	—	—	
51. Platinum—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	255.1	303A	—	—	
	AA furnace.	255.2	304	—	—	
52. Potassium—total[3], mg/L	Digestion followed by					
	AA direct aspiration,	258.1	303A	—	I-3630-84	33.103[2]
	inductively coupled plasma	—	—	—	—	200.7[4]
	Flame photometric, or	—	322B	D1428-82(A)	—	
	Colorimetric (Cobaltinitrite).	—	—	—	—	317B[10]

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 18th Ed.	ASTM	USGS[1]	Other
53. Residue—Total, mg/L	Gravimetric, 103-105°C.	160.3	209A	—	I-3750-84	
54. Residue—filterable, mg/L	Gravimetric, 180°C.	160.1	209B	—	I-1750-84	
55. Residue—nonfilterable, (TSS), mg/L	Gravimetric, 103-105°C post washing of residue.	160.2	209C	—	I-3765-84	
56. Residue—settleable, mg/L	Volumetric (Imhoff cone) or gravimetric.	160.5	209E	—	—	
57. Residue—volatile, mg/L	Gravimetric, 550°C.	160.4	209D	—	I-3753-84	
58. Rhodium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	265.1	303A	—	—	
	AA furnace.	265.2	304	—	—	
59. Ruthenium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	267.1	303A	—	—	
	AA furnace.	267.2	304	—	—	
60. Selenium—Total[3], mg/L	Digestion[3] followed by					
	AA furnace,	270.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	AA gaseous hydride.	270.3	303E	D3859-84(A)	I-3667-84	
61. Silica—Dissolved, mg/L	0.45 micron filtration followed by					
	Colorimetric, Manual or	370.1	425C	D859-80(B)	I-1700-84	
	Automated (Molybdosilicate), or	—	—	—	I-2700-84	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
62. Silver—Total[16], mg/L	Digestion[3] followed by					
	AA direct aspiration,	272.1	303A or B	—	I-3720-84	33.089[2], p.37[9]
	AA furnace,	272.2	304	—	—	
	Colorimetric (Dithizone), or	—	—	—	—	319B[16]
	Inductively coupled plasma.	—	—	—	—	200.7[4]
63. Sodium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	273.1	303A	—	I-3735-84	33.107[2]
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Flame photometric.	—	325B	D1428-82(A)	—	
64. Specific conductance, micromhos/cm at 25°C	Wheatstone bridge.	120.1	205	D1125-82(A)	I-1780-84	33.002[2]
65. Sulfate (as SO_4), mg/L	Automated colorimetric (barium chloranilate),	375.1	—	—	—	
	Gravimetric, or	375.3	426A or B	D516-82(A)	—	33.124[2]
	Turbidimetric.	375.4	—	D516-82(B)	—	426C[10]

Table 7.2. (continued)

Parameter and Units	Method	Reference (Method No. or Page)				
		EPA 1979	Std. Methods 16th Ed.	ASTM	USGS[1]	Other
66. Sulfide (as S), mg/L	Titrimetric (iodine) or	376.1	427D	—	I-3840-84	228A[30]
	Colorimetric (methylene blue).	376.2	427C	—	—	
67. Sulfite (as SO_3), mg/L	Titrimetric (iodine-iodate).	377.1	428A	D1339-84(C)	—	
68. Surfactants, mg/L	Colorimetric (methylene blue).	425.1	512B	D2330-82(A)	—	
69. Temperature, °C.	Thermometric.	170.1	212	—	—	Note 31
70. Thallium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	279.1	303A	—	—	
	AA furnace, or	279.2	304	—	—	
	Inductively coupled plasma.	—	—	—	—	200.7[4]
71. Tin—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	282.1	303A	—	I-3850-78[7]	
	AA furnace.	282.2	304	—	—	
72. Titanium—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration, or	283.1	303C	—	—	
	AA furnace.	283.2	304	—	—	
73. Turbidity, NTU	Nephelometric.	180.1	214A	D1889-81	I-3860-84	
74. Vanadium, Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	286.1	303C	—	—	
	AA furnace,	286.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Gallic acid).	—	327B	D3373-84(A)	—	
75. Zinc—Total[3], mg/L	Digestion[3] followed by					
	AA direct aspiration,	289.1	303A or B	D1691-84(C or D)	I-3900-84	33.089[3], p.37[9]
	AA furnace,	289.2	304	—	—	
	Inductively coupled plasma, or	—	—	—	—	200.7[4]
	Colorimetric (Dithizone) or	—	328C	—	—	
	(Zincon).	—	—	—	—	Note 32

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1986, unless otherwise stated.

[2] "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 14th ed. (1985).

[3] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979." One (Section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (Section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by the vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instruction and/or cautions.

NOTE: If the digestion included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion for dissolved metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample solution to be analyzed meets the following criteria:

Table 7.2. (continued)

a. has a low COD (<20)
b. is visibly transparent with a turbidity measurement of 1 NTU or less.
c. is colorless with no perceptable odor, and
d. is of one liquid phase and free of particulate or suspended matter following acidification.

[4]The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[5]Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[6]Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, NY, 10591

[7]The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," USGS TWRI, Book 5, Chapter A1 (1979).

[8]American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[9]"Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[10]The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[11]Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required, can the permittee report data using the nitrification inhibitor.

[12]OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[13]Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[14]The back titration method will be used to resolve controversy.

[15]Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

[16]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition, 1976.

[17]National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

[18]Copper, Biocinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[19]After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[20]Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, NY 10591.

[21]Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[22]Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.

[23]Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3, page 4 (1972).

[24]Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[25]Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[26]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 578-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

[27]R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," *Journal of Chromatography*, vol. 47, No. 3, pp. 421-426, 1970.

[28]Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[29]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 15th Edition.

[30]The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 13th Edition.

[31]Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water Resources Investigations, Book 1, Chapter D1, 1975.

[32]Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-233, Hach Chemical Company, Loveland, CO 80537.

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Table 7.3. List of approved test procedures for nonpesticide organic compounds (40 CFR, Part 136, July 1, 1987)

Parameter [1]	EPA Method Number [2, 3]			Other
	GC	GC/MS	HPLC	
1. Acenaphthene	610	625, 1625	610	
2. Acenaphthylene	610	625, 1625	610	
3. Acrolein	603	[4]624, 1624		
4. Acrylonitrile	603	[4]624, 1624		
5. Anthracene	610	625, 1625	610	
6. Benzene	602	624, 1624		
7. Benzidine		[5]625, 1625	605	Note 3, p. 1;
8. Benzo(a)anthracene	610	625, 1625	610	
9. Benzo(a)pyrene	610	625, 1625	610	
10. Benzo(b)fluoranthene	610	625, 1625	610	
11. Benzo(ghi)perylene	610	625, 1625	610	
12. Benzo(k)fluoranthene	610	625, 1625	610	
13. Benzyl Chloride				Note 3, p. 130; Note 6, p. S102.
14. Benzyl Butyl Phthalate	606	625, 1625		
15. Bis(2-chloroethoxy) methane	611	625, 1625		
16. Bis(2-chloroethyl) ether	611	625, 1625		
17. Bis(2-ethylhexyl) phthalate	606	625, 1625		
18. Bromodichloromethane	601	624, 1624		
19. Bromoform	601	624, 1624		
20. Bromomethane	601	624, 1624		
21. 4-Bromophenylphenyl ether	611	625, 1625		
22. Carbon tetrachloride	601	624, 1624		Note 3, p. 130;
23. 4-Chloro-3-methylphenol	604	625, 1625		
24. Chlorobenzene	601, 602	624, 1624		Note 3, p. 130;
25. Chloroethane	601	624, 1624		
26. 2-Chloroethylvinyl ether	601	624, 1624		
27. Chloroform	601	624, 1624		Note 3, p. 130;
28. Chloromethane	601	624, 1624		
29. 2-Chloronaphthalene	612	625, 1625		
30. 2-Chlorophenol	604	625, 1625		
31. 4-Chlorophenylphenyl ether	611	625, 1625		
32. Chrysene	610	625, 1625	610	
33. Dibenzo(a,h)anthracene	610	625, 1625	610	
34. Dibromochloromethane	601	624, 1624		
35. 1,2-Dichlorobenzene	601, 602, 612	624, 625, 1625		
36. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625		
37. 1,4-Dichlorobenzene	601, 602, 612	625, 1624, 1625		
38. 3,3-Dichlorobenzidine		625, 1625	605	
39. Dichlorodifluoromethane	601			
40. 1,1-Dichloroethane	601	624, 1624		
41. 1,2-Dichloroethane	601	624, 1624		
42. 1,1-Dichloroethene	601	624, 1624		
43. trans-1,2-Dichloroethene	601	624, 1624		
44. 2,4-Dichlorophenol	604	625, 1625		
45. 1,2-Dichloropropane	601	624, 1624		
46. cis-1,3-Dichloropropene	601	624, 1624		
47. trans-1,3-Dichloropropene	601	624, 1624		
48. Diethyl phthalate	606	625, 1625		
49. 2,4-Dimethylphenol	604	625, 1625		
50. Dimethyl phthalate	606	625, 1625		
51. Di-n-butyl phthalate	606	625, 1625		
52. Di-n-octyl phthalate	606	625, 1625		
53. 2,4-Dinitrophenol	604	625, 1625		
54. 2,4-Dinitrotoluene	609	625, 1625		
55. 2,6-Dinitrotoluene	609	625, 1625		
56. Epichlorohydrin				Note 3, p. 130; Note 6, p. S102.
57. Ethylbenzene	602	624, 1624		
58. Fluoranthene	610	625, 1625	610	
59. Fluorene	610	625, 1625	610	
60. Hexachlorobenzene	612	625, 1625		
61. Hexachlorobutadiene	612	625, 1625		
62. Hexachlorocyclopentadiene	612	[5]625, 1625		
63. Hexachloroethane	612	625, 1625		
64. Indeno(1,2,3-cd)pyrene	610	625, 1625	610	
65. Isophorone	609	625, 1625		
66. Methylene Chloride	601	624, 1624		Note 3, p. 130;
67. 2-Methyl-4,6-Dinitrophenol	604	625, 1625		
68. Naphthalene	610	625, 1625		
69. Nitrobenzene	609	625, 1625		
70. 2-Nitrophenol	604	625, 1625		
71. 4-Nitrophenol	604	625, 1625		
72. N-Nitrosodimethylamine	607	625, 1625		
73. N-Nitrosodi-n-propylamine	607	[5]625, 1625		
74. N-Nitrosodiphenylamine	607	[5]625, 1625		
75. 2,2-oxybis(1-chloropropane)	611	625, 1625		
76. PCB-1016	608		625,	Note 3, p. 43;
77. PCB-1221	608		625,	Note 3, p. 43;
78. PCB-1232	608		625,	Note 3, p. 43;
79. PCB-1242	608		625,	Note 3, p. 43;
80. PCB-1248	608		625,	Note 3, p. 43;
81. PCB-1254	608		625,	Note 3, p. 43;
82. PCB-1260	608		625,	Note 3, p. 43;
83. Pentachlorophenol	604	625, 1625		Note 3, p. 140;

Table 7.3. (continued)

Parameter [1]	EPA Method Number [2,3]			Other
	GC	GC/MS	HPLC	
84. Phenanthrene	610	625, 1625	610	
85. Phenol	604	625, 1625		
86. Pyrene	610	625, 1625	610	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin		[4] 613		
88. 1,1,2,2-Tetrachloroethane	601	624, 1624		Note 3, p. 130;
89. Tetrachloroethene	601	624, 1624		Note 3, p. 130;
90. Toluene	602	624, 1624		
91. 1,2,4-Trichlorobenzene	612	625, 1625		Note 3, p. 130;
92. 1,1,1-Trichloroethane	601	624, 1624		
93. 1,1,2-Trichloroethane	601	624, 1624		Note 3, p. 130;
94. Trichloroethene	601	624, 1624		
95. Trichlorofluoromethane	601	624,		
96. 2,4,6-Trichlorophenol	604	625, 1625		
97. Vinyl Chloride	601	624, 1624		

Table IC Notes

[1] All parameters are expressed in micrograms per liter (µg/L).

[2] The full text of Methods 601-613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Methods for Benzidine; Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[6] 625, Screening only.

[7] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[8] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

Note.—These warning limits are promulgated as an "interim final action with a request for comments."

Table 7.4. List of approved test procedures for pesticides[1] (40 CFR, Part 136, July 1, 1987)

Parameter µg/L)	Method	EPA[2,3]	Standard Methods 15th Ed	ASTM	Other
1. Aldrin	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
2. Ametryn	GC				Note 3, p. 83; Note 6, p. S88.
3. Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4. Atraton	GC				Note 3, p. 83; Note 6, p. S88.
5. Atrazine	GC				Note 3, p. 83; Note 6, p. S88.
6. Azinphos methyl	GC				Note 3, p. 25; Note 6, p. S51.
7. Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8. α-BHC	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[4] 625			
9. β-BHC	GC	608		D3086	
	GC/MS	625			
10. δ-BHC	GC	608		D3086	
	GC/MS	[4] 625			
11. γ-BHC (Lindane)	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
12. Captan	GC		509A		Note 3, p. 7.
13. Carbaryl	TLC				Note 3, p. 94; Note 6, p. S80.
14. Carbophenothion	GC				Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC	608	509A	D3086	Note 3, p. 7.
	GC-MS	625			
16. Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		509B		Note 3, p. 115; Note 4, p. 35.
18. 4,4'-DDD	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC-MS	625			
19. 4,4'-DDE	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
20. 4,4'-DDT	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
21. Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC				Note 3, p. 25; Note 6, p. S51.
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dicamba	GC				Note 3, p. 115.
25. Dichlorenthion	GC				Note 4, p. 30; Note 6, p. S73.
26. Dichloran	GC		509A		Note 3, p. 7.
27. Dicofol	GC			D3086	
28. Dieldrin	GC	608	509A		Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
29. Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30. Disulfoton	GC				Note 3, p. . Note 6, p. S51
31. Diuron	TLC				Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[4] 625			
33. Endosulfan II	GC	608	509A	D3086	Note 3, p. 7.
	GC/MS	[4] 625			
34. Endosulfan sulfate	GC	608			
	GC/MS	625			
35. Endrin	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	[4] 625			
36. Endrin aldehyde	GC	608			
	GC/MS	625			
37. Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38. Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39. Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
41. Heptachlor epoxide	GC	608	509A	D3086	Note 3, p. 7; Note 4, p. 30; Note 6, p. S73.
42. Isodrin	GC/MS	625			
	GC				Note 4, p. 30; Note 6, p. S73.
43. Linuron	TLC				Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		509A		Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
45. Methiocarb	TLC				Note 3, p. 94; Note 6, p. S80.
46. Methoxychlor	GC		509A	D3086	Note 3, p. 7; Note 4, p. 30.
47. Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S80.
48. Mirex	GC		509A		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
50. Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
51. Neburon	TLC				Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		509A		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		509A		Note 3, p. 25.
54. PCNB	GC		509A		Note 3, p. 7
55. Perthane	GC			D3086	
56. Prometon	GC				Note 3, p. 83; Note 6, p. S88.
57. Prometryn	GC				Note 3, p. 83; Note 6, p. S88.
58. Propazine	GC				Note 3, p. 83; Note 6, p. S88.
59. Propham	TLC				Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC				Note 3, p. 94; Note 6, p. S80.
61. Secbumeton	TLC				Note 3, p. 83; Note 6, p. S88.
62. Siduron	TLC				Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC				Note 3, p. 83; Note 6, p. S88.
64. Strobane	GC		509A		Note 3, p. 7.

Table 7.4. (continued)

Parameter (µg/L)	Method	EPA [2,1]	Standard Methods 15th Ed	ASTM	Other
65. Swep	TLC				Note 3, p. 104; Note 6, p. S64
66. 2,4,5-T	GC		509B		Note 3, p. 115, Note 4, p. 35.
67. 2,4,5-TP (Silvex)	GC		509B		Note 3, p. 115
68. Terbuthylazine	GC				Note 3, p. 83, Note 6, p. S66.
69. Toxaphene	GC	608	509A	D3086	Note 3, p. 7, Note 4, p. 30
	GC/MS	625 [1]			
70. Trifluralin	GC		509A [1]		Note 3, p. 7

Table ID Notes

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

[2] The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).

[5] The method may be extended to include α-BHC, δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

Note.—These warning limits are promulgated as an "interim final action with a request for comments."

Table 7.5. List of approved radiological test procedures
(40 CFR, Part 136, July 1, 1987)

Parameter and units	Methods	EPA [1]	Reference (method No. or page)		
			Standard Methods 15th Ed.	ASTM	USGS [2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	703	D1943-68	pp. 75 and 78.[3]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	703	D1943-68	p. 79.
3. Alpha-Counting error, pCi per liter	Proportional counter	900.0	703	D1890-68	pp. 75 and 78.[3]
4. Beta-Counting error, pCi per liter	Proportional counter	Appendix B	703	D1890-68	p. 79.
5. (a) Radium-Total, pCi per liter	Proportional counter	903.0	705	D2460-70	
(b) ^{226}Ra, pCi per liter	Scintillation counter	903.1	706	D3454-79	p. 81.

Table IE Notes

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.
[2] Frehman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).
[3] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

and blank/spike control. In methods not using surrogates such as metals, anions, and wet chemical analysis, a blank and a blank/spike (laboratory control sample) shall be analyzed. For pesticide/PCB methods, surrogates are often used. However, problems have been noted in surrogate recovery for the dibutyl chlorinate typically used. For pesticide/PCB analysis, a blank and a blank/spike shall be analyzed with each batch as separate samples. A pesticide or a PCB shall be used as the spiking compound.

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

All methods specified require calibration. In keeping with the method calibration requirements, 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, for the system performance check compounds (SPCCs), and for the 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 prior to sample analysis. The laboratory shall use the calibration check acceptance criteria specified by the method. The daily calibration acceptance criteria to be used for each method shall be documented in the laboratory QA plan or in the site-specific QA plan. The initial calibration curve shall be plotted and the correlation coefficient and response factors evaluated. The laboratory shall indicate in the laboratory QA plan or in the site-specific QA plan the acceptance criteria to be used for the initial calibration curve. The calibration shall include one standard at a concentration at the method detection limits. The calibration curve shall bracket all samples in the concentration range. If the samples are not within the calibration range, appropriate dilution shall be performed to bring the samples into the calibration 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 analysis 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.

For all GC methods used in level C QC, second column confirmation shall be used for all positive responses for the analytes of interest. In Level E, second column confirmation is not required.

In Level E, no matrix spikes or duplicates are required; only the initial and continuing calibration, method blank, and blank/spike are required.

7.2 DELIVERABLES

For Level D QC, a CLP data package shall be delivered. This shall include the summary package and the remainder of the package, which includes initial and continuing calibration, matrix spikes, matrix spike duplicates, blanks, duplicates, surrogate recoveries, chromatograms, mass spectra, and absorbance data. For methods which are not defined by CLP, the calibration information, method blanks, blank/spikes, the chromatograms, absorbance, matrix spikes, and matrix spike duplicates shall be reported. The control charts plotted per Sect. 4 associated with the blank/spikes shall be presented with the data.

For Level C QC, the method blanks, blank/spike, surrogates, matrix spikes, matrix spike duplicates, duplicates, and initial and continuing calibration data shall be reported. Table 7.6 lists the required deliverables. The forms referred to in Table 7.6 are from the current CLP for organics and metals/cyanide. The form numbers will be upgraded as new revisions occur in the CLP, which require changes in form content or numbering.

In Level E, the only information to be submitted is the sample data, method blank data, and the control chart from the blank/spike.

The deliverables shall be presented to the NCR. The forms shall be used when reporting any data in the MPR and in submitting the final data package prior to its inclusion in the appendix and summary tables of the final report. The final data deliverables shall be presented to the NCR at least three weeks prior to issuing the draft of the final report.

7.3 DATA VALIDATION

7.3.1 Level ~~E~~ D Validation

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

EPA, Hazardous Site Control Division, *Laboratory Data Validation Functional Guidelines for Evaluating Pesticides/PCB's Analyses*, R-582-5-5-01, May 28, 1985.

EPA, Hazardous Site Control Division, *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses*, R-582-5-5-01, May 28, 1985.

EPA, Office of Emergency and Remedial Response, *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses*, 1985.

Table 7.6. Data set deliverables for Level C QA

	Method requirements	Deliverables
Organics	- Method blank spikes with results and control charts. Run with each batch of samples processed.	Control chart
	- Results to be reported on CLP Form 1 or spreadsheet per Sect. 9. Sample results using CLP data flags.	Form 1 or Sect. 9 1/Sample chromatograms/and mass spectra
	- Surrogate recovery from samples reported on CLP Form 2. Surrogates to be used in volatiles, semivolatiles, pesticides/PCB. For volatiles by GC, the names of surrogates should be changed to reflect the surrogate used.	Form 2
	- Matrix spike/spike duplicate 1 spike and spike duplicate per 20 samples of similar matrix reported on Form 3.	Form 3
	- Method blank reported on CLP Form 4.	Form 4 or Sect. 9
	For volatiles by GC, a similar format will be used as CLP Form 4 for blanks.	
	- GC/MS tuning for volatiles/semivolatiles. Report results on Form 5.	Form 5
	- Initial calibration data reported on Form 6.	Form 6
	For volatiles by GC, the initial calibration data with response factors must be reported.	No Form
	For pesticide/PCB data Form 9 must be used for calibration data.	Form 9
	- Continuing calibration GC/MS data reported on Form 7.	Form 7
	For volatiles, GC data, the response factors and their percent differences from the initial must be reported.	No Form
	Internal Standard Area for Volatiles and Semivolatiles.	Form 8

Table 7.6. (continued)

	Method requirements	Deliverables
Organics (cont'd)	- For pesticides/PCB data, the CLP Form 9 must be presented.	Form 9
	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.	
	- Internal standard area for GC/MS analyses CLP Form VIII shall be supplied.	
	- Second column confirmation shall be done for all GC work when compounds are detected above reporting limits. Chromatograms of confirmation must be provided.	Chromatograms
Metals	- Level C, requirements	Deliverables
	- Sample results with CLP flagging system	CLP Form 1 or Sect. 9
	- Initial and continuing calibration	CLP Form 2, Part 1 only
	- Blanks 10% frequency	Form 3
	- Method blank taken through digestion (1/20 samples of same matrix)	Form 3 or Sect. 9
	- ICP interference check sample	Form 4
	- Matrix spike recovery (1 per 20 samples of similar matrix)	Form 5, Part 1
	- Postdigestion spike sample recovery for ICP metals. Only done if predigest spike recovery exceed CLP limits.	Form 5, Part 2 (never used for GFAA work)
	- Postdigest spike for GFAA	Recovery will be noted on raw data
	- Duplicates (1 per 20 samples will be split and digested as separate	Form 6 samples

Table 7.6. (continued)

	Method requirements	Deliverables
Metals (cont'd)	- Method blank spike information will be plotted on control chart, one per batch of samples processed.	Control chart
	- Standard addition. The decision process outlined in CLP page E-3 will be used to determine when standard additions are required.	Form 8
	Holding times	Form 10
Wet Chemistry	Level C	
	- Blank spike 1/batch	Control chart
	- Method Blank 1/batch	Report result No format
	- Sample results	Report result No format
	- Matrix spike/spike duplicate or calibration information	Report result if applicable
	- Calibration check report percent RSD or percent difference from initial calibration	Report percent or percent difference No format

7.3.2 Level C Data Validation Guidelines

Listed below are the validation criteria which will be utilized in evaluating the analytical data for a Level C QC site. For methods not listed here, a similar procedure will be submitted by the prime contractor and the laboratory which outlines validation of the holding times, initial calibration, continuing calibration, and blank-vs-sample results. The validation procedure will be approved by the NCR.

1. For Petroleum Hydrocarbons (418.1/SW-3540, EPA 418.1)

   Holding Times - Holding times are 28 days for water samples which are preserved and refrigerated. No holding times are cited for soils.

Calibration - Ensure that a three-to-five point curve bracketing the sample concentration is performed daily.

Blanks - A blank should be run with each batch. If the blank concentration exceeds the reporting limit, the reporting limit shall be raised and the data flagged as estimated (UJ).

2. Target Compound List (TCL) for VOAs (CLP Methods)

Holding Times - Samples must be analyzed within the holding times specified in Sect. 3 or the data should be marked as estimated (J).

GC/MS Tuning - Check that bromofluorobenzene tune is completed each 12-h shift of operation. Check that it meets the CLP criteria. Assure that each sample is associated with a tune.

Initial Calibration - The maximum relative standard deviation [(RSD) percent RSD] shall not be >30% for indicted CLP CCC. The maximum mean relative response factor (RRF) for SPCC shall be >0.300 (0.250 for bromoform). The SPCCs are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The CCC compounds are vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, and ethylbenzene.

Continuing Calibration - The minimum response factor for the SPCC components for VOAs analyses shall not be <0.300 (0.250 for bromoform). The maximum response factor percent deviation for indicated CLP CCC components from the mean initial calibration response factor shall not exceed 25%. If these criteria are exceeded, a new calibration for the compound shall be employed.

Blank/Spike Control Samples - Any control sample which exceeds the 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, the 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, the samples associated with the batch shall be reanalyzed and the data from reanalysis reported. If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples within the holding times or the data will be flagged with an "R," and the data are not usable.

Surrogates - If surrogates exceed the CLP limits, the data shall be flagged that the surrogates exceeded limits.

Method Blanks - A method blank should be run each day following the Continuing Calibration Standard. Common laboratory solvents should not be found in the blank at levels over five times the detection

limits. Other compounds should not be found in the blank at levels exceeding the detection limits. If common contaminant compounds are detected in samples at a concentration of <10 times the concentration found in the blank, or other compounds at <5 times the concentration in the blank, report those compounds as not detected. Adjust the sample quantitation limit to the value reported in the samples and flag the limit as estimated (UJ).

Matrix Spike/Spike Duplicate - Ensure that 1 out of 20 samples has been spiked in duplicate. The recoveries shall meet the CLP criteria. If the recoveries do not meet the criteria, examine the blank spike data. If the blank spike data exceed the limits and the matrix spikes exceed limits, the data shall be flagged as unusable (R). If the blank spike data from the batch are satisfactory, the data is usable, and the low recovery is discussed in the final report QA/QC and in the QC report sent to the NCR.

Field Trip and Equipment Blanks - If contaminant analytes are detected in samples at concentrations of <5 times the concentration found in the highest associated blank, the results are considered suspect and are reported as estimated.

3. TCL Semivolatile Organics (CLP Methods)

Holding Times - Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction. Any samples which do not meet these requirements must be flagged as estimated.

GC/MS Tune - Make certain that a decafluorotriphenylphosphine tune is completed every 12 h of sample analysis, that each sample is associated with a tune, and that each tune meets CLP requirements. Data are not reported if the instrument does not meet tune.

Initial Calibration - Ensure that a 5-point curve has been completed. The RRF of the BNA compounds shall be a minimum of 0.050 for the SPCC listed in the current revision of the CLP. The maximum RSD for the CCC listed in the CLP procedure is 30.0%. The minimum RRF for the SPCC is 0.050, and the maximum percent difference for the CCC is 25%. If these limits are exceeded, a new calibration curve shall be generated.

Continuing Calibration - The continuing calibration check will be performed once every 12 h during operation. The minimum RRF for the SPCC is 0.05, and the maximum percent difference from the initial calibration shall not exceed 25% for the CCC. If these limits are exceeded, a new calibration curve shall be generated.

Blank/Spike Control Samples - Any control sample which exceeds the 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, the data and the out-of-control point shall be discussed in the QC section of

the report. If problems are found in the analytical data, the samples associated with the batch shall be reanalyzed and the data from reanalysis reported. If holding times are exceeded in the reanalysis, both sets of data shall be presented.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples or the data will be flagged with an "R," and the data is not usable.

Surrogates - If surrogates exceed the CLP limits, the data shall be flagged that the surrogates exceeded limits.

Blanks - A method blank should be run each day following the Continuing Calibration Standard. Phthalate should not be found in the blank at levels over five times the detection limits. Other compounds should not be found in the blank at levels exceeding the detection limits. If common contaminant compounds are detected in samples at a concentration of <10 times the concentration found in the blank, or other compounds at <5 times the concentration in the blank, report those compounds as not detected. Adjust the sample quantitation limit to the value reported in the samples and flag the limit as estimated (UJ).

Matrix Spike/Spike Duplicate - Ensure that 1 out of 20 samples has been spiked in duplicate. The recoveries should meet the CLP criteria. If the recoveries do not meet the criteria, examine the blank spike data. If the blank spike data exceed the limits and the matrix spikes exceed limits, the data shall be flagged as unusable (R). If the blanks spike data from the batch is satisfactory, the data are usable, and the low recovery is discussed in the final QC report sent to the Analytical Environmental Support Section.

4. Metals

Holding Times - Samples must be analyzed within six months, except mercury shall be analyzed in 28 days from sample collection.

ICP Initial Calibration - A calibration blank and at least one standard must be analyzed daily. An initial calibration verification standard must be within 90 to 110% recovery or the samples should be reanalyzed. If it is not possible to perform reanalysis, the data are rejected and flagged with an "R."

AA Calibration - Calibration blank and at least three standards shall be used in establishing the curve prior to sample analysis. A curve shall be analyzed each day prior to sample analysis.

Calibration Verification - Verification using a standard obtained from a source other than that of the initial calibration shall be used and the result shall be within 90 to 110% of the true value for

both ICP and AA work. Calibration verification shall be done at a minimum frequency of 10% or every 2 h, whichever is more frequent, and shall be done at the end of the analytical run.

Method Blanks - At least one preparation blank shall be prepared with each batch of samples. The blanks shall contain less than the detection limit for all analytes. If the concentration of the associated blanks is above the detection limit and if the lowest analyte concentration is <10 times the blank, reanalysis of the sample must occur. If reanalysis is not done, the data shall be reported and flagged as estimated. The blank 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, the results are considered suspect and are reported as estimated.

Blank/Spike Laboratory Control Samples - Any laboratory control sample which exceeds the 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, 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, the samples associated with the batch shall be reanalyzed and the 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 blank/spike laboratory control sample is out of control shall be presented in the QC section of both the final report and the MPR.

If the blank/spike results are outside the internal laboratory limits and if the matrix spike results are outside the CLP limits, the laboratory will either reanalyze the samples or the data will be flagged with an "R," and the data are not usable.

8. MAINTAINING LABORATORY APPROVAL

Once a laboratory has received Navy approval to begin analysis of samples, maintaining that approval requires adherence to the QA plan and reporting of QA-related information. The performance and reporting requirements outlined below are essential to ensuring that data of known and defensible quality are being generated throughout the course of a site investigation. Topics covered include control samples, control charts, out-of-control events, corrective action reports, significant changes in the QA plan, and other reporting requirements.

8.1 MONTHLY PROGRESS REPORT

The primary means of communication from the laboratories to the NCR will be the MPR to be submitted by the laboratories to the NCR on the 15th of each month in which work for the Navy is performed. The following information is to be included in the MPR.

1. Site name and contract number.

2. Numbers, types and locations of samples collected and analyzed for Navy project only.

3. Data for blanks, spikes, laboratory duplicates and controls related to Navy samples.

4. New methods used for analysis and changes in old methods.

5. Copies of all control charts pertinent to Navy samples and to which results have been added over the reporting period.
6. Summaries of out-of-control incidents during the reporting period, including references to documentation and corrective action reports.

7. Descriptions of and justifications for significant changes in the QA.

8. Changes in LQAC personnel and other key technical personnel; resumes of new personnel must be submitted.

9. Completed sample data.

Much of the information presented in an MPR is incremental in nature and relates to changes and findings since the previous MPR.

1. Control charts from the minimizing control charts program and any additional control charts from monitoring matrix spikes, duplicates, or other QC parameters.

2. Personnel changes relating to QA responsibilities.

3. Method changes (e.g., a minor modification with an attached EPA variance).

4. Procedural changes in establishing control limits and/or the preparation and use of control charts.

Since 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.

8.2 FINAL REPORT

A draft of the final report shall be reviewed by the NCR prior to its release. This report is the final deliverable from the engineering subcontractor. An outline for a typical report is as follows.

1. Site name and Navy contract number.

2. Foreword--signed by those with major responsibilities for the QA program and by project management.

3. Executive Summary--brief review of the report.

4. Table of Contents--with specificity at approximately the same level as the Table of Contents in this Navy document.

5. Introduction--summarize the Navy field sites of interest, when the study occurred (dates of sampling, dates of analysis) and the objectives of the QA plan as they relate to the study.

6. Data Summary--summarize the results on a site-by-site basis.

7. Other Information--present any other information requested in the statement of work such as risk assessment, recommendation to perform more site characterization, or recommend site closure. This information was specified prior to beginning work and is directed by the Navy EIC.

8. The final report shall present the findings from the analytical, geological, and hydrogeological studies. The summary of analytical data will exclude non-detected compounds. No subtraction of blanks is allowed. Data will be flagged if blank contamination occurs. All data flags will follow the result in the summary.

9. QC Summary--the QC summary section will include a discussion of are data which flagged. Flagged data defined as data for which trip, field, or laboratory blanks were contaminated, matrix spike/spike duplicates exceed limits, calibration criteria are not met, and laboratory controls exceed limits. The QC summary will also discuss the results of laboratory blanks, matrix spikes/spike duplicates,

duplicates, control charts, surrogate holding times, field blanks, trip blanks, rinsates, and field duplicates. This section will also discuss precision, accuracy, and completeness.

10. Appendices--the appendices of the report shall include all field and analytical data. One appendix shall contain field logs and forms. A second appendix shall contain the laboratory data of each sample. These data shall be presented in a spreadsheet similar to the Format Section of this report. All trip, field, and laboratory blanks shall be marked so that each sample can be associated with the appropriate blanks.

    A third appendix shall include the method blank spike control charts, surrogate recoveries, matrix spike and duplicate, field, and laboratory duplicates for all spike samples.

8.3 FINAL QC DATA REPORT

A QC data report shall be sent to the NCR. This report shall contain the following.

For Level D QC, the contractor shall submit a subset of data from the CLP data packages. For 20% of the water and 20% of the soil samples, the subcontractor shall submit the full CLP package.

For Level C QC, the deliverables listed in Table 7.~~X~~6 will be presented.

For Level E QC, the initial and continuing calibration forms, method blank, and blank spike control chart are required.

The report shall indicate the duration and location of storage for the data. The stored data consists of all raw data, QC charts, corrective action, logs, sample lists, COC information, notebooks, work sheets, automated data processing system output, and calibration.

The report shall be delivered to the NCR three weeks prior to the final report.

9. DATA FORMAT

The data format refers to the format in the final report. The contractor may use its own format in the body of the report. However, in the appendices (which contain sample and blank data) a spreadsheet type of format may be used, or the CLP forms for reporting samples and equipment, trip, field, and method blanks may be used. The spreadsheet format allows for more samples per page and for more information on blanks and their association with samples. The spreadsheet format is not meant to be a rigid form. The information listed in Fig. 9.1 must be present. The contractor may add other information which will assist it in review. For calibration, tuning, spikes, surrogates, and duplicates, the current CLP forms are required for data presentation. If any other format is to be used, this shall be discussed with the NCR.

Sample Number	J25019	JS5020
Date Sampled	03-18-87	03-18-87
Sample Prep..Date	11-25-87	11-25-87
Sample Analysis Date	11-26-87	11-26-87
Sample Numbers of Associated Analytes, Field, Trip, and Equipment Blanks	J4455667	L4455667

	Detection limit	Sample	Results
VOLATILE ORGANIC COMPOUNDS (µg/kg)			
TETRACHLOROETHANE	5	50	50
CHLOROBENZENE	5		
SEMIVOLATILE ORGANIC COMPOUNDS (µg/kg)			
BIS(2-ETHYLHEXYL) PHTHALATE	330		750
2-METHYLNAPTHALENE	330		2500
INORGANIC COMPOUNDS (mg/kg)			
LEAD	10	360	25
HYDROCARBONS (2)			
PETROLEUM HYDROCARBON	1	0.611	0.268
OIL AND GREASE	1		

Sample Results

Analyte

Note: Petroleum hydrocarbon, oil, and grease results recorded in percent.

Fig. 9.1. Example of data format for final report.

GLOSSARY

Accuracy - The nearness of a result or the mean of a set of results to the true or accepted value.

Analyte - A chemical component of a sample to be determined or measured.

Analytical Method - Defines the samples preparation and instrumentation procedures or steps that must be performed to estimate the quantity of analyte in a sample.

Analytical Spike - The furnace postdigestion spike. The addition of a known amount of standard after digestion.

Background Correction - A technique to compensate for variable background contribution to the instrument signal and the determination of trace metals.

Calibration - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

Calibration Blank - A volume of acidified deionized/distilled water.

Comparability - is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions.

Completeness - Completeness is defined as the percentage of measurements made which are judged to be valid measurements. The completeness goal is to generate sufficient amount of valid data based on project needs.

Continuing Calibration - Analytical standard run every ten analytical samples or every 2 h, whichever is more frequent, to verify the calibration of the analytical system.

Control Limits - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Correlation Coefficient - A number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are, the closer the value to one. Determined on the basis of the least squares line.

Data Quality Objectives - are qualitative and quantitative statements which specify the quality of the data required to support decision during

GLOSSARY (continued)

remedial response activities. Data quality objectives are determined based on the end uses of the data to be collected.

Detection Limit - The minimum concentrations which must be accurately and precisely measured by the laboratory and/or specified in the quality assurance plan.

Dissolved Metals - Analyte elements which have not been digested prior to analysis and which will pass through a 0.45-µm filter.

Duplicates - Identical splits of individual samples which are analyzed by the laboratory to test for method reproducibility. In this case, samples are split in the laboratory.

Equipment Rinsates - The final analyte-free water rinse from equipment cleaning collected daily during a sampling event.

Field Blanks - Blanks are collected and analyzed to determine the level of contamination introduced into the sample due to sampling technique. They may consist of the source water used in decontamination and steam cleaning. At minimum, one sample from each event and each source of water must be collected and analyzed.

Field Duplicates/Splits - Samples that have been divided into two or more portions while in the field. Each portion is then carried through the remaining steps in the measurement process. A sample may be replicated in the field or at different points in the analytical process. For field replicated samples, precision information would be gained on homogeneity, handling, shipping, storage, preparation, and analysis.

Replicate samples divided into two portions and sent to different laboratories and subjected to the same environmental conditions and steps in the measurement process as the split samples.

Instrument Detection Limit - is defined in several ways. For example, (1) that concentration of analyte which produces an output signal twice the root mean square of the background noise may be determined under ideal conditions or (2) determined by multiplying by 3 the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x instrument detection limit on three nonconsecutive days with seven consecutive measurements per day.

Internal Standards - Compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatile), and sample extract (for semivolatile) at a known concentration prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

GLOSSARY (continued)

Laboratory Control Sample - A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for samples received.

Laboratory Quality Assurance Coordinator - An employee of a laboratory with no analysis or production responsibilities and who implements QA and QC. This person is responsible for ensuring all quality problems are resolved.

Matrix - The predominant material comprising the sample to be analyzed. The most common matrices are water, soil/sediment, and sludge.

Matrix Spike - An aliquot of a matrix (water or soil) spiked with known quantities of compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate - A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method.

Method Blank - A blank sample run to ensure reported analytical results are not the results of laboratory contamination.

Method Blank/Spike - Is the distilled and/or deionized water for soil or sand spiked with known compounds or elements. The method blank as defined by Contract Laboratory Protocol for organics and the laboratory control sample as defined by Contract Laboratory Protocol maybe use as the method blank/spike in the Navy Installation Restoration Program.

Method Detection Limits - Minimum concentrations of a substance that can be measured and reported with 99% confidence that the value is above zero. The sample is carried through the entire method under ideal conditions.

Method of Standard Additions - The addition of three increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept, and y-intercept are determined by least-squares analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (~10% of the volume). Standard addition may counteract matrix effects; it will not counteract spectral effects. It is also referred to as standard addition.

Out of Control - One or more of several conditions relating to the plotting of control data and indicating unacceptable results.

GLOSSARY (continued)

Percent Solids - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Precision - Measure of the reproducibility of a set of replicate results among themselves or the agreement among repeat observations made under the same conditions.

Preparation Blank (Reagent Blank, Method Blank) - An analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

Purge and Trap - An analytical technique used to isolate volatile (purgable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quality Assurance - A planned system of activities (program) whose purpose is to provide assurance of the reliability and defensibility of the data.

Quality Control - A routine application of procedures for controlling the monitoring process. QC is the responsibility of all those performing the hands-on operations in the field and in the laboratory.

Reagent Water - Water in which an analyte is not observed at or above the minimum quantitation limit of the parameters of interest.

Recovery - Usually expressed as a percent. The numerical ratio of the amount of analyte measured by the laboratory method divided by the known amount of analyte added to the matrix (i.e., spiked sample) to be analyzed.

Reporting Detection Limits - The same as method detection limits with consideration given for practical limitation such as sample size, matrix interferences, and dilutions.

Representativeness - Expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program.

Sample Holding Times - Times used to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis or sample preparation. Holding times may vary depending on the analysis, EPA regional preference, etc.

GLOSSARY (continued)

Semivolatile Compounds - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with base neutral acid or extractable compounds.

Serial Dilution - The dilution of a sample by a known factor. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Spikes - Known amounts of specific chemical constituents added by the laboratory to selected samples to test the appropriateness and recover efficiencies of specific analytical methods within the actual sample matrices.

Standard Deviation - The square root of the variance of a set of values.

Surrogates - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard and used to evaluate analytical efficiency of the method by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media. These are used typically in organic methods.

Tentative Identified Compounds - Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

Total Metals - Analyte elements which have been digested prior to analysis.

Variance - The sum of the squares of the difference between the individual values of a set and the arithmetic mean of the set, divided by one less than the number of values.

Volatile Compounds - Compounds amendable to analysis by the purge and trap techniques. Used synonymously with purgable compounds.

Data Qualifiers' Definitions as defined by the Contract Laboratory Protocol for Organic Analysis

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution and for percent moisture.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero.

GLOSSARY (continued)

C - This flag applies to pesticide results where the identification has been confirmed by gas chromatography/mass spectrometry. Single component pesticides ≥10 ng/ul in the final extract shall be confirmed by gas chromatography/mass spectrometry.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified TCL compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the gas chromatography/mass spectrometry instrument for that specific analysis. This flag will not apply to pesticides/PCBs analyzed by GC/EC methods. If one or more compounds have a response greater than full scale, the sample or extract must be diluted and reanalyzed. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is reanalyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" flag.

A - This flag indicates that a TIC is a suspected aldol-condensation product.

X - Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the Sample Data Summary Package and the Case Narrative. If more than one is required, use "Y" and "Z," as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "A," "B," and "D" flags for some sample.

R - Quality control indicates that data are not usable (compound may or may not be present). Resampling and reanalysis are necessary for verification.

Q - No analytical result.

GLOSSARY (continued)

Inorganic Data Qualifiers

E - The reported value is estimated because of the presence of interference. An explanatory note must be included under Comments on the cover page (if the problem applies to all samples) or on the specific FORM I-IN (if it is an isolated problem).

M - Duplicate injection precision not met.

N - Spiked sample recovery not within control limits.

S - The reported value was determined by the Method of Standard Additions.

W - Postdisgestion spike for Furnace Atomic Absorption analysis is out of control limits (85-115%), while sample absorbance is less than 50% of spike absorbance.

* - Duplicate analysis not within control limits.

+ - Correlation coefficient for the Method of Standard Addition is less than 0.995.

M (Method) Qualifier

"P" for ICP

"A" for Flame AA

"F" for Furnace AA

"CV" for Manual Cold Vapor AA

"AV" for Automated Cold Vapor AA

"AS" for Semiautomated Spectrophotometric

"C" for Manual Spectrophotometric

"T" for Titrimetric

"NR" if the analyte is not required to be analyzed

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Duties: Assist in the program management of the Installation Restoration Program.

Stephen Eikenberry, Manager

Duties: Manager of the Installation Restoration Program.