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PREPARATION AIDS

FOR THE DEVELOPMENT OF

CATEGORY I

QUALITY ASSURANCE PROJECT PLANS

by

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Foreword

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing the research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of a quality assurance outreach program that provides a vital communication link between the researcher and the user community.

Perhaps the greatest benefit to the users of this document comes from its emphasis on up-front planning -- do the right thing, right, the first time. While this sounds straightforward, managers know that determining the right course in a complex and uncertain situation is anything but simple. Determining customer requirements up-front, and then having the processes and procedures in place to accomplish them, averts costly mistakes. Resources are conserved in two ways: by avoiding rework to correct efforts that do not initially meet management or customer specifications; and by performing to the specifications required and not beyond them. In these ways, this "Preparation Aids" document can help management achieve its mission with more effective utilization of diminishing resources.

E. Timothy Oppelt, Director Risk Reduction Engineering Laboratory

Abstract

Data collection activities performed for the Risk Reduction Engineering Laboratory (RREL) of the U.S. Environmental Protection Agency are divided into four categories, depending on the intended use of the data. Quality Assurance (QA) Project Plans are written to ensure that project needs will be met and that quality control procedures are sufficient for obtaining data of known quality. The level of QA required, however, depends on the project category selected for a given project. Projects that are of sufficient scope and substance that their results could be used directly, without additional support, for compliance or other litigation are identified as Category I projects. Such projects are of critical importance to the Agency goals and must be able to withstand legal challenge. Accordingly, the QA requirements will be the most rigorous and detailed in order to ensure that such goals are met.

To assist professional scientists and engineers in preparing QA Project Plans, separate guidance manuals in an easy-to-read format have been developed for each category. The Category I manual contains detailed descriptions of each of the 15 required elements of a Category I QA Project Plan. Also included are definitions and explanations of frequently used terms, examples of QA forms and charts, sample equations, and numerous types of tables suggested for summarizing information.

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The extensive technical contributions of Dr. John R. Wallace of Maxwell Laboratories and the assistance of Robert Danner, Ann Kern, and William Mueller of the USEPA in reviewing the document are gratefully acknowledged. Grateful appreciation is, also, extended to the excellent editorial and technical comments provided by Dr. Monica Nees.

SECTION 0.0 INTRODUCTION

0.1 PURPOSE OF THE QUALITY ASSURANCE PROJECT PLAN (QAPjP)

The purpose of a QA Project Plan is to relate project objectives to specific measurements required to achieve those objectives. The QA Project Plan must provide sufficient detail to demonstrate the following:

- Intended measurements are appropriate for achieving project objectives;
- Quality control procedures are sufficient for obtaining data of known and adequate quality; and
- Such data will be defensible if challenged technically or legally.

Environmental projects require the coordinated efforts of numerous individuals, including regulators, managers, engineers, scientists, statisticians, and economists. The QA Project Plan must integrate the requirements of everyone involved, in a form that permits an easy and painless review. It must also provide unambiguous instructions to the sampling team, the analytical laboratory, and any other parties responsible for data generation. Finally, the QA Project Plan must provide sufficient detail to allow a thorough, detailed review by an independent party not involved in the project implementation.

0.2 QUALITY ASSURANCE CATEGORIES

Because the end use of the data determines the degree of quality assurance that is required, RREL uses four categories in its QA Program:

CATEGORY I PROJECTS require the most rigorous and detailed QA, since the resulting data must be both legally and scientifically defensible. Category I projects include enforcement actions and projects of significant national or congressional visibility. Such projects are typically monitored by the Administrator. Category I projects must produce results that are autonomous; that is, results that can prove or disprove a hypothesis without reference to complementary projects.

CATEGORY II PROJECTS are those producing results that complement other inputs. These projects are of sufficient scope and substance that their results could be combined with those from other projects of similar scope to produce information for making rules, regulations, or policies. In addition, projects that do not fit this pattern, but have high visibility, would also be included in this category.

CATEGORY III PROJECTS are those producing results used to evaluate and select basic options, or to perform feasibility studies or preliminary assessments of unexplored areas which might lead to further work.

CATEGORY IV PROJECTS are those producing results for the purpose of assessing suppositions.

The RREL Technical Project Manager is responsible for assigning the category that accurately reflects the intended use of the data and the type of work being done.

This document addresses only Category I requirements; separate documents are available for each of the remaining categories. Be sure to use the Preparation Aids document for the correct category.

0.3 UNDERLYING LOGIC AND ORGANIZATION OF A QA PROJECT PLAN

A QA Project Plan, regardless of category, must cover the following topics:

- Process being tested and the objectives of the test (i.e., the hypothesis to be tested);
- Measurements that will be taken to achieve those objectives;
- · Quality of data that will be required and how that quality will be obtained; and
- How data will be recorded, calculated, reviewed, and reported in a defensible manner.

The QA Project Plan is organized according to the topics discussed in the body of this report.

To be an effective communication tool, the QA Project Plan must be concise, but still contain essential experimental detail.

Be sure to include:

- Any project-specific information necessary for the sampling team, analytical laboratory, data reduction team, and all other project participants.
 However, assume these parties are familiar with standard methods.
- Any deviations from standard methods or procedures, or clarification of such documents whenever there are ambiguities.
- Equations for all project-specific calculations and data reductions.

But don't be repetitious:

- Do not discuss the same subject matter more than once in a QA Project Plan. Although the required elements of the QA Project Plan may appear to overlap in some cases, repetition leads to confusion, and to documents too long for effective communication. If you are unsure where a subject should be treated, discuss it once and cross-reference that discussion in the other sections.
- Do not repeat material from a Sampling Plan. If a separate Sampling Plan has been generated, reference the section and page number in the QA Project Plan, as needed.
- Do not repeat material from standard methods that are available from the EPA or the Code of Federal Regulations. Provide detailed citations and assume that the analyst and reviewer have these methods on-hand.

0.4 CATEGORY I FORMAT NOTES

All Category I QA Project Plans must incorporate the following format requirements:

- · Title page.
- QA Project Plan Approval Form (see Figure 0-1).
- Distribution list to ensure that key personnel will receive current copies and updates.
- Table of Contents as shown at the beginning of this document. If a given element is not applicable, the text of the QA Project Plan should explain its omission.
- Document control format. Section number, revision, date, and page should be recorded in an upper corner of each page. This format requirement is illustrated below.

 Section No.
 1.0

 Revision:
 0

 Date:
 October 20, 1989

 Page:
 3 of 5

0.5 QA PROJECT PLAN APPROVAL FORM (SIGNATURE PAGE)

It is important that the project principals understand and agree on the experimental approach. For this reason, the QA Project Plan Approval Form must be signed by key project personnel as well as key personnel from any subcontractors. These signatures—which must be obtained before the final QA Project Plan is submitted—indicate that the key personnel have read the appropriate sections of the QA Project Plan and are committed to implementing the plan.

QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM for RREL Contracts/IAGs/Cooperative Agreements/In-house Projects

Project Plan Title:	<u>.</u>		
		Revision Date:	
MMITMENT TO IMPLEMENT T	HE ABOVE QA PROJE	ECT PLAN:	
Contractor's Project/Task M	anager (print)	Signature	Date
Contractor's QA Manager (p	Print)	Signature	Date
Other as Appropriate/Affiliat	lon* (print)	Signature	Date
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* Commitment signature is r gathering support provider	equired for any ancillad by a subcontractor of	ary sampling, analytical, or da or RREL principal investigator	it a
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	ORDANCE TO THE AE		Date
ROVAL TO PROCEED IN ACCO	ORDANCE TO THE AE	BOVE QA PROJECT PLAN:	
ROVAL TO PROCEED IN ACCO	ager (print) ef (print)	BOVE QA PROJECT PLAN:	
ROVAL TO PROCEED IN ACCO	ef (print)	SOVE QA PROJECT PLAN: Signature	Date

Figure 0-1. Quality Assurance Project Plan Approval Form

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SECTION 1.0 PROJECT DESCRIPTION

This section describes the process or environmental system that is to be tested, the project objectives, a summary of the experimental design, and the proposed project schedule. This section typically contains the subsections listed below, plus others at the principal investigator's discretion.

1.1 GENERAL OVERVIEW

This section provides a brief synopsis of the overall project. In one or two paragraphs, describe the programmatic and regulatory setting in which the project is going to be carried out. The purpose(s) of the study should be described, along with the decisions that are to be made and the hypothesis to be tested. Other anticipated uses of the data should also be noted. Explain the consequences of drawing incorrect conclusions or making invalid decisions. The type of process or environmental system that is to be tested should also be described briefly.

Category I projects are typically preceded by various preliminary investigations of the site or process. Results of such preliminary tests are included in this section, with details presented in an appendix.

Example: Preliminary investigations of the test site indicated an average PCP concentration of 150 mg/kg, with a range of 10 to 500 mg/kg. A summary of individual analyses is contained in Appendix...

Category I projects use such preliminary data for selecting appropriate analytical methods, and for estimating the required number and types of samples.

Category I QA Project Plans must often demonstrate compliance with applicable regulations. Such regulations should be summarized if they are applicable to the project.

<u>Example</u>: According to TSCA regulations, PCB emissions from the stack shall be less than 1 mg/kg of PCB introduced into the incinerator. Combustion efficiency shall be greater than 99.9999 percent as defined in these regulations (40 CFR 761.70...). According to the State permit, stack emission rates of HCl must be less than

1.1.1 The Process, Site, Facility, or System

This section describes the process, site, facility, or environmental system that will be tested. Include flow diagrams, maps, charts, etc., as needed. Approximate mass or volumetric flow rates should be indicated to permit proper evaluation of the sampling and process monitoring procedures. Indicate sampling points with alphanumeric designations, either in this section or in Section 4.0 (Site Selection and Sampling Procedures). Additional diagrams are often included to unambiguously describe sampling points. Such detailed diagrams can be included either in this section or in Section 4.0.

This section should contain enough material to permit a technical reviewer who is unfamiliar with the specific project to assess the proposed sampling strategy.

1.1.2 Statement of Project Objectives

Category I projects typically have multiple objectives. These project objectives should be summarized and stated clearly in this section. Avoid scattering statements of project objectives throughout the sampling and analytical sections of the QA Project Plan, or among several documents. Statements of objectives, when scattered among various sections, often result in an unfocused effort.

Project objectives should be stated in numerical terms whenever possible:

<u>Poor Statement</u>: The bio-oxidation procedure will be characterized with respect to its ability to remove semivolatile organic compounds.

<u>Better Statement</u>: The major objective is to demonstrate a bio-oxidation efficiency of 95 percent for the compounds listed in Table...

<u>Best Statement</u>: The objective is to demonstrate a bio-oxidation efficiency of 90 percent or higher at a confidence level of 95 percent for the compounds listed in Table...

Some objectives cannot be stated in entirely quantitative terms. For example, one objective for a bio-treatment project might be to determine the dominant microbiological species in a reactor. Or, for a test of a new incinerator design, one might wish to determine the range of combustion conditions that result in a molten ("slagging") ash. However, such qualitative objectives are

more commonly found in developmental projects; the major objectives of a Category I project must be stated in numerical terms.

Clear project objectives form the basis for designing the sampling, analysis, and quality control strategies. Thus, it is essential that both the decision makers and technical staff understand and agree to these objectives.

It is common to rank project objectives according to importance (e.g., into primary and secondary objectives). Although this ranking is not essential, it does help focus effort on the primary goals of the project.

1.2 EXPERIMENTAL DESIGN

List all measurements that will be made during the project, then classify them as critical or noncritical measurements. Critical measurements are those that are necessary to achieve project objectives; they may include either on-site process measurements or chemical measurements. Thus, for a test of an electric arc furnace as a means of incinerating organics, the rates of consumption of shield gas (e.g., argon) and electric power are as important as the feed rate and concentration of pollutant. Noncritical measurements are those used for process control or background information. Table 1-1 and Table 1-2 illustrate two different means of designating critical and noncritical measurements.

It is common practice to test pollution control equipment or processes in different operating modes or stages. For example, an incinerator might be operated under warm-up conditions, followed by incineration tests at three different feed rates. The various test conditions should be summarized in this section. Indicate the length of time anticipated for each test condition. When appropriate, indicate how equilibrium conditions will be established before sample collection begins.

<u>Example</u>: Total hydrocarbons in the stack gas will be monitored continuously. Sampling will not begin until THC concentrations are stable within \pm 5 percent over a three-hour period.

Summarize in tabular form all measurements that are planned for each sample. Ideally, this table should indicate the total number of samples for each sample point, including quality control and reserve samples, as illustrated in Table 1-3 for a hypothetical chemical treatment project. Relate

Table 1-1. Summary of Critical and Noncritical Measurements for the Evaluation of an Hypothetical Electric Arc Incinerator

A. CRITICAL MEASUREMENTS

Chemical

Critical SVOCs^a in all feed and discharge streams

PCBs in all feed and discharge streams

Particulates, NO_x, and SO₂ in stock gas

TCLP/critical metals^b in slag and feed

TCLP/critical SVOCs^a in slag

Alkalinity of scrubber water

HCl in stack gas

Organic carbon in feed

Process

Electrical power consumption of arc furnace
Argon and oxygen consumption (rate and total)
Mass feed rate of soil
Total mass of slag discharged
Cooling requirements (temperatures and flow rates of cooling water)

B. NONCRITICAL MEASUREMENTS

Chemical

Noncritical SVOCs in all feed and discharge streams Permanent gases in stack: N₂, O₂, CO₂, CO Percent moisture in stack Ash fusion temperature of slag

Process

All temperatures and flows not listed above Opacity of stack gas Operating pressures

Critical SVOCs (semivolatile organic compounds) are pentachlorophenol, the three tetrachlorophenol isomers, chlorophenol, 1- and 2-methylnaphthalene, anthracene, acenaphthalene, and acetophene. These compounds have been detected during preliminary studies on the untreated wastes or are suspected decomposition products. Noncritical SVOCs are all additional compounds detected by Method 8270, as described in Section ____ of this QA Project Plan.

b Critical metals are As, Ba, Cd, Cr, Pb, Hg, Ni, Se, and Ag.

Table 1-2. Summary of Physical Measurements for the Evaluation of an Hypothetical Electric Arc Incinerator

Measurement	Measurement Classification	Measurement Site (Designation) ^a	Measurement Frequency
Electric Power to Arc Furn	nace	Power leads to furnace (P1)	
Current	Critical		15 minutes
Voltage	Critical	•	15 minutes
kWh	Critical	••	Before/after each run
Ar and O ₂ Flow	Critical	Feeds to furnace (Q1)	15 minutes
Mass of Soil/Slag	Critical	Drum balances @ feeder and discharge (M1, M2)	Each batch
•	• •	•	• ***
•	••	•	
• ,	•	• A Company of the Co	• • • • • • • • • • • • • • • • • • • •
Opacity of Stack Gas	Noncritical	Stack (S3)	Continuous
remperatures	Noncritical	(Test points T3-T12)	5 minutes
• *	•	•	•
•	•		•.
•	•	•	•
•	•	•	•

^a Alphanumeric designations of test points are shown in Figure ____.

Table 1-3. Summary of Planned Analyses (Including QC) for Chemical Treatment of Water

		Numb	er of Tests Perfe	ormeda	
		Influent Water	Effluent Water	Sludge	4
	Preliminary Samples	C1	Sample Points: E2		Total
emivolatile Organic Compounds (SVO	<u>C)</u> b				*
Non-QC (Primary)	3	60	60	10	133
Field Sampling Blank ^C	1	1	0	0	2
Field Duplicates	0	0	0	. 5	5
Laboratory Duplicates	0	0	0	0	0
Matrix Spikes (MSs)	1	3	10	2	16
Matrix Spike Duplicates (MSDs)	1	3	10	.2	16
Spare Samplesd	2	10	10	0	22
Independent Check Standard	0	1	0	0	_1
			Gra	and Total	195
<u>Metals</u> e				•	
Non-QC (Primary)	3	60	60	10	133
Field Sampling Blank ^C	1	'. 1	0	0	-2
Field Duplicates	0	0	0	5	5
Laboratory Duplicates	1	3	10	2	16
Matrix Spikes (MSs)	1	3	10	2	16
Matrix Spike Duplicates (MSDs)	0	. 0	0	0	0
Independent Check Standard	0	1	0	0	1
Spare Samples ^d	2	10	10	0	22
			Gra	and Total	195

Samples will be divided evenly among the ten anticipated runs. More QC samples are planned for the effluent than for the influent, since the former samples are expected to exhibit more variability. A matrix spike/matrix spike duplicate or a laboratory duplicate/matrix spike of the effluent stream will be determined for each treatment condition, because the effluent matrix may vary significantly with each treatment.

b Refers to the twelve compounds listed in Table ____.

c Trip blanks will be collected but not analyzed unless field blanks indicate a contamination problem.

d Not analyzed unless required.

e Refers to the eight metals in Table ____.

the individual samples to the sampling points shown in the process diagram, if applicable. For projects involving a large number of samples or analyses, it may not be possible to include all QC and reserve samples in a single table. For such cases, two or more tables may be necessary, the first to summarize the primary (non-QC) samples, and the others to show which QC samples are associated with which analyses. See Tables 1-4 and 1-5 for an example of a hypothetical project involving the solidification of solid wastes. Information on the total number of all sample types is needed both to perform a comprehensive review and to estimate analytical costs.

1.3 SCHEDULE

Indicate start-up and ending dates, including those for preliminary studies and field and laboratory activities.

Be sure to:

- Include enough information in this section to permit a technical person unfamiliar with your project to evaluate the sampling and analytical approach.
- Avoid repeating material from a sampling or analytical plan. Complete a
 necessary description once, then cite it in all other documents. Repeating
 material inevitably leads to confusion and wastes the valuable time of
 project participants, typists, and reviewers. Most important, essential
 details are readily overlooked when buried in repetitious or overly lengthy
 documentation.
- Cite applicable regulations, if any.

Table 1-4. Total Number of Analyses (Not Including QC) for Each Treatment Condition: Hypothetical Solidification Process

·			Treatmen	t Condition		-
Measurement ^a	Measurement Classification	Raw Soil	Treated Soil (28 Days)	Reagent Mix	Long-Tern Treated Soils	
On-Site Tests Slump of Portland Cement Concrete	Noncritical	• • ;	ANC	-	-	
Homogeneity of Mixing	Noncritical	-	9	•	•	
Leaching Tests/Analyses ^d					•	
TCLP/Metals	Critical	9	9	1	15	
TCLP-ZHE/VOCs	Critical	9	. 9	1	15	,
WILT/TOC + VOC + Metalsd	Critical	0	3	0	15	
ANS-16.1/TOC + VOC + Metals ^C	Critical	Ö	3	0	15	,
Chemical Tests	•	,				
pH	Critical	9	9	1	-	
ŤŌC	Noncritical	9	9	1		
Metals - Total	Critical	9	9	1	_	
VOCs - Total	Critical	9	9	ī	-	
Engineering Tests						
Particle Size Analysis	Noncritical	⁵ 3	0	0		
Bulk Density	Critical	3	3	Õ	15	
Freeze/Thaw Durability	Critical	Õ	6	ŏ	15	*
Unconfined Compressive Strength	Critical	Õ	6	ŏ	15	

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VOC = Volatile Organic Compounds
TCLP = Toxicity Characteristic Leaching Procedure
TCLP-ZHE = Zero headspace modification of TCLP
WILT = Waste Interface Leaching Test

= American Nuclear Society 16.1. ANS-16.1

= Total Organic Carbon

b Test performed at 6, 24, and 60 months. Five samples will be analyzed at the end of each period.

⁼ As needed until adequate consistency is achieved.

d Those leaching procedures each yield several separate leachate solutions. However, as described in the text, samples will be composited, resulting in two sets of analyses per leaching procedure.

Table 1-5. Expanded Sample Summary for Non-QC and QC Samples

Measurement	Туре	Raw Soil	Treated Soil (28 Days)	Reagent Mix	Long-Term Treated Soils	Total
TCLP-Metals	Non-QC Samples	9	9	1	15	34
	Leachate Duplicate	1	í	Ō	3	5
•	Leachate Blank	1	ī	Ö	3	5
	IRM ^a	1	1	Ö	3	5
	Matrix Spike	0	1	0	0	1
	•	•	•	•	•	
	•	•	D	•	•	•
	•	• .	•	•	•	
VOCs-Total	Non-QC Samples	9	9	1	0	19
	Sample Duplicate	3	0	0	0	3
	Sample Equipment Blank	1	1	0	0	2
	Method Blank	1	1	0	. 0	2
	Independent Check Standard	1	1	0	0	2
	Matrix Spike	1	1	0	0	2
	Matrix Spike Duplicate	1	. 1	0	0	2
•	•	. •			•	•
•	•	•	•	•	•	•
• .	•	•	• .	•	•	

 $a_{IRM} = Independent reference material$

. .

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SECTION 2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section demonstrates that the project organization is adequate to accomplish project goals, and that all responsibilities have been assigned.

Provide a table or chart illustrating project organization and lines of authority. For each organizational entity or subcontractor, identify by name all key personnel and give their geographic locations and phone numbers. The organizational chart should also include all subcontractors and their key points of contact. Separate organizational charts for subcontractors may also be needed. The organizational chart should identify QA Managers, including those of subcontractors, and should illustrate their relationship to other project personnel. The QA Managers should be organizationally independent of the project management so that the risk of conflict of interest is minimized.

Describe the responsibilities of all project participants, including QA Managers. Be sure to indicate responsibility for each type of analysis, physical measurement, and process measurement. This summary should designate responsibility for planning, coordination, sample collection, sample custody, analysis, review, and report preparation.

Any relevant certifications (e.g., OSHA 40-hour course for Hazardous Waste Site Operations) held by members of the project team should be noted. In some cases, these certifications augment relevant educational and work experience. In other cases, certification may be required by law.

Describe the frequency and mechanisms of communications among the contractor, the contractor's QA Manager, and the EPA Project Manager, as well as among contractor and subcontractors. Give the regular schedules for progress reports, site visits, and teleconferences, and describe which special occurrences would trigger additional communication.

Communication is a key element in achieving project goals. Because success largely depends on the prime contractor, effective monitoring of all project activities, including those of subcontractors, is vital. Procedures for communicating and monitoring all levels of activity must be clearly delineated.

Be sure to:

- Demonstrate that your QA Manager is independent of project organization.
- Provide names, locations, organizational affiliations, and telephone numbers for key personnel.

Figure 2-1 illustrates a typical project organizational chart with an independent QA Manager.

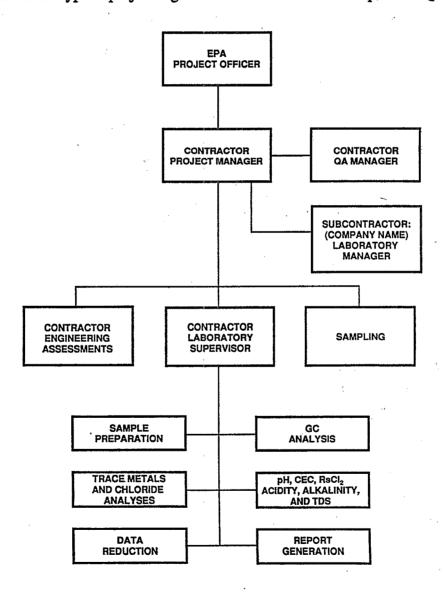


Figure 2-1. Project organization and names of responsible individuals

SECTION 3.0 OUALITY ASSURANCE OBJECTIVES

Quality assurance objectives are specifications that measurements must meet in order to achieve project objectives. For example, in ordering a pump for a chemical plant, factors such as capacity, pressure, and materials of construction must be specified. Similarly, precision, accuracy, detection limits, and completeness must be specified for physical/chemical measurements. Additional analytical requirements are described qualitatively in terms of representativeness and comparability. Quality assurance objectives are needed for all critical measurements (see Section 1.2) and for each type of sample matrix (soil, water, biota, etc.). Be sure to include quality assurance objectives for physical as well as chemical measurements.

Once QA objectives are set, the measurement systems are designed to meet them. The project manager, analytical chemists, and other principals must agree on the feasibility and appropriateness of these objectives.

3.1 DETERMINING QA OBJECTIVES

QA objectives must be defined in terms of project requirements, and not in terms of the capabilities of the intended test methods. Of course, the QA objectives must be achievable by available methods, and for this reason it is important that the laboratory review the QA objectives. When QA objectives exceed the capabilities of available methods, either the methods must be modified or the test plan must compensate for these deficiencies. Modifications may often be as simple as collecting a larger sample. However, if nonstandard or significantly modified test methods are required, the QA Project Plan must include laboratory validation data, to prove that the method is capable of achieving the desired performance.

The following are examples of how QA objectives can be determined:

Example 1: Biological treatment of pentachlorophenol (PCP).

Previous experience with this treatment process has indicated that the output concentration typically varies by a factor of two under stable operating conditions with uniform feed. To avoid contributing additional error, the precision of the analytical methods should be significantly less than this value, or approximately ± 30 percent or less.

<u>Example 2</u>: Determination of destruction removal efficiency (DRE) for incineration of a Dinoseb formulation containing 20 percent w/v Dinoseb.

The purpose of this test is to demonstrate a DRE of 99.99 percent or better. Dinoseb in stack gases will be collected on a Modified Method 5 sampling train. Extracts of all components of this train will be combined and reduced to 0.5-mL volume. The following parameters were used to estimate the required detection limit.

Feed rate of 20% w/v Dinoseb Formulation	(120 mL/min)
Dinoseb Feed Rate	24 g/min
Stack Gas Flow Rate	16 dry m³/min
Expected Concentration of Dinoseb in Stack Gas if DRE = 0 %	1.5 g/m^3
Maximum Permitted Concentration of Dinoseb, DRE = 99.99 %	$1.5 \times 10^{-4} \text{ g/m}^3$
Expected Volume of MM-5 Sample (3 Hours at 1.5 L/min)	$2.7 dry m^3$
Dinoseb Collected at DRE = 99.99 %	400 µg/sample

To allow a reasonable margin of error, the detection limit required for the Dinoseb determination will be 40 µg/MM5 train. Previous experience with the determination of Dinoseb on XAD resin has shown that this detection limit can be achieved routinely with the method attached as Appendix B. Previous experience has also shown that Dinoseb can be recovered routinely from a MM5 train with a recovery \geq 50 percent and a precision of \geq 30 percent relative percent difference (RPD), and deviations beyond these ranges indicate analytical problems. Results of a methods validation study performed in our laboratory are attached as Appendix C.

Category I projects, as part of the data quality objectives process, should develop quantitative QA objectives based upon project requirements. At a minimum, quantitative QA objectives should be specified for accuracy, precision, completeness, and method detection limit.

3.2 QUANTITATIVE QA OBJECTIVES: PRECISION, ACCURACY, METHOD DETECTION LIMIT, AND COMPLETENESS

QA objectives for precision, accuracy, method detection limit, and completeness should be presented in a QA objectives table similar to those shown in Tables 3-1 through 3-3. Be sure to include QA objectives for all matrix types and to indicate the units in which these QA objectives are given. Summary tables are very helpful to the laboratory which must meet these objectives.

Table 3-1. QA Objectives for Precision, Accuracy, and Method Detection Limits (MDL)

Critical Measurement	Matrix	Method	Reporting Units	MDL	$\begin{array}{ccc} & Accuracy \\ & (\% \ Recovery)^b & Completeness^c \end{array}$	Accuracy (% Recovery) ^b	Completeness ^c
Semivolatile organic compounds ^d Water (SVOC)	Water Soil	8270 8270	ug/L ug/kG	800 800	s 30 s 40	50-150 50-150	200
Volatile chlorinated organic compounds	Water	601	ug/L	(e)	<50	50-150	300
Metals As Ba Cd Cr Pb Flow rate	Soil/water Soil/water Soil/water Soil/water Water/air	7060 (GFAA) 7080 (FAA) 7131 (GFAA) 7190 (FAA) 7421 (GFAA) Rotameter	uglt uglt uglt uglt uglt .c	$^{56}_{16}$	£35 £35 £35 £35 	80-120 80-120 80-120 80-120 80-120 2 ^h	200 200 200 200 100

a Given as Relative Percent Difference of laboratory duplicates, unless otherwise indicated.

This table is meant to illustrate typical format.
The QA objectives listed are NOT necessarily appropriate for any particular project.

b As percent recovery of matrix spike, unless otherwise indicated.

c Based on the number of measurements needed to achieve 4 level of confidence in decision making. (Extra samples will be collected to permit reanalysis, as required.)

All of the Principal Organic Hazardous Compounds (POHCs) of interest to this project will be required to meet these objectives. Other compounds determined by this method need not satisfy these objectives.

MDLs are given in Table ____. (See example Table 3-3 in this document.)

MDL for water samples. MDL for soils is 100 times greater. Reporting units for soils will be mg/kg.

Maximum permitted deviation against volumetric standard.

h Absolute deviation against ASTM thermometer.

Table 3-2. QA Objectives for Precision, Accuracy, and Detection Limits - Alternate Form

Data Quality Parameter	Method of Determination	Frequency	Required Objective ^{a,(1)}
	Semivolatile Orga	nic Compounds	a .
Precision		,	4
- Water	Field duplicate	1/test condition	RPD <50
- TCLP leachates	Duplicate leaching of laboratory - split sample	1/test condition	RPD <60
Accuracy			
- Water & TCLP leachates	Laboratory matrix	1 water/test	Recovery = 50-150%
,	spike	1 leachate/test condition	Recovery = 50-150%
- Water & TCLP leachates	Surrogate	All samples	(b)
- Water	NIST standard reference materials	1/project	Recovery = 50-150%
- MM5 train	Spike of XAD resin	3/project	Recovery = 50-150%
Detection limits			
- Water	7 analyses of spiked clean water	1 before project	MDL \leq 10 μ g/L, neutrals MDL \leq 20 μ g/L, phenols MDL \leq 100 μ g/L, bases
- MM5 train	3 analyses of spiked XAD resin ⁽²⁾	1 before project	MDL ≤ 20 μ g

a RPD = relative percent difference.MDL = method detection limit.

Note (1) Objectives must be met for all critical measurements. (See Section 1.2 of this document.)

Note (2) MDLs must be calculated according to Equation (8), Section 12.0 of this document, which takes into account the number of low-level samples analyzed.

b As specified in Method 8270.

Table 3-3. Required Detection Limits for Volatile Chlorinated Organic Compounds^a

Compound	Regulatory Threshold (µg/L)	Required MDL (µg/L)
1,1,1-Trichloroethane	5	0,5
1,1-Dichloroethane	5	0.5
1,1-Dichloroethene	5	0.5
Vinyl chloride	2	0.2
1,2-Dichloroethane	1	0.1
Perchloroethylene	5	0.5

Method detection limits for these compounds are critical and will be determined experimentally by the laboratory before sample collection is started. MDLs must be well below the Regulatory Threshold in order to demonstrate that treated waters meet discharge limits.

This table is meant to illustrate typical format.

The tests listed are NOT necessarily those that would be employed for a process of this type.

Because precision and accuracy can be measured in various ways, explain the method to be used. If precision, for instance, is to be determined by duplicates, explain whether sample splitting will occur in the laboratory, during sampling, or at some other stage. Then summarize all such information in either text or tabular format.

The following statements are examples of descriptions for precision, accuracy, method detection limits, and completeness:

- Precision objectives for all the listed methods except pH are presented as relative percent difference (RPD) of field duplicates. Precision objectives for pH are listed in pH units and expressed as limits for field duplicates.
- Precision objectives for unconfined compressive strength are given as relative standard deviation for triplicate sets.
- Accuracy objectives for organic compounds and metals are given as percent recovery range of laboratory matrix spikes. Accuracy objectives for temperature measurements are absolute deviations in °C.

- Detection limits are defined as the method detection limit (MDL) multiplied by the dilution factor required to analyze the sample. MDLs will be determined by replicate extraction and analysis of seven identical spiked samples of XAD resin.
- Completeness is defined as the number of measurements judged valid compared to the number of measurements needed to achieve a specified level of confidence in decision making. It has been estimated in Section _._ that 10 valid measurements should suffice to demonstrate that the arsenic concentration in the discharge is less than 50 µg/L at a confidence level of 90 percent. To allow for a margin of error in the estimated number of samples, 15 valid measurements are planned, resulting in a completeness objective of 150 percent. An additional 6 spare samples will be collected but not analyzed, unless needed to achieve the desired confidence level.

When analyzing for a large number of organic compounds by GC or GC/MS, it is usually unnecessary to list QA objectives for each compound separately. Instead, list QA objectives according to compound type, as was done for the semivolatile detection limits in Table 3-2. In other cases, for example, where detection limits are derived from applicable regulations, list detection limits for individual compounds, as in Table 3-3.

Finally, the author of the QA Project Plan must explain how the QA objectives are to be interpreted in a statistical sense. QA objectives are often interpreted in a sense that all data must fall within these goals; for such projects any data that fail to satisfy the QA objectives are rejected and corrective action is undertaken. However, other interpretations are possible. For example, the project requirements may be satisfied if the average recovery is within the objectives; that is, excursions beyond the objectives might be permitted, but the average recovery would have to satisfy the goals described in this table. Whatever the case, it is important to describe in this section how tabulated QA objectives will be interpreted.

3.3 QUALITATIVE QA OBJECTIVES: COMPARABILITY AND REPRESENTATIVENESS

Comparability is the degree to which one data set can be compared to another. For instance, to evaluate an environmental cleanup process, analyses of the feed and discharge streams must be comparable. Similarly, to perform a nationwide environmental survey, methods used at different locations must be comparable. Comparability is achieved by the use of consistent methods and by traceability of standards to a reliable source.

Representativeness is the degree to which a sample or group of samples is indicative of the population being studied. An environmental sample is representative for a parameter of interest when the average value obtained from a group of such samples tends towards the true value of that parameter in the actual environment, as the number of representative samples is increased. Representativeness is normally achieved by collecting a sufficiently large number of unbiased samples.

The QA Project Plan should demonstrate that adequate comparability and representativeness will be achieved.

3.4 OTHER QA OBJECTIVES

Some projects may require additional QA objectives, such as mass balances. Requirements for all additional QA measurements should be stated in this section.

Example: Soil Washing for Removal of PCP.

A mass balance will be calculated according to the following expression:

Mass Balance = Mass of PCP in output streams/mass of PCP in input streams

For this test to be successful, mass balance should be between 50 and 150 percent. The specific equations for calculating the influent and effluent masses in terms of the measured quantities are given in Section __ of this QA Project Plan.

3.5 WHAT IF QA OBJECTIVES ARE NOT MET?

This section must include a discussion of the impact of not meeting one or more QA objectives. Will the project be a complete loss? Will some, but not all, of the project goals still be realized? Will the statistical confidence level be reduced? Are there legal or regulatory ramifications? Answers to such questions help provide a critical perspective on the QA Program.

•

SECTION 4.0 SITE SELECTION AND SAMPLING PROCEDURES

This section must describe a plan for site selection and sampling which is responsive to those project objectives stated in Section 1.1.2. A detailed discussion of the sampling plan development process is presented in Volume 2 of SW-846, Test Methods for Evaluating Solid Waste (3rd Ed.). Since SW-846 will likely undergo revisions, be sure to refer to the latest revision or edition.

This section explains the overall sampling strategy, the specific sampling procedures that will be employed, sample custody, record keeping, and shipping requirements.

4.1 SAMPLING SITE SELECTION

For most Category I projects, the sampling sites will have been identified already, and preliminary data will be available. However, for certain projects such as those involving surveys at a number of geographically separate sites, it may be impossible to explicitly delineate sampling locations in the QA Project Plan. For these projects, explain how sampling sites will be selected during the project. The following information should be provided:

- A qualitative statement of the sample population to be represented by the samples;
- A description of the statistical method or scientific rationale to be used in selecting sample sites;
- Descriptions of the type of sampling strategy (e.g., simple, stratified, systematic random sampling);
- A description of the sample types (air, water, soil, biota);
- A qualitative statement regarding potential sources of sample contamination;
- Guidelines for determining sampling frequency and number for each sample type;
- A discussion of the extent to which site selection will affect the validity of the resulting data and project objectives; and

• In some cases, especially those involving enforcement procedures, a discussion of site access should be included. It may be necessary for contractors to obtain an EPA letter of authorization to gain access to the site.

4.2 SAMPLING SITE DESCRIPTION

This section includes charts, maps, sampling grids, or tables specifying the exact sampling sites. Note any site modifications or additions that will be needed prior to sampling, such as the extension of duct work or the addition of special valves. Also describe all locations and access points for critical process measurements such as flow rates, pressures, or temperatures. Discuss any site-specific factors that may affect sampling procedures.

For each analyte and each sampling point, list the frequency of sample collection and the total number of samples collected. This summary can best be prepared in tabular form, as shown in Table 4-1. Note that the numbers given in this table may differ from the number of analyses, since some samples may be analyzed in replicate, while others may be analyzed only on a contingency basis. Explain the statistical basis for the sampling scheme, as necessary.

4.3 SAMPLING PROCEDURES

This section describes the specific procedures that will be used for collecting and preserving samples. It is vital that each of the following points be completely and clearly addressed to ensure that activities undertaken are adequate to meet project objectives.

- Discuss each sampling procedure that will be employed. For EPA-approved procedures, a reference is sufficient. Other sampling procedures should be summarized in the text, and additional details should be provided in an Appendix. (Copies of ASTM sampling procedures are frequently appended.)
- Prepare a list of analytes, sample volumes to be collected, and the amount of sample that is required for each analysis (see Table 4-2). Note whether the required amount is intended for matrix spike/matrix spike duplicate determinations or for a single determination only. Be sure to have your laboratory manager review this table to ensure that the sample volume or mass is sufficient for all intended analyses.
- Describe any compositing or sample splitting procedures that will be employed in the field or laboratory. Note whether the host facility requires additional split samples for its own analyses.

Table 4-1. Summary of Number of Samples Required for Hypothetical Incineration Test

Description/Use	Slurry Feed (F1)	Scrubber Sump (S1)	Scrubber Blowdown (S2)	Ash (S3)	Stack (S4)	Total
SVOCsa						
Non-QC Samples + MS + M	ISD ^b 10	10	10	5	0	35
Sample Duplicates	3	0	0	ő	Ö	3
Sample Blanks	1	1	0	Ö	Ö	2
Spare Samples ^C	5	5	5	5	Ö	20
Modified Method 5						
Non-QC Samples	0	0	0	0	4	4
Sample Blanks (see text)	0	0	0	0	i	i
VOCs ^a						
Non-QC Samples	20	20	20	10	0	70
Sample Blanks	1	. 1	1	1	0 -	4
Trip Blanks ^d	1	1	1	1	Ô	4
Spare Samples ^C	20	20	20	10	ŏ	70
•	•		•			,,
•	•	•		•	•	
•	•	•	•	•	•	

a As designated in Section ____.

This table is meant to illustrate typical format.

The tests listed are NOT necessarily those that would be employed for a process of this type.

b Each sample will provide enough material for an original sample plus a matrix spike and a matrix spike duplicate.

Not analyzed unless first analysis fails.

Not analyzed unless sample blank is contaminated.

Table 4-2. Summary of Laboratory Analyses and Sample Quantity Requirements

Stream	Sampling Method	Analysis Parameter	Container Size Rec	Sample Quantity quired for Analysis (1)
Test soil	Shelby tubes	Semivolatiles Volatiles Metals scan Dioxins/furans EP toxicity TCLP Higher heating value Chlorine Moisture	250 mL 250 mL 250 mL 250 mL 250 mL 250 mL 250 mL 250 mL 250 mL	50 g 50 g 100 g 50 g 250 g 500 g 250 g 250 g 100 g
Treated soil	ASTM-C172/ composite ⁽²⁾	Semivolatiles Volatiles Metals scan Dioxins/furans TCLP	250 mL 250 mL 250 mL 250 mL 250 mL	50 g 50 g 100 g 50 g 500 g
Scrubber makeup scrubber liquor	Grab	Semivolatiles Volatiles Metals scan Dioxins/furans	1 L 40 mL VOA v 1 L 1 L	1 L 40 mL VOA vial 1 L 1 L
Scrubber solids	Thief	Semivolatiles Volatiles Metals scan Dioxins/furans	250 mL 250 mL 250 mL 250 mL	50 g 50 g 100 g 50 g
Stack	Method 5 Midget M-5(2) Metals train(2) Modified Method 5 NIOSH-1003(2)	Particulate HCI Metals Semivolatiles Dioxins/furans Volatiles		900 L 900 L 900 L 3000 L 3000 L 20 L

Note (1) Indicate whether the listed quantity suffices for replicates, spikes, and other QC purposes, or if it is sufficient for only a single analysis. In the latter case, indicate how extra material will be provided for QC purposes.

This table is meant to illustrate typical format.

The tests listed are NOT necessarily those that would be employed for a process of this type.

Note (2) Copies of these methods would have to be appended because they are not readily available.

- Describe any sampling equipment that will be used, and how this equipment will be calibrated.
- Explain how sample containers will be cleaned to prevent sample contamination, and how new sample containers will be checked for contaminants.
- Describe the numbering sequence which ensures that each sample will be assigned a unique number.
- Describe the containers used for sample collection, transport, and storage for each sample type. Include sample preservation methods, noting specific reagents, equipment, supplies, etc., required for sample preservation, and the specific time requirements for shipping samples to the laboratory. Note refrigeration conditions and holding times that will be employed. (See, for example, Table 4-3.)
- Describe the procedures used to record sample history, sampling conditions, and any other pertinent information; include examples of forms that will be employed. In non-erasable waterproof ink, record all aspects of sample collection—field data and observations, problems encountered, and action taken to resolve the problems—in a bound notebook with consecutively numbered pages. Pages should never be removed from this logbook.
- Include an example of the sample label to be used.

How are you doing so far? At the completion of this section, you should be able to easily calculate the required number of each type of sample container.

Table 4-3. Required Containers, Preservation Techniques, and Holding Times

Measurement	Type ^a	Preservation ^b	Maximum Holding Times ^c
Extractable organics	G Teflon-lined septum	Cool to 4°C, protect from light	7 days until extraction, 40 days after extraction
Pesticides, PCBs	G Teflon-lined septum	Cool to 4°C,~ pH 5-9	7 days until extraction, 40 days after extraction
Metals (except mercury and chromium VI)	P, G	HNO ₃ to pH <2	6 months
Mercury	P, G	HNO ₃ to pH <2	28 days
Chromium VI	P, G	Cool, 4°C	24 hours
pН	P, G	None required	Analyze water immediately (on site); none specified for soil
Residue	P, G	Cool, 4°C	7 days
Organic carbon, total	P, G	Cool, 4°C, HCl or H ₂ SO ₄ to pH <2	28 days
Sulfide	P, G	Cool to 4°C; add zinc acetate plus NaOH to pH >9	7 days

a Polyethylene (P) or glass (G).

This table is meant to illustrate typical format.

Preservation requirements, although typical, may not be appropriate for every project.

b Sample will be preserved immediately upon sample collection.

Samples will be analyzed as soon as possible after collection. The times listed are the maximum times that samples will be held before analysis and still be considered valid. All data obtained beyond the maximum holding times will be flagged.

SECTION 5.0 SAMPLE CUSTODY

Occasionally samples are spilled, contaminated, accidentally evaporated to dryness, or otherwise compromised before or during sampling and analysis. Sample custody allows detection of such problems should they occur and minimizes such occurrences by assigning responsibility for all stages of sample handling. Sample custody is maintained when the samples are in a secure area, or in the view of, or under the control of, a particular individual. Records of everyone handling samples are maintained so that a sample history can be reconstructed later, should the need arise.

This section describes how sample custody will be maintained and recorded.

- Give the names of all sample custodians in the field and in each of the laboratories.
- Give examples of forms that will be used to maintain sample custody in the field, during shipping, and in the laboratory.
- Seal shipping containers with chain-of-custody seals. Give an example of the seal that will be used.
- Describe procedures that will be used to maintain chain of custody during transfer from the field to the laboratory, within the laboratory, and among contractors and subcontractors.
- Provide for the archiving of all shipping documents and other paperwork received at the laboratory with the samples.
- Discuss procedures which will ensure sample security. For example, samples should always be stored in locked refrigerators.
- Describe procedures for within-laboratory chain of custody. Such procedures should allow for unambiguous tracing of the samples through the laboratory. This "paper trail" should include a record of the individuals responsible for custody of samples, extracts, digests, etc., at all times in the laboratory. Finally, disposal or consumption of samples should be documented.

Figures 5-1 and 5-2 give examples of satisfactory chain-of-custody records, official sample seals, and sample labels. See also Chapter 9 of SW-846 (3rd Ed.) for additional discussion of shipping and custody procedures. Since SW-846 will likely undergo revisions, be sure to refer to the latest revision or edition.

Do you want to save time and money?

Use Standard Operating Procedures (SOPs)! Certain information required in this section -- such as sample custody procedures, forms, labels, and sampling methods -- is not project-specific, but applies to a wide range of sampling efforts. Typically, a sampling team or a laboratory will use the same chain-of-custody procedures from one project to the next. Such procedures may be written as SOPs, which can then be appended to subsequent QA Project Plans. Be sure to use document control format in the SOPs to alert the reviewer to any recent changes in the procedure.

Chain of Custody Record

W.O. No.		Project Name:	 						K		
Samplers: (Signature)	(Signature)					Sample Type	Type of Containers				Remarks
Sta. No.	Date	Time	S	Station Description	ription		<u> </u>				
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Figure 5-1. Example of a chain-of-custody record.

Sample Label

Samula Description:	(Name of Sampling Organization)	
Sample Description.		
Date:	Location:	
Media:	Station:	
Sample Type:	Preservative:	
		.
Sampled by:		
Sample ID No.:		
		ka:
Lab No.		Remarks:
	•	či. I I I

Custody Seal

Signature	AND THE STATES	CUSTODY SEAL
Date	å ==== ≥ i ====== ÷	Date
CUSTODY SEAL	STAR GELINI. THE PROTECTION	Signature

Figure 5-2. Examples of a typical sample label and a custody seal.

SECTION 6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section covers calibration procedures for each analytical or measurement system used to obtain data for critical measurements. Each description should include the specific calibration procedure to be used and the frequency of calibration verification.

- In the case of standard EPA-approved methods that include calibration procedures, a reference to those methods suffices. Simply list the required frequency and acceptance criteria in a summary table, such as that shown in Table 9-1. For nonstandard methods that are appended to the QA Project Plan, reference can be made to the SOP. Describe all other calibration procedures in detail.
- A list of calibration standards, including source, traceability, and verification of purity, must be included.
- For process measurements (e.g., flow, mass, etc.) and for chemical or physical analyses, specify the planned frequency of initial and continuing calibration checks and the acceptance criteria for all calibration measurements. Table 6-1 illustrates a typical summary for process measurements. For routine, scheduled calibrations, include the calibration frequency and acceptance criteria in Section 9.0, along with a summary of QC requirements.

For physical measurements such as temperature and pressure, calibration statements can be quite brief.

<u>Example</u>: All thermocouples intended for use in the range 50°C to 350°C will be calibrated versus an NIST-traceable thermometer at a minimum of two temperatures, by placing both in an oven simultaneously and noting any difference between readings. The thermocouple readout must be within 2°C of the corrected mercury thermometer or the thermocouple device will be replaced or corrected.

Don't forget:

- Include procedures for both physical and chemical measurement processes to be carried out in either the field or the laboratory.
- Provide whatever information is needed to complement the referenced procedure, but do not repeat material from the procedure itself. Consult your analytical chemist or other measurement specialist for details on the laboratory methods you will use to achieve your project objectives.

Table 6-1. Typical Summary Table of Calibration Requirements for Physical Process Measurements

Parameter	Measurement Classification	Device	Calibration Procedure	Frequency	Acceptance ^a Criteria
Flow rate of dopant feed	Critical	Mass flow meter	Compare to cali- brated dry test meter	Before/after field test; weekly	5%
Secondary volume standard	Critical	Dry test meter	NIST-traceable spirometer	Before/after field test	2%
Secondary time standard	Critical	Stopwatch	NIST time base	Before field test	0.05 sec/min
Temperature (50-300°C)	Noncritical	Thermometer	Comparison to certified thermometer @ 2 temperatures	Before project initiation	2°C
Temperature (300-800°C)	Critical	Thermocouple	Comparison to NIST-calibrated thermocouple @ 2 temperatures	Before field test	5°C
Flow rate in stack	Critical	Pitot tube and manometer	Measure pitot orifice with NIST- traceable micro- meter; compare manometer mark- ings to NIST- calibrated meter stick	Before field test	1%

^a Maximum allowable deviation from standard.

This table is meant to illustrate typical format.

The tests listed are NOT necessarily those that would be employed for a process of this type.

SECTION 7.0 ANALYTICAL PROCEDURES AND CALIBRATION

This section of the QA Project Plan must describe all of the field and laboratory procedures used for both chemical and physical measurements. Sample preparation methods and cleanup procedures (such as extraction, digestion, column cleaning) should also be included.

All methods must be appropriate for their intended use and be described in sufficient detail. This section, when coupled with QC procedures described in Section 9.0, should provide enough detail to permit the analytical chemists or other measurement specialists to carry out their procedures unambiguously.

Requirements of this section can often be satisfied by referencing appropriate standard methods. Most RREL projects rely heavily on EPA-approved methods that have been validated for environmental samples. Standardized procedures from other organizations, such as the ASTM and the American Public Health Association, are also commonly employed when approved validated EPA methods are unavailable. When standard methods are unavailable, nonstandard methods must be used and described in detail. It is essential to consult the analytical laboratory and other measurement specialists for guidance during preparation of this section because they know the intricacies and limitations of the methods.

Begin this section with a summary table of all measurements to be made. For projects involving many kinds of measurements, consider providing separate tables for each type of test. Specify the parameter to be measured, the sample type, the method number (when available), the title, the method type, and a reference. (See Table 7-1 as an example.) Note the revision number of the method or the edition number of a publication, since nominally identical methods may be modified by these updates. Provide additional project-specific information in the text of this section.

Since Category I projects are often related to regulatory or enforcement issues, the Code of Federal Regulations (CFR) should be consulted to ascertain whether certain methods have been promulgated. Where no method has been specified within a regulatory act, use a standard, validated method when possible.

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Parameter	Sample Type ^a	Method Number	Method Title	Method Type	Source (Reference) ^b
On-Site Tests					
Slump of Portland Cement Concrete	v	ASTM-C143	Slump of Portland Cement Concrete	Vertical distance	ASTM (1)
Homogeneity of Mixing	v	ASTM-C136/C142	Sieve Analysis of Fine and Coarse Aggregates/Clay Lumps and Friable Particles in Aggregates	Sieve/gravimetric	ASTM (1)
Leaching Tests					
TCLP-ZHE	U/RM/T/LT	TCLP	Toxicity Characteristic Leaching Procedure	Leaching	40 CFR Part 268 Appendix I
MWEP '	LT.	MWEP	Monofill Waste Extraction Procedure	Leaching	USEPA (2)
ANS 16.1	⊱ —	ANS 16.1	American Nuclear Society 16.1 Test	Leaching	American Nuclear Society (3)
				:	(Continued)

Table 7-1. (Continued)

Parameter	Sample Type ^a	Method Number	Method Title	Method Type	Reference ^b
Polychlorinated Biphenyls	ולרער	EPA Method 3520	Continuous Liquid-Liquid Extraction	Extraction	SW-846 (4)
	U/RM/T	EPA Method 3540/3620	Soxhlet Extraction/Florisil Column Cleanup	Extraction/ Cleanup	SW-846 (4)
	3520/3540 Extracts	EPA Method 8080	Organochlorine Pesticides and PCBs	G¢/E¢D	SW-846 (4)
	3520/3540 Extracts	EPA Method 680 (Backup)	Determination of Pesticide PCBs in Water and Soil/ Sediment by GC/MS	GC/MS	BPA (5)
Engineering/Geotechnical Tests	ical Tests				
Particle Size	D	ASTM D422	Particle Size Analysis	Sieve/hydrometer	ASTM (1)
Bulk Density	n.	ASA-13-2	Bulk Density-Excavation Method	Gravimetric/ Volumetric	ASA (6)

Treated waste, while still a slurry Untreated waste

The methods listed are NOT necessanily appropriate for any particular project. This table is meant to illustrate typical format.

Reagent mix

Treated water

Treated water, long-term monitoring

Leachate Leachate, long-term monitoring.

b Complete citations should be provided either here or in Section 15.0 of the QA Project Plan.

7.1 EPA-APPROVED OR OTHER VALIDATED STANDARD METHODS

EPA-approved or similar validated methods can be incorporated by reference, thereby greatly minimizing the effort required to complete this section. Once a method is cited, do not repeat information that is already found in the method. Some additional information is almost always required, however, to assure that project-specific requirements will be met. The following considerations apply:

- Some EPA-promulgated methods contain only general procedure descriptions and lack specific QC requirements or applicable validation data. For example, EPA Method 18 provides general requirements for GC analysis of stack gases, but contains no QC requirements. For such methods, validation data pertinent to the specific project must be appended. Alternately, a preliminary method validation can be specified as a subtask of the project; in that case, however, specific procedures and acceptance criteria for method validation must also be included as part of the QA Project Plan.
- Other EPA-approved standard methods, such as those found in Standard Methods for the Examination of Water and Wastewater, give operating procedures but omit most QC and calibration requirements. This missing information must be provided either in this section or in Section 9.0 of the QA Project Plan. As a minimum, be sure to specify the frequency, acceptance criteria, and corrective action plans for all QC procedures and calibrations.
- It is not necessary to include copies of methods or sections of methods from SW-846, the Code of Federal Regulations, Standard Methods for the Examination of Water and Wastewater, or Methods for Chemical Analysis of Water and Waste because these sources are readily available. However, do append ASTM and NIOSH procedures because they may not be so readily available to other project principals or reviewers.
- EPA-approved or similarly validated methods that are significantly modified are considered to be unvalidated methods and are treated as described in Section 7.2.
- Certain EPA methods such as those found in SW-846 specify most operating details, including quality control and calibration requirements. Such procedures, however, frequently allow the user to specify certain other options

to satisfy project objectives. For example, for multianalyte methods such as GC/MS, the user will typically specify the target compound list that is required for the project. Matrix spike compounds are chosen from the compounds of interest to the particular project, and are not necessarily those recommended in the method. Also list the project-specific target compounds to be used as calibration check compounds and matrix spike compounds. Specify the acceptance criteria for matrix spike compounds. In certain cases, isotopically labeled forms of the project analytes may be included as surrogates.

The following is an example of how one might specify various options allowed by Method 8080, a validated method from SW-846 which contains QC and calibration requirements:

Example: Method 8080.

This method will be employed to determine the 19 pesticides listed in Table ____ of this QA Project Plan. Although PCBs can also be determined by this method, the GC will not be calibrated for these products unless they are observed. The matrix spike compounds will be the six critical pesticides listed previously in this QA Project Plan. The surrogates will be tetrachlorometaxylene (not dibutyl-chlorendate). Detection will be by ECD. Quantitation will be by external calibration. Acceptance criteria for surrogate recovery will not be determined by control charts, but must be within a 50-150 percent range. Acceptance criteria for matrix spike/matrix spike duplicates are those stated in Table ___ of this QA Project Plan. Extraction and cleanup procedures are described below...

7.2 NONSTANDARD OR MODIFIED METHODS

Any nonstandard procedure must be described in detail in the form of a Standard Operating Procedure (SOP) that is appended to the QA Project Plan. Validation data applicable to the expected samples must also be included. Validation data must demonstrate that the analytes of interest can be determined without interferences in the expected matrices, and that precision, accuracy, and detection limits will be adequate for the intended use of the data. The method is validated only for samples expected from the specific project, and not for general environmental usage. Once the SOP is written and the validation data are accepted, the method is validated only for use on that specific project and on the specific sample matrix associated with that project. This validation process for a project-specific analysis does not constitute EPA approval for other projects or matrix types.

For Category I projects, all nonstandard methods must be validated prior to approval of the QA Project Plan.

The following are minimum guidelines for validating a nonstandard method for a Category I project:

- Determine method detection limits by preparing and analyzing replicates of artificially prepared samples in a matrix similar to the samples of interest. Detection limits should be calculated according to the procedure described in Section 12.1.4 of this document.
- Accuracy should be demonstrated by analyzing spiked samples in the matrix of
 interest that contains appreciable levels of known or suspected interferences.
 Accuracy may also be demonstrated by analyzing appropriate Standard
 Reference Materials (SRMs), if available.
- Demonstrate method precision by replicate analysis on realistic samples.

However, each project must be considered on a case-by-case basis, and more stringent validation techniques may be required. For example, for some projects it might be necessary to perform a comparative analysis with a different analytical technique.

SECTION 8.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section describes how data will be reduced, validated and reported. Deliverables that will be required from the analytical laboratory must also be specified.

Begin this section with an overall schematic of data flow, such as shown in Figure 8-1. This flow chart indicates the entire process of data handling, collection, transfer, storage, recovery, and review for both field and laboratory operations.

8.1 DATA REDUCTION

- · Name the individuals responsible for data reduction.
- Summarize the data reduction procedures that are specific to this project. Data reduction procedures that are part of standard methods or SOPs cited in Section 7.0 should not be repeated here other than to note any deviations.
- Summarize the planned statistical approach including formulas, units, and definition of terms. Do not simply reference a "standard text."
- Explain how results from blanks will be treated in calculations.

8.2 DATA VALIDATION

- Name the individuals responsible for data validation at each stage of data reduction.
- Describe the procedures that will be used for determining outliers. Describe the guidelines that will be employed for flagging or validating data. (This requirement is normally satisfied by Section 9.0 with respect to routine quality control.) In Section 8.0, discuss criteria that are not covered in Section 9.0.

8.3 DATA REPORTING

- Name the individuals responsible for the various types of reports.
- Indicate the units for each measurement and each matrix, and whether data will be reported on a wet, dry, or some other reduced basis. If this requirement has been covered in other sections (e.g., Table 3-1), do not repeat it here.

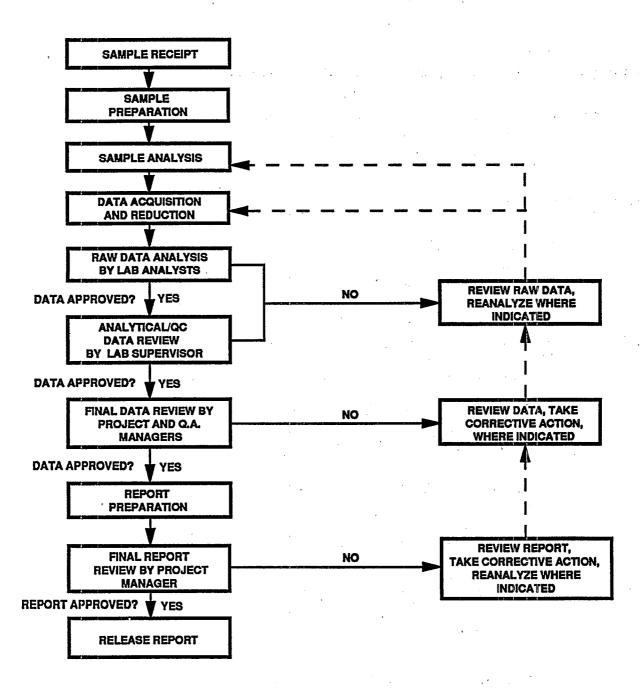


Figure 8-1. Example of a data reduction, validation, and reporting scheme.

- Indicate data storage requirements that will be expected of the laboratory once the project is complete. Will the laboratory need to maintain a complete set of raw data for six months? One year? Five years? Also indicate how long the actual samples will be stored, in case reanalysis is required.
- List the deliverables that will be expected from the laboratory and from the field operations. Will the data package include all raw data sufficient to recalculate any result, if need be? What type of QC data will be reported? Reporting requirements may vary, depending on the intended use of the data.
- Summarize the data that will be included in the final report. What QC data
 will be included? Will analytical and other measurement data be partially
 reduced before they are reported, or will all individual measurements be
 reported?

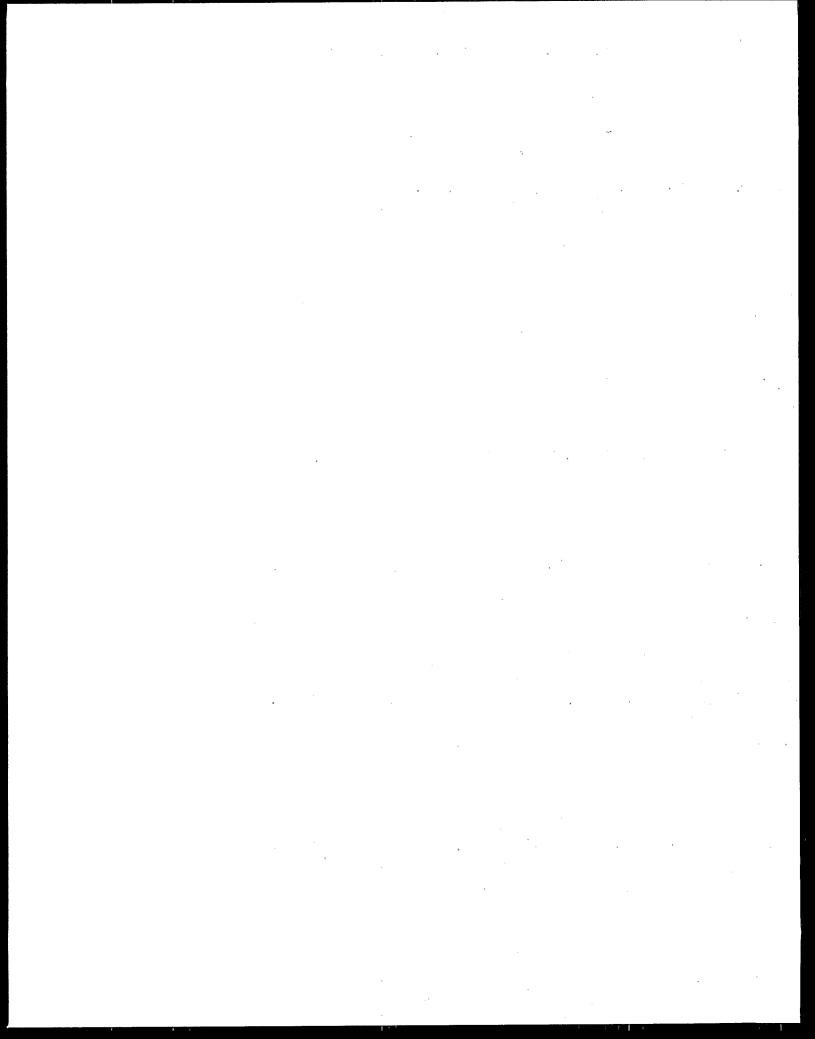
Example: The final report will contain the following analytical data:

All analytical results from all primary samples. Data judged to be outliers will be included, along with a justification for excluding this information from further interpretation.

All individual results from standard reference materials, independent check samples, replicates, and matrix spikes, including initial and final concentrations.

Data from the continuous emission monitors will be reported as 15-minute averages and, when appropriate, will be summarized graphically.

For Category I projects, this section should contain a statement that the final report will include a QA section that documents QA/QC activities and results. These results must be readily correlated to the primary data and must clearly indicate the limitations of the data and the range of validity of the conclusions. The final report should also include a summary of the original QA objectives, and a statement regarding whether these objectives were met. If QA objectives were not met, include an explanation of the impact of not meeting the project's QA objectives.



SECTION 9.0 INTERNAL QUALITY CONTROL CHECKS

This section describes all internal quality control (QC) checks that will be used throughout the project, including field and laboratory activities of all organizations involved. The QC procedures that are specified should follow from the QA objectives stated in Section 3.0. Thus, Section 3.0 specifies the analytical requirements, while Section 9.0 describes how these specifications will be met.

9.1 TYPES OF QC CHECKS

Examples of QC checks that should be considered include the following:

- Samples
 - Collocated, split, replicate
- Spikes
 - Matrix spikes and matrix spike duplicates
 - Spiked blanks
 - Surrogates and internal standards
- Blanks
 - Sampling, field, trip, method, reagent, instrument
 - Zero and span gases
- Others
 - Standard reference materials (complex natural materials, pure solutions)
 - Mass tuning for mass analysis
 - Confirmation on second column for gas chromatographic analyses
 - Control charts
 - Independent check standard
 - Determinations of detection limits
 - Calibration standards
 - Proficiency testing of analysts
 - Any additional checks required by the special needs of your project

Include QC checks for process measurements as well.

Be sure to:

- Identify the stage at which replication and spiking occur. Avoid using terms such as "sample replicate" without explaining how such replication will be performed.
- · Explain exactly how blanks will be prepared.

9.2 ITEMS TO INCLUDE

Most information for this section can be summarized in a table, as shown in Table 9-1. This table should designate the types of QC procedures, required frequencies, associated acceptance criteria, and corrective action that will occur if the acceptance criteria are not met. When QC procedures are referenced to a standard method that describes an exact procedure, additional discussion is not normally needed. However, standard methods lacking exact QC procedures or nonstandard methods require a detailed explanation, either in this section of the QA Project Plan or in an appended Standard Operating Procedure.

The tabular format shown in Table 9-1 is also convenient for summarizing routine and ongoing calibration requirements. If routine calibration is summarized in this section, a reference to that effect should be included in Section 7.0.

The accompanying text must assure that there are no ambiguities. Particularly troublesome terms are "duplicate" or "replicate." The text should explain precisely how and when replicates are taken. Do replicate samples, for instance, refer to samples collected simultaneously or sequentially in the field; to samples collected at the same sample point but at different times; to samples that are split upon receipt in the laboratory? The term "QC check sample" must also be carefully defined. Indicate at which point matrix spiking occurs.

Exact procedures for preparing the numerous kinds of blanks must be described fully in the text. Never assume that a term such as "field blank" will mean the same to the sampling team or to a reviewer that it does to you.

Table 9-1. Scheduled QC and Calibration

	Procedure	Frequency of QC Procedure	Acceptance Criteria	Corrective Action
PA	SVOCs (EPA 8270)			
	DFTPP tune	12 hours	Table 3/8270	Re-run before sample analysis
	Inertness for DDT	12 hours	DDE, DDD <20% of DDT	Repair before sample analysis
	5-pt ICAL	Initially and as needed	RSD of CCC compounds <30% RRTs within 0.06 RRF for all SPCCs ≥ .05	Re-calibrate
	Continuing calibration	12 hours	RF for all SPCCs > .05 RF for all CCCs within 30% of ICAL RT of IS within 30 sec of last CCC Area of IS within factor of two of last CCC	Correct before analysis, otherwise repeat ICAL
	Matrix spike	Each batch ≤20	Table 6	Run independent (C reference standard If reference standard okay, accept data, otherwise reject data.
	Replicate spike	Each batch <20	None for duplicates	None
	Surrogate recovery	Each sample	From control charts Default = Table 8	Repeat analysis
	Method blank	Before any samples; each blank	No significant interference to target analytes.	Find and remove interference
	Proficiency test	Each new analyst	Table 6	Re-do before samples are analyzed

Either in this section or in Section 7.0, be sure to specify what compounds or elements will be employed as matrix spikes and surrogates. Some standard methods recommend surrogate and matrix spike compounds, which may be incorporated by reference when appropriate. More typically for RREL projects, some of the matrix spike compounds must be selected on a project-specific basis.

In some cases, it may also be necessary to provide additional discussion of potential problems that might be expected with certain QC procedures, along with the proposed solutions. For example, spiking samples in the field is frequently less reliable and more difficult than spiking in the laboratory, due to contamination and less controlled conditions. If field spiking is required, then a discussion of procedures that minimize such problems is also required.

Because standard methods often include extensive QC requirements, why must such QC procedures be summarized in this section? Why not simply state that QC will be performed as required in the method? In some cases, EPA standard methods are sufficiently complete for this approach, but frequently they are not. First, many EPA-approved methods do not include specific QC procedures. Second, even the more complete EPA methods allow options, such as the choice of matrix spike compounds, or the use of either control charts or fixed acceptance limits. Third, the analytical and measurement requirements of RREL are often project-specific and require project-specific QC guidelines.

Thus, this section of the QA Project Plan must provide an unambiguous description of QC procedures. There is no need, however, to repeat in the text any material that is already described in a standard method.

SECTION 10.0 PERFORMANCE AND SYSTEMS AUDITS

Each Category I QA Project Plan should describe the QA audits planned for monitoring the system to be used for obtaining critical measurements. A schedule of all contractor-planned audits should be included, along with identification of the responsible personnel. This section should also indicate what audit reports will be generated and who will receive these reports. If no audits are planned, include an explanation.

QA audits for RREL projects may include one or more Technical Systems Audits (TSAs), Performance Evaluation Audits (PEAs), and Audits of Data Quality (ADQs).

A TSA is a qualitative evaluation of all components of the total measurement system, including technical personnel and QA management. This type of audit includes a careful evaluation of both field and laboratory QC procedures. TSAs are normally performed before, or shortly after, measurement systems are operational; they should also be performed on a regularly scheduled basis throughout the lifetime of the project.

After measurement systems are operational and begin generating data, PEAs are conducted periodically to determine the bias of the total measurement system(s) or component parts. As part of a PEA, the laboratory analyzes a performance evaluation sample. QA Project Plans should also indicate any scheduled participation in other interlaboratory performance evaluation studies. Long-term projects should provide for regularly scheduled PEAs.

ADQs are retrospective evaluations of data. Typically, a representative portion of the results in an analytical report is reviewed in detail, starting with raw data and chromatograms, and proceeding through the calculation of final results. ADQs are often used to resolve specific questions regarding the quality of a data set.

SECTION 11.0 PREVENTIVE MAINTENANCE

For most RREL projects, instrument performance is checked immediately before and after use, and in such instances preventive maintenance is not an issue. On long-term monitoring programs, preventive maintenance can become an important tool, especially assuring that QA requirements are met. All Category I projects must provide a brief summary of preventive maintenance, or should state why it is not relevant to the project.

This section should include a summary description of preventive maintenance procedures, a schedule for performing these procedures, a list of major spare parts, and lists of maintenance contracts for critical measurement systems. For convenience, these items may be presented in tabular format.

SECTION 12.0 CALCULATION OF DATA QUALITY INDICATORS

This section describes how data quality indicators will be calculated and reported. As a minimum, equations must be provided for precision, accuracy, completeness, and method detection limits. In addition, equations must be given for other project-specific calculations, such as mass balance, emission rates, confidence ranges, etc.

Make sure that this section complements Section 3.0 (QA Objectives) and Section 9.0 (Internal QC Checks) of the QA Project Plan. Section 3.0 specifies which particular data quality indicators will be employed. Section 9.0 then uses these specific indicators to generate acceptance criteria. Because groups can use different equations to calculate data quality indicators, Section 12.0 must provide the exact equations to avoid misunderstandings among future data users.

Listed below are general guidelines for calculating the more common data quality indicators.

12.1 COMMON DATA QUALITY INDICATORS

12.1.1 Precision

If calculated from duplicate measurements, relative percent difference is the normal measure of precision:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2)/2}$$
 (1)

where:

RPD = relative percent difference

C₁ = larger of the two observed values
 C₂ = smaller of the two observed values.

If calculated from three or more replicates, use relative standard deviation rather than RPD:

RSD =
$$(s/\bar{y}) \times 100\%$$
 (2)

where:

RSD = relative standard deviation

s = standard deviation

 \overline{y} = mean of replicate analyses.

Standard deviation is defined as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n-1}}{(3)}}$$

where:

s = standard deviation

 y_i = measured value of the *i*th replicate

 \overline{y} = mean of replicate measurements

n = number of replicates.

For measurements, such as pH, where the absolute variation is more appropriate, precision is usually reported as the absolute range, D, of duplicate measurements:

$$D = |m_1 - m_2| \tag{4}$$

where:

D = absolute range

 m_1 = first measurement

 m_2 = second measurement

The standard deviation, s, given above, can also be used.

12.1.2 Accuracy

For measurements where matrix spikes are used, calculate the percent recovery as follows:

$$R = 100\% \times \left(\frac{S - U}{C_{sa}} \right)$$
 (5)

where:

R = percent recovery

S = measured concentration in spiked aliquot

U = measured concentration in unspiked aliquot

 C_{Sa} = actual concentration of spike added.

When a standard reference material (SRM) is used:

$$R = 100\% \times \left(\frac{C_{m}}{C_{srm}}\right)$$
 (6)

where:

%R = percent recovery

 C_m = measured concentration of SRM

C_{srm} = actual concentration of SRM.

12.1.3 <u>Completeness</u>

Completeness is defined as follows for all measurements:

$$C = 100% \times \left(\frac{V}{n}\right) \tag{7}$$

where:

C = percent completeness

V = number of measurements judged valid

n = total number of measurements necessary to achieve a specified level of confidence in decision making.

Note: This more rigorous definition of completeness is an improvement on the conventional definition in which "n" is replaced by "T," the total number of measurements.

12.1.4 Method Detection Limit (MDL)

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1, 1-\alpha=0.99)} \times s$$
 (8)

where:

MDL = method detection limit

s = standard deviation of the replicate analyses

 $t_{(n-1, 1-\alpha=0.99)}$ = students' t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

12.2 PROJECT-SPECIFIC INDICATORS

RREL projects frequently incorporate data quality indicators in addition to those discussed in the previous sections. The following is an example of a project-specific data quality indicator:

<u>Example</u>: Mass balance calculation for a soil washing process being tested with PCP-contaminated soils. Mass balance (MB) will be calculated according to

$$MB = M_{out}/M_{in} \tag{9}$$

where M_{out} and M_{in} denote the total mass of PCP in the output and input streams for each test condition.

SECTION 13.0 CORRECTIVE ACTION

Each QA Project Plan must incorporate a corrective action plan. This corrective action plan must include the predetermined acceptance limits, the corrective action to be initiated whenever such acceptance criteria are not met, and the names of the individuals responsible for implementing the plan.

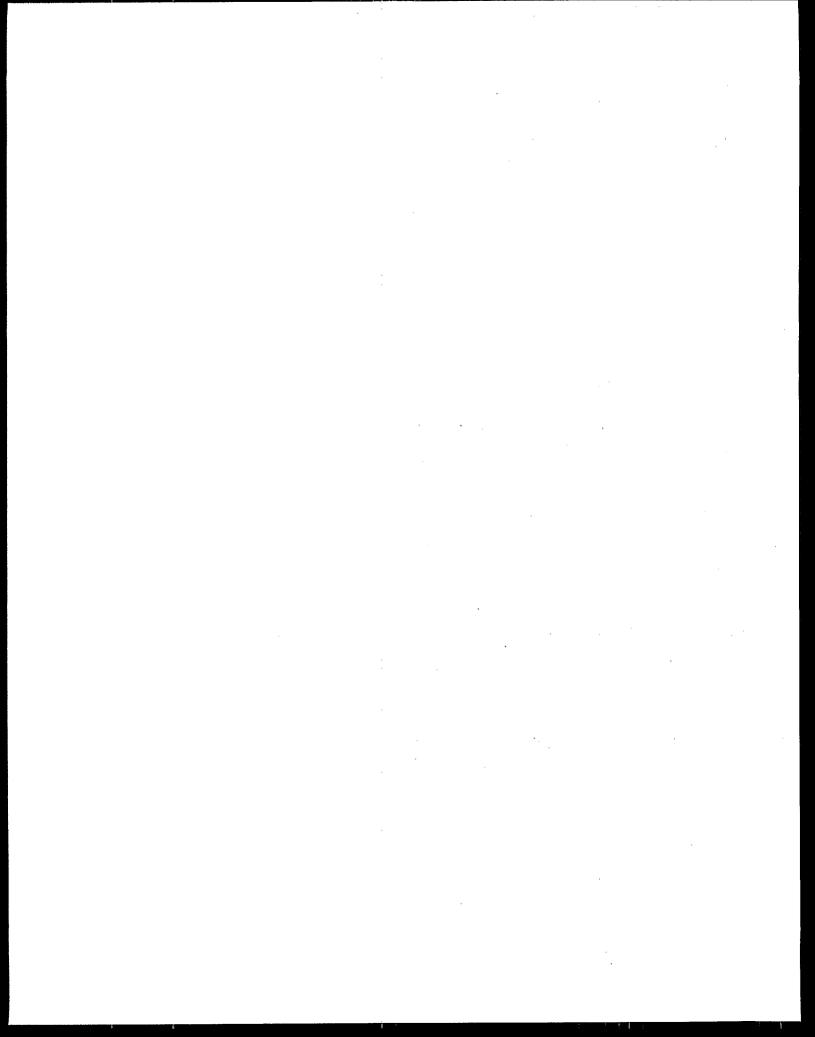
Routine QC procedures already included in Section 9.0 need not be repeated here. This section is reserved primarily for <u>nonroutine</u> corrective action not described elsewhere. Nonroutine corrective action may result from common monitoring activities, such as:

- Performance evaluation audits,
- Technical systems audits, and
- Interlaboratory comparison studies.

It may also arise from conditions unique to specific projects, as seen below.

<u>Example</u>: Bio-oxidation study of hazardous waste in municipal sludge carried out over a one-year period.

The prime contractor's Quality Assurance Manager will submit to the laboratory a blind performance evaluation (PE) sample containing some or all of the target analytes before any analytical work begins and on a monthly basis thereafter. The average percent recovery of all target analytes must be between 80 and 120 percent, with no outliers less than 50 or greater than 150 percent. If these limits are exceeded, analytical work will stop until the problems are identified and solved. Before work is restarted, another blind PE sample must be analyzed and results must meet the acceptance criteria. Results of these PE samples will be included in the final report.



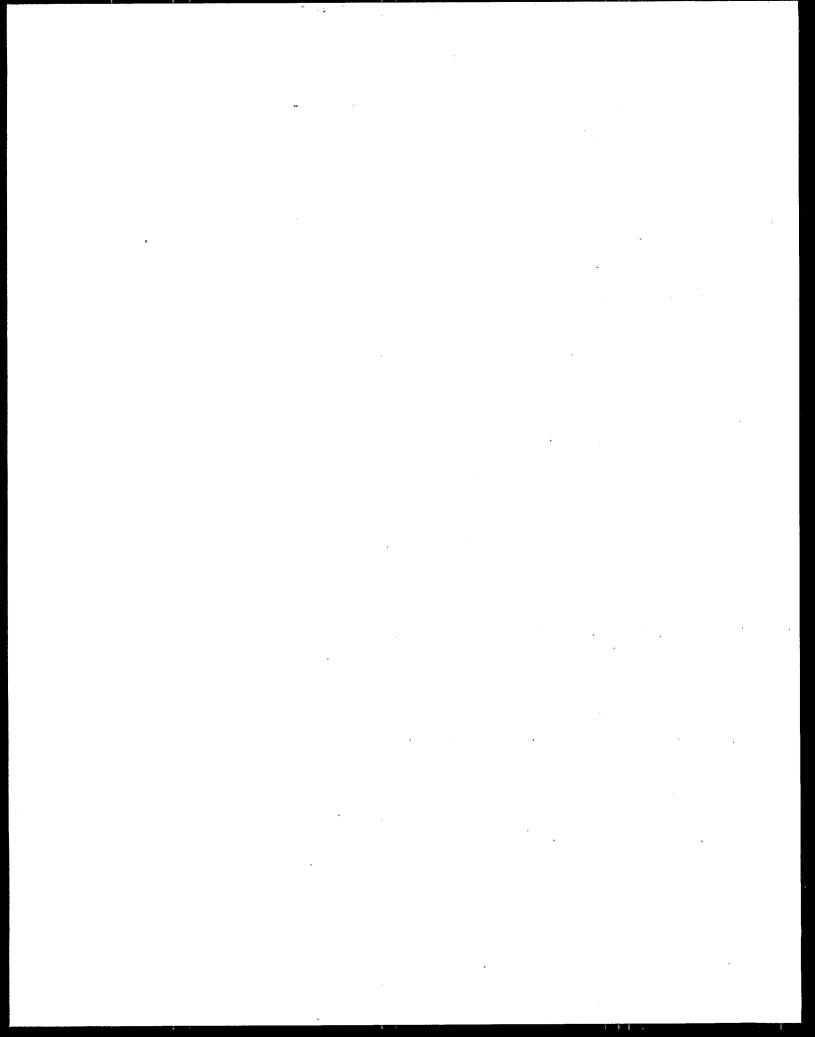
SECTION 14.0 QUALITY CONTROL REPORTS TO MANAGEMENT

This section of a Category I QA Project Plan identifies the individuals responsible for QA reports, and describes the type and frequency of reports (weekly oral presentations and discussions, monthly written reports, etc.) that will be used to keep project management informed. As a minimum, such reports include:

- Changes in the QA Project Plan
- Summary of QA/QC programs, training, and accomplishments
- · Results of technical systems and performance evaluation audits
- Significant QA/QC programs, recommended solutions, and results of corrective actions
- Data quality assessment in terms of precision, accuracy, representativeness, completeness, comparability, and method detection limit
- Discussion of whether the QA objectives were met, and the resulting impact on decision making
- Limitations on use of the measurement data.

Managers receiving these detailed reports will then be able to monitor data quality easily and effectively.

If subcontractors are used, include a discussion which specifies the QC reporting from the subcontractor to the prime contractor, along with the mechanism and frequency of such reporting. Regular QC reports from subcontractors during the course of a project are important in keeping project and QA management informed on progress and on potential problems which may require corrective action.

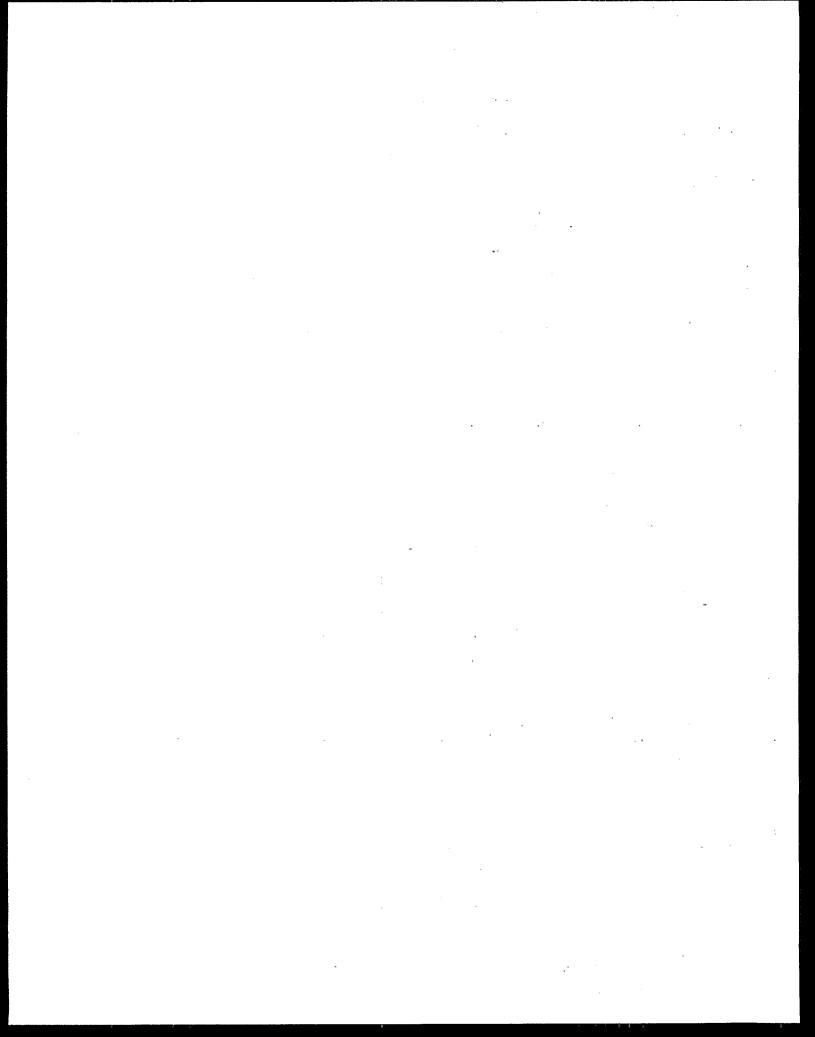


SECTION 15.0 REFERENCES

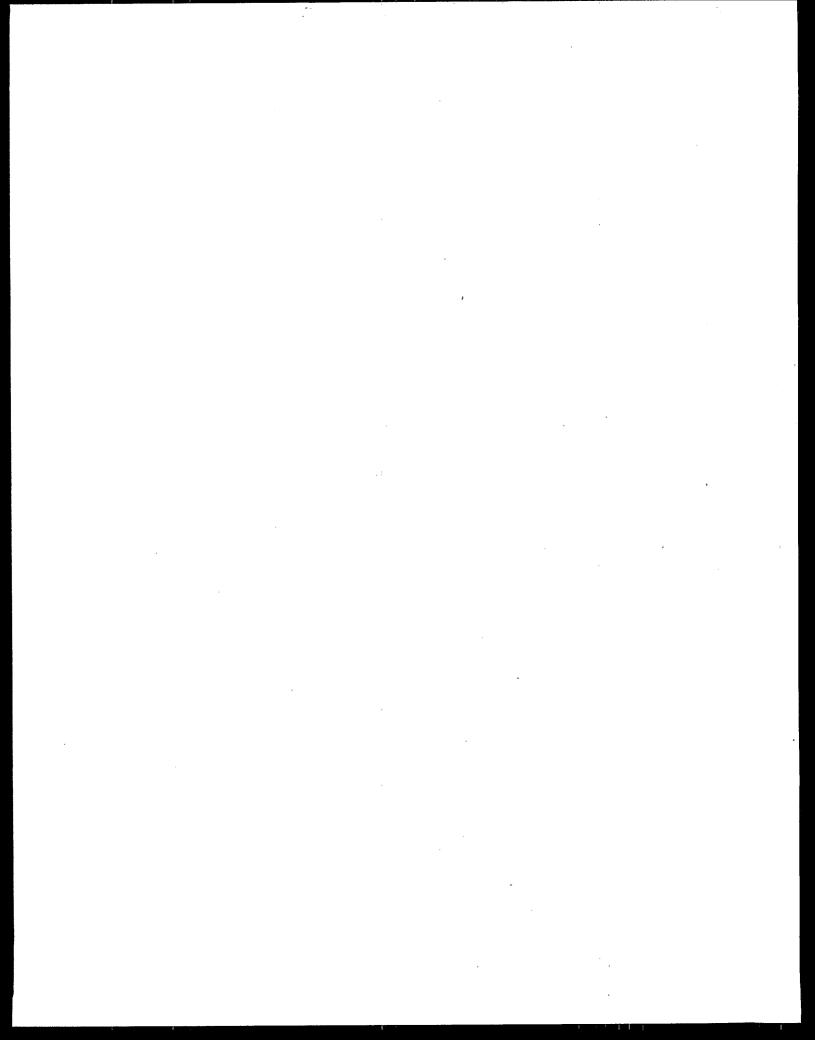
References, if any, can be included in the body of the text, as footnotes, or collected in this section. If a reference is not readily available, attach a copy to the QA Project Plan. References must uniquely identify the cited material. In particular, when citing various compendia of standard methods published by the EPA, ASTM, American Public Health Association, etc., be sure to include the edition number, since such methods can change substantially from one edition to the next.

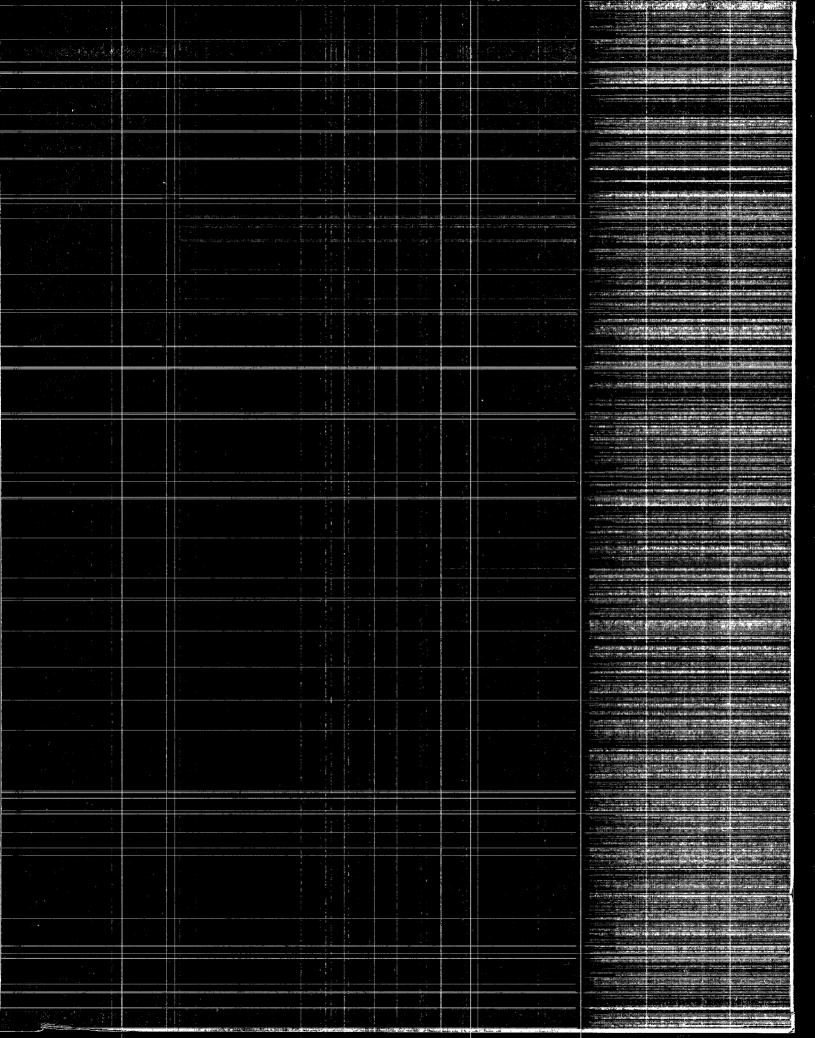
For example, the following are references applying to Table 7-1 of this document.

- 1. American Society for Testing and Materials. Annual Book of Standards. ASTM. Philadelphia, PA. Parts 14 and 19 (updated yearly).
- 2. U.S. Environmental Protection Agency. "A Procedure for Estimating Monofilled Solid Waste Leachate Composition." Technical Resource Document SW-924, 2nd ed. Hazardous Waste Engineering Research Laboratory, Office of Research and Development. Cincinnati, OH, and Office of Solid Waste and Emergency Response. Washington, D.C., 1986.
- 3. American Nuclear Society. ANSI/ANS-16.1-1988. American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure. American Nuclear Society. LaGrange Park, IL, 1986.
- 4. U.S. Environmental Protection Agency. Office of Solid Waste. Test Methods for Evaluating Solid Waste, 3rd ed. Available from U.S. Government Printing Office. Washington, D.C., 1986.
- 5. Alford-Stevens, A., T. A. Bellar, J. W. Eichelberger, and W. L. Budde. Method 680, Determination of Pesticides and PCB in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry. Available from the U.S. Environmental Protection Agency. Cincinnati, OH, 1985.
- 6. Klute, A. (Ed.). Methods of Soil Analysis, Part I. American Society of Agronomy. Madison, WI, 1986.









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