Quality Assurance Guidelines
for Environmental Measurements

Prepared by
QA/QC Implementation Work Group
1994

Revised
August 2003

UNITED STATES DEPARTMENT OF THE INTERIOR ★ BUREAU OF RECLAMATION

Mission Statements

The mission of the U.S. Department of the Interior is to protect and provide access to our Nation's natural and cultural heritage and honor our trust responsibilities to Indian Tribes and our commitments to island communities.

The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

Quality Assurance Guidelines

for Environmental Measurements

Prepared by
QA/QC Implementation Work Group
1994

Revised
August 1998
August 2003

UNITED STATES DEPARTMENT OF THE INTERIOR ★ BUREAU OF RECLAMATION

Sampling the environment for environmental data is no trivial exercise — it is most easily, and more likely to be, performed incorrectly.

United States Department of the Interior

BUREAU OF RECLAMATION
PO Box 25007
Denver, Colorado 80225-0007

IN REPLY REFER TO:

D-8000
ENV-1.10

SEP 18 2003

MEMORANDUM

To: Commissioner
Attention: W-1000
Regional Director, PN, MP, LC, UC, GP
Attention: PN-1000, MP-100, LC-1000, UC-100, GP-1000
Area and Project Offices (See Attached List)

From: Michael J. Roluti
Director, Technical Service Center

Subject: "Quality Assurance (QA) Guidelines for Environmental Measurements"

Attached is "Quality Assurance Guidelines for Environmental Measurements" ("Guidelines") prepared by Reclamation's QA Implementation Work Group in 1994, but never released. The original "Guidelines," distributed in 1991, revised in 1994 and 1998, has been further revised in 2003 to reflect the application of the guides to environmental data collection quality measures for both field and laboratory activities and Reclamation's current organizational capability.

The objectives of "Guidelines" are to improve the usability of data produced by Reclamation projects and improve the communication between data providers and data users. The 1994 Implementation Work Group was composed of Reclamation planners, environmental specialists, and laboratory and project managers. The 1998 revision was performed by D-8570, with peer review by QA/QC professionals throughout Reclamation.

QA principles and practices in "Guidelines" are intended to be used in all Reclamation projects and laboratories. Your dedicated adherence to these practices and your comments about them will help Reclamation perfect the policies that best meet our data needs. I invite you to submit any comments about "Guidelines" to the Reclamation Technical Service Center, code D-8010.

A directory of Reclamation's regional chemistry laboratories and the Denver Technical Service Center chemistry laboratory, listing testing capabilities and a management contact, is included to facilitate networking for technical support.

The "Guidelines" document will be available on the Reclamation Intranet.

Attachment

cc: Director, Operations, Attention: W-6000
Director, Office of Program and Policy Services, Attention: D-5000
(w/att to each)

bc: D-8100-8180, D-8200-8290, D-8300-8340, D-8400-8470, D-8500-8580
(w/att to each)

Area and Project Offices Distribution List:

Area Manager, Snake River Area Office - West, Boise ID
Attention: SRA-1000
Power Manager, Grand Coulee Power Office, Grand Coulee WA
Attention: GCP-1000
Area Manager, Upper Columbia Area Office, Yakima WA
Attention: UCA-1000
Area Manager, Lower Columbia Area Office, Portland OR
Attention: LCA-1000
Chief, Facility O&M, Lower Columbia Field Office, Bend OR
Attention: LCA-3200
Project Construction Engineer, Pacific Northwest Construction Office, Yakima WA
Attention: NCO-3100
Chief Facility O&M, Bend Field Office, Bend OR
Attention: BFO-3000
Manager, Umatilla Field Office, Hermiston OR
Attention: UMA-4000
Program Coordinator, Elwha Restoration Project Office, Port Angeles WA
Attention: ELW-1000A
Manager, Rogue River Field Office, Medford OR
Attention: RRFO-6200
Project Superintendent, Hungry Horse Field Office, Hungry Horse MT
Attention: HH-3000
Deputy Area Manager, Ephrata Field Office, Ephrata WA
Attention: EPH-2000
Manager, Yakima Field Office, Yakima WA
Attention: YAK-5000
Deputy Area Manager, Snake River Area Office - East, Burley ID
Attention: SRA-6300
Center Director, Columbia Basin Job Corps Civilian Conservation Center, Moses Lake WA
Attention: CBCCC-100
Center Director, Ft. Simcoe Job Corps Civilian Conservation Center, White Swan WA
Attention: FSJCCCC-100
Center Director, Centennial Job Corps Civilian Conservation Center, Nampa ID
Attention: CJCCCC-100
Area Manager, Klamath Basin Area Office, Klamath Falls OR
Attention: KO-100
Area Manager, Lahontan Basin Area Office, Carson City NV
Attention: LO-100
Office Manager, Fallon Field Office, Fallon NV
Attention: LO-930
Area Manager, Northern California Area Office, Shasta Lake CA
Attention: NC-100
Program Manager, Trinity River Restoration Division, Weaverville CA
Attention: NC-150

Area Manager, Central California Area Office, Folsom CA
Attention: CC-100
Resource Manager, Lake Berryessa Resource Center, Napa CA
Attention: CC-420
Resource Manager, New Melones Resource Center, Sonora CA
Attention: CC-460
Supervisory General Engineer, New Melones Powerplant, Jamestown CA
Attention: CC-670
Area Manager, South-Central California Area Office, Fresno CA
Attention: SCC-100
Water Operations Supervisor, Friant Dam Branch, Friant CA
Attention: SCC-439
Maintenance Operations Supervisor, Friant Dam Branch, Friant CA
Attention: SCC-438
Irrigation Systems Operator, Cachuma Field Branch, Santa Barbara CA
Attention: SCC-432
Supervisory General Engineer, Tracy Office, Byron CA
Attention: TO-400
Construction Engineer, MP Construction Office, Willows CA
Attention: MPCO-100
Operations Manager, Central Valley Operations Office, Sacramento CA
Attention: CVO-100
Area Manager, Phoenix Area Office, Phoenix AZ
Attention: PXAO-1000
Deputy Area Manager, Phoenix Area Office, Phoenix AZ
Attention: PXAO-1100
Administrative Officer, Phoenix Area Office, Phoenix AZ
Attention: PXAO-9000
Civil Engineer, Tucson Field Office, Tucson AZ
Attention: PXAO-7005
Program Manager, Native American Affairs Office, Phoenix AZ
Attention: NAAO-1100
Area Manager, Lower Colorado Dams Office, Boulder City NV
Attention: LCD-1000
Facility Manager, Lower Colorado Dams Office, Boulder City NV
Attention: LCD-1050
Manager, Lower Colorado Dams Office, Bullhead City AZ
Attention: LCD-D10
Manager, Lower Colorado Dams Office, Parker Dam CA
Attention: LCD-P10
Foreman, Headgate Rock Dam, Parker Dam CA
Attention: LCD-P52
Area Manager, Southern California Area Office, Temecula CA
Attention: SCAO-1000
Area Manager, Boulder Canyon Operations Office, Boulder City NV
Attention: BCOO-1000

Area Manager, Yuma Area Office, Yuma AZ
Attention: YAO-1000
Deputy Area Manager, Yuma Area Office, Yuma AZ
Attention: YAO-1100
Director, Technical Support Office, Yuma Area Office, Yuma AZ
Attention: YAO-2000
Director, Administration Support Office, Yuma Area Office, Yuma AZ
Attention: YAO-3000
Director, Operations and Maintenance Office, Yuma Area Office, Yuma AZ
Attention: YAO-4000
Director, Program Management Office, Yuma Area Office, Yuma AZ
Attention: YAO-6000
Director, Resource Management Office, Yuma Area Office, Yuma AZ
Attention: YAO-7000
Area Manager, Albuquerque Area Office, Albuquerque NM
Attention: ALB-100
Manager, Alamosa Field Division, Alamosa CO
Attention: AFD-400
Facilities Coordination Specialist, Carlsbad Office, Carlsbad NM
Attention: CB-410
Manager, Chama Field Division, Chama NM
Attention: C-10
Manager, Elephant Butte Field Division, Truth or Consequences NM
Attention: EB-600
Manager, El Paso Field Division, El Paso TX
Attention: EP-100
Manager, Socorro Field Division, Socorro NM
Attention: S-10
Center Director, Collbran Job Corps Civilian Conservation Center, Collbran CO
Attention: CJC-100
Construction Manager, Farmington Construction Office, Farmington NM
Attention: FCO-100
Manager, Power Office, Salt Lake City UT
Attention: UC-600
Manager, Glen Canyon Field Division, Page AZ
Attention: GC-100
Manager, Curecanti Field Division, Montrose CO
Attention: CCI-100
Manager, Flaming Gorge Field Division, Dutch John UT
Attention: FG-100
Area Manager, Provo Area Office, Provo UT
Attention: PRO-100
Center Director, Weber Basin Job Corps Civilian Conservation Center, Ogden UT
Attention: WBJC
Area Manager, Western Colorado Area Office, Grand Junction CO
Attention: WCG-CDeAngelis

Manager, Western Colorado Area Office - Four Corners Division, Durango CO
Attention: WCD-PSchumacher
Area Manager, Dakotas Area Office, Bismarck ND
Attention: DK-100
Pierre Field Office, Pierre SD
Attention: DK-200P
Manager, Rapid City Field Office, Rapid City SD
Attention: DK-700R
Area Manager, Eastern Colorado Area Office, Loveland CO
Attention: EC-1000
Powerplant Supervisor II, Estes Powerplant, Estes Park CO
Attention: EC-1830
Powerplant Supervisor III, Flatiron Powerplant, Loveland CO
Attention: EC-1820
Green Mountain Powerplant, Silverthorne CO
Attention: EC-1863
Resident Engineer, Horsetooth Field Office, Fort Collins CO
Attention: GP-2400
Water Treatment Supervisor II, Leadville Powerplant, Leadville CO
Attention: EC-1840
Supervisor Operation Manager, Loveland Control Center, Loveland CO
Attention: EC-1810
Electrician Foreman I, Meredith Powerplant, Basalt CO
Attention: EC-1862
Facility Manager, Mt. Elbert Powerplant, Granite CO
Attention: EC-1860
Facility Manager, Pueblo Dam Field Office, Pueblo CO
Attention: EC-1200
Area Manager, Nebraska-Kansas Area Office, Grand Island NE
Attention: NK-100
Manager, McCook Field Office, McCook NE
Attention: NK-400
Area Manager, Oklahoma-Texas Area Office, Austin TX
Attention: TX-Walkoviak
Deputy Area Manager, Oklahoma City Field Office, Oklahoma City OK
Attention: OK-Moomaw
Area Manager, Montana Area Office, Billings MT
Attention: MT-100
Facilities Manager, Canyon Ferry Field Office, Helena MT
Attention: MT-680
Water and Land Rep, Clark Canyon Field Office, Dillon MT
Attention: MT-242
Engineering Equipment, Milk River Field Branch, Babb MT
Attention: MT-468
Facilities Manager, Tiber Field Office, Chester MT
Attention: MT-460

Hydraulics Facility Superintendent, Fresno Field Office, Havre MT
Attention: MT-466
Chief, Power Division, Yellowtail Field Office, Yellowtail MT
Attention: MT-600
Area Manager, Wyoming Area Office, Mills WY
Attention: WY-1000

TABLE OF CONTENTS

Preface iii

Acknowledgments iii

List of Abbreviations and Acronyms v

Glossary of Terms vii

INTRODUCTION 1
- The Objectives of “Guidelines" 1
- Quality 2
- Quality Assurance Concepts 2
- Reclamation's New Quality Assurance Program for Environmental Measurements (QA Program) 2
- “Guidelines” Content Selection Criteria and Update Process 3

PART 1 - GUIDANCE 5
- Outline of Quality Assurance Overview 6
- Outline of Quality Assurance Project Plan (QAPP) 7

Data Quality Objectives (DQOs) 9
- Potential Stages of a DQO Process 10
- Stage I: Presentation of the Question or the Decision to be Made 11
- Stage II: State the Requirements of the Information Needed 11
- Stage III: Design the Data Collection Process 11
- Beyond Stage III: Detailed Design of the Data Collection Activity 12
- Summary 12

Bibliography 15

Sampling and Analysis Plan (SAP/Technical Work Plan) 17

Sample Collection Design - Sampling Theory 19
- Planning 19
- Safety 20
- Sampling Protocols 20
- Sampling Design 23
- Other Samples 27

Bibliography 35

References 35

Data Acquisition and Analysis Assessment/Oversight 37
- Field Audit 37
- Laboratory Audit 37
- Office Audit 39
- Follow Up 39

Bibliography 41

Data Validation and Usability 43
- Data Quality Assessment (DQA) 43
- Data Usefulness 44
- Screening Data with Definitive Confirmation 45
- Definitive Data 45
- Data Category: Other 48
- Previous Data Classifications 48

Bibliography 49

TABLE OF CONTENTS - CONTINUED

PART 2 - LABORATORY GUIDANCE 51
Introduction 51
Organization 51
Facilities 51
Sample Receipt Policy 51
Instrumentation 51
Services 52
Quality Control/Quality Assurance 52
Bibliography 57
References 57

PART 3 - GENERAL GUIDANCE 59
Quality Assurance of Data Management 59
Responsibilities 59
Sample Accountability 60
Records Control 60
Data Security 61
Verification of Records 61
Reviews and Approval 61
Maintenance of Records 61
Computer Management 62
Bibliography 63
Selection of an Analytical Laboratory 65
Laboratory Assessment 65
Bibliography 73
References 73
Checklist for Performing a Quality System Review 75
Bibliography 79

PART 4 - FIELD SAMPLING METHODOLOGY 81
Surface Water Sampling 81
Groundwater Sampling 82
Bed Sediment Sampling 82
Biological Sampling 83
Soil Sampling 83
Bibliography 85

RECLAMATION LABORATORIES 87

PREFACE

The Bureau of Reclamation's (Reclamation) Quality Assurance Program was established by the Commissioner in 1989 on the recommendation of the Permanent Management Committee to prepare a consensus guidance document for Reclamation and contracted laboratories doing environmental testing. The purpose of this guidance document is to update and expand the scope of the original guidance document. This document offers a standard and consistent basis for performing environmental sampling and testing. "Quality Assurance Guidelines For Environmental Measurements" was originally published in 1991 and revised (but never published) in 1994 by a work group of regional, area office, and Technical Service Center (TSC) staff. The group of chemists, laboratory and program managers, and environmental specialists was selected by regional directors and TSC managers to establish Reclamation-wide representation.

ACKNOWLEDGMENTS

The members of the 1994 work group that prepared and reviewed the 1994 text that served as the basis for this update of the guidance were:

Stanley Conway, Technical Service Center, Denver, Colorado

Douglas Craft, Technical Service Center, Denver, Colorado

John Ellis, Technical Service Center, Denver, Colorado

Juli Fahy, Technical Service Center, Denver, Colorado

John Fields, Mid-Pacific Region, Sacramento, California

Robert George, Technical Service Center, Denver, Colorado

David Hemphill, Lower Colorado Region, Boulder City, Nevada

Patti Ivey, Dakotas Area Office, Bismarck, North Dakota

Ernest Loya, Mid-Pacific Region, Sacramento, California

Jeffrey Lucero, Great Plains Region, Billings, Montana

Robert Radtke, Upper Colorado Region, Salt Lake City, Utah

Kerry Rae, Mid-Pacific Region, Sacramento, California

Bill Stroud, Pacific Northwest Region, Boise, Idaho

Evelyn Vigil, Alamosa Area Office, Alamosa, Colorado

The Reclamation staff involved in the opportunity to peer review the update of this document are:

Steven Bowser, D-8570, Denver, Colorado

John Ellis / Doug Craft, D-8240, Denver, Colorado

Samuel Schaefer, D-8550, Denver, Colorado

John Fields, MP-470, Sacramento, California

Robert Radtke, UC-289, Salt Lake City, Utah

Bill Stroud, PN-3210, Boise, Idaho

David Hemphill, LC-2121, Boulder City, Nevada

Jeffrey Lucero, GP-2500, Billings, Montana

LIST OF ABBREVIATIONS AND ACRONYMS

ACS	American Chemical Society
ANSI	American National Standards Institute, http://web.ansi.org/
AOAC	Association of Official Analytical Chemists
ASQ	American Society for Quality [Control], http://www.asqc.org/ (formerly ASQC)
ASTM	American Society for Testing and Materials, http://www.astm.org/
CCV	Continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
CLP	Contract Laboratory Program (EPA)
CRM	Certified reference material
CV	Coefficient of Variation
DL	Detection limit
DQA	Data quality assessment
DQO	Data quality objective
EPA	Environmental Protection Agency, http://www.epa.gov/
EQL	Estimated quantitation limit
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FSP	Field Sampling Plan
GLP	Good laboratory practices
GMP	Good measurement practices
ICB	Initial calibration blank
ICV	Initial calibration verification
IDL	Instrument detection limit
IEC	International Electrotechnical Commission
ISO	International Standardization Organization (ISO 9000 / 14000)
LCS	Laboratory control sample
LOD	Limit of detection
LOQ	Limit of quantification
MDL	Method detection limit
NAWQA	National Water-Quality Assessment Program
NEPA	National Environmental Policy Act
NPL	National Priority List
NPDES	National Pollutant Discharge Elimination System
NIST	National Institute of Standards and Technology, http://www.nist.gov/
NRCS	National Resource Conservation Service (formerly Soil Conservation Service)
PE	Performance evaluation sample
PQL	Practical quantitation limit
QA	Quality assurance
QAP	Quality assurance plan
QAPP	Quality assurance project plan
QMP	Quality (assurance) management plan
QC	Quality control
RPD	Relative percent difference
RSD	relative standard deviation
SAP	Sampling and analysis plan
SAS	Special analytical services
SOP	Standard operating procedure
SRM	Standard reference material (see NIST, et al.)
ULSA	Unique laboratory services agreement (replaces CLP special analytical services, see SAS)
USGS	U.S. Geological Survey

GLOSSARY OF TERMS

This glossary of terms provides definitions for some of the more specialized technical terms used in these guidelines. The use of these terms by project and laboratory staff is encouraged to improve the effectiveness of discussions about environmental investigations and testing. See also "http://www.epa.gov/emfjulte/html/qa_terms.html."

Accuracy - The degree of agreement between an observed value and an accepted reference or true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are caused by sampling and analytical operations. See **Bias** and **Precision**, below.

$$\text{Accuracy \%} = \frac{\text{Observed value}}{\text{True value}} \times 100$$

Aliquant - A subsample derived by a divisor that divides a sample into a number of equal parts but leaves a remainder; a subsample resulting from such a divisor.

Aliquot - A subsample derived by a divisor that divides a sample into a number of equal parts and leaves no remainder; a subsample resulting from such a division.

Analyte - The chemical element, compound, or form that an analysis seeks to determine; the element or compound of interest.

Analytical method - Any approved or reference body of systematic procedures and techniques of sample collection and/or analysis for a characteristic of interest.

Audit - A systematic evaluation to determine the conformance to specifications of some function or activity.

Bias - A systematic displacement of all the observations in a sample set from the true or accepted value; a systematic and consistent error in test results.

$$\text{Bias \%} = \frac{\text{Observed value - True value}}{\text{True value}} \times 100$$

Blank sample - A sample of medium known or presumed not to be contaminated with the analyte(s) of interest, processed to estimate the contamination during sampling and shipping of the associated samples, or a performance evaluation sample used to check for potential contamination introduced in the analysis system. The test result for a constituent measured in a blank sample is reported in the same units as the associated samples. See **Field blank**, **Method blank**, **Trip blank**, and **Rinse blank**.

Blind (single-blind) sample - A subsample submitted for analysis with a composition and type (regular subsample or quality control sample) known to the submitter, but with the composition unknown to the analyst, and used to test the analyst's or laboratory's proficiency in the execution of the measurement process (e.g., a field replicate subsample identified as a replicate, but without reference to the matching sample or samples). Used to prod the laboratory into quality consciousness. See **Double-blind sample**.

Calibration - The determination, by measurement or comparison with a standard, of the correct value of each scale reading on an instrument meter, or other device, or the correct value for each setting of a control knob. The adjustment of a measurement device such that the measurements it subsequently provides are true and accurate (precise and unbiased within the design capability of the instrument) under the environmental conditions at the time of calibration.

Chain of Custody (COC) - A record of possession or accountability that tracks sample transfers and attempts to assure the physical integrity and security of samples, data, and records.

Check sample - An uncontaminated sample matrix spiked with known amounts of analytes usually from the same source as the calibration standards. It is generally used to establish the stability of the analytical system, but may also be used to assess the performance of all or a portion of the measurement system. See also **Quality control sample**.

Coefficient of variation (CV) - A measure of relative variation (precision). The ratio, expressed as a decimal, of the standard deviation of a set of values divided by the average value. When the decimal is multiplied by 100, the CV becomes the relative standard deviation (RSD).

Comparability - The degree to which different methods, data sets, and/or decisions can be represented as similar and be compared.

Completeness - The amount of valid data obtained compared to the amount expected, expressed as a percentage.

Composite sample - A population subsample consisting of multiple discrete increments integrated into one uniquely identified sample submission. A minimum of 30 increments per sample is recommended to reduce the grouping and segregation error.

Continuing calibration verification (CCV) - Analysis of an internal, or other, standard at regular intervals during an analysis run to verify the calibration of the analytical system.

Control limits - A range of statistical uncertainty within which specified measurement results must exist. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring noncompliant data be identified.

Control sample - See **Check sample** and **Quality control sample**.

Corrective action - An action specified in a method, procedure, or quality control plan taken to document and stop the production of data of poor or unacceptable quality and retroactively correct the process to produce data of acceptable quality.

Data quality - The totality of features and characteristics of data that bears on their ability to satisfy a given purpose. The characteristics often of major importance are accuracy, precision, completeness, representativeness, and comparability.

Data Quality Objectives (DQOs) - Statements which specify the overall level of uncertainty that a decisionmaker is willing to accept in results or decisions derived from environmental data. DQOs provide the statistical framework for planning and managing environmental data operations consistent with the data users needs.

Data reduction - The process of changing original data by calculations, standard curves, concentration factors, etc., to a more useful form.

Detection limit (DL) - NO CONSISTENT DEFINITION EXISTS WITHIN THE QA COMMUNITY. For Bureau of Reclamation (Reclamation) work, the following definition is appropriate: the lowest concentration or amount of the target analyte that, when processed through the complete method, can be determined to be different from zero by a single measurement at a stated level of probability (EPA). See **Estimated quantitation limit**, **Method detection limit**, and **Limit of quantification**.

Instrument detection limit (IDL) - IDL is based on the signal-to-noise ratio of the instrument involved.
Limit of detection (LOD) - LOD is three times the standard deviation of a fixed number of successive analytical runs (7, 15, or 21).
Limit of quantitation (LOQ) - LOQ is 10 times the standard deviation of a fixed number of successive analytical runs.
Method detection limit (MDL) - MDL is based on a precision measurement process in which all steps in the given method are employed.
Practical quantitation limit (PQL) - PQL is an MDL based on the laboratory experience in the specific sample matrix.

If your DQOs specify detection limits, be sure you understand how the terms are defined and that all parties agree on the definitions.

Digestion - Preparation of the sample (by heating with acid or other specified process) to enable the analysis system to measure the process-specific analyte concentration.

Dissolved constituents - The operational definition for the constituents in a water sample that pass through a 0.45-micrometer membrane filter or glass fiber filter for analysis. The named chemical constituents dissolved in water as identified by laboratory analysis.

Document control - A systematic procedure for indexing documents by unique number, date, and revision number for tracking, archiving, storage, and retrieval.

Double-blind sample - A sample submitted to evaluate performance with concentration unknown and type disguised to the analyst (e.g., a duplicate subsample identified as just another regular subsample). Used to eliminate preferential sample handling and analysis of quality control samples.

Dry weight basis - The expression of constituent concentration relative to the mass of water-free sample. The constituent concentration based on dry sample weight is determined directly from a water-free (dried) sample, or may be calculated by dividing the constituent concentration determined from the total (undried) sample by the absolute difference between the percent moisture of the sample, expressed as a decimal, and the number one.

Duplicate (for Reclamation purposes) - An additional field subsample of a population taken by duplicating the sampling method (same location and manner, different time) used to acquire the original subsample, used to verify population characteristics or assess the effect of different sampling personnel. In the laboratory, a second or subsequent subsample properly split from the original sample used to verify sample handling, processing, and analysis processes. See **Replicate**.

Estimated quantitation limit (EQL) - The lowest concentration measurement that can be reliably achieved within specified limits of bias and precision during routine laboratory operating conditions. Method EQLs are highly matrix dependent (EPA). See **Detection limit**.

External QC - The evaluation of analytical performance on a batch of samples independent of the laboratory by (including but not limited to): (1) comparing the results of analysis of control samples submitted with the samples to the known, or true, values; (2) comparing sample holding times before analysis to recommended maximum holding times; (3) comparing the content of the report received with expectations; and (4) comparing the reported results with historical values.

External QC samples - Duplicate, spiked, reference material, and/or blank samples included in a sample batch submitted to a testing laboratory to provide a means, independent of the laboratory, for estimating the precision and bias of test results and the extent of contamination of the associated samples by the sampling and analysis process.

Field blank sample - A clean sample (e.g., distilled water or washed sand), carried to the sampling site, exposed to sampling conditions (e.g., bottle caps removed, passed through sampling device, preservatives added), and returned to the laboratory and treated as an environmental sample. Field blank samples check for analyte changes introduced by the combined sampling and analysis procedures. See **Rinse blank** and **Trip blank**.

Field sampling plan (FSP) - The operational plan for conducting field activities to obtain environmental measurements described in the sampling design. The procedures for collecting samples in a manner that is consistent with the assumptions upon which the sampling design is based. See **Sampling and analysis plan**.

Fractional alternate shoveling - Soil sample splitting technique that involves the shoveling or scooping of at least 30 fractional increments, randomly (equiprobable selection) chosen from the original sample, per subsample (split) created. For example, for a 60 cc sample of fine (passing the 200 sieve - <.074 mm) soil, split by extracting 60 1-cc increments alternately into two piles, each containing 30 increments. Thirty increments are necessary to reduce the grouping and segregation errors to acceptable levels. More increments may be required for coarser materials. If more split samples are required, or a smaller subsample mass is needed, use a smaller scoop/shovel; i.e., if the test portion is 5 cc, and six split samples are required for analysis and QC, divide the original 60-cc sample (subsample) into twelve 5-cc split piles using a 0.1667-cc (30 increments/5-cc) measure and discard six of the 5-cc split samples.

Good laboratory practice - Either general guidelines or formal regulations for performing basic laboratory operations or activities that are known or believed to improve the quality or integrity of the results (i.e., dishwashing, cleanliness, sterilization, etc.).

Grab sample - A population subsample consisting of one discrete, uniquely identified, single-increment sample. The grab sample is representative of the conditions of the sample at the place and time taken, but little else (of limited value). Grab sample data cannot be used with normal statistics to evaluate a population because it does not represent the population (unless the target population is limited to the sample collected, and then you have the entire population, and not a sample thereof - so why bother collecting it?).

Homogeneous - Uniform material characteristics (structure and composition).

Holding time - The time from sample collection to extraction, digestion, or analysis of the sample, as appropriate. Maximum allowable holding times for analytes in preserved samples are specified by EPA. (See Validation measures discussed in parts 1 through 3.)

Heterogeneous - Nonuniform material characteristics (structure and composition) or a mix of structures and compositions.

Instrument detection limit (IDL) - See **Detection limit**.

Internal standard - A nontarget analyte added to all investigative and control samples, and measured to determine when changes in the measurement system adversely affect the quantification of target compounds. See **Continuing calibration verification**.

Judgmental sampling - A subjective selection of samples based on the experience and knowledge of the site by an expert.

Laboratory QC - The in-house techniques to monitor and maintain the variables of the analytical processes within an established limit (ASTM).

Limit of detection (LOD) - See **Detection limit.**

Limit of quantification (LOQ) - See **Detection limit.**

Matrix - The chemical element(s) and/or compound(s) containing the analyte of interest. Elements in the matrix may interfere with the detection and/or accurate quantification of the analyte of interest.

Medium - The phase of the sample - liquid, solid, or gas. Usually water, soil, or air.

Method blank - A clean sample (e.g., distilled water, pure air, or clean sand) which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. Method blanks check for analyte changes introduced by the analysis procedures.

Method detection limit (MDL) - See **Detection limit**.

Nonprobabilistic sampling - Nonrandom sampling based upon professional judgment or opportunity not related to chance occurrence. Generally used for qualitative assessment of presence or absence and maximum contaminant and may not be acceptable as legally defensible data.

Opportunity sample - Samplers may be presented with an opportunity to collect unplanned samples; e.g., judgmental samples may become evident while grid sampling, or rain may produce runoff to sample during a soil sampling event. The SAP should specify need for and use of any opportunity samples that may be acquired and indicate when opportunity samples should not be taken.

Percent moisture (content) - The ratio of the mass of free water in a sample to the mass of the sample, expressed as a percentage; it can be calculated on a dry (the result of the **Dry weight basis** calculation may also be referred to as moisture content) or wet weight basis, as follows:

$$\text{(Wet)} \qquad \text{Moisture (\%)} = \frac{\text{Mass of total sample - Mass of dried sample}}{\text{Mass of total sample}} \times 100$$

$$\text{(Dry)} \quad \text{Moisture (\%)} = \frac{\text{Mass of total sample - Mass of dried sample}}{\text{Mass of dried sample}} \times 100$$

Performance audit - A check of a measurement system in which quantitative data are independently obtained and used to evaluate the proficiency of an analyst or laboratory. May also include a site visit, inspection, and check of procedure and documentation. See also **Round-robin testing**.

Performance evaluation (PE) sample - Any sample or subsample submitted to the laboratory to evaluate analytical performance and estimate data quality.

Practical quantitation limit (PQL) - See **Detection limit**.

Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions - a data quality indicator. Precision is usually expressed as standard deviation, variance, or range in either absolute or relative terms. See also **Standard deviation** and **Variance**.

Procedure - A set of systematic, step-wise instructions for performing an operation. See **SOP**.

Probabilistic sampling (random sampling) - The selection of sampling points due to chance (probability) that allows the results from a set of samples to be generalized to the entire site (i.e., lay out a square grid and randomly sample at some of the nodes). See **Judgmental**, **Systematic**, and **Opportunity sampling**.

Protocols - Protocols are detailed, written, standardized procedures for field and/or laboratory operations that must be strictly adhered to.

QA1/QA2/QA3 - Former EPA levels of quality assurance for Superfund work - there are currently only two data quality objectives considered in the EPA data validation process.

Quality assurance (QA) -

> The total integrated process for assuring the reliability of decisions based on monitoring and measurement data.
>
> An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence (EPA).
>
> The planned system of activities to provide assurance that the quality control system is performing adequately.
>
> **Laboratory QA** - A program of techniques and procedures to assure that the reported results are of satisfactory utility (ASTM).

Quality assurance plan (QAP) - An orderly assembly of management policies, objectives, principles, and general procedures by which an agency or laboratory describes how it intends to produce known quality data.

Quality assurance project plan (QAPP) - A formal element of a technical work plan that describes in comprehensive detail the necessary **OA**, **QC**, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

Quality control (QC) - The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users (EPA).

Quality control chart - A graph of some measurement plotted over time or a graph of a sequence of sampling, together with statistically determined warning and control limit(s) and, usually, a central line.

Quality control sample - An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intralaboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. See also **Check sample**.

Quality System Review/Technical System Audit - A qualitative, onsite assessment and report of a laboratory (or field) operation including organization and staffing; facilities, equipment, and instrumentation; methods; recordkeeping and reporting; and implementation of system controls for assuring the quality of the final product.

Records system - A plan documenting the types of records; how they are produced, stored, and retained; and the circumstances for destruction or other disposition. See also **Document control**.

Recovery - With regard to analysis, the measured amount of a target analyte divided by the actual concentration expressed in percent (%R). The ability of the analysis system to accurately determine a true analyte concentration in a reference material sample or spiked sample is a measure of the instrument accuracy and/or the analyst's skill. Procedural error, sample contamination, analyte loss or absorption, and analysis interferences can cause other than 100-percent recovery. See also **Spiked sample**.

$$\text{Recovery (\%R)} = \frac{\text{Observed value}}{\text{True value}} \times 100$$

Reference material sample - A sample of a homogenous material with specific properties, such as purity, identity, and potency, that has been measured and certified by a qualified and recognized organization. Reference material samples are used to determine the ability of an analytical procedure to measure analyte concentrations in a specific sample material (e.g., water, soil, biota).

Relative percent difference (RPD) - A precision value which is the absolute difference between the initial and duplicate measurements of an analyte in a sample expressed as a percent of the mean of the two values.

$$\text{RPD} = \frac{\text{Sample - Duplicate}}{(\text{Sample + Duplicate})/2} \times 100$$

Replicate (for Reclamation purposes) - A second field sample taken at the same time and place as the original sample, representing the same population characteristics, and used to determine small (sampling) scale variance in the sampled population or sampling method (also known as a colocated sample); or subsequent runs of the same analytical sample used to determine analysis precision. See **Duplicate**.

Representativeness - The degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition.

Representative sample - A sample or group of samples selected from a larger population to typify the larger population, or a sample or collection of samples that answers a question about a population with a specified confidence.

Reproducibility - The extent to which a method, test, or experiment yields the same or similar results when performed on subsamples of the same sample by different analysts or laboratories. See also **Precision**.

Rinse (rinsate) blank sample - A sample of water or soil material collected by exposing contaminant free water or soil (sand) to the sampling instrument(s), following the final rinse in the cleaning process of the sample collection device. The rinsate blank sample is submitted for analysis with the associated samples. The test results from a rinsate blank provide a means of estimating the adequacy of the decontamination of the sampling device and the potential for cross-contamination between sampling sites.

Risk Assessment - Evaluating the potential for physical harm, environmental damage, and/or economic loss as a result of a condition, an action, or inaction. The evaluation is limited by the availability and quality of the data needed to perform the evaluation.

Round-robin testing - An interlaboratory program in which subsets of samples with known and/or unknown analyte concentration(s) are distributed to laboratories, analyzed, and the results evaluated to validate methodology or measure laboratory analytical accuracy.

Sample (subsample) increment - An equal (volume or weight) portion of a subsample when the subsample is created by compositing multiple equal portions from different (random/nonrandom) locations during the same sampling event.

Sample population - **The most important concept when designing a sampling plan**. The sample population must be defined before sampling occurs. The sample population is the mass/volume that will be represented (if properly sampled) by the sample(s) collected. The sample population must be defined in project terms in order to be properly sampled. If the project entails soil removal on the basis of construction zones (i.e., front yard, side yard, back yard, garden, etc.), then the sample populations should coincide with the construction zones. If you randomly sample the entire property as your population, no defensible decisions can be made from the data regarding replacement of discrete portions of the property, such as a back yard or a garden area, on the basis of the data collected.

Sampling and Analysis Plan (SAP) - A formal stand-alone document or an integral part of a QAPP that specifies the processes and defines responsibilities for obtaining environmental data of sufficient quantity and quality to satisfy the project objectives.

Sampling error - The difference between an estimate of a population value and the true value. Sampling errors are due to compositional and distributional heterogeneity. Sampling errors can be broken into six general categories: fundamental error, grouping and segregation error, materialization error (includes both delimitation and extraction errors), preparation error, trends errors, and cycle errors. **All sampling errors are selection errors (caused by observing an inadequate limited number and/or the incorrect limited number of the total possible values), except for preparation errors, which occur after sampling and before analysis.**

Screening test - A quick test for coarsely assessing the types and levels of a variable of interest. May only be qualitative (is it there?) as opposed to quantitative (how much is there?).

Sediment - Particulate matter derived from weathered rock and deposited by flowing ice or water within current or former stream channels or in the oceans, or remaining suspended in the water column. The minimum particle size is 45 microns.

SOP - See **Standard operating procedure** below.

Spiked sample - A subsample, extract, digestate, or blank to which a known concentration of analyte has been added. The spiked sample test result is compared to the unspiked sample result to determine recovery (%R) of the added analyte.

$$\%R = \frac{\text{Spiked sample concentration - Un-spiked sample concentration}}{\text{Actual concentration of spike added}} \times 100$$

Split samples - Two or more portions or subsamples derived from the physical process of properly separating (splitting) one sample into two or more samples. Cone and quartering methods and riffle splitters are widely used BUT NOT RECOMMENDED. Rotary riffling and fractional alternate shoveling are recommended. Split samples are used to compare sample preparation methods and to check analytical precision. See **Duplicate**, **Replicate**, and **Splitting samples**.

Splitting samples - The physical process by which one sample or subsample becomes two or more samples or subsamples; i.e., subsampling a sample. Fractional shoveling and vertical pipette extraction (and equivalent) are the preferred methods for soil and water subsampling. See **Fractional alternate shoveling** and **Vertical pipette extraction**.

Standard - A material or solution having a property that has been derived from a material of certified concentration or purity. Standards are used to establish or verify the response of a measuring device to a specific constituent concentration (calibration).

Standard deviation - The most common statistical measure of the dispersion or imprecision of observed values expressed as the positive square root of the sample variance.

Standard operating procedure (SOP) - A document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed (see **Protocols** above) and which is accepted as the method for performing certain routine or repetitive tasks.

Subsample - A representative (or not) portion of a larger sample or population. See also **Aliquot**, **Aliquant**, and **Split samples**.

Surrogate compounds - Nontarget compounds that are added at prescribed concentrations to all investigative and control samples and measured to verify the extraction and measurement efficiency of the organic analysis method.

Systematic sampling - The selection of samples or sample collection sites by a geometric design (e.g., lay out a square grid and sample at every node). See **Probabilistic sampling**.

Total constituent - The concentration of constituent determined by an analytical procedure which measures at least 95 percent of the constituent present in the whole sample. In a water-suspended sediment sample, the constituent present, regardless of the constituent's chemical or physical form (USGS).

Total dissolved solids (TDS) - Determined from a filtered water sample. Filterable solids at 180° C (EPA 160.1) or residue which passes a (0.45 micron, usually) glass fiber filter and remains after evaporation at 180° C. TDS is expressed as a weight of passing material (mg) divided by the total volume (liter) of the sample. See also **Dissolved constituents** and **Total suspended solids** for distinctions.

Total measurement error - The sum of all the errors that occur from the taking of the sample through the reporting of the results; the difference between the reported result and the true value of the population that was to have been sampled (EPA).

Total recoverable constituent - The procedurally defined concentration of constituent in the whole sample. In water, the concentration of constituent measured in an unfiltered sample which is heated with dilute acid to substantially reduce its volume, filtered, and diluted to its original volume (EPA).

Total suspended solids (TSS) - Determined from an unfiltered water sample. Nonfilterable solids, at 105° C (EPA method 160.2), or by Standard Methods 2540 D, 1992; also, residue which does not pass a (0.45 micron, usually) glass fiber filter. See also **Dissolved constituents** and **Total dissolved solids** for distinctions.

Trip blank - An uncontaminated sample of medium that is carried to the sampling site and transported with the samples to the laboratory without having been exposed to the sampling procedures. The results of analysis of the trip blank are used to estimate the contamination of the associated samples during transport and storage prior to analysis. Applies to QC of sampling for volatile organic compounds (VOCs).

Validation (data) - NO CONSISTENT DEFINITION EXISTS WITHIN THE QA COMMUNITY. For Reclamation work, the following definition is appropriate: A systematic process for reviewing a body of data against a set of criteria to assess the adequacy of that data for their intended use. Data (verified) validation consists of evaluating specified performance criteria (QC measures) and Data Quality Assessment (DQA) results (including determining compliance with DQOs) and results in data validated as "Accepted," "Qualified," or "Rejected." The DQA section should specify the personnel and process. Accepted and qualified (qualifications noted and assessed) data may be used for the intended purpose.

Variance (statistical) - A measure of the dispersion of a set of values. Determined by calculating 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 in the set, or the standard deviation of the set of values squared.

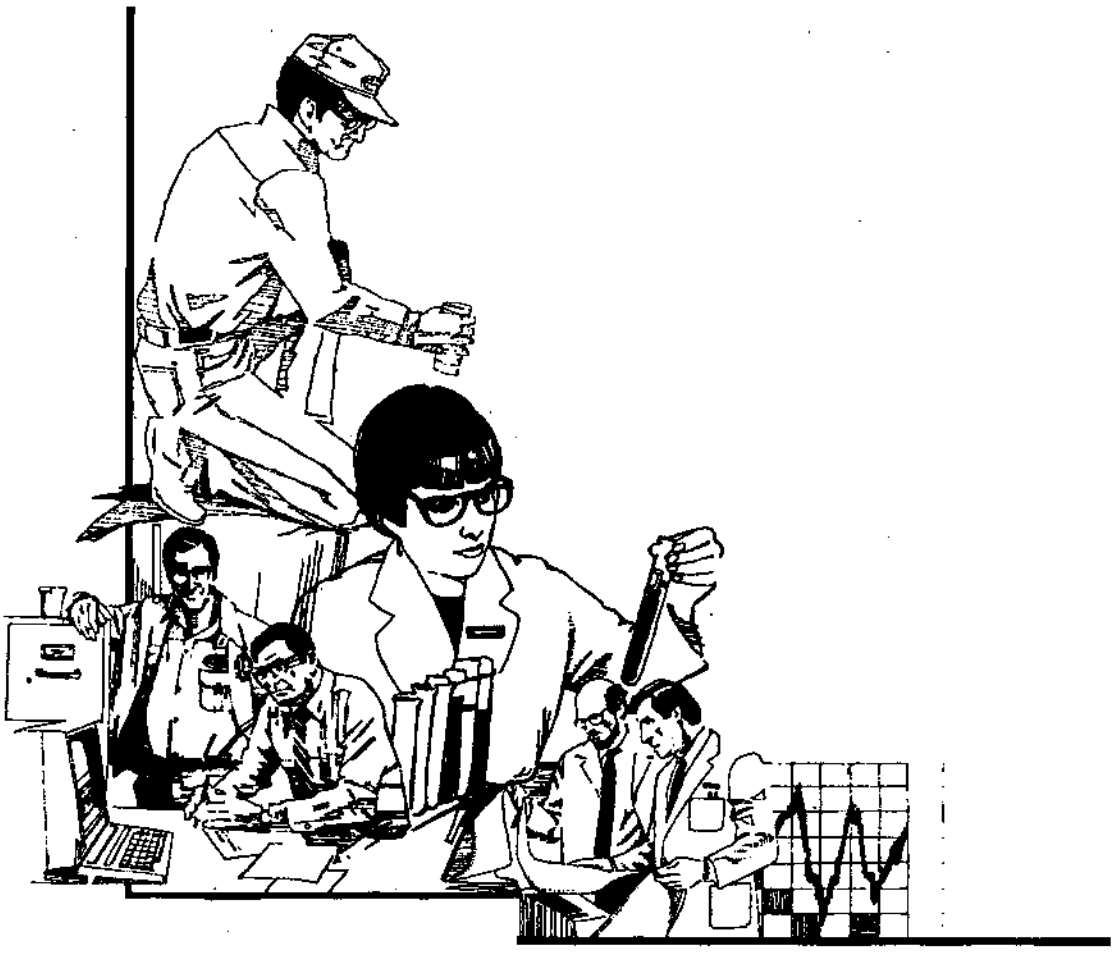
Verification (data) - NO CONSISTENT DEFINITION EXISTS WITHIN THE QA COMMUNITY. For Reclamation work, the following definition is appropriate: The initial process (prior to the validation process) of substantiating all written data records and report content. Verification includes checks for completeness, appropriateness, and accuracy. ANYTIME numbers are manually transferred or derived from a calculation, the transfer and/or equation requires a check to verify that the number was not transposed, or entered in the wrong column/space, or improperly calculated (accuracy). Checks are also required to verify that all data are accounted for (completeness) and that the data and original sample are correctly matched (appropriateness). Verified data are ready for validation.

Vertical pipette extraction - Liquid sample splitting technique that involves the extraction of split samples from a beaker or drum of original sample. Most appropriate for immiscible, density stratified liquids. The pipette (or equivalent) requires vertical insertion through the entire column to the bottom to ensure complete representation selection from the original sample. Multiple extractions may be necessary to achieve required test portion volume.

Wet weight basis - The expression of sample constituent concentration relative to the total (undried or at natural moisture content) mass of the sample.

Work plan - A general or detailed description of activities to be performed. General work plans may define the scope of services, level-of-effort, costs, and the schedule for performance. Also see **Sampling and Analysis Plan**.

INTRODUCTION



INTRODUCTION

The mission of the Bureau of Reclamation (Reclamation) often requires precise knowledge of the chemical and physical state of the environment (data) to support a variety of projects from water quality impacts of irrigation return flows to hazardous waste remediation to research on acid rain impacts on watersheds to land classification. This data requirement necessitates the measurement of environmental parameters (air, biota, soil, and water conditions; constituents; and contaminants). Reclamation projects performing environmental measurements generally must adhere to guidance document requirements for sampling, analysis, and use of environmental data and may be required to strictly observe regulatory requirements for sampling methods and sample tracking, custody, holding times, analysis, and reporting. Requirements have historically been scattered among many documents. "Quality Assurance Guidelines for Environmental Measurements" ("Guidelines") was prepared to assist Reclamation project managers, team leaders, and laboratory managers by consolidating guidance and requirements for small projects and offering references and resources for larger, more complicated investigations and studies. Adherence to the industry accepted standards of practice for environmental measurements found in "Guidelines" by Reclamation and its contractors is intended to effect well informed, defensible decisions by decisionmakers provided with known quality data from environmental measurement activities. The publication, dissemination, and recommended adherence to "Guidelines" and the designation of a Quality Assurance (QA) Manager represent the implementation of Reclamation's overall or umbrella Quality Assurance Plan (QAP).

The Objectives of "Guidelines"

"Guidelines" has been prepared and distributed throughout Reclamation to assist project managers in planning for and obtaining quality, cost-effective information from environmental measurements. Consensus-based, Reclamation-wide guidance will improve the usability of information produced by Reclamation projects and improve cooperation and communication between data providers and data users. Standards and criteria provided in "Guidelines" are intended to foster good practices and encourage their use by project managers.

"Guidelines" provides information about project planning, sample acquisition and control, laboratory selection and evaluation, data verification and validation, and records management. Recommendations are based on accepted industry standards and regulatory requirements. These recommendations can be applied to environmental measurements of air, biota, soils, water, and other materials. The quality assurance/quality control (QA/QC) practices endorsed in "Guidelines" are promoted in Reclamation project and laboratory operations by virtue of their value as standards of professional practice.

"Guidelines" encourages Reclamation's chemistry laboratories to budget for participation, by consensus, in the recommendations and processes described in "Guidelines," including outside performance sample exchanges and audits. Consensus analytical methods, specified control criteria, and regular performance reviews and performance sample audits ensure that the quality of analytical services meets the requirements of Reclamation and its clients. "Guidelines" includes a directory of Reclamation's analytical chemistry laboratories listing their testing capabilities and a management contact to facilitate communication and networking for technical support.

Quality

Quality is generally defined as the totality of features and characteristics of a product or service that bear on its ability to meet the stated or implied needs and expectations of the user. Therefore, the quality of environmental measurement data is **absolutely dependent** on the ability of the project manager or data user to define data requirements in precise quality control (QC) terms.

Quality should not be equated to perfection, unless perfection is a stated user requirement (initial expectations may require adjustment - explore early).

The argument for quality (meeting the stated needs) is simply that it is less expensive than the alternative and represents the only course for competent professionals. All the equipment, materials, and methods used by Reclamation personnel and/or Reclamation contractor personnel to produce environmental data are part of the quality equation.

Quality Assurance Concepts

Quality assurance (QA) is an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of a type and quality needed and expected by the end user (client/team leader). QA seeks to improve the consistency and reduce the uncertainty of environmental measurement data.

QA integrates Data Quality Objectives (DQOs), Standardized Operating Procedures (SOPs), and approved methodology (protocols) with written descriptions detailing these features and delineating responsibilities in a QA Project Plan (QAPP). The QAPP communicates the quality objectives and activities to everyone involved with the project and increases the reliability and completeness of the product. Routine or simple tasks may only require a brief statement of the processes and quality requirements, while complex tasks may require a comprehensive plan including resources, detailed procedures, and controls. In either case, QA must begin at the *conception* of a project and extend through the final product. QA is Reclamation's effort to maximize the effectiveness of the dollars spent, so the data we get counts, and the acquisition team is accountable.

Quality control (QC) is *not* the same as QA. QC asks if we are doing things correctly; QA asks if we are doing the correct things. QCs are operational methods and techniques, such as equipment rinsate samples and initial/continuing instrument calibration, that measure and fulfill the quality requirements of specific activities such as sampling, analysis, data reduction, and reporting in the data production process. Quality control techniques must be continuously applied and must be capable of detecting and quantifying the effects of changes in the activity. The evaluation of these effects determines the usability of the resulting information and, ultimately, the quality of decisions based on that information.

Reclamation's New Quality Assurance Program for Environmental Measurements (QA Program)

The original Reclamation QA Program was established to ensure the reliability and usability of environmental data produced by or for Reclamation's projects. The goal encompassed the entire data collection activity, from the establishment of project objectives and project design, through sample

collection and laboratory analysis, to reporting and records management. The program was structured to provide direction to project managers for QA procedures within the standards of good practice and regulatory requirements.

Under the original program, a work group composed of regional, laboratory, and Technical Services Center (TSC) staff addressed QA issues related to obtaining environmental measurements. The original program established consensus quality-system standards for all Reclamation chemistry laboratories, and laboratory operations were regularly reviewed.

Past members of the work group and update writers and reviewers are listed in the Preface. The TSC's QA manager is available to assist in coordinating the Reclamation-wide program. Quality Assurance Officers (QAOs) in each region and the TSC will assist project managers in incorporating QA practices into environmental measurement activities. Guidance for the individual responsibilities in environmental data collection activities is in Part 3 - General Guidance.

The new Reclamation QA program for environmental measurements seeks to build on past practices and philosophy within the current framework of the Government Performance Review Act, reorganization, and lack of program fiscal support. Reclamation recognizes the importance of QA practices but can no longer support the program at previous levels. Instead, QA has become the responsibility of each employee. Reclamation staff have received, and will continue to receive, training on project QA and sampling procedures. Laboratory managers are encouraged to budget for, and continue with, the performance reviews, internal audits, and other program measures that directly promote quality products and maintain current standards of professional practice. The new Reclamation QA Program consists of the publication of "Guidelines" and reliance upon individual employee and supervisory commitment to, and responsibility for, assured quality environmental measurement products.

"Guidelines" Content Selection Criteria and Update Process

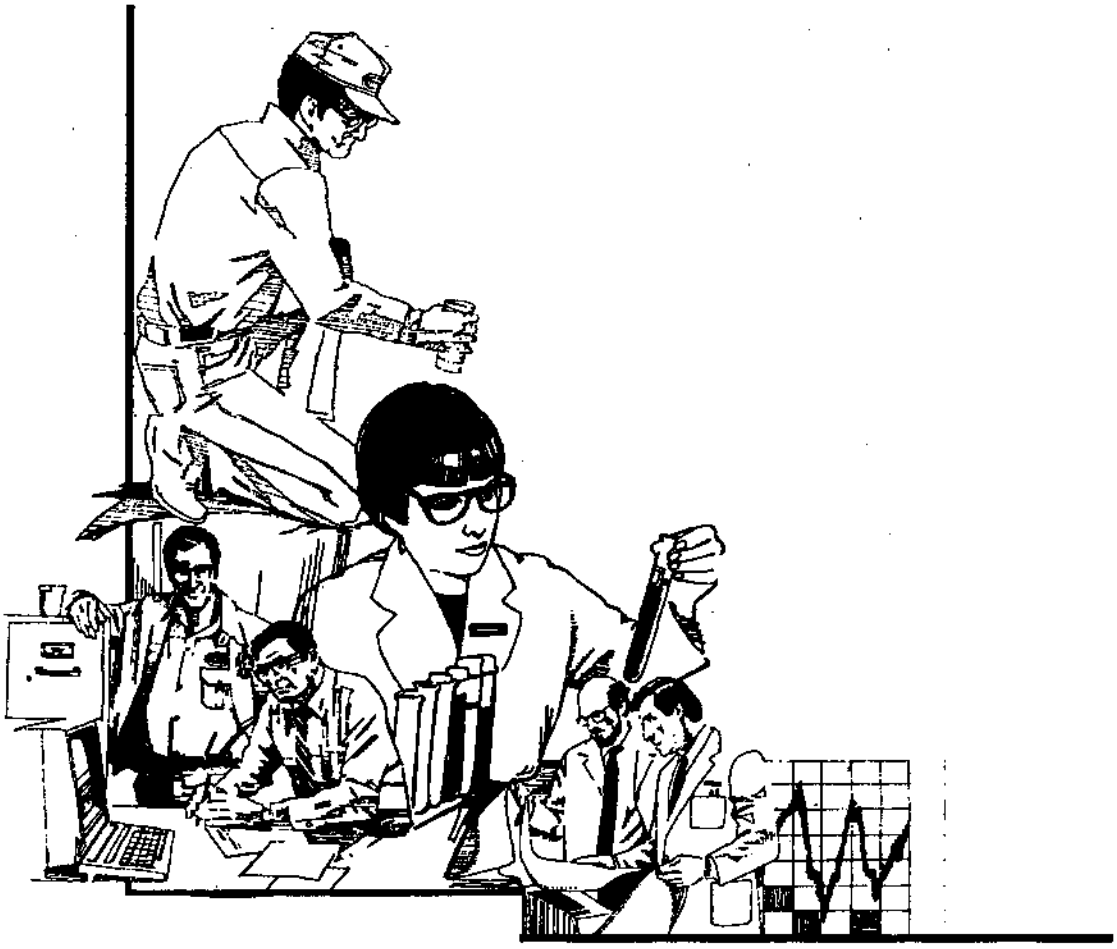
Analytical capabilities, regulations, and project needs are constantly changing. As improvements in QA practice occur, "Guidelines" will be revised. Reclamation's laboratory supervisors and investigators will be informed of new consensus practice by written revisions to the "Guidelines" and peer communication with the regions and QA officers.

Suggestions and comments about "Guidelines" may be made to the Quality Management Services Division of the Client Business Services Office, Mail Code D-8010, Bureau of Reclamation, PO Box 25007, Denver, Colorado 80225.

This version of "Guidelines" replaces the August 1998 edition.

"Guidelines" is divided into four parts. The first part is devoted to guidance on field measurements and sampling, the second part is devoted to guidance on Reclamation laboratory QAP content, the third part contains general QA guidance information, and the fourth part contains the methodology of field sampling. All parts end with part-specific references and/or bibliographies.

PART 1 - GUIDANCE



PART 1 - GUIDANCE

Part 1 contains outlines, information, and references related to the field activities that generally produce the samples for laboratory analysis. Part 1 is arranged from the broadest perspective, the overview, down through the outlined subcategories of interest. Whenever an outline precedes a new section or sections, the new sections have been underlined in the preceding outlines to indicate the position of the following sections within the process as a whole. This is done to provide the user with a logical vision of the way the pieces fit together into a complete plan for the acquisition and delivery of samples of known quality to the laboratory for analysis.

Field guidance begins with an outline of the Quality (Assurance) Management Plan (QMP). The QMP is the overall vision of any project that includes a data acquisition function in order to satisfy a design data requirement or a decision data requirement.

Following the QMP outline, is the Quality Assurance Project Plan (QAPP) outline. The creation of a QAPP is (should be) the FIRST order of business for any new project, following scoping and funding. The elements of the QAPP are listed under four headings: Project Management, Measurement / Data Acquisition, Data Acquisition & Analysis Assessment / Oversight, and Data Verification and Validation (Usability Determination). Beneath the Project Management heading is the subheading, *Data Quality Objectives* (DQOs). While the other subheadings are generally self-explanatory, DQOs (as indicated by the underline) are the subject of the next section. Identification of DQOs is the primary task of the project manager and those who assist the project manager. Once the data quality objectives are clearly defined, the subsequent tasks are effectively constrained.

The *Sampling and Analysis Plan* (SAP) is developed after the DQOs are defined. Some guidance for the development of the SAP (as indicated by the underline in the QAPP outline) is the subject of the section following the DQO section. For small projects, the SAP may be the bulk of the project plan, but ignoring the other elements is usually a mistake.

Data Acquisition & Analysis Assessment and *Data Verification & Validation* (as indicated by the underline in the QAPP outline) sections follow the SAP section. These sections offer advice on planning for assessing the samples collected and validating their associated data. This involves a determination of the usefulness of the data for its intended purpose and ultimately a determination of the success of the field effort.

Reclamation's intent is that a project manager can be reasonably confident that the field effort will yield the data he needs to make the decision, or go to design, after preparing a QAPP following the guidance here in Part 1, before a single sample has been collected and submitted to a laboratory for analysis, if everything occurs according to the QAPP and any revisions thereto.

Outline of Quality Assurance Overview

Quality (Assurance) Management Plan (QMP)

- Project
  - Scoping
    - Goals and Objectives
    - Cost Estimate

  - **Quality Assurance Project Plan (QAPP) [EPA QA/R-5]**
    - **Project Management**
      - **Data Quality Objectives (DQOs) [EPA QA/G-4]**

    - **Measurement / Data Acquisition**
      - **Sampling and Analysis Plan (SAP)**
        - **Sampling QA/QC Plan**
          - **Sampling SOPs**
        - **Analysis QA/QC Plan**
          - **Analysis SOPs**

    - **Data Acquisition & Analysis Assessment / Oversight**
      - **Field Compliance Audit**
      - **Laboratory Compliance Audit**

    - **Data Verification and Validation (Usability Determination)**
      - **Data Quality Assessment (DQA) [EPA QA/G-9]**

  - Site Safety and Health Plan (SSHP), as needed, or required by 29 CFR 1910.120

  - Investigation (Air/Biota/Earth/Water)
    - Environmental Sampling
    - Sample Analysis
    - Data Reduction and Validation
    - Report

  - Design/Decision

  - Construction/Action

The development of subitems should be appropriate to the level of effort of the project. Avoid unnecessary duplication. EPA guidance documents provided in brackets - if sampling is for EPA, you MUST follow their requirements (reference EPA QA/G-5).

Outline of Quality Assurance Project Plan (QAPP)

Project Management
- Title, and approval sheet (if required)
- Table of contents
- Project / task organization *(*roles and responsibilities*)*
- Problem definition / background
- Project / task description
- **DQOs for measurement data *(see next section)***
- Schedule *(is there time to gather / process all needed data?)*
- Resource issues *(is manpower / equipment available? - access rights?)*
- Cost tracking (EPA may require a particular system and daily cost accounting)
- Communication of QAPP to, and coordination of QAPP with organization & lab

SAP (Data Acquisition / Measurement) [goal is to minimize error potential]
- Sampling and Analysis Plan (SAP)
- Sampling methods / requirements *(*probabilistic, judgmental, opportunity / grab, composite, etc.*)*
- Sample handling / shipping method / tracking / custody requirements *(minimize manual data transfers with preprinted labels and forms)*
- Analytical methods / requirements
- Quality control requirements *(blanks, duplicates, spikes, reference standards, etc.)*
- Instrument calibration and frequency (field / lab)
- Data report method / format / deliverables

Data Acquisition and Analysis Assessment / Oversight
- Activities (field / lab audit) to assess compliance of implemented activities with SAP, and reconciliation methods *(QA officer to perform if required)*

Data Verification and Validation (Usability Determination)
- Data Quality Assessment (DQA)
  - Data review, verification, and validation requirements
  - Verification and validation methods *(EPA credible data/other, data reduction equations used, data transfer checks, referee laboratory)*
  - Reconciliation with DQOs
    - State how limitations on data use and data qualifiers will be reported
    - State how results of assessments and data evaluations may be considered in decisionmaking *(qualified ("bad") data may be useful - if the problems with the data are understood, proper judgment can be applied to their use)*
- Data Usefulness (Accept, Qualify, Reject)
  - Can the data be used to make/support the decision
    - DQOs met
    - Reconciliations acceptable

DATA QUALITY OBJECTIVES (DQOs)

Data quality objectives (DQOs) are qualitative and quantitative statements which specify the quality of data needed to support decisionmaking. Determining DQOs should be the first step in initiating any significant data collection program. In large programs, separate DQOs may be needed for phases, by media type, or for special sampling efforts.

The DQO process is a series of planning steps that are designed to ensure that the type, quantity, and quality of environmental data used in decisionmaking are appropriate for the intended application. The DQO process requires that project managers determine limits on decision errors so that acceptable goals for the quality of the necessary data can be established.

Once DQOs have been determined, a well-defined SAP is prepared which includes the appropriate quality elements in the data collection program. Once the data collection activity has been well described, the DQOs assist the decisionmaker in assessing whether the data to be collected by this plan will be suitable for the defined purpose.

DQOs are determined by clear statements of the following:

- decision(s) to be made
- questions to be answered before the decision(s) can be made
- specific data collection activities / tests planned / analysis methods planned
- intended data uses / decisions for each measurement activity / test
- quality and type of data needed to support intended uses / decisions
- identity of data users / decisionmakers
- applicable regulatory or other standards / permits needed
- physical and temporal study boundaries / units
- applicable reference levels / standards and decision rules (action levels / regulatory limits / permit requirements)
- identification of tolerable limits on decision errors (if any)
- form of data used for decisionmaking (e.g., compare average to standards / permits)
- accuracy, representativeness, completeness, and comparability objectives for each measurement activity / test

---

Communication of DQOs between data providers (field samplers and laboratory analysts) and data users (regulators and decisionmakers) in project planning is essential to meeting project data collection objectives. This is a key concept which must be understood by everyone involved in the process.

Potential Stages of a DQO Process

A DQO process is outlined in figure 1. It consists of three stages with several steps in each stage. Stages I and II result in statements of the program objectives and requirements of the data collection activity. Stage III results in a fully documented data collection design that meets the requirements determined in Stages I and II.

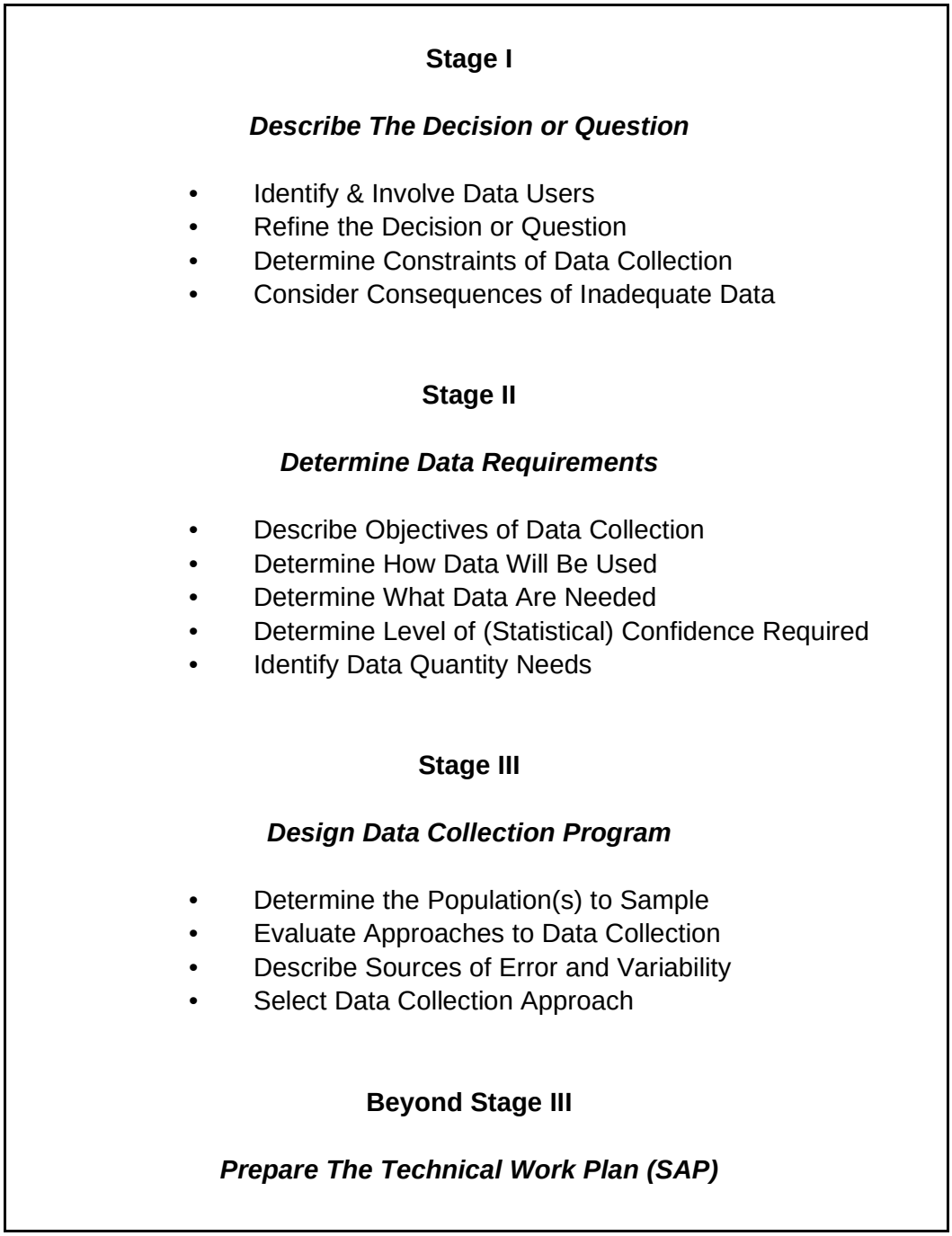


Figure 1.—A DQO three-stage process.

Stage I: Presentation of the Question or the Decision to be Made

In Stage I, the decisionmaker describes his/her perceptions of what information is needed and why, how the information will be used, and the consequences of obtaining inadequate or wrong information. Initial decisions regarding the time and resources available to accomplish the decisionmaking activity are made. The level of detail with which these concepts can be discussed will depend on how well the problem or question can be defined at this time.

Stage II: State the Requirements of the Information Needed

In Stage II, the senior staff or team consisting of both management and technical personnel (with periodic involvement of the decisionmaker) examine the question in detail to determine the specific requirements of the data collection activity. Qualitative and quantitative statements are then written that describe the needed quality control measures for the data. Discussion of issues such as the following will aid in clarifying the initial concepts:

- What ultimate products or actions are anticipated, and what decisions will be made? (Products might be specific regulations or standards; actions might include litigation, issuance of water service contracts, or certification of irrigation suitability.)
- What is the role of data collection in making the decisions?
- What will be the impact on the decision if inadequate or inaccurate data are collected? What is the statistical confidence required in the decisions?
- What criteria exist for making decisions based on conclusions from the data (e.g., action levels, agency policies, or previous experience with similar questions)?
- What alternative sources of suitable data exist? Are new data required?
- Will the collected data be applied to other similar decisions or, by inference or extrapolation, to sites not in the sample? Are new data required to be comparable with existing data? (This factor may substantially influence the sample size and the selection of sampling sites.)

Clear statements of the objectives of the data collection activity are carefully described for use by the technical staff in Stage III. These statements will prescribe the consideration and evaluation of various approaches to collecting data which will meet the given constraints.

Stage III: Design the Data Collection Program

The technical staff is primarily responsible for designing and preparing a technical work plan, but management and the decisionmaker are also involved in ensuring that the requirements stated in Stage II are clearly understood. The objective of Stage III is to develop a data collection plan that meets the criteria and constraints determined in Stage II.

Data collection options are evaluated with respect to sample population, technical feasibility, time and cost requirements, usability of data, and associated limitations. Field (sampling network design and sample collection procedures) and laboratory activities (sample handling, analytical procedures, and data manipulation) have a direct impact on the selection of an optimum data collection design. The design team selects the data collection option that provides the best balance between time and resource constraints and the statistical level of confidence required by the program objectives.

An example will illustrate the tradeoffs involved: The confidence level of an estimate depends on the number of samples collected and analyzed. Decreasing the level of confidence in an estimate allows cost and time savings because fewer samples are collected and analyzed, but it increases the risk of incorrectly deciding to take some action based on the estimate. Oversampling may consume resources needed in other project phases without much value added.

In developing alternative approaches to data collection, the technical staff will need to analyze options for each data collection step in terms of the tradeoffs described above. In order to perform this analysis, the staff must have information on the amount of variability expected, the capability of present methodology to measure those characteristics, and the types of error to which the measurements are subject. The staff's ability to analyze options properly and thoroughly will depend on adequate information. If it is not available, professional consultation, specific pilot studies, or new method development may be required prior to designing the full-scale study or plan. Evaluating the elements of data quality in terms of accuracy (precision and bias), representativeness, comparability, and completeness for each data collection option allows the staff to determine the overall data quality that can be expected from each approach. The staff presents the alternative approaches to the decisionmaker in an objective, documented way, characterizing each approach in terms of the factors being balanced: the time and resources required for data collection versus data quality and the risk of being incorrect. Given this information, the decisionmaker can select the approach to be used with substantial knowledge of the expected quality and utility of the resulting data.

Beyond Stage III: Detailed Design of the Data Collection Activity

The detailed design process is primarily the responsibility of the technical staff. At this point, the *extensive iterations* between technical staff and decisionmaker have been essentially completed, and the staff has enough information to complete the planning and prepare the technical work plan. However, if planning or pilot studies reveal unanticipated problems that will affect data quality, adjustments in the study design may be required. The implications of those changes must be discussed with the decisionmaker by the technical staff. If serious problems arise, it may be necessary to return to Stage III and change both the approach and the data quality objectives.

The technical work plan, also called the Sampling and Analysis Plan, documents the tasks to be performed and specifies the procedures to be used in collecting samples and performing analyses for environmental measurements. The work plan includes the QA/QC elements necessary to ensure that the project objectives will be met.

Summary

In the preparation of technical guidance for data collectors, DQOs are used to develop explicit qualitative and quantitative statements of the sources and types of errors that will be controlled, the

level to which those errors will be controlled, and the information that will be collected to characterize all known sources of error. These statements are known as *data quality indicators*. They are needed to select appropriate methods for sample collection, laboratory analysis, and statistical analysis.

DQOs are a planning tool, a thought process, a methodology, a basis for negotiation, and a contractual obligation. *DQOs are ultimately the basis for ensuring the suitability of data for the intended purpose*.

BIBLIOGRAPHY

EPA, *Interim Final Guidance on Data Quality Objectives Process For Superfund*, Publication No. 9355.9-01, Office of Emergency and Remedial Response, Washington, DC, September 1993.

——, *The Volunteer Monitor's Guide to Quality Assurance Project Plans*, Office of Wetlands, Oceans and Watersheds, EPA-841-B-96-003, September 1996.

——, OSWER Directive No. 9355.9-02, September 30, 1993.

SAMPLING AND ANALYSIS PLAN
(SAP/TECHNICAL WORK PLAN)

The DQO process can be considered to be the preliminary step in the development of the technical work plan, also called the Sampling and Analysis Plan (SAP). The SAP is the document which specifies the task and provides the technical procedures to be used in collecting samples and performing analyses for environmental measurements so that the quality objectives determined in the DQO planning process are met.

Figure 2 lists the elements of a SAP. These elements should be described in sufficient detail to demonstrate that:

- The intended measurements or data acquisition are appropriate for achieving the project objectives.
- Assessment procedures, including QA/QC, are sufficient for obtaining data of the type and quality needed and expected.
- Any limitations on the use of the data are known and documented.

Items numbered 4 through 7 are topics covered in subsequent sections of this or the remaining parts.

The QA/QC elements of the SAP specify the required processes for controlling the quality and documenting field and laboratory activities performed in conjunction with the SAP. Some of the QA/QC elements are incorporated into specific technical performance activities of the work plan. Other elements, such as project management, will relate to the entire task activity.

It is important that either all QA/QC elements be addressed in the SAP or a brief statement of the reasoning be provided explaining why an element is not applicable.

1. Introduction
   - Project summary and objectives
   - Information requirements
   - Scope of plan
   - Rationale for the data collection process
   - Use of data collected
   - Constraints

2. Site Description
   - Site map
   - Previous uses
   - Structures
   - Hazards

3. Previous Investigations
   - Summaries and conclusions
   - Existing sampling sites
   - Use of historic data in SAP development

4. Sample Collection Design
   - Safety (Perform a hazard evaluation and determine appropriate mitigation measures to minimize exposures.)
   - Sampling theory (population, number of samples, method, type)
   - Sampling Method (random/probabilistic, judgment, opportunity)
   - Sample types (grab, composite)
   - Media to sample (air, water, rock, soil, sediment, etc.)
   - Collection schedule (conflicts?, alternatives, weather impacts)
   - Sampling site locations (map / description)
   - Preparation for sampling (preprinted labels, supplies, schedule samplers, access?)
   - Equipment required (list, available?, operational?)
   - Field tests to perform (frequency, calibration, SOP)
   - Field testing procedure references (Include the standard operating procedure (SOP) for each type field measurement in an appendix.)
   - Sample collection reference (include an SOP for sampling each sample type - filtering requirements / preservatives, etc., in an appendix.)
   - Shipment of samples (COC, log, schedule, Friday OK?, Fedex?, SOP.)

5. Sample Analysis Design
   - Samples to analyze
   - Constituents of interest
   - Sample preparation references (Include the SOP or method for each preparation by sample type in an appendix.)
   - Analysis procedure references (Include the SOP or method for each analysis of each constituent by sample type in an appendix.)
   - Specify the initial QC level / requirements needed to meet the DQOs
   - Specify laboratory to be utilized (name, receiving address, contact name, and telephone number)
   - Specify sample transit conditions (intact, preservatives, temperature)
   - Specify holding times (extraction / analysis)

6. Data Validation (QA officer function)
   - Verify sample receipt condition (intact, preservatives, temperature)
   - Verify holding times met
   - Audit results (blanks, duplicates, replicates, recovery %, performance evaluation samples, etc.)
   - QC results (equipment calibrations error determination, standards, CCV)
   - Additional instrument QC
   - Overall assessment of data
   - If EPA - Use their requirements
   - Option - Use a referee laboratory and minimize performance sample submission to the contracted lab

7. Data Analysis
   - Estimate total error
   - Statistical evaluation

[1] Sections 1, 2, 3, and 6 are unnecessary when the SAP is an integral part of a QAPP (as it should be).

Figure 2.—Elements of a Stand-Alone Sampling and Analysis Plan[1]

SAMPLE COLLECTION DESIGN - SAMPLING THEORY

Incorrect sampling design yields poor sample collection. Poor sample collection yields samples that are not representative of the population of interest, are of little use, seriously compromise the purpose of sampling, and contribute to the uncertainty of the analytical results. Furthermore, sampling and analytical errors occur independently of each other, so sampling-related errors cannot be accounted for by laboratory blanks or control samples. There are seven potential sources of major sampling error (fundamental error, grouping and segregation error, two materialization errors, preparation error, trend error, and cycle error) that will be discussed later in this section.

This section discusses **planning, performance, and control of field sampling** activities based on the principles of sampling summarized by the American Chemical Society (ACS) Committee on Environmental Improvement and others. Since data may be used for purposes other than initially planned, guidelines for **evidentiary documentation** of sampling suggested by the EPA National Enforcement Investigations Center are included. **References** and a **bibliography** specific to the guidance in this section follow the text. Figure 3 is a **"Chain of Custody Record**" form (form E7-2518) which may be used to record possession and transfer of critical samples.

A looseleaf **compendium** of 39 articles, guidance, procedures, and a bibliography was prepared in 1994 (but not currently maintained) to provide project managers with specific information about how to sample environmental materials. The compendium entitled **"Sampling Guides**" is organized into the following sections: **Planning** for Sampling; **Surface Water** Sampling; **Soils** Sampling; **Sediment** Sampling; **Groundwater** Sampling; **Air** Sampling; and **Waste** Sampling. Some sections and references may still contain useful information. The compendium - or copies of sections - is available from the Denver Technical Service Center Quality Assurance Manager or from the regional and area office staff listed in the Acknowledgments.

Planning

DQOs provide information about the confidence required in the conclusions drawn from the data produced by the whole project. The objectives determine the total degree of variability that can be tolerated in data. The sample plan must clearly reflect the **stated objectives** of the sampling effort and the quality of data required. The **limits of variability** are incorporated into the sampling and analysis plan and must be achievable by using detailed sampling and analysis protocols.

Planners should resolve the **allowable sampling error** relative to the total project error. Sampling error may be reduced through proper selection of sampling methods, types, and devices; field audits; training; and strict adherence to protocols. Early detection of procedural errors may allow resampling with minimal cost impact. In planning, determine the **method of analyzing the samples and analyzing the data from sampling** because the analysis methods will affect, and be affected by, the objectives and design of the sampling program. The types and numbers of quality control samples to be taken will depend directly on the nature and importance of the errors to be assessed and their impact on the confidence in the decision.

The sampling plan needs to balance the desired DQOs with other factors, such as **time and resources** available. Planners must also recognize the possibility that data and conclusions resulting from sample collection may be used for purposes other than those originally envisioned. **Involve the data users with the samplers and analysts early** to increase perspectives and avoid restricted sampling programs that prohibit otherwise unrecognized wider data use potential.

In the **sampling plan**, describe the location and timing of sampling, type of samples (including QC), analytes to be measured, recordkeeping requirements, and shipping information. Restate these in the sampling and shipping protocol(s) with the reasons for the decisions.

Trained and experienced samplers who are involved in the planning stage can assist the planner and better support the collection of good samples.

Safety

The act of collecting samples and taking measurements for environmental investigations exposes personnel to safety and health hazards. **Sources of risks** include the physical environment, equipment, and chemicals used in measurements and sample preservation. Describe the hazards to sampling personnel and require that procedures to reduce accidents and exposures be included in the sampling plan.

If work will be performed within the boundaries of a recognized or suspected hazardous waste site, Occupational Health and Safety Administration (OSHA) training, medical surveillance, and documentation (Site Safety and Health Plan, medical records, training records) requirements per 29 CFR 1910.120 must be understood and complied with.

Sampling Protocols

Sampling protocols are detailed **written procedures** to be followed for sample collection, handling, storage, and documentation. Specific protocols, when adhered to, control errors and minimize incomplete data.

The plan should identify the sampling locations and all **information needed for sampling** in protocols, including:

- types, numbers, and sizes of containers
- labels or tags
- field logs
- sampling devices
- numbers and types of blanks, sample splits, and spikes
- volumes of sample
- specifics on compositing samples
- **preservation** instructions and **holding times** for each type of sample (see table 1)
- field preparations and measurements
- **timing**
- the format of **reports**

Table 1.—EPA Recommended Preservation Methods and Holding Times for Water and Wastewater Samples[1]

Constituent	Preservation method	Container[2]	Maximum holding time
Acidity/Alkalinity	Store at 4 °C	P,G	14 days
Ammonia	H_2SO_4 to pH<2, Store at 4 °C	P,G	28 days
Arsenic	HNO_3 to pH<2	P,G	6 months
BOD	Store at 4 °C	P,G	48 hours
COD	H_2SO_4 to pH<2, Store at 4 °C	P,G	28 days
Chloride	None	P,G	28 days
Residual chlorine	None	P,G	Analyze immediately
Chromium, hexavalent	Store at 4 °C	P,G	24 hours
Cyanide	NaOH to pH>12 0.6 g ascorbic acid[3] Store at 4 °C	P,G (amber)	14 days (24 hours if sulfide present)
Dissolved oxygen	None	G	Analyze immediately
Fluoride	None	P	28 days
Inorganic metals[11]	HNO_3 to pH<2	P,G	180 days
Mercury	HNO_3 to pH<2	P,G	28 days
Nitrate	Store at 4 °C	P,G	48 hours
Nitrite	Store at 4 °C	P,G	48 hours
Nitrate + nitrite	H_2SO_4 to pH<2, Store at 4 °C	P,G	28 days
Oil and grease	H_2SO_4 to pH<2, Store at 4 °C	G	28 days
Total organic carbon	H_2SO_4 to pH<2, Store at 4 °C	P,G	28 days
pH	None	P,G	Analyze immediately
Phenolics	H_2SO_4 to pH<2, Store at 4 °C	G	28 days
Ortho-phosphate, total	Store at 4 °C	P,G	48 hours
Phosphorus, total	H_2SO_4 to pH<2, Store at 4 °C	P,G	28 days
Residue, filterable and nonfilterable	Store at 4 °C	P,G	7 days
Selenium	HNO_3 to pH<2	P,G	6 months
Silica	Store at 4 °C	P	28 days
Specific conductance	Store at 4 °C	P,G	28 days
Sulfate	Store at 4 °C	P,G	28 days
Sulfide	Add zinc acetate/NaOH to pH>9, Store at 4 °C	P,G	7 days
Kjeldahl nitrogen	H_2SO_4 to pH<2 Store at 4 °C	P,G	28 days
Turbidity	Store at 4 °C	P,G	48 hours
Purgeable Hydrocarbons	HCl to pH 2 [6]Store at 4 °C	G, Teflon-lined septum	14 days

Table 1.—EPA Recommended Preservation Methods and Holding Times for Water and Wastewater Samples[1] - continued

Constituent	Preservation method	Container[2]	Maximum holding time
Purgeable aromatic hydrocarbons	[6]Store at 4 °C	G, Teflon-lined septum	14 days[7]
Phenols[8]	[6]Store at 4 °C	G, Teflon-lined cap	7 days until extraction/ 40 days after extraction
PCBs[8]	[6]Store at 4 °C	G, Teflon-lined cap	Same as above
Phthalate esters[8]	[6]Store at 4 °C Store in dark	G, Teflon-lined cap	Same as above
Nitrosoamines[8,9]	[6]Store at 4 °C Store in dark	G, Teflon-lined cap	Same as above
Nitroaromatics and Isophorone[8,9]	[6]Store at 4 °C Store in dark	G, Teflon-lined cap	Same as above
Polynuclear aromatic hydrocarbons[8]	[6]Store at 4 °C Store in dark	G, Teflon-lined cap	Same as above
Chlorinated hydrocarbons[8]	[6]Store at 4 °C	G, Teflon-lined cap	Same as above
Pesticides[8]	[10]Store at 4 °C, pH 5-9	G, Teflon-lined cap	Same as above

Notes:

[1] Source: 40 CFR 136, July 1, 1990.

[2] P = High density polyethylene; G = Glass.

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

[4] Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment 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.

[5] For samples of nonchlorinated drinking water supplies, concentrated H_2SO_4 should be added to lower sample pH to <2. The sample should be analyzed before 14 days.

[6] If residual chlorine is present, 0.008-percent $Na_2S_2O_3$ should be added.

[7] Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

[8] When the extractable analytes of concern fall within a single category, the specified preservative and maximum holding times should be observed. When analytes fall within two or more categories, the sample may be preserved by cooling to 4 °C, reducing the residual chlorine with 0.008 percent sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction.

[9] For the analysis of diphenynitrosoamine, add 0.008-percent $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[10] 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 aldrin, add 0.008 percent $Na_2S_2O_3$.

[11] Al, Ag, Au, Cd, Cr, Cu, Fe, Pb, Mg, Mn, Sr, Vn, Zn

Specific protocols also need to describe the **documentation** requirements for site observations, calibrations and equipment checks, variables to be measured at the time of sampling, chain-of-custody, and sample transportation (electronic notes, logbooks, COC forms, etc.). The plan, or specific protocols, should also address **responsibilities** of personnel for samples, equipment, and records.

In sampling information, the plan should include the requirements of the **analytical methods** (sample volumes, preservation), sensitivities, and limitations (detection limits, precision) to help samplers make better decisions when unforeseen circumstances require changes in the sampling protocol.

Sampling Design

Much of the information presented here is from the seminar "Defensible Environmental Decisions" (Ramsey, 1998). There are two basic criteria for proper sampling: how to collect the sample and how much sample material (mass) to collect. It is also helpful to keep in mind that **all** SAMPLING ERRORS are the result of heterogeneity in the environment. There are two types of heterogeneity: compositional and distributional. Compositional heterogeneity (applied only to the analyte of interest - refers to the difference in composition of the particles that make up the population) leads to sampling fundamental error (FE), which is the only error that can be estimated before sampling from the equation

$$FE^2 = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] cfgd^3, \text{ or } FE^2 = \frac{clfgd^3}{M_s}, \text{ for } M_L >> M_S$$

but CANNOT be **eliminated** (see Pitard 1992 for a discussion of variables and application — FE should be kept below 17 percent and consistent with DQOs). For a given sample mass and state of comminution, the fundamental error is the smallest possible sampling error – even if everything is done perfectly. Distributional heterogeneity is the nonrandom distribution of particles in time and space (segregation), where those particles may have different sizes, shapes, and concentrations, causing grouping and segregation error (GSE).

A good sampling plan or design properly addresses both forms of heterogeneity and minimizes sampling errors when required by the DQOs. A good plan controls compositional heterogeneity, the source of data variability, by the size (mass) of the sample, and distributional heterogeneity by randomization (many random samples through the heterogeneity) and provides representative samples resulting in statistically normal data that can meet DQOs and data requirements.

The sampling plan or design must be based on the population to be sampled and the data analysis to be employed. The population(s) should be determined prior to or during the DQO process on the basis of the project data requirements. Sampling the wrong population properly provides no useful data for decisionmaking. If the decision to be made on the basis of the data collected is whether or not to remove contaminated soil, the designer needs to know the areal extent (population) of the potential removal. If soil will be removed on the basis of small construction zones in a residential neighborhood (i.e., front, back, and side yards), these discrete areas need to be individually sampled. If garden areas are to be treated separately, they must be sampled separately. If the decision will be to remove **all** contaminated soil, then the soil profile from surface to the molten mantle interface will

need to be sampled. If more reasonably, up to the top 2 feet of contaminated soil will require removal, the sampling plan needs to address samples representing the top 2 feet of each construction zone. But if the removal will occur in two stages, the first 12 inches (if contaminated), and then the next 12 inches (if contaminated), the sampling plan must discretely sample both layers (not necessarily at the same time) to make the correct decision about removal. Other questions that need resolution include:

- Does soil under cement flatwork count?
- Is organic material included?
- What particle size should be collected?
- Will plants and trees require removal?

Until the data requirement population(s) is/are specified in great detail in the sampling plan, sampling should not begin.

The data analysis method should also influence the data collection design. If the mean, standard deviation, median, or other normal statistical parameters are of no interest, or if the contaminant distribution is known to be non-normal (poisson, bimodal, binomial, log-normal), there may be no need to acquire large size samples in an attempt to control variability. If only the limits of surficial contamination or maximum contaminant levels are needed, a nonrandom systematic or judgmental approach may be most appropriate. But if the decision will be made on the basis of the (statistical) mean contaminant level of the population, statistically normal and random data will be needed for a legally defensible decision.

Sampling a population may be accomplished by **probabilistic** (random/statistical/systematic) methods, **nonprobabilistic** (systematic/judgmental/authoritative) methods, or through phased combinations of each method (see Glossary of Terms). The data objectives, costs, allowable error, legal requirements, and professional judgment will determine an appropriate approach. You should consider prior knowledge about the site characteristics, analyte concentrations, medium (water, soil, sediments, biota, air, wastes), and transport mechanisms in your sampling approach selection. Generally, probabilistic sampling is the preferred method because, when properly executed, probabilistic samples allow inference to the whole population characteristics (random samples provide normally distributed data that are scientifically and legally defensible), as opposed to nonrandom sampling that provides information about only the samples themselves, with no population inference potential. Systematic sampling, such as sampling at regular intervals along a line, is useful for targets that are spatially evenly distributed or for determining the areal extent of contamination. Judgmental methods may work best to identify “hot spots” for analytes of concern that have special characteristics that cause concentrated deposits related to known physical conditions or settings or provide sensory faculty clues (odors, surface staining, unique taste, temperature difference).

Samples may be specified within a collection design method (probabilistic or nonprobabilistic method) as either composite for probabilistic sampling or grab (discrete) for probabilistic or nonprobabilistic sampling. Individual grab samples are representative of only the sample (matrix and medium) itself at the time and location of the sample; **no** statistical population inferences are warranted or legitimate for individual or a statistically insignificant number of grab samples. Composite samples are composed of two or more discrete increment samples. Samples may be composited spatially (x/y/z) from different locations, elevations, and/or strata, and/or temporally

from different times. Composite data are useful for providing qualitative and quantitative data economically when the average population property is desired. After selection of the method and type of samples to collect, the next consideration is the choice of sampling equipment.

Due to potentially large materialization errors (ME), the selection of the proper sampling tool or instrument is very important. Materialization error manifests itself as either improper sample "shape" extraction (delimitation error (DE)), due to the exclusion of some particles because of their size or shape, or poor extraction (extraction error (EE)) due to loss of some portion of the "ideal sample shape." Random selection requires equiprobable selection of all particles in the sample population. If particles larger than 1-inch diameter are not considered part of the sample population, they could be excluded by the sampling device by choosing a device with an opening that excludes 1-inch particles and above. However, if particles below 1-inch diameter that are considered part of the target population are also excluded due to plugging by larger particles (may be better to collect and hand sort out larger fraction), materialization errors will adversely affect the data results. Generally, round-opening sampling devices should have an opening three times (or more, depending on plugging potential) the size of the maximum diameter of any particle of interest. Always consider the sample population characteristics when determining the proper sampling device or instrument (shape and size), with equiprobable selection in mind, and always maintain the sample integrity for full sample extraction. Ease of sampling device or instrument decontamination should be the other selection criteria. Generally, disposable is best, all other considerations being equal (if you are using disposable plastic spoons to collect samples in zip-lock bags, include the spoon in the sample bag for later use).

Final sampling plan considerations are how many samples to take and how much mass per sample is needed. In many cases, the number of samples to take is dictated by the analytical budget divided by the laboratory cost per sample analysis - this is the wrong approach. Through the use of composite sampling and the correct identification of the sample population, the number of samples requiring analysis can be reduced while better representing the population characteristics through increased sample mass. EPA method SW-846 suggests that the correct number of samples to take is defined by

$$n = \frac{t_{.2}^2 s^2}{\Delta^2}$$ ($t_{.2}$ = two sided students t value for n-1 degrees of freedom, s = sample standard

deviation, and Δ = difference between regulatory threshold and mean), which assumes a normal distribution, requires an estimate of the mean and variance, and is very sensitive to variability. A better estimate for composite samples is **at least** 10 increments per sample, with each sample representing a well defined population, to "ensure" that the GSE is less than the FE, and **at least** 30 increments per sample to reduce the grouping and segregation error to a point where its contribution to total sampling error is not significant. If large heterogeneity is suspected, more increments should be collected.

Sample increment mass collected is often a function of the sampling device. If a 2-foot shelby tube is pushed into clay, and the entire thickness is part of the population, the entire core removed in the tube is the sample (increment), unless it is properly subsampled (if part of the core is not recovered, you introduce a materialization error). Sample mass is related to FE by the relationship $FE^2 = 22.5(d^3)/m_s$ (where m_s is mass of sample, d is maximum particle diameter, 22.5 mg/mm^3 is a density/conversion factor, and FE is fundamental error in percent). For an FE of 15 percent and a maximum particle size of 9.5mm, $m_s = 22.5(9.5^3)/.15^2 = 857$ grams. Work performed in support of sampling theory proves that greater, rather than lesser, sample mass (resulting from additional

increments) provides for more representative samples and reduced sampling error (Ramsey 1998, Pitard 1992). Most laboratories are **not** prepared to deal with increased sample mass, and you may experience large preparation error (PE) if the laboratory incorrectly subsamples from your samples for analytical test portions.

Trend (CE_2) and cycle (CE_3) errors should be considered in the sampling plan design but are not covered in this guidance. Preparation error (PE) is the last major source of sampling error covered in this guidance. Preparation error results from incorrect subsampling technique. When a sample is field prepared or submitted to a laboratory with excess sample mass when compared to the amount required for a test portion, the original sample mass is reduced to the test portion requirement by some splitting technique or by simple extraction of the test portion from the entire original sample mass (liquid samples are affected by splitting technique only when they contain density stratified or immiscible/nonaqueous phase components). If the entire sample were homogeneous at the extraction mass scale, there would not be a problem – this is **never** the case. With larger sample mass, as advocated, the problem is compounded. Typical sample splitting techniques include cone and quartering, riffle splitting, and test portion extraction (a spoonful from the center of a pile). Samples will differ in their sensitivity to splitting techniques, but generally you can expect to introduce up to 50-percent (or greater) error (PE) by using the spoonful from the center approach, 19.2-percent error by using cone and quartering, 3.7-percent error with a riffle (chute) splitter, and 0.9-percent error by rotary riffle splitter (add this error alone to a 15-percent fundamental error, and the data may no longer be useful). While the rotary splitter is very efficient, and a preferred method, not everyone (or every lab) will have access to one. An alternative available to everyone, with approximately the same precision as the rotary riffle splitter, is fractional alternate shoveling (see Glossary). Fractional shoveling is also well suited for subsampling samples with greater mass.

Fractional shoveling is accomplished by scooping increments from the original sample into alternate smaller piles. The number of smaller piles and scoop/shovel size are determined by the desired final subsample size and the need to make at least 30 fractional contributions to each subsample pile. All dehydration (drying) and comminution (particle size reduction) necessary for analysis should occur prior to splitting to ensure equal distribution of large particle content contribution to overall contaminant concentration. Mixing prior to fractional shoveling may improve the accuracy of the split. Flattening a pile prior to shoveling may also improve subsample to sample representation, but the ultimate goal is for each scoop/shovelful to be randomly chosen from the original sample, for all subsamples to be of equal mass and contain at least 30 increments, and for all of the original sample to be used in the process. You should be certain that sample preparation is performed by someone familiar with this technique. It may be necessary to take the initial project samples to the lab and train the personnel responsible for your sample preparation in order to ensure a minimal PE.

Therefore, there are at least four important issues for sampling plans: correct, detailed, identification of the sample population(s); accurate sample specification (correct selection of sampling tools (DE) and correct use of sampling tools (EE)); precise sample specification (enough sample mass to keep FE below 17 percent) and enough random increments to reduce GSE below concern; and proper sample preparation (correct subsampling/splitting techniques to minimize PE) prior to submission or analysis.

Other Samples

Control Samples - Control samples should be part of the sampling plan when they will provide useful or necessary information. Control samples are taken at locations outside the site boundary to determine the "normal" (unaffected or background) concentrations of the analyte(s) of interest. Control samples are often used to determine target cleanup levels or relative impact. Water samples upstream from suspected contamination or loading sources may also be considered control samples.

Sample collection must not significantly disturb the environment being sampled or the results may be biased. Removing significant confining overburden from a volatile organic compound (VOC) contaminated site may be dangerous to samplers, cause an unlawful release, and/or result in sample concentration loss. Methods and materials used to collect, store, and transport samples must be selected to avoid causing changes in or contaminating the sample, especially where the analytes of interest are in low concentration, reactive, or absorptive. The best current reference for information about how to sample specific environmental materials is Pitard (1992).

Sampling Quality Control

QC Samples - The preconceived goal of field quality control (QC) is not only to identify, measure, and control the variability of the data collection process, but also to document the uncertainty of the information collected. However, most QC samples are of limited value either because of their inability to accurately determine the source of error or contamination they indicate or because they suffer from the same sampling and preparation errors as the samples they are intended to be a control for. QC samples **cannot be used** to show that there are no problems; they can only be used to estimate some errors. A good sampling plan/design that is well implemented should require minimal QC type samples. Be certain you understand the need for and actual meaning of QC sample results before specifying and acquiring them.

There are basically eight types of field quality control samples: duplicates, replicates (colocated), splits, reference materials, trip blanks, field blanks, rinsate blanks (field decontamination check samples), and spiked field samples. The eight basic types may be permutated into additional types through the use of single- and double-blind labeling and/or appearance disguises. Some sampling errors may be estimated or controlled with appropriate numbers and types of one or more of these QC samples and through field audits and strict adherence to SOPs and protocols. The project manager / QA officer should suggest QC samples based on potential errors of critical project importance.

Any sample may be submitted to the laboratory from the field as either a correctly identified sample or a blind sample. **Blind** samples are so designated because the receiving laboratory cannot tell the sample composition from the sample identification information — the sample identity and composition are known only to the submitter. Blind samples may be submitted as either single-blind or double-blind. Single-blind samples are usually recognized by the laboratory as quality control samples by virtue of their distinct appearance or identification relative to the project environmental samples, but the composition cannot be determined visually. Double-blind samples are identified in the same way and appear the same as project environmental samples. They are unrecognizable to the laboratory as QC check samples. Single-blind sample masking is used to assess laboratory or analyst measurement proficiency. Double-blind samples are used to assure that QC samples do not receive special attention by the laboratory/analyst. Control samples for water matrices are easily disguised in sample batches; however, solid matrices such as vegetation, soil, sediment, or biological tissue may

be difficult to disguise. Solid matrix control samples can still be incorporated in sample batches where the laboratory will suspect they are controls, but they will not know the actual concentrations of the constituents of interest (spiked single-blind samples). **Caution** - when samples are not properly identified (on labels and/or COC forms), they may be difficult to trace during verification and validation - work out a sample identification, recording, and labeling scheme in advance that allows single- and double-blind sample analysis data to be correctly matched back to the original samples.

A **field duplicate sample**, for Reclamation purposes, is another sample (subsample) from the same sample population collected by duplicating the original sampling episode method a second time. Duplicate samples, as opposed to split samples, are taken serially from the target population to attempt to assess the sampling error. Duplicate samples are the only QC type sample capable of providing an estimate of sampling error by indicating the repeatability (precision) of collecting a sample. The judicial use of duplicates is suggested to prove that the sampling plan adequately provides a reasonable estimate of the sample population characteristics.

A **field replicate sample**, for Reclamation purposes, is a second sample (subsample) from the same sample location(s) (collocated) and sampling event (population) that is intended to be as representative of the characteristics of the population sampled as the initial sample. Replicate samples provide an estimate of the "nugget effect," or small scale variability for variograms/geostatistics. The samples must be taken separately (separate containers) at the same time and place, with the same type of sampling instrument. If variograms or geostatistics are not part of the analysis, there is no reason to collect replicate samples.

Field split samples are the best field initiated laboratory QC type samples (when correctly prepared) and the only type recommended for use. Field split samples are subsamples of an original sample (population subsample) resulting from the accurate (as discussed above relative to preparation error) splitting of the original sample into two or more indistinguishable subsamples (original sample subsamples).

Split samples, as opposed to duplicate samples, are taken simultaneously from the field sample to try to provide identical split samples independent of sampling error.

For sampling programs that will involve many samples with the same or similar-in-appearance matrix, such as a medium brown ML soil, the first sample mass may be collected in great excess, from which many split samples may be derived. One of the resulting split samples can be submitted as the first sample, and another split from the first sample can be included with each batch of samples thereafter (as a double-blind QC sample), providing any holding time requirements are met, as a means of assessing the analytical laboratory's ability to repeat the measurement on successive samples (blind CCV). Split samples may also be used for submission to referee laboratories to confirm the primary laboratory's analysis. Split sample results should be within accepted tolerances, by medium, matrix, analyte, detection limit, and/or method (check with your laboratory manager for specific guidance on allowable split sample data differences). Provide conflicting data evidence to your laboratory for reconciliation as soon as possible. Independent third party laboratories may be required to establish consensus results if primary and referee laboratory results significantly disagree, and neither primary laboratory nor referee laboratory can find a problem with their respective systems.

Split sample analytical results may be incorrectly used to validate, qualify, or reject affected data. If split sample results will be used in data validation, determine in advance the trigger or performance deviation that will initiate concern, the extent of data that will be considered affected, the corrective action to be taken when triggered, and the effect on the data quality or usability for possible corrective action outcomes.

If split sample results agree, it may mean only that the splitting procedure was well done and the laboratory analytical system offered repeatable results — the original sample may have missed any contamination or analyte of concern (good data, bad decision). If split sample data do not agree, it may be an indication that the original sampling was insufficient to determine the average population property, that the splitting procedure was not done properly, or that the laboratory did not provide repeatable results.

Standard reference material samples may be submitted to the laboratory in an attempt to gage analytical accuracy; however, most laboratories are familiar with the limited number of available materials and will repeat their analyses, if necessary, until the known correct values are "determined." Certified reference materials are very expensive, and this is not a recommended use for them.

Blanks are an important QC tool. Blank samples are clean (do not contain any trace of the target analyte(s)) samples of a medium (gas, biota, soil, or water). There are three types of blanks used in field QC — trip blanks, field or sampling blanks, and equipment or rinsate blanks. A **trip blank** is clean, distilled, de-ionized (DDI) water; clean soil; or clean sand that is carried to the field and returned unopened (unexposed to sampling procedures) to the laboratory. Trip blanks are used to measure background contamination from site volatile organic compounds (VOCs) of VOC samples or contamination of samples during shipment. Trip blanks are not commonly used for Reclamation projects.

Field blanks are blank samples exposed to all of the steps of the sampling and analysis process exactly as any other sample and are used to assess the occurrence of incidental contamination from procedures, filters, traps, and/or sample containers. Alone, field sample blanks are not capable of isolating the incidental contamination source (field vs. lab). When used in conjunction with laboratory blank sample results from the same and sequential analyses batches, the source of contamination (field vs. lab) should be clear - re-analysis of samples may be necessary to determine the source if lab preparation or instrument carryover is the culprit. Field sample blanks may be useful for mercury studies or other similar elements but, again, are not generally needed for most Reclamation projects.

An equipment or **rinsate (rinse) blank** is DDI water which has been poured over the decontaminated sampling apparatus, preserved (as required), and analyzed to measure the effectiveness of the decontamination cleaning procedure. A rinsate blank assesses the potential for carryover between samples. Certified clean sea sand or matrix soil may also be used this way to create a double-blind, blank sample (see caution above). Equipment rinsate blanks are commonly used in Reclamation hazardous waste investigation projects where disposable sampling equipment is not feasible.

Contaminated blanks should trigger a corrective action loop (audit / methods and procedure review, detecting and eliminating the source of contamination, and initiating steps to control future occurrences) that documents the problem(s) and determines the need for resampling or reanalysis of the affected submitted samples. A data quality objective might be the submission of one rinsate

blank per sampling team per collection apparatus per day. For teams collecting many samples per day that require very expensive analytical methods, more frequent blanks may be indicated to better isolate affected samples.

Field (matrix) spiked samples are samples of verified composition to which a known amount of the constituent of interest is added ("spiked ") to the sample prior to delivery of the sample to the laboratory. (See Glossary of Terms.) Certified spiking standards are strongly suggested, and good practice includes pipet calibration and pipet measurement logging. Results from field spiked samples are used to indicate the suitability of the total sample handling and analysis process for the associated sample matrix. Spiked sample results may indicate a need to change analysis methods or adjust results due to matrix absorption or interference with the detection of the analyte of interest. Submit spiked samples early in the program, whenever the sample matrix changes, and at regular plan intervals. Advise your laboratory of adverse results immediately to resolve the source(s) of bias an/or imprecision.

Field spiked samples sound useful but, in actual use, are very difficult to adequately prepare in the field, and because of differences between spike and contaminant species and matrix effects, the results from spiked samples may be difficult to interpret. Field spiked samples are not recommended for use.

Judicious use of recommended field QC samples and types, according to the data quality objectives, allows one means of assessment of the quality of data produced. A common rule of thumb is to include at least one set or type of quality control sample (blank, duplicate, and spiked sample) with every 10 samples collected (10 percent) or per batch, whichever is more frequent. The field QC sample analysis **results** should be used only to initiate corrective action, or along with laboratory QC data, to assess project data quality, when you are sure you understand what the results actually mean.

Project managers should be aware that for investigations with rigorous quality requirements, especially those with small sample submission batches (only one or two environmental samples per batch), QC samples may double, triple, or quadruple the analysis cost for the investigation. DOQs should support the need for QC samples.

Field Quality Assurance

Specific QA guidelines for good sampling practice are prescribed by EPA's Office of Emergency and Remedial Response in training documentation for sampling hazardous materials. Key elements of the training are listed below. Including these elements in the **Technical Work Plan** (Sampling and Analysis Plan) of the **Quality Assurance Project Plan (QAPP)** will ensure that the quality of the samples collected can be researched and possibly determined.

Field Logbook

Keep **original notes** of field observations, calibration records, sample information, and photos, etc., for a specific site or program in a bound notebook, preferably maintained by one team member. Describe site locations in the logbook using maps or sketches and measurements from **permanent features**. Make all entries in a logical manner in **permanent ink** and sign and date each page.

Assign a second team member to review and verify the completeness of all entries. To be legally defensible, the logbook may need to be in your custody (on your person or secured) at ALL times, as well as formatted to preclude out of sequence entries and changes to the original text.

Data Entry

Record all data at the time of collection. **Read back recorded data** to the data generator and **certify** the check of data by signature. Use calculators and **record formulas** for derived values. Maintain cross-reference listings for single- and double-blind samples. Write legibly, print, or keep the log electronically (special legal considerations may apply). Make it easy to follow a "paper trail" from sample collection through packaging and shipping (record tracking numbers).

Field Equipment

Designate an individual or group to **maintain** equipment and provide **training** and reviews on its operation, calibration, and limitations. Keep instruction manuals, calibration solutions, and extra batteries with instruments used in the field. **Record calibration data** and field data in the field logbook. Keep an **equipment logbook** for maintenance records, operational checks, and calibration schedules.

Sampling

Collection – Document the sample **bottle cleaning** procedure and certify the cleaning of specific bottles by signature in the field logbook or purchase certified clean containers (certified clean containers may not actually be clean) and retain the certification sheet with field records. Include **sample requirements** (types and volumes) in the sampling plan and field logbook. Label sample containers at the sampling location (point of use) or verify preprinted labels before use. Use care in capping containers. Totally complete the recording, sampling, labeling, and cleanup of equipment before moving to the next sample site to avoid cross-contamination and data loss.

Preservation - Verify preservation and holding time requirements with the analytical laboratory and **describe the requirements** in the sampling plan and field logbook. Preplan **shipping** times for collected samples - samples should NOT be arriving at the laboratory late Friday afternoon without warning and approval. Preserve and label samples individually. Take preservative reagents to the field site and replace regularly.

Labeling – Use unique identifications for site designations and sample types (e.g., soil = s, water = w, groundwater = gw). Use numbered **sample tags or labels** which are formatted to receive necessary sample information to prompt record completeness. Complete sample tags immediately prior to attachment on the sample container. Record tag number and corresponding site identification and testing requirements in the **field logbook** (especially important for blind samples).

Decontamination

Document the decontamination procedure in the sampling plan and field logbook. Use **disposable equipment** at each site or use equipment which is easily disassembled for cleaning before moving to the next sampling site. Certify by a statement and signature that the decontamination procedure was performed at each site. Collect a **rinse or equipment blank** at least daily to check the decontamination process. Properly store and use decontamination chemicals. Send blanks with the other samples for analysis.

Recognition of Loss of Quality Control

Review field logbooks, sample labels, and analysis results regularly. Loss of QC is shown by incomplete or unsigned entries in logbooks and sample tags, missing calibration and instrument performance checks, and contaminated blanks.

Legal Considerations

The possibility that conclusions based on the results of sampling and analysis will be legally challenged is greater today than it was in the past because of the visibility of environmental projects and the economic consequences of court decisions on environmental damages. Personnel planning, performing, and managing investigations should observe these guidelines for defensibility, as suggested by the National Enforcement Investigations Center of EPA.

Observations

Personnel should recognize the constant need to make records of observations in the field logbook which are **signed and dated in permanent ink**. (Use strike out for deletions and initial and date all changes.) Records of observations may include tape recordings, photographs, subject matter and data, and shipping records.

Equipment Operation

Demonstrate by **performance checks** that instruments are operating correctly and record check data and **operator names**. Use experienced personnel to operate, provide training, and review the operation of field measurement devices.

Standard Practices

Use **recognized references** (ASTM, EPA, USGS) in describing procedures for making measurements, performing sampling, and preserving samples. (See “Sampling Guides” compendium for accepted procedures.) Observe established technical or legal limitations on holding times for measurements. Record any variation from recognized reference methods.

Site Locations

Physically mark the sample collection sites and describe the locations in the field logbook using maps and references to permanent features (take photographs).

Chain of Custody

When legally required or prudent, prepare and carefully follow a chain-of-custody process for samples from the time of collection to disposal. Figure 3 is an example of an electronic **Chain of Custody Record** (form E7-2518) that is available in WordPerfect® format. This form is available for download from the intranet at http://intra.usbr.gov/tcg/publist/guidelines/COC.htm.

An ad hoc TSC team has also developed an electronic chain-of-custody form in Microsoft® Excel that is linked to a Microsoft® Access database. This portable system, or something equivalent, is highly recommended for large, ongoing field sampling efforts. The TSC system is compatible with an internal sample tracking system in use in the Denver laboratory. Inquiries regarding the field tracking system should be directed to Mike Szygielski, Don Serina, or Sam Schaefer. Inquiries regarding the laboratory tracking system should be directed to the TSC laboratory manager (D-8240) or the laboratory computer specialist. The Reclamation TSC QA manager may also be contacted for information concerning either application.

You must provide for secure, verifiable custody of **all records** of the data collection activity, including procedures, logbooks, sample tags, equipment, chain-of-custody forms, and personnel records.

NOTE: The use of chain-of-custody forms is advisable for any data acquisition project. They provide a convenient, generally comprehensive method of recordkeeping and data tracking and may be used without the need for a signature custody chain for many Reclamation projects.

Figure 3. Chain of Custody Record

E7-2518 (1997)

Bureau of Reclamation

UNITED STATES

DEPARTMENT OF THE INTERIOR
BUREAU OF RECLAMATION

Chain of Custody Record

Project Manager	Project Name	Sheet ____ of ____ Batch Identification _____

Sampler Name and Title (printed below, signature above)					Number of Containers	Analysis Requested										Remarks	Initials and date sample destroyed
Destination Laboratory																	
Sample Field Identification	Date Collected	Time Collected	Laboratory Identifier	Sample Type													

Relinquished by (signature)	Date	Time	Relinquished by (signature)	Date	Time	Relinquished by (signature)	Date	Time
Received by (signature)	Date	Time	Received by (signature)	Date	Time	Received by (signature)	Date	Time

Point of Contact (Client): _____ WOID or 18-digit Account _____

Remarks: _____ Return Shipment Container to Shipper Yes ____ No ____

DISTRIBUTION: Print a copy for sampler, for contract laboratory (if any), and to include with shipment; electronic copy for files and electronically mailed to laboratory

SHIPPED VIA:

- ☐ Fedex
- ☐ UPS
- ☐ Bus
- ☐ Express Mail
- ☐ Priority Mail
- ☐ Other_____

BIBLIOGRAPHY

ASTM, *Standard Guide for Sampling Chain of Custody Procedures*, D 4840-95, American Society of Testing and Materials, Philadelphia, Pennsylvania, 1997.

Bureau of Reclamation, *Sampling Guides* (Compendium of selections on sampling environmental materials), Denver, Colorado, 1993.

EPA, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods*, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC, 1986.

———, *Sampling for Hazardous Materials*, Course 165.9, Office of Emergency and Remedial Response, Cincinnati, Ohio.

———, *Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents*, EPA QA/G-6, EPA/600/R-96/027, Office of Research and Development, Washington, DC, November 1995.

Keith, L.H., ed., *Principles of Environmental Sampling*, American Chemical Society, Washington, DC, 1988.

REFERENCES

Pitard, Francis F., *Pierre Gy's Sampling Theory and Sampling Practice: Volumes I and II, Heterogeneity and Sampling*, CRC Press, Boca Raton, Florida, 1992.

Ramsey, Charles., *Defensible Environmental Decisions Seminar Notes*, Envirostat Inc., PO Box 1354, Eugene, Oregon, 97440-1354, USBR, Denver, Colorado, February 1998.

DATA ACQUISITION AND ANALYSIS ASSESSMENT/OVERSIGHT

Concurrent with field sampling, activities to assess compliance of implemented activities with the SAP (SOPs and QC measures) should be undertaken by the QA officer. These measures are generally performed as audits of practices, methods, and techniques, and can be divided into three categories - field, laboratory, and office.

Field Audit

When the DQOs require fully assured, quality controlled field data, the QA officer must perform a number of field audits of sampling methods and equipment use, calibration, and decontamination. The QA officer will review SOPs and observe field practices for deviation from SOPs. The QA officer should review the field log for instrument calibration entries and notes on problems affecting quality. The initial audit should occur early in the schedule, preferably the first day, so that problems are detected and corrected early. SOPs may require adjustment as a result of field audit(s). Spot inspections may follow an initial, intensive investigation and review.

Field audit methods also include the submission of quality control samples. The QA officer will ensure that the requisite field performance evaluation samples - blank (trip / equipment / field) and duplicate (blind) - are collected / prepared and shipped along with the investigation samples. The preceding SAP QA/QC section provides an indepth discussion of field performance evaluation sample submission options and recommendations. The results of these field performance measurement efforts are considered in the validation process, along with laboratory audit measures, to determine whether data are acceptable, need qualification, or are rejected as unusable for the intended purpose.

Laboratory Audit

Field quality control measures can include laboratory audits. Laboratory audits can occur prior to the field investigation (for laboratory selection, see Part 3 - Selection of an Analytical Laboratory) and during sample submission after selection. When the DQOs require quality assured/controlled laboratory data, the SAP needs to specify the necessary external QC measures by sample type (organic / inorganic / radionuclide / volatile / etc.) and analysis method. Laboratory audit measures can include: (1) comparison of the analysis results to known values of **external QC samples** incorporated into each batch of samples and submitted to the laboratory for analysis, (2) an assessment of whether **holding times** were met for testing, (3) an assessment of the **completeness** of the analysis report, (4) a review of the internal laboratory QC sample results, and (5) a **comparative review** of analysis results from split samples and/or with historical data from the same site. The primary method of project data analysis assessment and oversight is through the submission of **external** (field) **QC samples**. Results for QC samples incorporated in batches of field samples submitted to the laboratory provide the means, independent of the laboratory, for estimating the precision and bias of the associated sample data and for assessing the extent of contamination in the sample handling and analysis processes. Duplicate, spiked, split, blank, and reference material samples are control samples which may be included with field samples as appropriate to the QA objectives of the data gathering project. Control samples are defined in the Glossary of Terms. The QA officer should have assured that the requisite laboratory performance evaluation samples - duplicate (double-blind), spike (matrix), standard reference material (SRM), and split samples - were

collected, prepared, and shipped along with the investigation samples. Double-blind duplicate samples not compromised by poor collection/split technique or matrix heterogeneity can be used to assess a laboratory's repeatability performance. Matrix spike control samples measure detection (as affected by matrix interference) performance, and SRM samples measure discrete analyte measurement performance. **Split samples** provide an independent means of confirming laboratory performance.

Internal QC checks which are implemented and reviewed by the analytical laboratory are discussed in *Part 2 - Laboratory Quality Guidance* of these guidelines.

External Quality Control Samples

Review the results for QC control samples incorporated in batches of field samples and submitted to the laboratory to estimate the precision and accuracy of the associated sample data and the extent of contamination in the sample handling and analysis processes. Duplicate, spiked, blank, and reference material sample acceptable deviation varies by analyte, media, control sample type, and analysis method. Project managers and QA officers should seek professional counsel for acceptable deviation in specific cases, and DQOs should state the required project quality requirements within the bounds of accepted practice. Some guidance is available from the CLP reference regarding example method criteria. Fresh certified NIST standard reference material (SRM) should be analyzed to within the certified standard tolerances for the specific SRM. Quality control samples that fail to meet expectations can necessitate rerunning the batch of samples, adjusting sample values by the error percentage (and recording methods and reasons), or eliminating the results for the real samples from project consideration. The responsible person must document the reconciliation methods and provide them to the project manager and/or QA officer.

Holding Times

The length of time between sample collection and analysis may be an important factor in assessing the quality of analytical results for a batch of samples. Results may be compromised when recommended holding times are exceeded, especially where the concentrations of constituents of interest are very low or are subjected to biologic, chemical, or physical changes. Table 1 lists sample holding time guidelines for analysis of specific constituents when preserved according to EPA methods. When holding times are exceeded, the data may be suspect (and requires qualification) or unusable, depending on the DQOs.

Completeness

Completeness in analytical performance refers to the timely processing of and accounting for all samples; a statement of the estimated uncertainty of test results in reports, where relevant; willingness to repeat analyses, if necessary; and accuracy in cost accounting and control. Acceptable analytical performance on any specific batch of samples may be judged by completion of analyses and reports as expected by the client according to a predefined schedule.

Site Specific Historical Data

If a historical data base exists for sites under investigation, a comparison can be made of the results of recent samples with past data (provided that methods and detection limits are comparable) from the same site to determine if any of the new values vary significantly from previous data. Similarity of site conditions (i.e., season and flow) and contaminants of interest must be considered in evaluating the significance of any variations.

Split Samples

Split sample analysis results are compared for agreement within tolerances defined by the analysis method, sample type, and sample medium. Check with both laboratories for guidance on agreement tolerances in each specific case. Differing results need to be reconciled by the QA officer. Reconciliation may be accomplished by reanalysis of the samples or submission to a referee laboratory. Split sample analysis results that are in agreement provide a measure of confidence in unsplit sample results.

The assessment of analytical performance for any specific batch of samples can be made based on an evaluation process that compares any or all of the parameters from the preceding paragraphs to specified performance criteria. If the results of the assessment process determine that the project QA objectives for the data have been met, the data user can be reasonably confident that the analytical data for the associated field samples are useful. If the assessment process determines that the QA objectives of the analytical data have not been met, the usability of data for the associated field samples is questionable and must be reported with appropriate qualifiers. Data quality problems identified by the assessment process must be addressed with the field crew and/or laboratory immediately. Resampling or reanalysis of the affected samples may be requested but may not provide much information in the case of reanalysis because holding times will most likely be exceeded.

Office Audit

When field and/or laboratory analytical data require postprocessing (graphing, mapping, mathematical/statistical manipulation, etc.) for interpretive convenience and/or visual presentation, the QA officer should review the processed data for method applicability and accuracy. Review methods will vary with postprocessing technique but should be specified in the QAPP, if appropriate. Errors should be corrected prior to data release for validation.

Follow Up

The point of this section is that it is not enough to simply put QA measures into place and then trust that acceptable quality data will be produced. The plan needs to include followup measures. The QA officer needs to diligently pursue the plan quality requirements by confirming that all QA measures occur on schedule. The QA officer then needs to assimilate the results from QA measures, as they become available, and make any required corrections. QA is an iterative process, not an act.

BIBLIOGRAPHY

EPA, *Laboratory Data Validation – Functional Guidelines For Evaluating Inorganic Analyses*, Office of Emergency and Remedial Response, Washington, DC, 1985.

Taylor, J.K., and T.W. Stanley, *Quality Assurance for Environmental Measurements*, ASTM Special Technical Publication 867, Philadelphia, Pennsylvania, 1985.

DATA VALIDATION AND USABILITY[1]

Data Quality Assessment (DQA)

The quality of data is assessed by the QA officer or the project manager against prescribed requirements or specifications (DQOs) in order to determine whether the data are useful and appropriate to assist in making the decision or answering the question with confidence. Results from the assessment detailed in the previous section are used along with data review and verification methods to establish the quality criteria of the data produced. Then the relationship between the (actual) collected data and the data requested is determined. This comparison of produced data with requested data culminates in a quality of data determination and data validation within a framework that incorporates the intended use of the data.

Prior to validation, data needs to be reviewed and verified. The plan should state how the data review will be performed, who will perform the review, and what method or criteria will be used in the review. The QA officer should review the data for completeness. That is, the QA officer should determine that all planned environmental and QC samples were acquired and accounted for. The QA officer should then verify the accuracy of the data.

Verification Methods - The QA officer should track each sample (follow the written paper trail) from acquisition, through preparation, packaging and shipping, and the laboratory analysis report, checking and verifying the accuracy of all recorded information. Errors and omissions should be fixed or noted according to the plan specifications for verification procedures.

Following verification, the data from field, laboratory, and/or office efforts must be compared to the Data Quality Objectives set forth in the QAPP. Data that conforms to the DQOs can be validated.

Reconciliation with DQOs - The QAPP needs to state how limitations on data use will be reported. The QA officer should include a section in the data report detailing any deficiencies in the data and how these deficiencies may or should affect the usefulness of the data in providing a sound basis for the decision; qualified ("bad") data may be useful. If the problems with the data are understood, proper judgment can be applied to their use. If the quantity of data of acceptable quality is insufficient, additional sampling may be required to support the decision.

When the data has been fully examined through the preceding processes, it is ready for validation to determine the usefulness of the data.

Validation is the process of substantiating specified quality criteria (i.e., that data satisfy the requirements specified by the user). The results of the assessment, review, and verification will determine the quality criteria of the data. Comparison of the data with DQOs should determine the quality of the data for the intended purpose. Data validation statements can be either "validated" (good to use) or "validated with qualifications" (good to use but requires qualifiers due to certain shortcomings). Data that cannot be validated should be rejected (unusable). The entire data package may be validated as a whole, or discrete packages may be individually validated (more common).

[1] When performing work for EPA, use EPA validation specifications and methods as described in the last section, EPA validation methods.

There can be up to three steps - the validation of field data, the validation of laboratory analytical data, and the validation of postprocessed data.

Validation Methods - Field data are validated through a review of any qualifiers generated by the assessment, review, and verification process and comparison with the DQOs. When this review concludes that no data qualifiers are present and the produced data are equivalent to the requested data, the QA officer or project manager will validate the data for use without qualification by stamping "validated" on the data report or otherwise effectively indicating the judgment. When problems with the field data exist, the QA officer or project manager will validate the data with qualifications or reject the data as unusable, based upon the examiner's knowledge of the problems and his/her professional judgment. Validated data that contains acceptable qualifications on the use of some or all of the data will be stamped "validated - qualified." Data that contains unacceptable errors or qualifications should be stamped "rejected" on every page.

Laboratory analytical data validation is accomplished in the same manner as the field data validation, except that internal laboratory QC results would also require integration into the review. Where the continuous quality of data is important to the decisionmaking process, a validation of analytical data could be conducted on every batch of samples submitted to a laboratory upon laboratory release of the data.

Postprocessed data prepared in the office from field and laboratory data are validated with reviews for method appropriateness, accuracy, and statistical parameters. When only a portion of the data are presented in some processed format, or manipulations include numerical adjustments, these liberties must be fully documented on the data format. Processed data are validated through its acceptance, publication, and use after the review. No formal stamp or designation is required, other than appropriate qualifiers in the legend/notes/etc.

The extent of data assessment and validation should be appropriate to and depend upon how the data will be used. Data collected for a screening objective (is there anything of interest out there) may not be assessed for validation to the same extent as data collected for a constituent-specific or a concentration-specific objective (exactly where and how much of something of interest is out there). The elements necessary for the evaluation of data based on how it will be used are described in guidance from EPA's Office of Emergency and Remedial Response. The assessment and validation requirements defined in the QAPP should be appropriate for the DQOs defined in the QAPP.

Data Usefulness

The data user (client) will always determine the usefulness of the data. Validated data should always be useful, qualified data may be useful, and some use could be found for rejected data, but the determination will always lie with the data user. Therefore, if the data user is not involved early and throughout the project, the chances for failure (unusable data) are very good.

The data user should be presented with all the data, appropriately identified according to validity (i.e., with all defined qualifiers resulting from assessment, review, and verification, for the user's determination of usefulness).

Data produced for one client/project that are subsequently applied to another unrelated project must be independently validated for the second project. Independently validated data must still be determined useful by the data user, who assumes all risks.

The EPA had previously defined three levels of objectives for validation (EPA/540/G-90/004 (NTIS Publication PB90-274481)) as QA1, QA2, and QA3. The EPA currently defines TWO levels of QA objectives. The objectives and the minimum validation elements necessary to substantiate data collected for these objectives are listed below. Specify an EPA designation for EPA (Superfund) work. Note that neither of these two levels requires 100-percent data checking.

The following EPA QA objectives are associated with minimum validation elements necessary to provide data of known quality.

Superfund Data Categories - The Superfund program developed the following two descriptive data categories to help meet the National Contingency Plan (NCP) requirement that Superfund environmental data be of known quality:

1. Screening data with definitive confirmation
2. Definitive data

These two data categories are defined in the document **Interim Final Guidance on** Data Quality Objectives Process for Superfund (see References). This guidance is to be used to implement Superfund data collection activities according to OSWER Directive No. 9355.9-02. The directive was effective September 30, 1993.

The **Interim Final Guidance on** Data Quality Objectives Process for Superfund of September 1993 (Superfund DQO Guidance) explains the two data categories in section 7.4 (pp. 42-44). These data categories are associated with specific QA/QC elements and may be generated using a wide range of analytical test methods. The particular type of data to be generated depends on the needs of the project. The project data needs are expressed as the qualitative and quantitative project DQOs. The definitions of the two data categories are presented below, and the corresponding QA/QC elements are shown in the table 2.

Screening Data with Definitive Confirmation

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent instead of elaborate extraction or digestion and cleanup. Screening data provide analyte identification and quantification, although the quantification may be relatively imprecise. For the category of "screening data with definitive confirmation," at least 10 percent of the screening data are confirmed using analytical methods and QA/QC procedures and criteria associated with "definitive data" (see below). Screening data without the associated confirmation data are not considered to be data of known quality.

Definitive Data

Definitive data are generated using rigorous analytical methods such as approved EPA reference methods and are analyte specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., spectra, chromatograms, and digital values) in the form of paper printouts or computer generated electronic files. Definitive data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. The current Superfund DQO guidance (interim final) states that for the data to be definitive, either analytical or total measurement

Table 2.—QA/QC Elements and Criteria Associated With Each Data Category

QA/QC elements and criteria	Screening data with definitive confirmation[1]	Definitive data
Sample documentation (location, date and time collected, etc.)[2]	X	X
Chain-of-Custody[3]	X	X
Sampling design approach (systematic, simple, stratified random, judgmental, etc.) documented in approved QA Plan[4]	X	X
Determination and documentation of detection limits[5]	X	X
Initial and continuing calibration	X	X
Analyte(s) identification[6]	X	X
Analyte(s) quantification[6]	X	X
QC Blanks (trip, method, rinsate)		X
Matrix spike recoveries		X
Performance Evaluation (PE) samples (when specified)		X
Analytical or measurement error determination[7]	X	X
Rigorous analytical methods (e.g., approved EPA reference methods)		X
Confirmation of analyte identity and concentration[8]		X
Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of printouts or computer files[9]		X

Table 2.—QA/QC Elements and Criteria Associated With Each Data Category

[1] The Superfund DQO Guidance states that for definitive confirmation, at least 10 percent of the screening data must be confirmed with definitive data. And, as a minimum, at least three screening samples reported above the action level (if any) and three screening samples reported below the action level (or as non-detects, ND) should be randomly selected from the appropriate group and confirmed.

Note that the 10 percent confirmation rate may be modified if the modification is demonstrated to be appropriate for a particular project. For example, adequate information may already have been generated to support the correlation of the screening method with the confirmation results. (This existing information must be applicable to the project analytes and sample matrices.) In addition, the correlation data generated as a project progresses may support a decision to decrease the initial 10 percent confirmation rate once an acceptable correlation has been established for the analytes and sample matrix. If the 10 percent rate called for in the "Guidance" is modified in the project, information supporting the change should be documented along with the rationale for the change.

[2] This documentation is typically provided in the field records.

[3] For some analytical methods, such as those that are made *in situ,* the main chain-of-custody documentation may be the field logbook.

[4] The rationale for the selection of sampling strategy, methods, sample numbers, types, location, frequency, etc., should be addressed in the approved QA Plan (e.g., QAPP). The rationale should link the selections to fulfilling stated project objectives.

[5] For certain measurements, such as temperature and pH, it is the resolution sensitivity of the instrument that is determined (e.g., pH measured to 0.1 unit and temperature to 0.5 degrees).

[6] The details of how analyte identification and quantification requirements are met depend on the analytical method used. For this QA/QC element to be met, the approved QA Plan typically should specify use of appropriate standards containing the analyte and bracketing the expected concentration for calibration; demonstration of instrument control; proper dilution procedures to avoid diluting the analyte below the detection limit; and, for some methods, techniques to correct for the presence of other elements/compounds in the samples that might interfere with analyte identification.

[7] There are several types of QC samples and techniques that can be used to determine various aspects/types of analytical or measurement error. The information regarding error that needs to be collected depends on the project DQOs. Sample measurement error can occur at every stage of the data generation process, such as during selection of sampling location, collection of sample, analysis of sample, and reporting of results. There may also be error associated with the heterogeneity of the sampling medium itself. If the measurement error that occurred throughout the process was "added up," it would represent the total measurement error. QC samples can be introduced at any stage of the process to "capture" the error information needed to evaluate the quality of the data produced. For example, if information on laboratory sample handling and analytical error is needed, the approved QA Plan could require that the laboratory homogenize and split one sample into two portions, analyze the two portions, and calculate the precision in terms of relative percent difference. If field sampling error is also of interest, duplicate field samples might be appropriate. Note that because many terms such as duplicates and splits are used loosely, it is important for the QAPP to describe what these samples are intended to represent and to provide an operational definition of how these QC samples are to be collected and processed. The types of measurement errors that need to be determined in the data generation process may depend on how likely it is that the types of error will affect the decision that may be made with the data.

[8] Typically, the confirmation step is more applicable to the analysis of organic analytes. An example of confirmation would be second-column confirmation of gas chromatograph (GC) results.

[9] The methods used must be capable of producing tangible raw data. The raw data must be available for review (i.e., archived), but whether or not the raw data are reviewed or used to validate the data depends on the project DQOs.

This information was provided by EPA Region 8 in January 1998. Check with your specific EPA region for possible deviation, modification, or revision.

error must be determined. The final version of the guidance will emphasize the importance of determining the significant sources of error (e.g., errors in sample collection) that make up the total measurement error and not just the analytical portion of the error. (Please refer to footnote 7 of table 2 for a discussion of measurement error.)

Data Category: Other

Most Superfund environmental data should meet the criteria of one of the two categories, whichever is appropriate. There may be some data collected that do not fit exactly into either of the two categories. For example, the pH, temperature, and conductivity measurements that are taken during groundwater well purging do not fall neatly into either category. This "other" type of data is generally not used directly for site decisionmaking.

Previous Data Classifications

The two Superfund data categories in the 1993 Superfund DQO Guidance **replaced** references to the five analytical levels (Levels I-V) in the 1987 remedial DQO guidance, the quality assurance objectives (QA1-QA3) in the removal QA/QC guidance, and the data use categories (DUCs) in the site inspection guidance. The major guidance documents that were impacted by the revision to two data categories are:

- *Data Quality Objective Guidance for Remedial Response Activities: Development Process and Case Studies:* EPA/540/G-87/003 and 004, OSWER Directive 9355.0-7B

- *Quality Assurance/Quality Control Guidance for Removal Activities: Sampling QA/QC Plan and Data Validation Procedures:* EPA/540/G-90/004, OSWER Directive 9360.4-01 April 1990

- *Guidance for Performing Site Inspections under CERCLA*, OSWER Directive 9345.1-05, August 1992

Although most data previously generated as "Level IV" would probably meet the criteria for definitive data, there is no easy way to automatically translate between all of the previous data classifications and the two current Superfund data categories. For data generated under the previous classifications, the available support documentation (e.g., QA/QC records) would probably have to be examined to determine its category under the current system. Contact the regional QA program for assistance in using the two data categories or incorporating them into ongoing projects. QA program staff can also provide assistance in determining whether the data generation activities planned will produce data that meet the criteria of the specified data category.

BIBLIOGRAPHY

EPA, *Guidance for Data Usability in Risk Assessment*, EPA/540/2-90/008, Office of Emergency and Remedial Response, Washington, DC, October 1990.

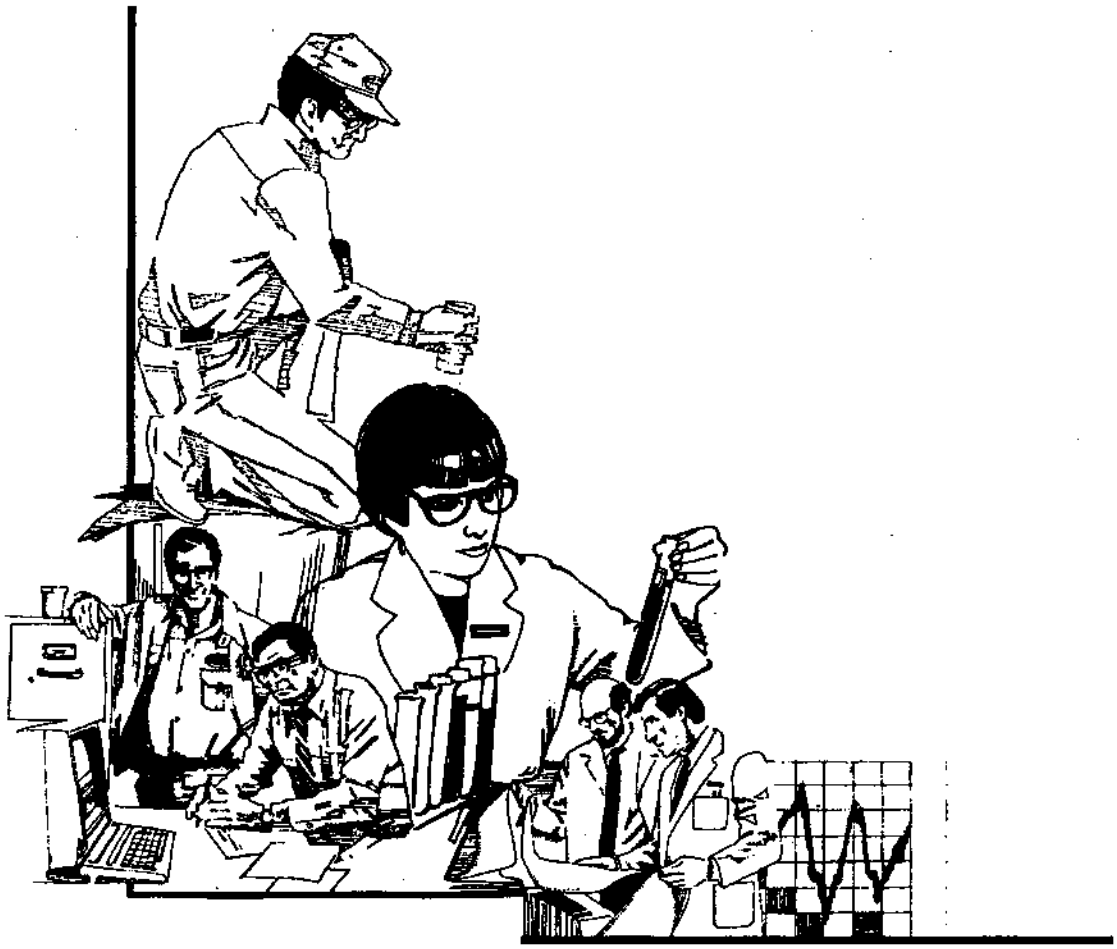
———, *National Functional Guidelines For Organic Data Review*, Office of Emergency and Remedial Response, Washington, DC, draft, June 1991.

———, *Interim Final Guidance on Data Quality Objectives Process For Superfund*, Publication No. 9355.9-01, Office of Emergency and Remedial Response, Washington, DC, September1993.

———, *Guidance for Data Quality Assessment - Practical Methods for Data Analysis*, EPA QA/G-9, EPA/600/R-96/084, Office of Research and Development, Washington, DC, July 1996.

Wagner, R.E., ed., *Guide to Environmental Analytical Methods*, Genuim Publishing Corp., New York, 1996.

PART 2 - LABORATORY GUIDANCE



PART 2 - LABORATORY GUIDANCE

All Reclamation laboratories should have a QAP in place. The general features of a laboratory QAP are listed below. The listed features are intended to provide a minimum content level, and laboratory managers are encouraged to expand content and add sections as needed.

Introduction

The introduction may contain any pertinent background or historical information the laboratory might wish to convey. This might include policy statements, limitations on clientele, and/or QAP version data.

Organization

This section should provide the reader with a current functional organizational breakdown, including contact information (telephone number, e-mail address, pager number, cellular telephone number, emergency/after hours contact information), personnel classifications, responsibilities, brief personnel resumès, and other pertinent organizational / personnel data.

Facilities

This section should provide the reader with all necessary location and physical plant (facilities) information. The postal and overnight delivery address should be specified, along with any specific information regarding access, such as locked gates, loading dock height, bells/buzzers/signals to alert personnel of deliveries, special security measures, proper delivery door/dock, and acceptable sample receipt days and times.

Sample Receipt Policy

This section should specify the laboratory sample receipt policy regarding unexpected samples, unacceptable sample containers, unacceptable sample shipment containers, unacceptable sample shipment container condition, and unacceptable documentation accompanying samples. There should be a clear statement concerning the laboratory's obligation to accept sample shipments and the intended disposition of rejected sample submissions.

Instrumentation

This section should specify the onsite equipment available for performing sample analysis. The list should include instrument analysis type, instrument name, model, manufacture date, and any accessories attached or modifications made.

Services

This section should include a list of services provided by the laboratory, including a listing of analytical methods the laboratory can perform. Analytical services should be identified as in-house or by contract. Other services to consider for inclusion are consulting; SAP/QAPP preparation; referee laboratory service; and supply or preparation of sample containers, blank media, and spike samples. Include for consideration any special analytical services and acceptance of special sample types, such as hazardous waste site samples.

Quality Control/Quality Assurance

Quality Control

The quality of analytical results is generally controlled by two factors - preparation error and analytical error. Preparation error includes things like sample log-in, sample storage, sample handling, holding times, sample preservation, sample mixing, and test portion subsampling. In general, the largest preparation error will occur in test portion subsampling. Analytical error includes things like weighing/volumetric measurement, solvent/reagent purity, extraction efficiency, analyte isolation (interferences), actual reference standard purity, calibration practices, analysis procedure (method), standard operating procedures (SOPs), data analysis (misidentification, integration, etc.), differences among analysts, laboratory environmental conditions, and data verification/validation. Both factors require due diligence to minimize errors.

Uncertainty (bias, variability) statements provided with methods from EPA, ASTM, and other recognized sources of test procedures generally define the quality of data that can be produced by the method and can assist the laboratory in selecting analytical methodology which is appropriate to the objectives of the data collection activity. Quality control (QC) of analytical tests is necessary to control the uncertainty of the analytical process within these established limits.

The guidance presented here is intended to provide a recommended minimum process to be incorporated into the laboratory QAP for substantiating the quality of data produced by Reclamation laboratories and contractors. **IF samples have been collected and handled correctly prior to receipt by the laboratory**, the minimum process quality control (QC) should provide for a comparable level of data quality from laboratories Reclamation-wide.

The International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC) jointly prepared ISO/IEC Guide 25-1982, "General Requirements for the Competence of Testing Laboratories." A later revision, ISO/IEC Guide 25-1990 (*Guide 25*), includes calibration laboratories and addresses ISO 9000 series standards. The recommended QC guidance for environmental measurements is based on this guide. Where methods do not require specific QC functions, this guidance provides the minimum QC for compliance with EPA-accepted practice.

QC analysis must be performed at the required frequency and within the acceptable criteria specified in the analytical method and associated project plan. QC analyses generally include instrument calibrations, calibration verifications, laboratory control samples, blanks, spiked samples, duplicates, and sample-related QC such as surrogates and internal standards for organic sample analyses.

All QC data that support data reports should be available for reference and inspection. This data should be retained by the laboratory for at least 2 years after project completion.

As part of an overall laboratory QC program, Reclamation and contracted laboratories should participate in recognized interlaboratory performance evaluation programs. These programs can assist the laboratory management in identifying and correcting weaknesses in analytical processes.

The following guidance is for the <u>minimum</u> QC to be practiced in the laboratory:

QC of Inorganic Analyses

- Instrument logbooks and maintenance records, including SRM/CRM dates (freshness) and sources.

- An approved SOP for each analytical method and sample preparation procedure (SOPs should be under continual development, by consensus (internal and external), and reviewed at least annually for possible formal revision).

- Quality control samples (with appropriate maintenance and use of control charts):

  Calibration standards
  Initial calibration verification (with appropriate traceable CRM)
  Method blanks (5 percent)
  Lab control samples (LCS)
  Instrument duplicates
  Matrix spikes, as appropriate
  Continuing calibration verification at end of run

- Corrective action loop - identify failed QC sample reconciliation methods.

QC of Organic Analyses

- Instrument logbooks and maintenance records, including SRM/CRM dates (freshness) and sources.

- An approved SOP for each analytical method and sample preparation procedure (SOPs should be under continual development, by consensus (internal and external), and reviewed at least annually for possible formal revision).

- Quality control samples (with appropriate maintenance and use of control charts):

  Calibration standards
  Initial calibration verification (with appropriate traceable CRM)
  Method blanks (5%)
  Lab control samples (LCS)
  Instrument duplicates
  Matrix spikes, as appropriate
  Surrogate spikes, as appropriate
  Continuing calibration verification at end of run

- Corrective action loop - identify failed QC sample reconciliation methods.

Organic analyses are very complex. The project quality requirements for organic sample analysis should be discussed by the project manager with his laboratory client representative (laboratory staff member assigned to project and responsible for the project manager's laboratory data product) and a qualified organic chemist, if the representative is not a qualified organic chemist. Where analytical services are obtained from a commercial laboratory, Reclamation laboratory staff should review the data as necessary to assure the project manager that QC measures specified in the QAPP were conveyed to the commercial laboratory and were performed.

Quality Assurance

This section should contain the laboratory provisions for assuring complete and appropriate data and internal verification of all recorded, calculated, and transferred data. At a minimum, complete data packages should be reviewed by a laboratory QA officer prior to release to the client.

An **ideal** laboratory QA system could begin with the designation of a qualified laboratory staff member as the client representative, or client contact person, who is personally responsible to the client for the laboratory project data, from receipt of the samples to release of the data report. The client representative should be responsible for performing a thorough check (every number, every calculation, every manual data entry or transfer) of the complete data package. Following the client representative review of the data package, the laboratory QA officer, or another designated employee with QA training not involved with the production of the data, should review the data for completeness and coherence, spot checking every third or fourth line in the data package. The laboratory manager should perform the final review and acceptance of the data package, spot checking every ninth or tenth data entry for accuracy. Any problem discovered at the second or final review level should send the package back to the client representative for resolution and initiate a second cycle of the complete review process. The **actual** system in use should be written, well defined (model source cited), and available to the client.

Following a successful review, a data report should be issued under the client representative's signature. The data report should include an explanation of all data qualifiers and the effect of those qualifiers on the usefulness of the data to the client.

Laboratory policy regarding release of data prior to the final certified data package should be explained in detail. The QAP should indicate the minimum content and standard format of the data report, including any willingness to provide data in the user's preferred format.

Subcontracting

If the Reclamation laboratory relies on outside contract laboratory support for some analysis work, such as mercury quantitation for a trace metals suite, the contract laboratory should provide enough information to satisfy any QA/QC concerns a client may have regarding the portion of analytical work to be performed by contract. If the contract work product is critical to the success of the project, the client (project manager) may wish to include (additional) control samples. Therefore, the client must be aware of any contract laboratory relationship that impacts the project.

Training

The QAP should include a statement of laboratory policy regarding maintenance of staff capability and internal instrument/method certification, including any annual or recurring training or certification required for technicians or managers.

Rates and Response

The laboratory rates for services should be available in a separate document that is kept current. The sample submission to data-report-release response time (turnaround) should also be included in the rate, and any special rates for accelerated (priority) analysis and data reporting should be included.

Reference Data

The laboratory QAP should include any useful references or tables that educate or inform potential clients regarding analytical methods, sample preservation methods, recommended container types and sizes, allowable holding times by analyte, etc. Any required or useful forms may also be included.

BIBLIOGRAPHY

ANSI/ASQC, *Quality Management and Quality System Elements for Laboratory - Guidelines*, American National Standard ANSI/ASQC Q2-1991, American National Standards Institute/American Society for Quality Control, Milwaukee, Wisconsin, 1991.

Dux, James P., *Handbook of Quality Assurance for the Analytical Chemistry Laboratory*, Van Nostrand Reinhold Co., Inc., New York, 1986.

Eichelberger, T.W., et al., *Analytical Chemistry*, 47, 1975, 995-1000.

EPA, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, U.S. Environmental Protection Agency, National Environmental Research Center, Cincinnati, Ohio, 1972.

———, *Methods of Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, revised March 1983.

———, *Test Methods for Evaluating Solid Waste*, 3rd ed., SW-846, Office of Solid Waste and Emergency Response, Washington, DC, 1986.

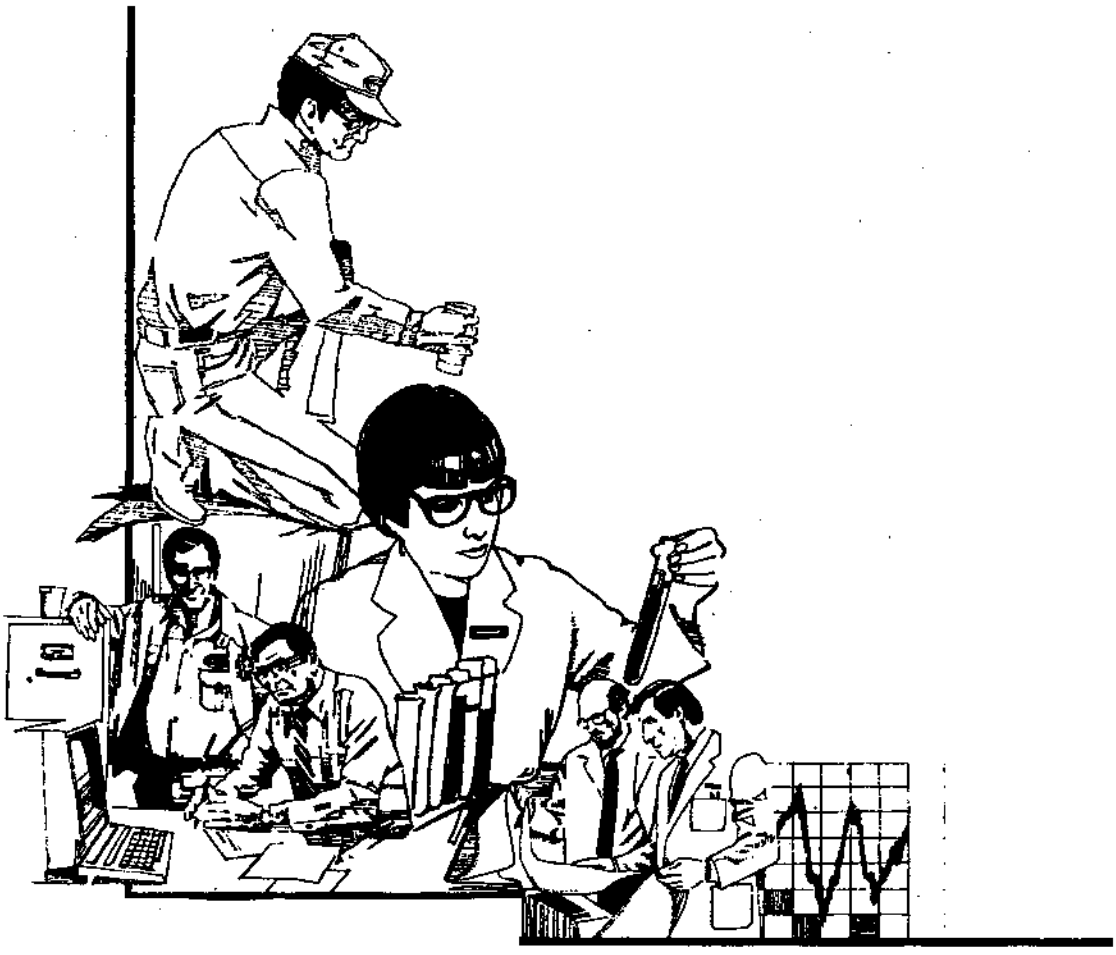
Garfield, Frederick M., *Quality Assurance Principles for Analytical Laboratories*, Association of Official Analytical Chemists, Arlington, Virginia, 1984.

Taylor, John K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, Michigan, 1987.

REFERENCES

ISO/IEC, General Requirements for the Competence of Calibration and Testing Laboratories, *Guide 25*, 3rd ed., International Organization for Standardization (Case postale 56, CH-1211), Geneve, Switzerland, 1990.

PART 3 - GENERAL GUIDANCE



PART 3 - GENERAL GUIDANCE

QUALITY ASSURANCE OF DATA MANAGEMENT

QA of data management is the planned system of activities used to ensure sound and defensible recording, storage, and retrieval of all essential data. Quality management practices require that all activities having an impact on data quality or process operations shall be documented explicitly so that activities are performed consistently and efficiently by all personnel.

A data collection project cannot be substantiated without a records program. For projects engaged in regulatory activities, the records program must be explicit for chain-of-custody and sample information if the findings are to stand the test of close examination in legal proceedings.

Field and laboratory QA practices for producing and maintaining accurate records should be described in standard operating procedures (SOPs).

Responsibilities

Specific responsibilities of project managers, supervisors, and field and laboratory staff for QA of environmental investigations are identified throughout these guidelines. Each project participant has responsibilities for data management as described in written laboratory and field QAPs and determined by the Project's DQOs (data quality objectives). The suggested responsibilities for control of data management are summarized as follows:

Project manager - The project manager is responsible for the records which document the overall quality of project activities. It is also the manager's responsibility to develop and describe project DQOs (in conjunction with the data user), communicate the objectives to project participants, and ensure that the resulting data meets the needs of the project.

QA officer - The QA officer is responsible for daily assurance of project field data record quality and interim record management. The QA officer delivers complete data records to the Project Manager with recommendations as to quality improvement methods and any shortcomings in completed records. The Project Manager and the QA officer may be the same person for smaller projects.

Laboratory staff - The staff of the testing laboratory is responsible for implementing the quality system of the laboratory. The system evaluates and controls the performance of the laboratory's record management system. The staff implements the records control measures and continually evaluates the process by the use of check data, system audits, staff evaluations, and specific written performance requirements. It is the staff's responsibility to maintain the records which document the quality of the laboratory's performance.

Field staff - The field staff is responsible for knowledge of the requirements of recordkeeping and control of records as described in the project QAP. The Field Staff is also responsible for the performance of QC measures as specified in written procedures (SOPs), the accuracy and completeness of their observations and transcriptions, and the security of records in their possession.

Sample Accountability

Specific SOPs prescribe the activities involved with movement of a sample from time of collection until time of disposal. Documentation of sample collection, chain-of-custody, sample analysis, tracking, storage, and disposal of samples includes specific recordkeeping requirements and provides for record filing and security. Sample labels and field records in logbooks must fully and unquestionably identify the sample. Formatted sample labels and field worksheets ensure completeness. Use appropriate logbooks and forms which provide for signatures and dates of regular supervisory reviews of records.

Assign record custodian duties to specific field and laboratory personnel. Responsibilities of the field custodian include maintenance of logbooks and records for sampling and field testing, sample shipment and chain-of-custody records, and field standard operating procedures. The laboratory custodian is responsible for maintaining and securing records of sample receipt, inspection and verification of information on sample documents, and records of performance of sample tracking, storage, and security systems.

Records Control

Handwritten records are best made in bound books which establish the sequence of insertion of data. However, to increase efficiency and convenience of operation and minimize transposition, forms for worksheets and labels are an acceptable alternative. Serialize forms and use carbons or photocopies to submit partially completed worksheets. Three-ring binders and folders can be used to store and protect sampling and analytical records. If notebooks are used, have a standardized notebook control procedure. Serialize notebooks and control their distribution.

Describe documentation requirements in field and laboratory SOPs. Fully identify personnel responsible for recording data and initial and date all entries. Use permanent ink to record all data. Make changes by lining out the original entry and writing in the revision. Erasures, use of liquid paper, and removal of pages are not acceptable. Initial and date all changes and state the reason for the change. Retain all original records for the statutory period, 1 year minimum.

Field data and observations are important project records, as are the records of QA checks, method development, research references and data, training, seminar and meeting information, and daily logs of activities. Keep equipment logs to record instrument checks, preventive maintenance, repairs, calibration data, standard checks, and other information relative to equipment performance.

Document the procedures for completing, checking, and retaining all records. The procedures must identify how the record is produced, reviewed, distributed, and filed. Include copies of blank data forms and the format for final reports.

Data Security

Data and other project records must be safeguarded by field and laboratory staff. Establish chain-of-custody requirements for tracking and storing laboratory and field notebooks, manuals, equipment logs, data sheets, forms, worksheets, and reports. Chain-of-custody requirements for records must include the name and location of the person to whom the manual, report, or notebook has been assigned and the dates of assignment and return.

Verification of Records

Verification procedures for data recording are described in each activity SOP which describes acceptance and rejection criteria and lists corrective action to apply. Verify field measurement records with second-party reviews or laboratory checks of field produced data, when possible. The laboratory staff shall certify that all analytical records meet the requirements described in the laboratory or project QAP. Verify that records provided by contractors are complete and clear and meet specified data management requirements.

Document the verification of all data reduction and handling processes. Use dummy data and compare produced results against expected or hand calculated results. Data reduction errors may occur only under special conditions, so careful and thorough checks must be made on a continuing basis to reduce errors.

Reviews and Approval

Document review and approval criteria for accepting sample data information, laboratory work, and reports. All records must be critically reviewed by the laboratory or field supervisor or his designee for specific items; such as complete sample information, including the person producing the record; checks of analytical testing and transcription accuracy; and completeness of reports. The review and acceptance of reports are documented on the report by the full signature of the supervisor along with the date of the review.

File and maintain signed copies of all records and reports in the laboratory or project office. Maintain distribution lists as supplements to the report. Only individuals who are authorized to review and verify data for reporting purposes should release records.

Maintenance of Records

Poor records maintenance reflects poor quality control in other areas of operations.

The record maintenance system describes, in writing, the types of records; the steps required to produce and review the records; their distribution, storage location, and length of storage; and conditions for destruction of records (including sample disposal). Establish a filing system for storage of all records. Keep original records of project activities in a secure location. Assign responsibility to specific individuals for filing records. Designate authority to remove or release records to only specific individuals.

Establish security of records by using a sign-out system to track records and to ensure that records are returned to their proper locations.

Establish a system for reviewing records maintenance. Identify records that must be updated on a routine basis and determine a time line for updating those records. The laboratory supervisor or project manager is ultimately responsible for all records produced and stored as a result of laboratory or project activities. Field staff and analysts are responsible for maintaining records for their specific areas of responsibility.

Computer Management

Computers are being used more and more to produce and control records. Security of computer operations, software, and file storage requires adequate procedures for control and review.

Describe methods for confirming that only accurate data have been entered in the computer. Data entry methods should incorporate check systems and prompts to force the computer user to consider the validity of the data and to avoid entry of obviously erroneous data. The check system should prohibit the use of a sample identification number for more than one sample and warn of data that does not fall into a normal range. Include questions to validate replacing previously entered data or deleting data. The computer SOP designates specific responsibilities for sample log-in, data entry and verification, and preparation of reports.

Describe computer data handling and record storage processes. Use document checks to ensure accurate transfer of data into records with no loss of data or desired sensitivity. The computer software must ensure that data entries which would destroy information without deliberate action are not accepted.

State the check criteria and documentation requirements for regular review of computer processes. The review process should check every operation performed on data by the computer.

Specify and perform hard disk backups once a week, or more frequently if significant amounts of data entry occur. Store backup disks in a designated remote and secure location. Use three to four sets of backup disks to ensure minimal loss of data in case of a “head crash” or computer theft.

Restrict access to computers by use of passwords. Describe the password system and its maintenance. Describe procedures to ensure security against inadvertent destruction or misrepresentation of data or deliberate tampering with computer processes. Control access to storage devices to prevent tampering with computer files.

BIBLIOGRAPHY

Dux, James P., *Handbook of Quality Assurance for the Analytical Chemistry Laboratory*, Van Nostrand Reinhold Co., Inc., New York, 1986.

Garfield, Frederick M., *Quality Assurance Principles for Analytical Laboratories,* Association of Official Analytical Chemists, Arlington, Virginia, 1984.

Taylor, John K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, Michigan, 1987.

SELECTION OF AN ANALYTICAL LABORATORY

After sampling, the performance of the analytical laboratory most directly influences the quality of decisions based on analysis results. The evaluation and selection of a laboratory to perform analytical services is an important task and requires a **planned approach** and an **objective rating criteria**. The evaluation process results in a series of products which reflect a laboratory's general overall **performance capability** and the quality of its analytical services. It is a means to select the most qualified laboratories to perform analytical tests.

Laboratory Assessment

The assessment process for selecting an analytical testing laboratory begins with a **review of general information** provided by the laboratory to determine if the project's data quality objectives (DQOs) can be met. The review includes the laboratory's analytical capabilities, its methods, quality control and reporting processes, and costs. If the DQOs can be met, the laboratory's **quality assurance plan (QAP)** is reviewed, and a **performance evaluation** is conducted. The final step in the assessment process is an **onsite review** of the laboratory operation to verify conformance of the laboratory activities with the laboratory's QA plan.

The selection process can be accomplished by project personnel with the cooperation of laboratory staff from the regional or area offices or personnel of the Technical Service Center. (See Preface.)

A detailed description of the selection process follows.

Step 1. Solicitation of General Information

The first step in selecting the most qualified laboratory from several competing laboratories is to determine which of the laboratories can meet the data collection program's DQOs. This is determined by reviewing each laboratory's analytical capabilities.

In order of importance, the initial review must confirm that a laboratory has the capability to:

- Analyze the constituents of interest
- Measure the constituents at the concentration ranges required
- Produce measurements with an acceptable level of confidence
- Provide results within the time requirements
- Generate a data report which is acceptable in content and format (computer disk or hard copy)
- Conduct the work within the analytical budget

Results from participation in interlaboratory performance evaluation programs or proficiency tests can provide a measure of a laboratory's general analytical capability and the comparability of its results with those of other laboratories. Interlaboratory evaluation programs are conducted by EPA and the U.S. Geological Survey (USGS) to qualify laboratories performing testing for these agencies.

This review can be used to eliminate from further consideration laboratories that are not capable of producing data that meet the program's objectives. The next step is to request and review QA plans from the remaining laboratories.

Step 2. Review of Laboratory QA Plans

A QA plan or manual should describe the processes and documentation by which a laboratory produces and supports the quality of its analytical data. The QA plan should reflect the commitment of the organization and staff to support and provide what is expected.

A laboratory's QA plan should be evaluated in terms of thoroughness and completeness and should be specifically examined for the following items:

- **QA policy** – The plan should state the laboratory's commitment to analytical data which are accurate, reliable, and adequate for the intended purpose and produced in a cost efficient manner under a planned and documented quality system of activities.

- **Organizational structure** – The plan should identify the laboratory's organizational structure and line of authority. Key individuals, including the laboratory director and the quality assurance officer (QAO), should be listed. The responsibilities of the key members of the organization should be described. The QAO should be independent of the laboratory supervisor and report directly to the laboratory director.

- **Qualifications of staff** – The number and types of positions as well as the educational background and professional experience of the staff should be described. The plan should state that training records documenting staff development should be maintained.

- **Laboratory facilities** – The plan should include a laboratory floor plan or describe the amount of floor and bench space available for testing activities. It should describe the adequacy of the facilities for sample preparations and storage, as well as the number of laboratory sinks, hoods, ovens, freezers, and refrigerators. Office, laboratory, and file security and safety at the work site should be addressed.

- **Laboratory instrumentation** – The plan should list major analytical instruments and identify the model and manufacturer, the year purchased, and whether the equipment is under service contracts.

- **Laboratory environment** – The plan should describe the environmental conditions (i.e., air and humidity controls and water and electrical service) and the housekeeping policies of the laboratory.

- **Methods and procedures** – The plan should state the specific analytical methods used by the laboratory, by name, and reference each to a single published procedure of the scientific community (e.g., ASTM, EPA, USGS, AOAC). Analytical methods cited in

the Code of Federal Regulations (40 CFR 136) are acceptable. Any method modifications (e.g., lower detection limit, application to other sample types) must be supported by adequate documentation.

- **Sample handling** – The plan should describe the laboratory's sample control process. This process description should include sample identification and tracking, handling, storage, security, and disposal. Management processes should provide assurance that holding times can be met for low concentration level analytes in samples. (See Table 1 of section on Sample Collection Design.)

- **Quality control (QC)** – A description of the QC practices used by the laboratory for establishing the quality of test results should be a major component of the plan. The QC practices described should include verification of the purity of calibration standards, equipment performance and calibration checks, and data quality checks (blanks, duplicates, spikes, and control samples). The process for review of test results, including acceptance criteria, sample holding times, and application of corrective actions should be described. Quality system reviews and participation in performance evaluation sample programs should be described.

- **Data reports** – The plan should describe the content and format of testing reports provided to clients. Data quality indicators of accuracy, precision, detection limits, and method references for each constituent, as well as the signature of the laboratory supervisor authorizing release of the report, should be included with the report. The data management process should include how the laboratory retains and controls original data and laboratory notebooks which support data reports.

- **Laboratory procedures manual** – The plan should state that all laboratory procedures are described in detail in a laboratory procedures manual. This manual should include all the step-wise standard operating procedures (SOPs) for the analytical methods used by the laboratory. The SOPs should include all the sample handling, analysis, and quality control processes and be consistent with statements in the QA plan. The QA plan should describe the responsibilities and authorities for preparing, approving, and modifying SOPs.

- **Subcontracting** – The QA plan should indicate the laboratory's regular and incidental use of a contract laboratory (or laboratories) to perform specific analyses, and the plan should include the pertinent contract laboratory QA/QC disclosures regarding the specific analyses performed by the contracted laboratory for the client's laboratory.

- **Corrective action loop** – The QAP should contain references to corrective action loops. These corrective actions should clearly state the logical steps to be taken if samples fail to meet QC specifications or tolerances and how other QA/QC measure shortcomings will be addressed. Certain options, such as rejecting an entire batch of samples instead of running the QC sample again, could be very costly for a client. Not having predefined corrective actions postpones decisions to critical decision periods and introduces new sources of variability in data.

Laboratories that do not submit a QA plan for review or those that submit a plan that does not adequately address the items listed above should be eliminated from the selection process. The remaining laboratories should be ranked according to the completeness of their QA plan.

Step 3. Performance Evaluation

Following the review and acceptance of the laboratory's QA plan, the third step in the selection process is to conduct a performance evaluation. A performance evaluation is a means of assessing a laboratory's ability to measure the constituents of interest to the necessary quality level.

The evaluation is accomplished by submitting performance test samples to the laboratory for analysis and evaluation of the results against actual values. The laboratory may or may not know that the samples are designed to test the laboratory's analytical abilities (single or double blinds). The laboratory analyzes the constituents of interest according to their stated procedures and reports the results to the prospective client or evaluator. The evaluator assesses the data quality based on the actual values and the requirements of the project's DQOs. An analytical laboratory will normally participate in a performance evaluation at no cost to the potential client.

The number of performance test samples submitted to a laboratory will depend on the requirements of the data collection program. Samples should be similar in type and composition to the project samples. A laboratory's abilities may be masked if performance samples do not closely match the composition of the project samples. One or two project samples may be submitted in duplicate to assess the reproducibility of constituent analyses.

Performance samples can be natural or prepared reference materials that have been analyzed to such a degree that the true (actual) values for constituent concentrations have been established. Vendors of laboratory supplies offer a wide variety of evaluation sample types and constituents and may also provide for interlaboratory comparisons of test results. The National Institute of Standards and Technology (NIST) Standard Reference Materials (SRM) program can provide a wide variety of materials for which true values of constituents have been certified. The USGS (Branch of Quality Assurance, Box 25046, Denver, Colorado 80225) sponsors a semiannual, interlaboratory testing program which can provide nine natural-material reference sample types, including water and sediments, to USGS and non-USGS laboratories by request. Performance samples are provided to laboratories participating in State and Federal drinking water or wastewater testing programs required by the EPA. Participating laboratories may volunteer results of these programs.

Alternately, for water, spiked and unspiked project samples may be used as performance evaluation samples.

The laboratory reporting results for performance evaluation samples should use the laboratory's reporting format, which should include method references, detection limits, and data review and approval signatures.

For performance evaluation, the accuracy of a test result, **percent recovery (%R)**, is determined by comparing the reported analyte concentration to the actual concentration by the following formula:

$$\text{Percent recovery (\%R)} = \left(\frac{\text{Reported concentration}}{\text{Actual concentration}} \right) x\ 100$$

For spiked samples, the difference between the reported constituent results for the spiked and unspiked samples is compared to the concentration of constituent actually added ("spiked ") to determine the percent recovery (%R) of the spike by the following formula:

$$\text{Percent spike recovery (\%R)} = \left(\frac{\text{Spiked concentration} - \text{un-spiked concentration}}{\text{Spike concentration added}} \right) X\,100$$

Another way to evaluate analysis accuracy is to perform *spike recovery* determinations. Spike recovery can be used to evaluate matrix effects (analyte signal supression or enhancement) from other analytes present in the sample (as in spectroscopic methods such as AA or ICP) or to assess potential analyte losses during extraction or digestion (for organic analyses that require extraction or inorganic tests that require digestion). In general, spike recovery is determined by comparing the spiked sample concentration of spike analyte to the recovered concentration of sample spike analyte.

There are two general approaches to preparing spikes. The "sample + spike" approach is performed by adding (or spiking) a *known* volume of a standard containing a *known* concentration of the analyte of interest into a *known* volume subsample of a randomly selected real sample. Note that *known* means that the spiking standard is a fresh, certified, standard solution traceable to an accepted SRM and that sample and spiking volumes are calibrated and quantitative. Calculate percent spike recovery for the sample + spike method as above. Percent recovery values less than 100 percent suggest analyte signal suppression or loss, and %R > 100 suggests analyte signal enhancement or addition (as with contamination).

The alternative to this approach, called "blank + spike," is to spike a blank along with a randomly selected real sample. This method is performed by adding identical volumes of spiking solution (usually with a calibrated auto-pipet) to sampler cuvettes containing a deionized water blank and sample of equal volume. The benefits of this approach are that the spiked concentration is measured directly (rather than being calculated), and a certified, fresh spiking solution is not required.

An important consideration when evaluating spike recoveries is whether the spike concentration is appropriate relative to the sample concentration. The CLP *Inorganic Statement of Work* (EPA 1991) suggests that the spike concentration should be at least 25 percent of the analyte concentration. There are also problems if the spike concentration is greater than several times sample analyte concentration. Percent spike recovery values calculated when sample and spike concentrations are outside these limits should not be calculated or used to evaluate, qualify, or reject data. Refer to the project QAP or applicable guidance documents (SW-846 or the CLP *Statements of Work*) for recommendations on appropriate spiking levels.

The precision of a test result may be determined by calculating the **relative percent difference (RPD)** for measurements of the same analyte on duplicate samples by the following formula:

$$\text{Relative percent difference (RPD)} = \left(\frac{\text{Duplicate}_1 - \text{Duplicate}_2}{(\text{Duplicate}_1 + \text{Duplicate}_2) / 2} \right) X\,100$$

Remember, if the sample chosen by the analysis lab for RPD determination contains analyte below the limit of detection (LOD), RPD precision should not be calculated. Also, if the analyte concentration is less than five-times the LOD, the RPD should not be calculated or used to judge analysis precision, to reject or to qualify results. If the analyte concentration is less than five times the LOD but greater than one-times the LOD, duplicate samples should be within ± one times the LOD.

The adequacy of a laboratory's capabilities should be evaluated against the project's objectives. Project sample types other than water or with complex matrices may require special considerations. Generally, for water, the following **acceptance criteria** for performance sample results are reasonable (consult with your lab on specific cases).

Accuracy

Reference material samples - If the reported result for a performance sample is greater than 20 times the method's detection limit, the result is acceptable if the percent recovery (%R) is within **80 to 120 percent**. If the reported result is less than or equal to 20 times the detection limit, the result is acceptable if it is in the range of the actual concentration plus or minus two times the detection limit.

Spiked samples - The accuracy of the analysis of a spiked sample is generally acceptable if the percent recovery (%R) is within **75 to 125 percent**. Consult your qualified laboratory QA personnel for specific acceptable ranges. Performance limits do not apply when the concentration of the analyte in the unspiked sample exceeds four times the spike added.

Precision

Duplicate samples - If the average result of an analysis on duplicate samples is greater than five times the method's statistical detection limit, acceptable precision is (generally) **not more than 20 percent (i.e. ≤20%) RPD**. If the average result of duplicate sample analyses is less than five times the detection limit, the precision of the duplicate results should be within plus or minus the value of the method's detection limit.

Acceptance criteria provided herein cannot apply to all analytes, sample types, matrices, or methods of analysis. Project managers are encouraged to discuss analytical requirements with the laboratory in the DQO and sampling planning stages.

Competing laboratories should be ranked according to the evaluation of the performance sample results for the constituents of interest. Laboratories unable to analyze performance samples with the accuracy and precision required for the project objectives should be eliminated from further consideration.

The performance sample ranking should be combined with the assessment of the laboratories' QA plans to develop a list of the most qualified laboratories. Prior to a final laboratory selection, a Quality System Review should be conducted on one or more of the top candidates.

Step 4. Quality System Review

The final step in the laboratory selection process is an inspection of the laboratory operation. The onsite "system audit" verifies the laboratory's conformance with the procedures and policies described in its QA plan. The visit provides assurance that the organization and personnel are qualified to complete the analytical requirements as expected, that adequate facilities and instrumentation are available, and that the necessary quality control and data handling procedures are used. The system review should be conducted for the project by technical staff who are familiar with the laboratory aspects of the project's requirements.

A checklist for performing a Quality System Review follows. The checklist serves as a guide for the review team to ensure that the scope of the review is adequately covered. It also provides a place for the reviewer to document the items inspected and the observations. The review checklist can be used to prepare a list of questions to be asked during the review which can assist the reviewer in understanding laboratory processes.

Following the system review, the selection of a qualified laboratory can be made with the assurance that a reasoned approach has been applied to the selection process. If more than one laboratory is qualified, the restrictions of time and/or budget may be considered to make a selection decision.

BIBLIOGRAPHY

ASTM, D 3856, Standard Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water, *Annual Book of ASTM Standards*, vol. 11.01, American Society for Testing and Materials, Philadelphia, Pennsylvania, 1988.

EPA, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA-600/ 4-79-019, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 1983.

Friedman, Linda, and David Erdmann, Quality Assurance Practices for the Chemical and Biological Analyses of Water and Fluvial Sediments, Chapter 6, Book 5 of *Techniques of Water-Resources Investigations of the U.S. Geological Survey*, USGS, U.S. Government Printing Office, Washington, DC, 1982.

ISO/IEC, General Requirements for the Competence of Calibration and Testing Laboratories, *Guide 25*, 3rd. ed., International Organization for Standardization (Case postale 56, CH-1211), Geneve, Switzerland, 1990.

REFERENCES

EPA, *Statement of Work for Inorganic Analysis, Multi Media, Multi-Concentration*, Attachment A, Document Number ILM01.01, USEPA Contract Laboratory Program, 1991.

CHECKLIST FOR PERFORMING A QUALITY SYSTEM REVIEW

Informally meet with laboratory personnel.

Summarize the system review process with Laboratory management.

Determine laboratory capabilities:

- Sample types
- Analytical and preparative processes
- Sampling, contracting, research, data interpretation, technical assistance

Determine the extent of the laboratory's control of its operation:

- Management involvement
- Defined responsibilities and organizational structure
- Independent reviews of performance
- Client and sampler relationships
- Training from written procedures by experienced personnel
- Limitations on the Laboratory's ability to perform under GLP

Determine the personnel resources available:

- Background, experience, and training
- Capabilities of staff to advance and train in new areas
- Quality measures included in performance reviews
- Staff turnover
- (Interviews) Staff understanding of QC and techniques

Determine the extent of the laboratory's analytical facility:

- Adequacy of space, environmental controls, sample/reagent storage conditions
- Equipment condition and protection against excessive down time
- Safety program and Material Safety Data Sheets (MSDS)
- Hazard containment (sprinklers, alarms, personnel protection)
- Offices and files (security, protection, accessibility)

Determine the laboratory workload:

- Sample types and numbers (percent each of total)
- Constituents determined (percent metals, nutrients, inorganic, organic of total)
- Percent of total time spent on routine, varied, and unusual samples
- Limitations (policies, facilities, staffing, seasonal workloads)

Determine the existence of an external QC program:

- Frequency and number of splits, reference material samples, duplicates, and spiked samples submitted with samples
- Project communication with Laboratory regarding results of external QC
- Certification and/or evaluation sample program participation

Evaluation sample program/certification comments.

Determine presence of a preventive maintenance program:

- Equipment operation manuals available
- Ventilation hoods regularly checked
- Equipment condition regularly established and certified
- Service contracts in place
- Staff responsibilities defined and assigned
- Balances serviced and certified

Determine the adequacy of sampling control:

- Field responsibilities defined and reviewed
- Sample traceability (chain of custody)
- Use of forms, labels, seals, and carbon copies (completeness)
- Sample security

Examine the sample processing system:

- Log-in procedures and sample storage conditions
- Laboratory ID of samples
- Record of sample condition on receipt
- Verification of sample label and submittal information
- Sample disposition and disposal
- SOPs for extractions and digestions
- Processing of soils, sediments, biota
- Standard references to processes

- QC requirements specified in SOPs (reference material samples, method blanks, spiked samples, and duplicates; number and frequency; acceptance criteria)

Processing SOPs reviewed / comments.

Determine the adequacy of sample analysis procedures:

- Calibration and verification
- Reagent purity or traceability to standards
- Frequency and documentation of calibrations
- Independent verification of calibration accuracy; acceptance limits
- Use of control charts for performance criteria
- Blanks required to be less than the detection limit
- Documentation on original data (preparation/analysis date, analyst name, and acceptance certification)
- Holding times met

Methods:

- Acceptable, referenced methods
- SOPs available and followed
- Process for validation of lab-developed procedures
- QC criteria and corrective action specified in SOPs

Methods reviewed / comments.

Determine the adequacy of data control procedures:

- SOPs for data review and records handling
- Data transcriptions checked
- Data reduction software processes documented and verified
- Original data retained and accessible
- Analyst and data reviewer identified
- Records systematically retained and controlled

Records reviewed / comments.

Determine adequacy of reports:

- Format: disk, electronic, paper (fax)
- Includes laboratory name and supervisory signature
- DQIs (data quality indicators) with data: reference method, precision, bias, detection limit, holding time compliance
- Lab number traceable to sample identification
- Work request retained
- Report clear and complete

Determine the adequacy of QC in field sampling (if applicable):

- Field SOPs and quality controls
- Training records
- QC documentation (calibrations, check sample results, field records)
- Regular review of procedures, performance, and documentation

Conduct closing meeting with Laboratory management to summarize conclusions of review:

- Overall perception of laboratory quality system
- Adequacy of laboratory QA Plan
- Conformance of laboratory processes to QA Plan
- Commitment of staff and management to meet client expectations
- Communications / TQM: involvement of staff, management, project staff
- Facilities: safety and security
- Adequacy of internal QA/QC
- External QC performance
- Implementation of corrective actions
- Recordkeeping
- Recommendations
- Timeframe and distribution of review report

BIBLIOGRAPHY

Dux, James P., *Handbook of Quality Assurance for the Analytical Chemistry Laboratory*, Van Nostrand Reinhold Co., Inc., New York, 1986.

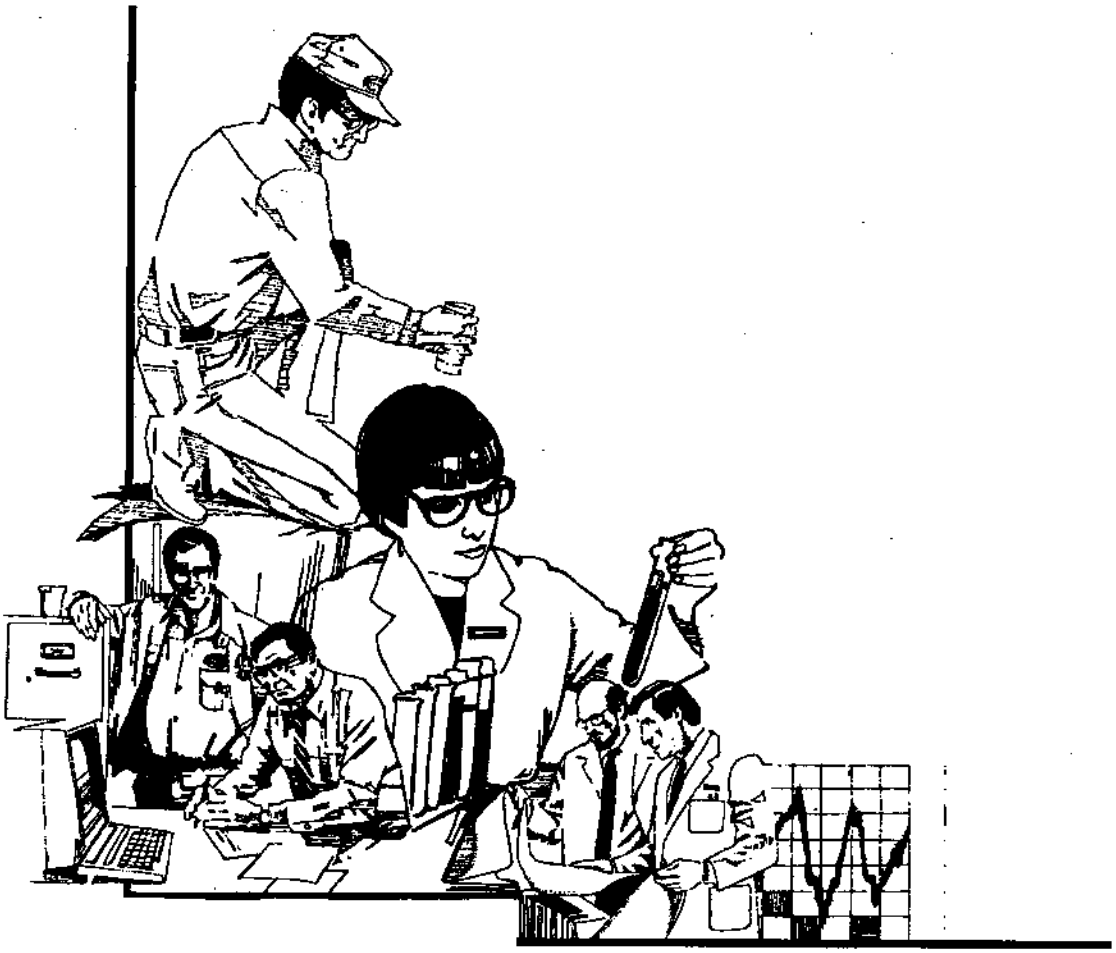
EPA URL - http://www.epa.gov/r10earth/offices/oea/qaindex.htm

EPA, *EPA Guidance for Quality Assurance Project Plans - External Working Draft*, EPA QA/G-5, Quality Assurance Division, Washington, DC, November 1996.

Garfield, Frederick M., *Quality Assurance Principles for Analytical Laboratories*, Association of Official Analytical Chemists, Arlington, Virginia, 1984.

Taylor, John K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, Michigan, 1987.

PART 4 - FIELD SAMPLING METHODOLOGY



PART 4 - FIELD SAMPLING METHODOLOGY

An important aspect of obtaining accurate, representative water quality data is to define the data needs (parameters), the hydrologic conditions (high, low, and annual variation) that need to be sampled, and the area of interest (watershed, tributary, specific point in stream, etc.) that the data will represent. A sampling plan is developed to cover the data to be collected and the physical sampling areas that will answer the identified questions and concerns. The samples that are submitted to the laboratory for analysis should be representative of the conditions in the field and collected using protocols that will ensure collection of accurate, representative samples that have been protected from contamination during the collection process, during transportation to the laboratory, and during laboratory analysis.

A sampling plan is established to ensure that field personnel have adequate direction to obtain the required sample volumes, that samples are collected at proper locations, and that appropriate field information is recorded. Care must be taken to ensure that samples represent the quality of the media being sampled. For example, large water bodies usually require that many samples be collected to represent the entire water body location at the time of sampling. Different procedures are usually required for sampling different media and for obtaining different data. Much has been written on sampling procedures and the steps that need to be taken to ensure that representative data are obtained. The main sources of procedures are the USGS and EPA. They currently support a National Water Quality Monitoring Council (NWQMC) which has representation from States and Federal agencies, industry, and environmental groups. The council is developing data collection guidelines and working with data collection entities to ensure that data collection results are representative of existing water quality and that the data can be shared and used by others without major QA/QC concerns. Council membership, objectives, and work progress can be obtained from their website at http://water.usgs.gov/wicp/acwi/monitoring/.

The USGS National Water-Quality Assessment Program (NAWQA) has established sampling protocols and written reports to provide guidance to NAWQA study teams. These reports are referenced for additional guidance and are briefly summarized in this document. The guidance covers most types of water related data such as water chemistry; sediment quantity and quality; benthic organisms, numbers and diversity, and toxic chemical content; algae numbers and species; and fish species and toxic chemical buildup. The referenced reports also contain additional references that provide more detailed information.

Surface Water Sampling

When sampling surface water, the objective is to obtain a sample that will represent the quality of the water body at that instant of time and location. To accomplish this, special procedures are often required. The USGS (Shelton, 1994) provides procedures to follow when sampling streams to get representative samples for chemical analysis. USGS discusses equipment needs and sampling procedures such as Equal-Width-Increment sampling, Equal-Discharge-Increment sampling, and nonstandard sampling. For all sampling activities, field logs should be maintained to record field data and any deviation from the established protocol. It is best to provide streamflow data associated with all stream quality samples. If there isn't a gaging station nearby with flow data, an estimate is better than no flow information at all. If flow is estimated, the method used should be described in the field notes. Also, field data should be collected for air and stream temperature, pH, electrical conductivity, and dissolved oxygen. A written physical description of the stream color, flow,

sediment, algal conditions, and any other important or unusual conditions that can be identified should be recorded. Sampling of lakes and reservoirs should cover the above information as well as information about the water surface elevation and inflow and outflow conditions.

Groundwater Sampling

Three types of groundwater quality studies are typically conducted: study unit or area water quality surveys, land use impact water quality studies, and flow path water quality studies. Each study type can require different well requirements to ensure that the proper data are collected for the intended purposes. Well selection is based on the study type and the information known about the wells and the aquifer. The well sampling process must be able to produce the data required to best answer the questions identified for the study. In area studies, the wells must be distributed over the identified study area. The study area should be subdivided into three to five subunits, based on physiographic and hydrogeologic features. Land use studies require the area to be divided into appropriate land uses. Wells must be established to sample the uppermost part of the aquifer in each identified land use area. Flow path studies are generally conducted in unconsolidated shallow aquifers along the inferred flow path. The studies commonly measure interactions of groundwater and surface water. Wells are required at various locations and depths along the inferred flow path and must be aerially distributed in the vicinity of the believed flow path. Additional information concerning groundwater investigations can be found in the USGS Open-file Report 95-398, by Lapham and others (1995).

Water level measurements are required each time a well is sampled unless the well construction makes it impossible to obtain the measurement. Before collecting the water sample, the well should be pumped until the measured temperature and specific conductance are constant. When aquifer yield limits the amount of water available for sampling, the well should be pumped until a volume of water equal to the volume of the casing and the filter pack around the well screen has been removed. The well is then allowed to recover before obtaining the water sample for analysis.

Bed Sediment Sampling

Bed sediment samples are collected to determine concentrations of trace elements and hydrophobic organic contaminants at a particular site. Bed sediments in depositional environments of streams can provide a time integrated sample of particulate matter transported by the stream. The concentrations of contaminants in streambed materials are strongly affected by the size distribution of sediment particles in the sample. Generally, the concentration of trace elements in streambed materials increases as particle size decreases. However, the concentration of organic contaminants in bed sediments is not significantly affected by sediment particle-size distribution and is more a function of the amount of organic matter in the sediment. To increase the probability of detecting trace elements and to enhance the comparability of data between sites, bed-sediment samples should be sieved and the fine-grained fraction analyzed for the contaminants of interest. For trace elements, the silt-clay fraction smaller than 63 μm should be saved for analysis. For pesticides and other organic contaminants, the sand and silt-clay fraction smaller than 2.0 mm should be saved for analysis.

The appropriate season and the optimum hydrologic conditions for sampling streambed sediment are determined by current and antecedent discharge conditions. These conditions are usually during low flow, which provides maximum direct access to the streambed and minimizes seasonal streamflow variability. These variables should be taken into account in establishing the sampling plan. Shelton

and Capel (1994) provide more information to help develop the sampling plan, including a potential parameter list for the study, the number and location of samples, and the required equipment for proper sample collection.

Biological Sampling

Biological tissue sampling to determine contaminant levels in water can help to describe pollution of streams and lakes. However, several difficulties are inherent in any study of contaminant concentrations based on organisms. Many tissue analysis programs have shown that, with rare exceptions, contaminant concentrations are comparable only within the same species and at the same life stage, reproductive condition, size, weight, and sex. For most surveillance studies, relatively sedentary organisms whose exposure can be linked to a particular site are preferable to organisms that move or migrate. The selection of species to sample is important, and a biologist familiar with biological contaminant accumulation and assessment should be consulted to ensure that study objectives are properly defined and can be met. Obtaining sufficient sample mass to ensure that all parameter concentrations can be determined by the laboratory may be difficult when conducting biological sampling for organic chemical testing.

Significant information concerning biological sampling, potential taxa that can be sampled to meet study objectives, and the sample size required can be obtained from the USGS NAWQA publication written be Crowford and Luoma (1993). They discuss how to sample to meet various study objectives and the appropriate biologic community to sample, depending on study objectives. Other reports are available that can provide additional guidance on sampling procedures, required permits, and sample sizes. Fish sampling is covered by Meador and others (1993). Guidelines for benthic invertebrates are covered by Cuffney and others (1993).

Soil Sampling

Soil samples should be collected by a soil scientist or someone familiar with soil surveys and land classification. After identification of the parameters of concern for the soil sampling, the existing soil survey information for the area should be reviewed to identify blocks of similar soil. The existing information should be studied to identify sampling sites that will best meet study objectives in defining concentrations of expected contaminants in the water movement paths. Each soil horizon should be sampled and analyzed individually, with care being taken to not contaminate one horizon's sample with material from another horizon. Coordination with the laboratory is needed to ensure that a sample is collected that is large enough that all physical and chemical tests can be performed. Two liters is usually enough. If trace elements are to be determined, special sample collection and handling procedures are required to prevent sample contamination. General sampling information can be found in the U.S. Department of Agriculture Handbook 18, Soil Survey Manual. Reclamation has a draft technical guide for land classification with respect to irrigation that involves soil sampling to determine soil physical characteristics and chemical content. Selenium, boron, arsenic, and other toxic chemicals are of concern as they relate to toxicity to plants, animals, and humans. Soil salinity is of concern to determine what crops can be grown and their expected yield for the given soil and irrigation water salinity. Toxic chemical paths can be directly from the soil to the plant, in the drainage water, and in surface runoff from the field. The quality of potential water sources for irrigation can be evaluated using the Department of Agriculture's "Handbook 60" and the United Nation's Food and Agriculture Organization's report entitled "Water Quality for Agriculture."

BIBLIOGRAPHY

Crawford, J. K., and S. N. Luoma, *Guidelines for Studies of Contaminants in Biological Tissues for the National Water-Quality Assessment Program,* U.S. Geological Survey Open-File Report 92-494, 1993.

Cuffney, T. F., M. E. Gurtz, and M. R. Meador, *Guidelines for the Processing and Quality Assurance of Benthic Invertebrate Samples Collected as Part of the National Water-Quality Assessment Program,* U.S. Geological Survey Open-File Report 93-407, 1993.

Meador, M. R. , T. F. Cuffney, and M. E. Gurtz, *Methods for Sampling Fish Communities as a Part of the National Water-Quality Assessment Program*, U.S. Geological Survey Open-File Report 93-104, 1993.

Porter, S. D., T. F. Cuffney, M. E. Gurtz, and M. R. Meador, *Methods for Collecting Algal Samples as Part of the National Water-Quality Assessment Program,* U.S. Geological Survey Open-File Report 93-409, 1993.

Shelton, Larry R., *Field Guide for Collecting and Processing Stream-Water Samples for the National Water-Quality Assessment Program,* U.S. Geological Survey Open-File Report 94-455, 1994.

Shelton, L. R., and P. D. Capel, *Guidelines for Collecting and Processing Samples of Stream Bed Sediment for Analysis of Trace Elements and Organic Contaminants for the National Water-Quality Assessment Program*, U.S. Geological Survey Open-File Report 94-458, 1993.

United Nations, Food and Agriculture Organization, *Water Quality for Agriculture*, 1985.

U.S. Bureau of Reclamation, *Land Classification Guide, Land Suitability and Water Quality*, Technical Directive, 2003.

U.S. Department of Agriculture, *Soil Survey Manual, Agricultural Research Administration, Bureau of Plant Industry, Soils, and Agricultural Engineering*, Handbook 18, 1951.

U.S. Salinity Laboratory Staff, *Diagnosis and Improvement of Saline and Alkali Soils,* U.S. Department of Agriculture, Handbook 60, edited by L. A. Richards, Washington DC, 1954.

RECLAMATION LABORATORIES

Reclamation now has five laboratories which perform environmental testing of water, soil, gases, and other materials to determine physical properties and inorganic, organic, and biologic constituents. The laboratories use methods of ASTM, EPA, the Natural Resource Conservation Service (NRCS), USGS, the Association of Official Analytical Chemists (AOAC), and other procedures accepted in the scientific community. The laboratories may provide sampling services and technical assistance as well.

The laboratories provide these analytical (and other) services primarily to Reclamation projects and programs of the regional and area offices. Under technical assistance agreements, services may be provided to other Federal and state agencies as either a primary analytical laboratory, a secondary/ special services laboratory, or a referee laboratory (when all necessary acceptance criteria are met and all required certifications are held). The Environmental Research Chemistry Laboratory, Technical Service Center, Denver, Colorado, can provide other related services, such as data validation and methods development, if requested by Reclamation regional, project, or area offices or other Federal or state agencies.

The following pages provide specific information about Reclamation's environmental testing laboratories. Further information about a laboratory's capabilities may be obtained by contacting the laboratory directly.

SOIL AND WATER LABORATORY
BUREAU OF RECLAMATION
DAKOTA AREA OFFICE
BISMARCK, NORTH DAKOTA

Supervisor

David Hartman

Mail Code DK-370
304 East Broadway Avenue
PO Box 1017
Bismarck ND 58502
(701) 250-4242 Ext. 3370

****

Laboratory Staff

Supervisory Chemist
One Chemist
Three Physical Science Technicians

Water Quality Testing Capabilities

PHYSICAL PROPERTIES

Specific Conductance
pH Value
Total Suspended Solids
Total Dissolved Solids

INORGANIC, NONMETALS

Alkalinity
Ammonia
Nitrate
Nitrite
Orthophosphate
Total phosphate
Total Kjeldahl Nitrogen
Dissolved oxygen
Chloride
Sulfate

INORGANIC METALS

Al, Ag, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Tl, V, Zn

Laboratory Instrumentation

Atomic Absorption Spectrophotometer/flame/ cold vapor/hydride/furnace
Auto Titrator
Carbon Analyzer
pH/Specific Ion Meters
Conductivity Meters
UV-Visible Spectrometer
Flow Injection Analyzer
Physical properties testing instruments for soils
ICP

Interlaboratory Performance Participation

USGS Proficiency Evaluation Program
EPA Proficiency Evaluation Program

Special Testing Capabilities

SOIL TESTING

Specific conductance
pH value
Settling volume
Particle size
Moisture retention
Hydraulic conductivity
Bulk density
Gypsum requirement
Sodium adsorption ratio (SAR)
Inorganic metals
Organic carbon
Lime requirements
Exchangeable acidity
Cation-exchange capacity (CEC)
Exchangeable cations

BIOLOGIC MATERIALS

Chlorophyll
Phaeophytin
Organic Carbon
Purgeable and Nonpurgeables

REGIONAL LABORATORY
BUREAU OF RECLAMATION
PACIFIC NORTHWEST REGION
BOISE, IDAHO

Supervisor

Bill Stroud

Mail Code PN-3210
300 East Garrison Road
Boise ID 83702
(208) 334-1540

******

Laboratory Staff

Supervisory Chemist
Two Chemists
One Physical Science Technician

Water Quality Testing Capabilities

PHYSICAL PROPERTIES

Chlorophyll-α
Specific Conductance
pH Value
Total Dissolved Solids
Total Suspended Solids
Turbidity

INORGANIC, NONMETALS

Chloride
Sulfate
Alkalinity
Fluoride
Silica
Orthophosphate
Total Phosphate
Ammonia
Total Kjeldahl Nitrogen
Nitrate/nitrite
Nitrite

INORGANIC METALS

As, B, Ca, Cd, Cr, Cu, Fe, Hg, K, Mg, Mn, Na, Pb, Se, Zn

BACTERIOLOGICAL/DEMANDS

Fecal, Fecal Strep, Total, ε-coli
TOC, BOD, COD

Laboratory Instrumentation

Auto Analyzers - segmented flow/flow injection
Carbon Analyzer
Conductivity Meters
pH/Specific Ion Meters
Atomic Absorption Spectrophotometers/
furnace/flame/hydride/cold vapor
Autoclaves
Incubators for bacteria testing
Soil physical properties test equipment
Specialized water sampling devices
Programmable auto samplers
Lake bottom sediment samplers
Streamflow measurement devices
Submersible recording thermographs
Total dissolved gas
Turbidimeter
Multi-parameter probe (six parameters)
Mobile testing laboratory
Power boats
UV-VIS spectrophotometer
Ion Chromatograph

Interlaboratory Performance Participation

USGS Proficiency Evaluation Program
(semiannually)
ERA sample audits
(semiannually)

REGIONAL LABORATORY
BUREAU OF RECLAMATION
PACIFIC NORTHWEST REGION
BOISE, IDAHO - continued

Special Testing Capabilities

ONSITE TESTING

DO, ORP, EC, pH, Hydrogen sulfide, Chlorine, Alkalinity, Bacteria, Dissolved gases, Streamflows

WATER SAMPLE COLLECTION

Deep or shallow well sampling
Integrated stream sampling
Reservoir profile sampling

SOIL TESTING

Following the procedures formerly described in “Reclamation Instructions,” physical and chemical analyses are performed on master site soils for land classification and drainage investigations.

REGIONAL LABORATORY
BUREAU OF RECLAMATION
LOWER COLORADO REGION
BOULDER CITY, NEVADA

Supervisor

David C. Hemphill

Mail Code LC-2550
400 Railroad Avenue
PO Box 61470
Boulder City NV 89006-1470
(702) 293-8655

****

Laboratory Staff

Supervisory Chemist
One Chemist
One Technician

Water Quality Testing Capabilities

PHYSICAL PROPERTIES

Specific Conductance
pH Value
Total Dissolved Solids
Total Suspended Solids

INORGANIC, NON-METALS

Sulfate
Chloride
Alkalinity
Fluoride
Silica
Nitrate
Ammonia
Boron
Orthophosphate
Total Phosphate

INORGANIC METALS

Na, K, Ca, Mg, Ag, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Se, Zn

BIOLOGICAL

Chlorophyll

Laboratory Instrumentation

Atomic Absorption Spectrophotometer/flame/furnace
UV-Visible Spectrometer
pH/Specific Ion Meters

Interlaboratory Performance Participation

USGS Proficiency Evaluation Program (semiannually)
ERA performance audits (semiannually)

Special Testing Capabilities

SOIL TESTING

Screening tests
1:5 tests – EC, pH, settling volume
Desert Research Institute salinity
Mechanical analysis (soil texture)
Saturated paste extracts
Exchangeable sodium percentage (ESP)
Gypsum saturation
Specific gravity
Unified Soil Classification System (engineering construction soil testing)

ENVIRONMENTAL RESEARCH CHEMISTRY LABORATORY
BUREAU OF RECLAMATION
TECHNICAL SERVICE CENTER
DENVER, COLORADO

Supervisor

Chris Holdren

Mail Code D-8220
Sixth Avenue and Kipling Street, Building 56
PO Box 25007
Denver CO 80225
(Call for sample delivery address)
(303) 445-2178

****

Laboratory Staff

Supervisory Chemist
Two Research Chemists
Two Chemists
Two Physical Science Technicians
One Physical Scientist

Water and Soil Quality Testing Capabilities

PHYSICAL PROPERTIES

Specific Conductance
pH Value
Total Suspended Solids
Total Dissolved Solids

INORGANIC, NON-METALS

Alkalinity
Ammonia
Bromide/Bromate
Chloride/Chlorate
Fluoride
Nitrate/Nitrite
Orthophosphate
Perchlorate
Sulfate
Total Kjeldahl Nitrogen
Total Phosphorous

INORGANIC METALS

Ca, Mg, Na, K, Si, Ag, Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, Li, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn

ORGANIC CONSTITUENTS

Chlorophyll *a*/Pheophytin
Volatiles
Semi-Volatiles
Non-Volatiles
PCBs, Herbicides, and Pesticides

Laboratory Instrumentation

Inductively Coupled Plasma/Emission Spectroscopy (ICP/ES)
Perstorp automated nutrient analyzer
Atomic Absorption Spectrophotometer/Zeeman furnace/flame/hydride/cold vapor
Gas Chromatograph (detectors: electron capture/Hall/flame ionization/N-P/flame photometric)
High Pressure Liquid Chromatograph
Gas Chromatograph/Mass Spectrometer
Ion Chromatograph
Auto Titrator
pH/Specific Ion Meters
Auto Analyzer
Mercury Fluorescence Analyzer
Microwave Digestion System
Turbidimeter
Organic Carbon Analyzer
Total Organic Halide (TOX) Analyzer
Soil testing equipment
UV-Visible Spectrometer
ISCO samplers

Interlaboratory Performance Participation

USGS Proficiency Evaluation Program (semiannually)

ENVIRONMENTAL RESEARCH CHEMISTRY LABORATORY
BUREAU OF RECLAMATION
TECHNICAL SERVICE CENTER
DENVER, COLORADO - continued

Special Testing Capabilities

Analysis of samples with low nutrient levels
Analysis of sediment and tissue samples
Microcosm studies

SEDIMENT TESTING

Selective extractions
Total sediment concentration
Turbidity

SOIL ANALYSIS

Leach tests – EC, pH, settling volume
Soil moisture
Saturated paste extracts
Cation exchange capacity (CEC)
Sodium adsorption ratio (SAR)
Exchangeable sodium percentage (ESP)
Gypsum saturation
1:5 Calcium chloride pH
Moisture retention (1/3 and 15 bar)

Research and Consulting

Contracting analytical services
Hazardous waste sampling/testing
Geochemistry of dam seepage
Statistical analysis of data
Corrosion evaluations
Sampling design
Water chemistry modeling
Water quality data interpretation