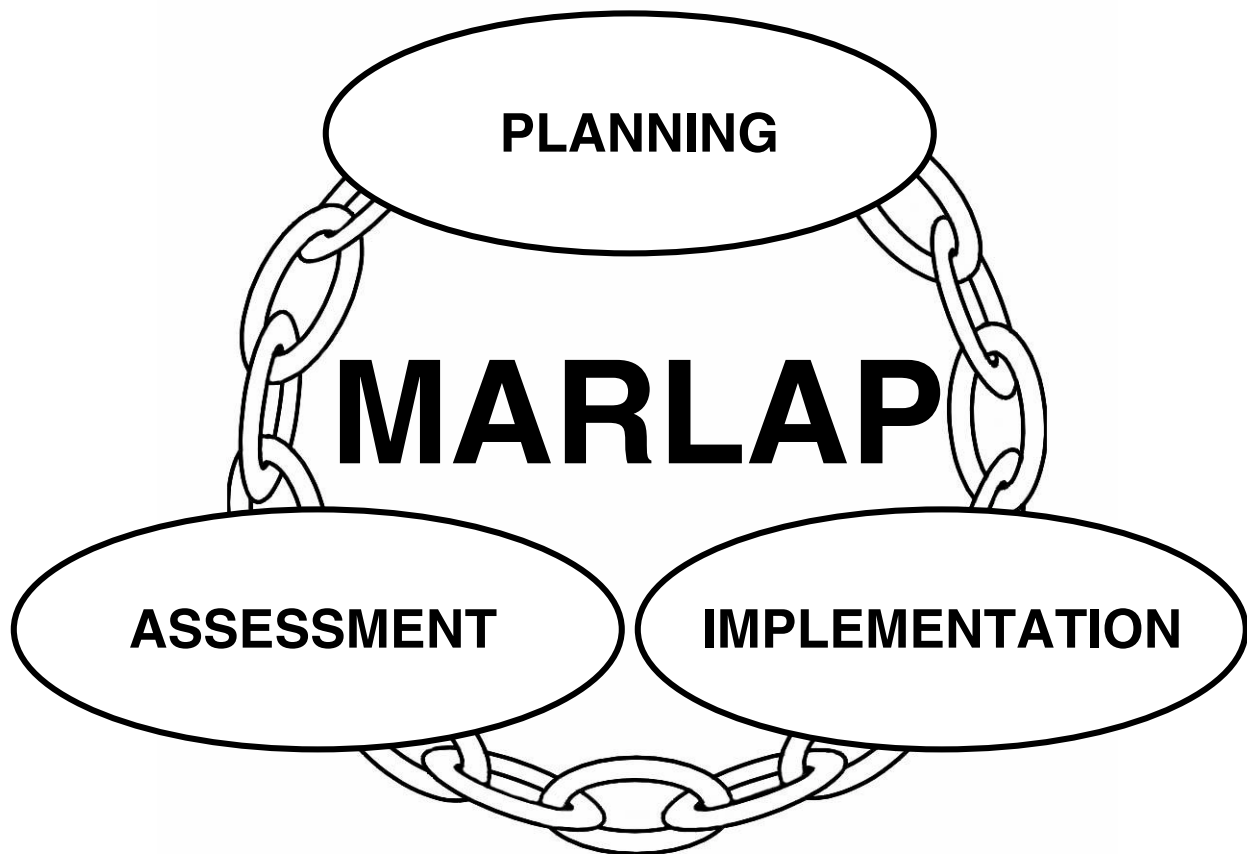


Multi-Agency Radiological Laboratory Analytical Protocols Manual



ABSTRACT

The Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) manual provides guidance for the planning, implementation, and assessment of projects that require the laboratory analysis of radionuclides. MARLAP's basic goal is to provide guidance and a framework for project planners, managers, and laboratory personnel to ensure that radioanalytical laboratory data will meet a project's or program's data requirements. To attain this goal, the manual is intended to provide the guidance necessary for national consistency in the form of a performance-based approach for meeting a project's data requirements. The guidance in MARLAP is designed to help ensure the generation of radioanalytical data of known quality, appropriate for its intended use.

MARLAP was developed by a workgroup that included representatives from the U.S. Environmental Protection Agency (EPA), Department of Energy (DOE), Department of Defense (DOD), Nuclear Regulatory Commission (NRC), National Institute of Standards and Technology (NIST), U.S. Geological Survey (USGS), and U.S. Food and Drug Administration (FDA). State participation in the development of the manual involved contributions from representatives from the Commonwealth of Kentucky and the State of California. Contractors to EPA, DOE, and NRC, and members of the public, have been present during the open meetings of the MARLAP workgroup.

Examples of data collection activities that MARLAP supports include site characterization, site cleanup and compliance demonstration, decommissioning of nuclear facilities, remedial and removal actions, effluent monitoring of licensed facilities, environmental site monitoring, background studies, and waste management activities.

NOTICE

This draft manual being released for simultaneous public and peer review, and technical comments are solicited as described below. MARLAP has not been approved for use in part or in whole and should not be used, cited, or quoted except for the purposes of providing comments as requested by the agencies participating in its development.

MARLAP was developed by a workgroup that included representatives from the U.S. Environmental Protection Agency (EPA), Department of Energy (DOE), Department of Defense (DOD), Nuclear Regulatory Commission (NRC), National Institute of Standards and Technology (NIST), U.S. Geological Survey (USGS), and U.S. Food and Drug Administration (FDA). State participation in the development of the manual involved contributions from representatives from the Commonwealth of Kentucky and the State of California. Contractors to EPA, DOE, and NRC, and members of the public, have been present during the open meetings of the MARLAP workgroup.

Although Federal Government personnel are involved in the preparation of this document, the draft manual does not yet represent the official position of any participating agency. This review is a necessary step in the development of a multi-agency consensus manual. References within this manual to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement or recommendation by the United States Government.

Members of the public are invited and encouraged to submit comments to the following website <http://www.eml.doe.gov/marlap/>. Comments may also be submitted to *either*:

U.S. Environmental Protection Agency
ATTN: Air and Radiation Docket, Mail Stop 6102
Docket Number A-2001-16, Room M1500
401 M Street, SW
Washington, DC 20460

or

Chief, Rules and Directives Branch
Division of Administrative Services
Mail Stop T6D59
U.S. Nuclear Regulatory Commission
Washington, DC 20555-0001

All comments received will be reviewed by the entire MARLAP workgroup. Comments received by the date published in the *Federal Register Notice* announcing the availability of the document for public review will be considered. Comments received after that date will be considered if it is practical to do so, but no assurance can be given for consideration of late comments.

Copies of the draft MARLAP manual and all comments received may be examined or copied for a fee at the EPA Docket Room M1500, Docket Number A-2001-16, First Floor Waterside Mall, 401 M Street, SW, Washington, DC 20460; and the NRC Public Document Room, at U.S. Nuclear Regulatory Commission, Public Document Room, Washington, DC 20555. The document is also available through the National Technical Information Service (NTIS). The NTIS document number is PB2001-106745, and the NTIS Sales Desk can be reached between 8:30 a.m. and 6:00 p.m. Eastern Time, Monday through Friday at 1-800-553-6847; TDD (hearing impaired only) at (703) 487-4639.

In addition to providing comments on individual chapters and appendices, reviewers are also requested to address the following questions while reviewing the draft manual:

- (1) Is the performance-based approach used in MARLAP for the planning, implementation, and assessment phases of projects technically sound, and is the approach reasonable in terms of ease of implementation by project managers and laboratories? Does the approach effectively link the three phases of a project, and is the guidance on quality control appropriate and supportive of a performance-based approach?
- (2) Is the guidance on laboratory operations in Part II (Chapters 10-20) technically accurate and useful?
- (3) Are the concepts covered under measurement statistics—specifically measurement uncertainty, detection and quantification capability—presented accurately and appropriately?
- (4) Is the information understandable and presented in logical sequence? How can the presentation of material be modified to improve the manual?
- (5) Does MARLAP provide benefits that are not currently available through other approaches? What are the costs associated with implementing the guidance in MARLAP in comparison with currently available alternatives?

Commentors are encouraged to use the website, <http://www.eml.doe.gov/marlapp>, for their review. The website has detailed instructions on how to submit comments and has several

Notice

features that should aid the review process. Commentors also may submit written comments to either of the addresses listed on page IV of this Notice using the same general approach described in the MARLAP website. Comments should be accompanied by supporting details, rationale, or data. To ensure efficient and complete comment resolution, commentors are requested to reference the *page number* and the *line number* to which the comment refers. Comments corresponding to an entire chapter, section, or table should be referenced to the line number for the title of the chapter (always line number 1), section, or table. Comments on footnotes should be referenced to the line in the text where the footnote appears (footnotes do not have line numbers). Comments on figures should be referenced to the page on which the figure appears (figures do not have line numbers) and figure number. Comments on the entire manual should be referenced to the title page.

ACKNOWLEDGMENTS

The origin of the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) manual can be traced to the recognition by a number of agencies for the need to have a nationally consistent approach to producing radioanalytical data that meet a program's or project's needs. A multi-agency workgroup was formed with representatives from the U.S. Environmental Protection Agency (EPA), Department of Energy (DOE), Nuclear Regulatory Commission (NRC), Department of Defense (DOD), U.S. Geological Survey (USGS), National Institute of Standards and Technology (NIST), and Food and Drug Administration (FDA) to develop guidance for the planning, implementation, and assessment of projects that require the laboratory analysis of radionuclides. Representatives from the Commonwealth of Kentucky and the State of California also contributed to the development of the manual.

Of particular importance to the workgroup is that the guidance needs to be both scientifically rigorous and flexible enough to be applied to a diversity of projects and programs. The draft MARLAP manual is the result of a cooperative effort with these goals in mind.

MARLAP would not have been possible without the workgroup members who contributed their time, talent, and efforts to develop this guidance document:

John Griggs*, EPA, Chair

EPA: H. Benjamin Hull
Marianne Lynch*
Keith McCroan*
Eric Reynolds
Jon Richards

DOE: Emile Boulos*
Carl Gogolak
Pam Greenlaw*
Catherine Klusek*
Stan Morton*
Colin Sanderson*
Stephanie Woolf*

DOD: CPT Andrew Scott (Army)
Ronald Swatski* (Army)
Jan Dunker (Army Corps of Engineers)
Troy Blanton (Navy)
CAPT David Farrand (Navy)
Dale Thomas (Air Force)

NRC: Rateb (Boby) Abu Eid
Tin Mo
George Powers

USGS: Ann Mullin*

NIST: Kenneth G.W. Inn*

FDA: Edmond Baratta

* These workgroup members also served as chapter chairs.

Acknowledgments

Special recognition is given to John Volpe, Commonwealth of Kentucky, and Penny Leinwander, State of California, for their contributions to the development of the MARLAP manual. The following Federal Agency contractors provided assistance in developing the MARLAP manual:

EPA: N. Jay Bassin (Environmental Management Support, Inc.)
Diane Dopkin (Environmental Management Support, Inc.)
U. Hans Behling (S. Cohen & Associates, Inc.)
Richard Blanchard (S. Cohen & Associates, Inc.)
Harry Chmelynski (S. Cohen & Associates, Inc.)
Scott Hay (S. Cohen & Associates, Inc.)
Patrick Kelly (S. Cohen & Associates, Inc.)
Robert Litman (S. Cohen & Associates, Inc.)
Charles (Chick) Phillips (S. Cohen & Associates, Inc.)
William Richardson III (S. Cohen & Associates, Inc.)
Steven Schaffer (S. Cohen & Associates, Inc.)

DOE: David McCurdy (Duke Engineering & Services)
John Maney (Environmental Measurements Assessments)
Stan Blacker (MACTEC, Inc.)
Pat Harrington (MACTEC, Inc.)
Mike Miller (MACTEC, Inc.)
Lisa Smith (Argonne National Laboratory)

NRC: Eric W. Abelquist (ORISE)
Dale Condra (ORISE)

The MARLAP Workgroup was greatly aided in the development of the manual by the contributions and support provided by the individuals listed below.

David Bottrell (DOE)	David Friedman (EPA)	Kevin Miller (DOE)
Lloyd Currie (NIST)	LCDR Lino Fragoso (Navy)	Jim Mitchell (EPA)
Mike Carter (EPA)	Richard Graham (EPA)	Colleen Petullo (EPA)
Mary Clark (EPA)	Patricia Gowland (EPA)	Steve Pia (EPA)
Ron Colle (NIST)	Larry Jensen (EPA)	Phil Reed (NRC)
Mark Doehnert (EPA)	Jim Kotton (NRC)	Cheryl Trottier (NRC)
Steve Domotor (DOE)	Ed Messer (EPA)	John Warren (EPA)
Joan Fisk (EPA)		

CONTENTS

	<u>Page</u>
Abstract	III
Notice	IV
Acknowledgments	VII
Acronyms and Abbreviations	XLVII
1 Introduction to MARLAP	1-1
1.1 Overview	1-1
1.2 Purpose of the Manual	1-2
1.3 Use and Scope of the Manual	1-3
1.4 Key MARLAP Concepts and Terminology	1-4
1.4.1 Data Life Cycle	1-4
1.4.2 Directed Planning Process	1-5
1.4.3 Performance-Based Approach	1-6
1.4.4 Analytical Process	1-7
1.4.5 Analytical Protocol	1-8
1.4.6 Analytical Method	1-8
1.4.7 Uncertainty and Error	1-8
1.4.8 Precision, Bias, and Accuracy	1-10
1.4.9 Performance Objectives: Data Quality Objectives and Measurement Quality Objectives	1-11
1.4.10 Analytical Protocol Specifications	1-12
1.4.11 The Assessment Phase	1-13
1.5 The MARLAP Process	1-14
1.6 Structure of the Manual	1-15
1.6.1 Overview of Part I	1-17
1.6.2 Overview of Part II	1-17
1.6.3 Overview of the Appendices	1-19
1.7 References	1-20
2 Project Planning Process	2-1
2.1 Introduction	2-1
2.2 The Importance of Directed Project Planning	2-2
2.3 Directed Project Planning Processes	2-4
2.3.1 A Graded Approach to Project Planning	2-4

	<u>Page</u>
2.3.2 Guidance on Directed Planning Processes	2-4
2.3.3 Elements of Directed Planning Processes	2-6
2.4 The Project Planning Team	2-7
2.4.1 Team Representation	2-7
2.4.2 The Radioanalytical Specialists	2-8
2.5 Direct Planning Process and Role of the Radioanalytical Specialists	2-9
2.5.1 Define the Problem	2-12
2.5.2 Identify the Decision	2-13
2.5.2.1 Action Level	2-13
2.5.2.2 Scale of the Decision	2-14
2.5.2.3 Inputs and Boundaries to the Decision	2-15
2.5.2.4 Data Needs	2-15
2.5.3 Specify the Decision Rule and the Tolerable Decision Error Rates	2-15
2.5.4 Optimize the Strategy for Obtaining Data	2-17
2.5.4.1 Analytical Protocol Specifications	2-18
2.5.4.2 Measurement Quality Objectives	2-18
2.6 Results of the Directed Planning Process	2-19
2.6.1 Output Required by the Radioanalytical Laboratory: The Analytical Protocol Specifications	2-20
2.6.2 Chain of Custody	2-21
2.7 Project Planning and Project Implementation and Assessment	2-21
2.7.1 Documenting the Planning Process	2-21
2.7.2 Obtaining Analytical Services	2-22
2.7.3 Selecting Analytical Protocols	2-23
2.7.4 Assessment Plans	2-23
2.7.4.1 Data Verification	2-24
2.7.4.2 Data Validation	2-24
2.7.4.3 Data Quality Assessment	2-24
2.8 References	2-25
3 Key Analytical Planning Issues and Developing Analytical Protocol Specifications	3-1
3.1 Introduction	3-1
3.2 Overview of the Analytical Process	3-2
3.3 General Analytical Planning Issues	3-2
3.3.1 Develop Analyte List	3-4
3.3.2 Identify Concentration Ranges	3-6

	<u>Page</u>
3.3.3 Identify and Characterize Matrices of Concern	3-6
3.3.4 Determine Relationships Between the Radionuclides of Concern	3-7
3.3.5 Determine Available Project Resources and Deadlines	3-8
3.3.6 Refine Analyte List and Matrix List	3-8
3.3.7 Method Performance Characteristics and Measurement Quality Objectives ...	3-9
3.3.7.1 Develop MQOs for Select Method Performance Characteristics	3-11
3.3.7.2 The Role of MQOs in the Protocol Selection and Evaluation Process ...	3-16
3.3.7.3 The Role of MQOs in the Project’s Data Evaluation Process	3-16
3.3.8 Determine Any Limitations on Analysis Options	3-17
3.3.8.1 Gamma Spectrometry	3-18
3.3.8.2 Gross Alpha and Beta Analysis	3-19
3.3.8.3 Radiochemical Nuclide-Specific Analysis	3-19
3.3.9 Determine Method Availability	3-19
3.3.10 Determine the Type and Frequency of, and Evaluation Criteria for, Quality Control Samples	3-20
3.3.11 Determine Sample Tracking and Custody Requirements	3-21
3.3.12 Determine Data Reporting Requirements	3-21
3.4 Matrix-Specific Analytical Planning Issues	3-22
3.4.1 Solids	3-23
3.4.1.1 Homogenization and Subsampling	3-24
3.4.1.2 Removal of Unwanted Materials	3-24
3.4.2 Liquids	3-25
3.4.3 Filters and Wipes	3-26
3.5 Assembling the Analytical Protocol Specifications	3-26
3.6 Level of Protocol Performance Demonstration	3-27
3.7 Project Plan Documents	3-30
3.8 References	3-31
4 Project Plan Documents	4-1
4.1 Introduction	4-1
4.2 The Importance of Project Plan Documents	4-2
4.3 A Graded Approach to Project Plan Documents	4-3
4.4 Project Plan Documents	4-4
4.4.1 Guidance on Project Plan Documents	4-4
4.4.2 Approaches to Project Plan Documents	4-5
4.5 Elements of Project Plan Documents	4-6

	<u>Page</u>
4.5.1 Content of Project Plan Documents	4-7
4.5.2 Plan Documents Integration	4-9
4.5.3 Plan Content for Small Projects	4-10
4.6 Linking the Project Plan Documents and the Project Planning Process	4-10
4.6.1 Planning Process Report	4-15
4.6.2 Data Assessment	4-16
4.6.2.1 Data Verification	4-16
4.6.2.2 Data Validation	4-16
4.6.2.3 Data Quality Assessment	4-17
4.7 References	4-18
5 Obtaining Laboratory Services	5-1
5.1 Introduction	5-1
5.2 Importance of Writing a Technical and Contractual Specification Document	5-2
5.3 Statement of Work—Technical Requirements	5-2
5.3.1 Analytes	5-3
5.3.2 Matrix	5-3
5.3.3 Measurement Quality Objectives	5-3
5.3.4 Unique Analytical Process Requirements	5-4
5.3.5 Quality Control Samples and Participation in External Performance Evaluation Programs	5-4
5.3.6 Laboratory Radiological Holding and Turnaround Times	5-5
5.3.7 Number of Samples and Schedule	5-5
5.3.8 Quality System	5-6
5.3.9 Laboratory’s Proposed Methods	5-6
5.4 Request for Proposal—Generic Contractual Requirements	5-7
5.4.1 Sample Management	5-7
5.4.2 Licenses, Permits and Environmental Regulations	5-8
5.4.2.1 Licenses	5-8
5.4.2.2 Environmental and Transportation Regulations	5-9
5.4.3 Data Reporting and Communications	5-9
5.4.3.1 Data Deliverables	5-9
5.4.3.2 Software Verification and Control	5-10
5.4.3.3 Problem Notification and Communication	5-10
5.4.3.4 Status Reports	5-11
5.4.4 Sample Re-Analysis Requirements	5-11

	<u>Page</u>
5.4.5 Subcontracted Analyses	5-11
5.5 Laboratory Selection and Qualification Criteria	5-12
5.5.1 Technical Proposal Evaluation	5-12
5.5.1.1 Scoring and Evaluation Scheme	5-12
5.5.1.2 Scoring Elements	5-13
5.5.2 Pre-Award Proficiency Evaluation	5-15
5.5.3 Pre-Award Assessments and Audits	5-15
5.6 References	5-16
5.6.1 Cited References	5-16
5.6.2 Other Sources	5-17
6 Selection and Application of an Analytical Method	6-1
6.1 Introduction	6-1
6.2 Method Definition	6-2
6.3 Life Cycle of Method Application	6-6
6.4 Generic Considerations for Method Development and Selection	6-10
6.5 Project-Specific Consideration for Method Selection	6-13
6.5.1 Matrix and Analyte Identification	6-13
6.5.1.1 Matrices	6-13
6.5.1.2. Analytes and Potential Interferences	6-15
6.5.2 Process Knowledge	6-16
6.5.3 Radiological Holding and Turnaround Times	6-17
6.5.4 Unique Process Specifications	6-18
6.5.5 Measurement Quality Objectives	6-19
6.5.5.1 Method Uncertainty	6-19
6.5.5.2 Quantification Capability	6-20
6.5.5.3 Detection Capability	6-21
6.5.5.4 Applicable Analyte Concentration Range	6-23
6.5.5.5 Method Specificity	6-23
6.5.5.6 Method Ruggedness	6-24
6.5.5.7 Bias Considerations	6-24
6.6 Method Validation	6-25
6.6.1 Laboratory's Method Validation Protocol	6-26
6.6.2 Tiered Approach to Validation	6-27
6.6.2.1 Existing Methods Requiring No Additional Validation	6-29
6.6.2.2 Use of a Validated Method for Similar Matrices	6-30

	<u>Page</u>
6.6.2.3 New Application of a Validated Method	6-30
6.6.2.4 Newly Developed or Adapted Methods	6-32
6.6.4 Method Validation Documentation	6-32
6.7 Analyst Qualifications and Demonstrated Proficiency	6-33
6.8 Method Control	6-33
6.9 Continued Performance Assessment	6-34
6.10 Documentation To Be Sent to the Project Manager	6-36
6.11 References	6-37
7 Evaluating Methods and Laboratories	7-1
7.1 Introduction	7-1
7.2 Evaluation of Proposed Analytical Methods	7-2
7.2.1 Documentation of Required Method Performance	7-2
7.2.1.1 Method Validation Documentation	7-3
7.2.1.4 Method Experience, Previous Projects, and Clients	7-5
7.2.1.5 Internal and External Quality Assurance Assessments	7-5
7.2.2 Performance Requirements of the SOW—Analytical Protocol Specifications .	7-6
7.2.2.1 Matrix and Analyte Identification	7-6
7.2.2.2 Process Knowledge	7-7
7.2.2.3 Radiological Holding and Turnaround Times	7-7
7.2.2.4 Unique Processing Specifications	7-9
7.2.2.5 Measurement Quality Objectives	7-9
7.2.2.6 Bias Considerations	7-15
7.3 Initial Evaluation of a Laboratory	7-17
7.3.1 Review of Quality System Documents	7-17
7.3.2 Adequacy of Facilities, Instrumentation, and Staff Levels	7-19
7.3.3 Review of Applicable Prior Work	7-19
7.3.4 Review of Performance Indicators	7-20
7.3.4.1 Review of Internal QC Results	7-20
7.3.4.2 External PE Program Results	7-21
7.3.4.3 Internal and External Quality Assessment Reports	7-21
7.3.5 Initial Audit	7-22
7.4 Ongoing Evaluation of the Laboratory’s Performance	7-22
7.4.1 Quantitative Measures of Quality	7-23
7.4.1.1 MQO Compliance	7-24
7.4.1.2 Other Parameters	7-30

	<u>Page</u>
7.4.2 Operational Aspects	7-31
7.4.2.1 Desk Audits	7-31
7.4.2.2 Onsite Audits	7-33
7.5 References	7-36
8 Radiochemical Data Verification And Validation	8-1
8.1 Introduction	8-1
8.2 Data Assessment Process	8-2
8.2.1 Planning Phase of the Data Life Cycle	8-2
8.2.2 Implementation Phase of the Data Life Cycle	8-3
8.2.2.1 Project Objectives	8-4
8.2.2.2 Documenting Project Activities	8-4
8.2.2.3 QA/QC	8-4
8.2.3 Assessment Phase of the Data Life Cycle	8-5
8.3 Validation Plan	8-7
8.3.1 Technical and Quality Objectives of the Project	8-8
8.3.2 Validation Tests	8-9
8.3.3 Data Qualifiers	8-9
8.3.4 Reporting and Documentation	8-11
8.4 Other Essential Elements	8-11
8.4.1 Statement of Work	8-12
8.4.2 Verified Data Deliverables	8-12
8.5 Data Verification and Validation Process	8-13
8.5.1 The Sample Handling and Analysis System	8-14
8.5.1.1 Sample Descriptors	8-15
8.5.1.2 Aliquant Size	8-16
8.5.1.3 Dates of Sample Collection, Preparation, and Analysis	8-16
8.5.1.4 Preservation	8-17
8.5.1.5 Tracking	8-18
8.5.1.6 Traceability	8-18
8.5.1.7 QC Types and Linkages	8-18
8.5.1.8 Chemical Separation (Yield)	8-19
8.5.1.9 Self-Absorption (Residue)	8-20
8.5.1.10 Efficiency, Calibration Curves, and Instrument Background	8-20
8.5.1.11 Spectrometry Resolution	8-20
8.5.1.12 Dilution and Correction Factors	8-21

	<u>Page</u>
8.5.1.13 Counts and Count Time (Duration)	8-22
8.5.1.14 Result of Measurement, Uncertainty, Minimum Detectable Concentration, and Units	8-22
8.5.2 Quality Control Samples	8-22
8.5.2.1 Method Blank	8-24
8.5.2.2 Laboratory Control Samples	8-25
8.5.2.3 Laboratory Replicates	8-25
8.5.2.4 Matrix Spikes and Matrix Spike Duplicates	8-26
8.5.3 Tests of Detection and Unusual Uncertainty	8-26
8.5.3.1 Detection	8-26
8.5.3.2 Detection Capability	8-27
8.5.3.3 Large or Unusual Uncertainty	8-28
8.5.4 Final Qualification and Reporting	8-29
8.6 Validation Report	8-30
8.7 Other Sources of Information	8-32
9 Data Quality Assessment	9-1
9.1 Introduction	9-1
9.2 Assessment Phase	9-2
9.3 Graded Approach to Assessment	9-3
9.4 The Data Quality Assessment Team	9-4
9.5 Data Quality Assessment Plan	9-4
9.6 Data Quality Assessment Process	9-6
9.6.1 Review of Project Documents	9-8
9.6.1.1 The Project DQOs and MQOs	9-8
9.6.1.2 The DQA Plan	9-9
9.6.1.3 Summary of the DQA Review	9-9
9.6.2 Sample Representativeness	9-10
9.6.2.1 Review of the Sampling Plan	9-10
9.6.2.2 Sampling Plan Implementation	9-13
9.6.2.3 Data Considerations	9-14
9.6.3 Data Accuracy	9-16
9.6.3.1 Review of the Analytical Plan	9-19
9.6.3.2 Analytical Plan Implementation	9-21
9.6.4 Decisions and Tolerable Error Rates	9-22
9.6.4.1 Statistical Evaluation of Data	9-23

	<u>Page</u>
9.6.4.2 Evaluation of Decision Error Rates	9-26
9.7 Data Quality Assessment Report	9-27
9.8 References	9-29
9.8.1 Cited Sources	9-29
9.8.2 Other Sources	9-29
10 Field and Sampling Issues That Affect Laboratory Measurements	10-1
Part I: Generic Issues	10-1
10.1 Introduction	10-1
10.1.1 The Need for Establishing Channels of Communication	10-2
10.1.2 Developing Field Documentation	10-2
10.2 Field Sampling Plan: Non Matrix Specific Issues	10-3
10.2.1 Determination of Analytical Sample Size	10-3
10.2.2 Field Equipment and Supply Needs	10-3
10.2.3 Selection of Sample Containers	10-4
10.2.3.1 Container Material	10-4
10.2.3.2 Container Opening and Closure	10-5
10.2.3.3 Sealing Containers	10-5
10.2.3.4 Precleaned and Extra Containers	10-5
10.2.4 Container Label and Sample Identification Code	10-5
10.2.5 Field Data Documentation	10-6
10.2.6 Field Tracking, Custody, and Shipment Forms	10-8
10.2.7 Chain of Custody	10-9
10.2.8 Field Quality Control	10-9
10.2.9 Decontamination of Field Equipment	10-10
10.2.10 Packing and Shipping	10-11
10.2.11 Worker Health and Safety Plan	10-12
10.2.11.1 Physical Hazards	10-13
10.2.11.2 Biohazards	10-15
Part II: Matrix-Specific Issues That Impact Field Sample Collection, Processing, and Preservation	10-16
10.3 Liquid Samples	10-17
10.3.1 Liquid Sampling Methods	10-18
10.3.2 Liquid Sample Preparation: Filtration	10-18
10.3.2.1 EPA Guidance for Samples/Filtration	10-19
10.3.2.2 Filters	10-21

	<u>Page</u>
10.3.3 Field Preservation of Liquid Samples	10-22
10.3.3.1 Sample Acidification	10-22
10.3.3.2 Non-Acid Preservation Techniques	10-23
10.3.4 Liquid Samples: Special Cases	10-26
10.3.4.1 Radon-222 in Water	10-26
10.3.4.1 Milk	10-27
10.3.5 Non-aqueous Liquids and Mixtures	10-27
10.4 Solids	10-29
10.4.1 Soils	10-29
10.4.1.1 Soil Sample Preparation	10-30
10.4.1.2 Sample Ashing	10-31
10.4.2 Sediments	10-31
10.4.2.1 Initial Mixing and Transport Dispersion of Radionuclides Discharged to Water	10-31
10.4.2.2 Sediment Effect	10-32
10.4.2.3 Sample Preparation/Preservation	10-32
10.4.3 Other Solids	10-32
10.4.3.1 Structural Materials	10-32
10.4.3.2 Biota: Samples of Plant and Animal Products	10-33
10.5 Air Sampling	10-37
10.5.1 Sampler Components	10-37
10.5.2 Filter Selection Based on Destructive Versus Non-destructive Analysis ...	10-39
10.5.3 Sample Preservation and Storage	10-39
10.5.4 Special Cases: Collection of Gaseous and Volatile Air Contaminants	10-40
10.5.4.1 Radioiodines	10-40
10.5.4.2 Gases	10-41
10.5.4.3 Tritium Air Sampling	10-41
10.5.5 Radon	10-42
10.5.5.1 Radon Sampling Methods	10-43
10.5.5.2 Selecting a Radon Sampling Method Based on Data Quality Objectives	10-46
10.6 Wipe Sampling for Assessing Surface Contamination	10-47
10.6.1 Sample Collection Methods	10-48
10.6.1.1 Dry Wipes	10-48
10.6.1.2 Wet Wipes	10-48
10.6.2 Sample Handling	10-50

	<u>Page</u>
10.7	References 10-50
11	Sample Receipt, Inspection, and Tracking 11-1
11.1	Introduction 11-1
11.2	General Considerations 11-3
11.2.1	Communication Before Sample Receipt 11-3
11.2.2	Standard Operating Procedures 11-3
11.2.3	Laboratory License 11-4
11.2.4	Sample Chain-of-Custody 11-5
11.3	Sample Receipt 11-6
11.3.1	Package Receipt 11-6
11.3.2	Radiological Screening 11-7
11.3.3	Corrective Action 11-9
11.4	Sample Inspection 11-9
11.4.1	Physical Integrity of Package and Sample Containers 11-9
11.4.2	Sample Identity Confirmation 11-10
11.4.3	Confirmation of Field Preservation 11-11
11.4.4	Presence of Hazardous Materials 11-11
11.4.5	Corrective Action 11-12
11.5	Laboratory Sample Tracking 11-12
11.5.1	Sample Log-In 11-13
11.5.2	Sample Tracking During Analyses 11-13
11.5.3	Storage of Samples 11-14
11.6	References 11-14
12	Laboratory Sample Preparation 12-1
12.1	Introduction 12-1
12.2	General Guidance for Sample Preparation 12-2
12.2.1	Potential Sample Losses During Preparation 12-2
12.2.1.1	Losses as Dust or Particulates 12-2
12.2.1.2	Losses Through Volatilization 12-3
12.2.1.3	Losses Owing to Reactions Between Sample and Container 12-4
12.2.2	Contamination from Sources in the Laboratory 12-6
12.2.2.1	Airborne Contamination 12-6
12.2.2.2	Contamination of Reagents 12-7
12.2.2.3	Contamination of Glassware/Equipment 12-7

Contents

	<u>Page</u>
12.2.2.4 Contamination of Facilities	12-8
12.2.3 Cleaning of Labware, Glassware, and Equipment	12-8
12.2.3.1 Labware and Glassware	12-8
12.2.3.2 Equipment	12-10
12.3.1 General Procedures	12-14
12.3.1.1 Exclusion of Material	12-14
12.3.1.2 Principles of Drying Techniques	12-14
12.3.1.3 Obtaining a Constant Weight	12-24
12.3.1.4 Subsampling	12-26
12.3.2 Soil/Sediment Samples	12-30
12.3.2.1 Soils	12-31
12.3.2.2 Sediments	12-31
12.3.3.1 Biological Samples	12-32
12.3.3.2 Food	12-32
12.3.3.3 Vegetation	12-32
12.3.3.4 Bone and Tissue	12-33
12.3.4 Other Samples	12-33
12.4 Filters	12-33
12.5 Wipe Samples	12-34
12.6 Liquid Samples	12-35
12.6.1 Conductivity	12-35
12.6.2 Turbidity	12-36
12.6.3 Filtration	12-36
12.6.4 Aqueous Liquids	12-36
12.6.5 Nonaqueous Liquids	12-37
12.6.6 Mixtures	12-38
12.6.6.1 Liquid-Liquid Mixtures	12-38
12.6.6.2 Liquid-Solid Mixtures	12-39
12.7 Gases	12-39
12.8 Bioassay	12-40
12.9 References	12-41
12.9.1 Cited Sources	12-41
12.9.2 Other Sources	12-47
13 Sample Dissolution	13-1
13.1 Introduction	13-1

	<u>Page</u>
13.2 The Chemistry of Dissolution	13-2
13.2.1 Solubility and the Solubility Product Constant, K_{sp}	13-2
13.2.2 Chemical Exchange, Decomposition, and Simple Rearrangement Reactions .	13-3
13.2.3 Oxidation-Reduction Processes	13-4
13.2.4 Complexation	13-5
13.2.5 Equilibrium: Carriers and Tracers	13-6
13.3 Fusion Techniques	13-6
13.3.1 Alkali-Metal Hydroxide Fusions	13-10
13.3.2 Boron Fusions	13-11
13.3.3 Fluoride Fusions	13-12
13.4 Wet Ashing and Acid Dissolution Techniques	13-13
13.4.1 Acids and Oxidants	13-13
13.4.2 Acid Digestion Bombs	13-23
13.4.3 Is it Dissolved?	13-23
13.5 Microwave Digestion	13-24
13.5.1 Focused Open-Vessel Systems	13-25
13.5.2 Low-Pressure, Closed-Vessel Systems	13-25
13.5.3 High-Pressure, Closed-Vessel Systems	13-26
13.6 Special Matrix Considerations	13-26
13.6.1 Liquid Samples	13-26
13.6.1.1 Aqueous Samples	13-26
13.6.1.2 Nonaqueous Samples	13-27
13.6.2 Solid Samples	13-27
13.6.3 Filters	13-27
13.6.4 Wipe Samples	13-28
13.6.5 Liquid Scintillation Samples	13-28
13.6.5.1 Wet Oxidation	13-28
13.6.5.2 Dry Oxidation	13-29
13.7 Total Dissolution and Leaching	13-29
13.7.1 Acid Leaching	13-30
13.7.2 Total Dissolution through Fusion	13-31
13.7.3 Acid Digestion — Fusion Combined Approach	13-32
13.8 Examples of Decomposition Procedures	13-32
13.9 References	13-33
13.9.1 Cited References	13-33
13.9.2 Other Sources	13-36

	<u>Page</u>
14 Separation Techniques	14-1
14.1 Introduction	14-1
14.2 Oxidation/Reduction Processes	14-2
14.2.1 Introduction	14-2
14.2.2 Oxidation-Reduction Reactions	14-3
14.2.3 Common Oxidation States	14-6
14.2.4 Oxidation State in Solution	14-11
14.2.5 Common Oxidizing and Reducing Agents	14-12
14.2.6 Oxidation State and Radiochemical Analysis	14-14
14.3 Complexation	14-19
14.3.1 Introduction	14-19
14.3.2 Chelates	14-21
14.3.3 The Formation (Stability) Constant	14-24
14.3.4 Complexation and Radiochemical Analysis	14-25
14.3.4.1 Extraction of Laboratory Samples and Ores	14-25
14.3.4.2 Separation by Solvent Extraction and Ion-Exchange Chromatography	14-25
14.3.4.3 Formation and Dissolution of Precipitates	14-26
14.3.4.4 Stabilization of Ions in Solution	14-27
14.3.4.5 Detection and Determination	14-27
14.4 Solvent Extraction	14-27
14.4.1 Extraction Principles	14-27
14.4.2 Distribution Coefficient	14-28
14.4.3 Extraction Technique	14-30
14.4.4 Solvent Extraction and Radiochemical Analysis	14-33
14.4.5 Solid-Phase Extraction	14-35
14.4.5.1 Extraction Chromatography Columns	14-36
14.4.5.2 Extraction Membranes	14-37
14.4.6 Advantages and Disadvantages of Solvent Extraction	14-38
14.4.6.1 Advantages	14-38
14.4.6.2 Disadvantages	14-38
14.5 Volatilization and Distillation	14-39
14.5.1 Introduction	14-39
14.5.2 Volatilization Principles	14-40
14.5.3 Distillation Principles	14-42

	<u>Page</u>
14.5.4 Separations in Radiochemical Analysis	14-43
14.5.5 Advantages and Disadvantages of Volatilization	14-44
14.5.5.1 Advantages	14-44
14.5.5.2 Disadvantages	14-44
14.6 Electrodeposition	14-45
14.6.1 Electrodeposition Principles	14-45
14.6.2 Separation of Radionuclides	14-46
14.6.3 Preparation of Counting Sources	14-47
14.6.4 Advantages and Disadvantages of Electrodeposition	14-47
14.6.4.1 Advantages	14-47
14.6.4.2 Disadvantages	14-47
14.7 Chromatography	14-48
14.7.1 Chromatographic Principles	14-48
14.7.2 Gas-Liquid and Liquid-Liquid Phase Chromatography	14-49
14.7.3 Adsorption Chromatography	14-50
14.7.4 Ion-Exchange Chromatography	14-50
14.7.4.1 Principles of Ion Exchange	14-50
14.7.4.2 Resins	14-53
14.7.5 Affinity Chromatography	14-59
14.7.6 Gel-Filtration Chromatography	14-59
14.7.7 Chromatographic Laboratory Methods	14-60
14.7.8 Advantages and Disadvantages of Chromatographic Systems	14-61
14.8 Precipitation and Coprecipitation	14-61
14.8.1 Introduction	14-61
14.8.2 Solutions	14-62
14.8.3 Precipitation	14-64
14.8.3.1 Solubility and the Solubility Product Constant, K_{sp}	14-65
14.8.3.2 Factors Affecting Precipitation	14-70
14.8.3.3 Optimum Precipitation Conditions	14-75
14.8.4 Coprecipitation	14-76
14.8.4.1 Coprecipitation Processes	14-77
14.8.4.2 Water as an Impurity	14-81
14.8.4.3 Postprecipitation	14-81
14.8.4.4 Coprecipitation Methods	14-82
14.8.5 Colloidal Precipitates	14-86
14.8.6 Filterability of Precipitates	14-88

	<u>Page</u>
14.8.7 Advantages and Disadvantages of Precipitation and Coprecipitation	14-90
14.9 Carriers and Tracers	14-91
14.9.1 Introduction	14-91
14.9.2 Carriers	14-91
14.9.2.1 Isotopic Carriers	14-92
14.9.2.2 Nonisotopic Carriers	14-93
14.9.2.3 Common Carriers	14-94
14.9.2.4 Holdback Carriers	14-98
14.9.2.5 Yield (Recovery) of Isotopic Carriers	14-98
14.9.3 Tracers	14-99
14.9.3.1 Characteristics of Tracers	14-101
14.9.3.2 Coprecipitation	14-103
14.9.3.3 Deposition on Nonmetallic Solids	14-103
14.9.3.4 Radiocolloid Formation	14-103
14.9.3.5 Distribution (Partition) Behavior	14-105
14.9.3.6 Vaporization	14-105
14.9.3.7 Oxidation and Reduction	14-106
14.10 Radiochemical Equilibrium	14-107
14.10.1 Basic Principles of Equilibrium	14-107
14.10.2 Oxidation State	14-110
14.10.3 Hydrolysis	14-111
14.10.4 Polymerization	14-113
14.10.5 Complexation	14-114
14.10.6 Radiocolloid Interference	14-114
14.10.7 Isotope Dilution Analysis	14-115
14.10.8 Masking and Demasking	14-116
14.10.9 Review of Specific Radionuclides	14-120
14.10.9.1 Americium	14-120
14.10.9.2 Cesium	14-125
14.10.9.3 Cobalt	14-130
14.10.9.4 Iodine	14-136
14.10.9.5 Plutonium	14-144
14.10.9.6 Radium	14-153
14.10.9.7 Strontium	14-161
14.10.9.8 Technetium	14-167
14.10.9.9 Thorium	14-173

	<u>Page</u>
14.10.9.10 Tritium	14-180
14.10.9.11 Uranium	14-185
14.10.9.12 Zirconium	14-196
14.11 References	14-204
14.12 Selected Bibliography	14-222
14.12.1 Inorganic and Analytical Chemistry	14-222
14.12.2 General Radiochemistry	14-223
14.12.3 Radiochemical Methods of Separation	14-223
14.12.4 Radionuclides	14-223
14.12.5 Separation Methods	14-225
15 Nuclear Counting Instrumentation	15-1
15.1 Introduction	15-1
15.2 Alpha Counting	15-2
15.2.1 Introduction	15-2
15.2.2 Detectors for Alpha Counting	15-3
15.2.2.1 Ionization Chambers	15-3
15.2.2.2 Proportional Counters	15-3
15.2.2.3 Scintillation Counters	15-4
15.2.2.4 Liquid Scintillation Counters	15-5
15.2.2.5 Semiconductor Detectors	15-6
15.3 Beta Counting	15-7
15.3.1 Introduction	15-7
15.3.2 Proportional Counter	15-7
15.3.3 Liquid Scintillation	15-8
15.3.4 Solid Organic Scintillators	15-9
15.3.5 Beta Particle Counter	15-10
15.3.6 Associated Electronic Equipment	15-11
15.4 Gamma Counting	15-12
15.4.1 Introduction	15-12
15.4.2 Energy Efficiency Relationship	15-16
15.4.3 Sodium Iodide Detector Assembly	15-18
15.4.4 High Resolution Germanium Detectors	15-19
15.4.5 Low Background High Resolution Germanium Detectors	15-19
15.4.6 High Resolution Detectors for Low Energy Spectrometry	15-20
15.4.7 CsI(Tl) Detectors	15-20

	<u>Page</u>
15.4.8 CdZnTe Detectors	15-20
15.4.9 BGO Detectors	15-21
15.5 Spectrometry Systems	15-21
15.5.1 Alpha/Gamma Coincidence Systems	15-21
15.5.2 Beta/Gamma Coincidence Systems	15-21
15.5.3 Gamma/Gamma Coincidence Systems	15-21
15.5.4 Photon-Electron Rejecting Alpha Liquid Scintillation Systems	15-22
15.6 Special Instruments	15-22
15.6.1 4- π Counter	15-22
15.6.2 Low-Geometry Counters	15-23
15.6.3 Internal Gas Counters	15-23
15.7 Spectrometers and Energy-Dependent Detectors	15-24
15.7.1 Anti-Coincidence Counters	15-29
15.7.2 Coincidence Counters	15-30
15.8 Shielding	15-31
15.9 Instrument Calibration	15-31
15.10 Other Considerations	15-32
15.10.1 Alpha	15-32
15.10.1.1 Troubleshooting	15-32
15.10.1.2 Calibration	15-35
15.10.1.3 Costs	15-35
15.10.1.4 Quality Control	15-37
15.10.2 Beta	15-40
15.10.2.1 Introduction	15-40
15.10.2.2 Alpha Particle Interference and Beta Energy Resolution	15-41
15.10.2.3 Liquid Scintillation Quenching	15-42
15.10.2.4 Beta Particle Attenuation	15-42
15.10.2.5 Calibration	15-44
15.10.2.6 Costs	15-44
15.10.2.7 Quality Control	15-46
15.10.3 Gamma	15-46
15.10.3.1 Troubleshooting	15-46
15.10.3.2 Calibration	15-48
15.10.3.3 Software	15-49
15.10.3.4 Costs	15-50
15.10.3.5 Quality Control	15-50

	<u>Page</u>
15.10.4 Non-Nuclear Instrumentation	15-51
15.10.4.1 ICP-Mass Spectrometry	15-51
15.10.4.2 Laser	15-53
15.10.4.3 Radionuclides Analyzed By Neutron Activation	15-54
15.11 References	15-55
15.11.1 Cited References	15-55
15.11.2 Other Sources	15-61
 Attachment 15A Field Measurements	 15-63
15A.1 Introduction	15-63
15A.2 Analytical Level of Measurements	15-63
15A.3 Documentation of Methodology	15-65
15A.4 Instrument Operating Conditions	15-66
15A.5 Site Conditions/Limitations	15-66
15A.6 Interferences	15-67
15A.7 Calibration	15-67
15A.8 Minimum Detectable Concentrations	15-68
15A.9 Precision	15-69
15A.10 Accuracy	15-69
15A.11 Representativeness	15-69
15A.12 Completeness	15-70
15A.13 Comparability	15-71
15A.14 Reference Measurements	15-71
15A.15 Record Keeping	15-72
15A.16 Quality Improvement	15-72
15A.17 Management Assessment	15-73
15A.18 Combined Laboratory and Field Measurements	15-73
15A.19 References	15-73
 16 Instrument Calibration and Test Source Preparation	 16-1
16.1 Introduction	16-1
16.2 Instrument Calibration	16-2
16.2.1 Standards	16-3
16.2.2 Correspondence	16-3
16.2.3 Homogeneity	16-4
16.2.4 Uncertainty	16-4

	<u>Page</u>
16.3 General Test Source Characteristics	16-4
16.3.1 Geometrical Arrangement	16-5
16.3.2 Uniformity of Test Source Material	16-5
16.3.3 Self-Absorption and Scattering	16-6
16.3.4 Counting Planchets	16-8
16.4 Test Source Preparation and Calibration for Alpha Measurements	16-8
16.4.1 Proportional Counters	16-9
16.4.1.1 Alpha Test Source Preparation	16-9
16.4.1.2 Proportional Counter Calibration — Alpha	16-10
16.4.2 ZnS(Ag) Scintillation Counter	16-11
16.4.3 Alpha Spectrometry With Semiconductor Detectors	16-12
16.4.4 Liquid-Scintillation Spectrometer	16-13
16.5 Characteristics of Sources for Beta Measurements	16-13
16.5.1 Proportional Counters	16-13
16.5.1.1 Beta Test Source Preparation	16-14
16.5.1.2 Proportional Counter Calibration — Beta	16-14
16.5.2 Liquid-Scintillation Spectrometers	16-15
16.5.2.1 Liquid Scintillation Test Source Preparation	16-16
16.5.2.1.1 Liquid-Scintillation Spectrometer Calibration	16-17
16.6 Characteristics of Sources for Gamma-Ray Measurements	16-18
16.6.1 Gamma Test Source Preparation	16-18
16.6.2 Gamma Spectrometer Calibration	16-20
16.7 Methods of Test Source Preparation	16-20
16.7.1 Electrodeposition	16-20
16.7.2 Coprecipitation	16-23
16.7.3 Evaporation	16-25
16.7.4 Thermal Volatilization/Sublimation	16-26
16.7.5 Preparing Sources to Measure Radioactive Gases	16-27
16.7.6 Preparing Air Filters for Counting	16-29
16.7.7 Preparing Swipes/Smears for Counting	16-29
16.8 References	16-30
17 Data Acquisition, Reduction, and Reporting	17-1
17.1 Introduction	17-1
17.2 Data Acquisition	17-2
17.2.1 Generic Counting Parameter Selection	17-3

	<u>Page</u>
17.2.1.1 Counting Duration	17-4
17.2.1.2 Counting Geometry	17-5
17.2.1.3 Software	17-5
17.2.2 Basic Data Reduction Calculations	17-6
17.3 Data Reduction on Spectrometry Systems	17-8
17.3.1 Gamma Spectrometry	17-8
17.3.1.1 Peak Search or Identification	17-10
Regions of Interest (ROI) Method	17-11
Gaussian Function Derivative Method	17-12
Channel Differential Method	17-12
Correlation Method	17-12
17.3.1.2 Singlet/Multiplet Peaks	17-13
17.3.1.3 Definition of Peak Centroid and Energy	17-14
17.3.1.4 Peak Width Determination	17-14
17.3.1.5 Peak Area Determination	17-16
17.3.1.6 Calibration Reference File	17-19
17.3.1.7 Activity and Concentration	17-19
17.3.1.8 Summing Considerations	17-20
17.3.1.9 Uncertainty Calculation	17-22
17.3.2 Alpha Spectrometry	17-23
17.3.2.1 Radiochemical Yield	17-27
17.3.2.2 Uncertainty Calculation	17-27
17.3.3 Liquid Scintillation Spectrometry	17-28
17.3.3.1 Overview of Liquid Scintillation Counting	17-28
17.3.3.2 Liquid Scintillation Spectra	17-29
17.3.3.3 Pulse Characteristics	17-29
17.3.3.4 Coincidence Circuitry	17-30
17.3.3.5 Quenching	17-30
17.3.3.6 Luminescence	17-30
17.3.3.7 Test Source Vials	17-31
17.3.3.8 Data Reduction for Liquid Scintillation Counting	17-31
17.4 Data Reduction on Non-Spectrometry Systems	17-32
17.5 Reporting Data	17-37
17.5.1 Sample and Analysis Method Identification	17-37
17.5.2 Units and Radionuclide Identification	17-38
17.5.3 Values, Uncertainty, and Significant Figures	17-38

	<u>Page</u>
17.5.4 Other Information to be Provided on Request	17-38
17.6 Data Packages	17-39
17.7 Electronic Data Deliverables	17-39
17.8 References	17-40
17.8.1 Cited References	17-40
17.8.2 Other Sources	17-42
18 Laboratory Quality Control	18-1
18.1 Introduction	18-1
18.1.1 Organization of Chapter	18-2
18.1.2 Format	18-2
18.2 Quality Control	18-3
18.3 Evaluation of Performance Indicators	18-4
18.3.1 Importance of Evaluating Performance Indicators	18-4
18.3.2 Statistical Means of Evaluating Performance Indicators — Control Charts ..	18-5
18.3.3 Measurement Uncertainty	18-7
18.4 Radiochemistry Performance Indicators	18-9
18.4.1 Method and Reagent Blank	18-9
18.4.2 Laboratory Replicates	18-13
18.4.3 Laboratory Control Samples, Matrix Spikes, and Matrix Spike Duplicates .	18-16
18.4.4 Certified Reference Materials	18-18
18.4.5 Chemical/Tracer Yield	18-21
18.5 Instrumentation Performance Indicators	18-25
18.5.1 Instrument Background Measurements	18-25
18.5.2 Efficiency Calibrations	18-27
18.5.3 Spectrometry Systems	18-31
18.5.3.1 Energy Calibrations	18-31
18.5.3.2 Peak Resolution and Tailing	18-34
18.5.4 Gas Proportional Systems	18-38
18.5.4.1 Voltage Plateaus	18-38
18.5.4.2 Self-Absorption, Backscatter, and Crosstalk	18-39
18.5.5 Liquid Scintillation	18-41
18.5.6 Summary	18-41
18.6 Related Concerns	18-43
18.6.1 Detection Capability	18-43
18.6.2 Secular Equilibrium	18-44

	<u>Page</u>
18.6.3 Half-Life	18-47
18.6.4 Interferences	18-48
18.6.5 Negative Results	18-50
18.6.6 Blind Samples	18-51
18.6.7 Calibration of Apparatus Used for Weight and Volume Measurements	18-54
18.7 References	18-55
18.7.1 Cited Sources	18-55
18.7.2 Other Sources	18-57
Attachment 18A: Control Charts	18-59
18A.1 Introduction	18-59
18A.2 X Charts	18-59
18A.3 Charts	18-63
18A.4 R Charts	18-64
18A.5 Control Charts for Instrument Response	18-65
18A.6 References	18-70
Attachment 18B: Statistical Tests for QC Results	18-71
18B.1 Introduction	18-71
18B.2 Tests for Excess Variance in the Instrument Response	18-71
18B.3 Instrument Background Measurements	18-78
18B.3.1 Detection of Background Variability	18-78
18B.3.2 Comparing a Single Observation to Preset Limits	18-80
18B.3.3 Comparing the Results of Consecutive Measurements	18-84
18B.4 Negative Activities	18-86
18B.5 References	18-86
19 Measurement Statistics	19-1
19.1 Overview	19-1
19.2 Statistical Concepts and Terms	19-2
19.2.1 Basic Concepts	19-2
19.2.2 Summary of Terms	19-5
19.3 Measurement Uncertainty	19-7
19.3.1 Measurement, Error, and Uncertainty	19-7
19.3.2 The Measurement Process	19-8
19.3.3 Analysis of Measurement Uncertainty	19-10
19.3.4 Corrections for Systematic Effects	19-11
19.3.5 Counting Uncertainty	19-11

	<u>Page</u>
19.3.6 Expanded Uncertainty	19-11
19.3.7 Significant Figures	19-12
19.3.8 Reporting the Measurement Uncertainty	19-13
19.3.9 Recommendations	19-14
19.3.10 Summary of Terms	19-14
19.4 Detection and Quantification Capability	19-17
19.4.1 Analyte Detection Decisions	19-17
19.4.2 The Minimum Detectable Concentration	19-19
19.4.3 Differences between the ISO and IUPAC Definitions	19-22
19.4.4 Other Detection Terminologies	19-23
19.4.5 The Minimum Quantifiable Concentration	19-24
19.4.6 Recommendations	19-25
19.4.7 Summary of Terms	19-25
19.5 Procedures for Estimating Uncertainty	19-27
19.5.1 Identifying Sources of Uncertainty	19-27
19.5.2 Evaluation of Standard Uncertainties	19-28
19.5.2.1 Type A Evaluations	19-28
19.5.2.2 Type B Evaluations	19-31
19.5.3 Combined Standard Uncertainty	19-33
19.5.4 The Estimated Covariance of Two Output Estimates	19-37
19.5.5 Nonlinear Models	19-38
19.5.5.1 Uncertainty Propagation	19-38
19.5.5.2 Bias	19-40
19.5.5.3 Nominal Values	19-42
19.6 Radiation Measurement Uncertainty	19-43
19.6.1 Radioactive Decay	19-43
19.6.2 Radiation Counting	19-44
19.6.3 Count Rate	19-47
19.6.3.1 Dead Time	19-48
19.6.3.2 A Confidence Interval for the Count Rate	19-49
19.6.4 Instrument Background	19-50
19.6.5 Counting Efficiency	19-52
19.6.6 Radionuclide Half-life	19-56
19.6.7 Gamma Spectrometry	19-56
19.6.8 Balances	19-56
19.6.9 Pipets and Other Volumetric Apparatus	19-58

	<u>Page</u>
19.6.10 Digital Displays and Rounding	19-62
19.6.11 Subsampling	19-63
19.6.12 The Standard Uncertainty for a Hypothetical Measurement	19-64
19.7 Detection and Quantification Limits	19-65
19.7.1 Calculation of the Critical Value	19-65
19.7.1.1 Normally Distributed Signals	19-66
19.7.1.2 Poisson Counting	19-67
19.7.1.3 Reagent Blanks	19-70
19.7.2 Calculation of the Minimum Detectable Concentration	19-71
19.7.2.1 The Minimum Detectable Net Instrument Signal	19-72
19.7.2.2 Normally Distributed Signals	19-72
19.7.2.3 Poisson Counting	19-74
19.7.2.4 The MDC	19-75
19.7.2.5 Regulatory Requirements	19-76
19.7.2.6 Testing the MDC	19-77
19.7.3 Calculation of the Minimum Quantifiable Concentration	19-78
19.8 References	19-80
19.8.1 Cited Sources	19-80
19.8.2 Other Sources	19-84
Attachment 19A Distributions	19-85
19A.1 Introduction	19-85
19A.2 Normal Distributions	19-85
19A.3 Log-normal Distributions	19-86
19A.4 Chi-square Distributions	19-87
19A.5 T-Distributions	19-88
19A.6 Rectangular Distributions	19-90
19A.7 Trapezoidal and Triangular Distributions	19-91
19A.8 Exponential Distributions	19-92
19A.9 Binomial Distributions	19-92
19A.10 Poisson Distributions	19-93
19A.11 References	19-95
Attachment 19B Multicomponent Analyses	19-97
19B.1 Matrix Equations	19-97
19B.2 Random Vectors and Matrices	19-98
19B.3 Linear Least Squares	19-99
19B.4 General Least Squares	19-101

	<u>Page</u>
19B.5 The Covariance Matrix for a Least-Squares Solution	19-102
19B.6 Critical Values	19-103
19B.7 Detection and Quantification Limits	19-104
19B.8 References	19-104
Attachment 19C Estimation of Coverage Factors	19-105
19C.1 Introduction	19-105
19C.2 Procedure	19-105
19C.3 Poisson Counting Uncertainty	19-106
19C.4 References	19-107
Attachment 19D Low-Background Detection Limits	19-109
19D.1 Overview	19-109
19D.2 Calculation of the Critical Value	19-109
19D.2.1 Normally Distributed Signals	19-109
19D.2.2 Poisson Counting	19-110
19D.3 Calculation of the Minimum Detectable Concentration	19-123
19D.3.1 The Minimum Detectable Net Instrument Signal	19-123
19D.3.2 Normally Distributed Signals	19-123
19D.3.3 Poisson Counting	19-126
19D.4 References	19-132
Attachment 19E Example Calculations	19-135
19E.1 Overview	19-135
19E.2 Sample Collection and Analysis	19-135
19E.3 The Measurement Model	19-136
19E.4 The Combined Standard Uncertainty	19-138
19E.5 The Critical Net Count	19-140
19E.6 The Minimum Detectable Concentration	19-143
19E.7 The Minimum Quantifiable Concentration	19-148
Attachment 19F Tests for Normality	19-149
19F.1 Purpose	19-149
19F.2 Normal Probability Plots	19-149
19F.3 Filliben's Statistic	19-151
19F.4 References	19-155
Attachment 19G Balance Measurement Uncertainty	19-157
19G.1 Purpose	19-157
19G.2 Considerations	19-157
19G.3 Repeatability	19-157

	<u>Page</u>
19G.4 Environmental Factors	19-158
19G.5 Calibration	19-159
19G.6 Linearity	19-160
19G.7 Air Buoyancy Corrections	19-160
19G.8 Combining the Components	19-164
19G.9 References	19-165
20 Waste Management in a Radioanalytical Laboratory	20-1
20.1 Introduction	20-1
20.2 Types of Laboratory Wastes	20-1
20.3 Waste Management Program	20-2
20.3.1 Program Integration	20-3
20.3.2 Staff Involvement	20-3
20.4 Waste Minimization	20-4
20.5 Waste Determinations and Characterization	20-6
20.6 Specific Waste Management Requirements	20-7
20.6.1 Sample/Waste Exemptions	20-9
20.6.2 Storage	20-10
20.6.2.1 Container Requirements	20-11
20.6.2.2 Labeling Requirements	20-11
20.6.2.3 Time Constraints	20-11
20.6.2.4 Monitoring Requirements	20-12
20.6.3 Treatment	20-12
20.6.4 Disposal	20-13
20.7 Contents of a Laboratory Waste Management Plan/Certification Plan	20-14
20.7.1 Laboratory Waste Management Plan	20-14
20.7.2 Waste Certification Plan/Program	20-14
20.8 Useful Web Sites	20-16
20.9 References	20-17
20.9.1 Cited References	20-17
20.9.2 Other Sources	20-18

Glossary *to be added following public review*

Appendices

Appendix A: Directed Planning Approaches A-1

 A.1 Directed Planning Approaches A-1

 A.2 Elements Common to Directed Planning Approaches A-1

 A.3 Data Quality Objectives Process A-2

 A.4 Observational Approach A-3

 A.5 Streamlined Approach for Environmental Restoration A-4

 A.6 Technical Project Planning A-4

 A.7 Expedited Site Characterization A-5

 A.8 Value Engineering A-5

 A.9 Systems Engineering A-6

 A.10 Total Quality Management A-7

 A.11 Partnering A-7

 A.12 References A-8

 A.12.1 Data Quality Objectives A-8

 A.12.2 Observational Approach A-10

 A.12.3 Streamlined Approach for Environmental Restoration (Safer) A-10

 A.12.4 Technical Project Planning A-11

 A.12.5 Expedited Site Characterization A-11

 A.12.6 Value Engineering A-13

 A.12.7 Systems Engineering A-14

 A.12.8 Total Quality Management A-16

 A.12.9 Partnering A-17

Appendix B: The Data Quality Objectives Process B-1

 B1.0 Introduction B-1

 B2.0 Overview of the DQO Process B-2

 B3.0 The Seven Steps of the DQO Process B-3

 B3.1 DQO Process Step 1: State the Problem B-3

 B3.2 DQO Process Step 2: Identify the Decision B-4

 B3.3 DQO Process Step 3: Identify Inputs to the Decision B-5

 B3.4 DQO Process Step 4: Define the Study Boundaries B-7

 B3.5 Outputs of DQO Process Steps 1 to 4 Lead Into Steps 5 to 7 B-8

 B3.6 DQO Process Step 5: Develop a Decision Rule B-8

 B3.7 DQO Process Step 6: Specify the Limits on Decision Errors B-9

	<u>Page</u>
B3.8 DQO Process Step 7: Optimize the Design for Obtaining Data	B-12
B3.9 References	B-14
Attachment B-1 Decision Error Rates And The Gray Region	B-16
B-1.1 Introduction	B-16
B-1.2 The Region of Interest	B-16
B-1.3 Measurement Uncertainty at the Action Level	B-17
B-1.4 The Null Hypothesis	B-18
Case 1: Assume The True Concentration is Over 1.0	B-18
Case 2: Assume The True Concentration is 0.9	B-20
B-1.5 The Critical Region	B-20
B-1.6 The Gray Region	B-21
 Appendix C: Measurement Quality Objectives for Method Uncertainty And Detection and Quantification Capability	 C-1
C.1 Introduction	C-1
C.2 Hypothesis Testing	C-2
C.3 Development of MQOs for Analytical Protocol Selection	C-4
C.4 The Role of the MQO for Method Uncertainty in Data Evaluation	C-9
C.4.1 Uncertainty Requirements at Various Concentrations	C-9
C.4.2 Acceptance Criteria for Quality Control Samples	C-12
C.5 References	C-19
 Appendix D Content of Project Plan Documents	 D-1
D1.0 Introduction	D-1
D2.0 Group A: Project Management	D-3
D2.1 Project Management (A1): Title and Approval Sheet	D-6
D2.2 Project Management (A2): Table of Contents	D-7
D2.3 Project Management (A3): Distribution List	D-7
D2.4 Project Management (A4): Project/Task Organization	D-8
D2.5 Project Management (A5): Problem Definition/Background	D-8
D2.6 Project Management (A6): Project/Task Description	D-10
D2.7 Project Management (A7): Quality Objectives and Criteria for Measurement Data	 D-12
D2.7.1 Project's Quality Objectives	D-12
D2.7.2 Specifying Measurement Quality Objectives	D-13
D2.7.3 Relation between the Project DQOs, MQOs, and QC Requirements ...	D-14

	<u>Page</u>
D2.8 Project Management (A8): Special Training Requirements/Certification . . .	D-14
D2.9 Project Management (A9): Documentation and Record	D-14
D3.0 Group B: Measurement/Data Acquisition	D-16
D3.1 Measurement/Data Acquisition (B1): Sampling Process Design	D-16
D3.2 Measurement/Data Acquisition (B2): Sampling Methods Requirements . . .	D-18
D3.3 Measurement/Data Acquisition (B3): Sample Handling and Custody Requirements	D-20
D3.4 Measurement/Data Acquisition (B4): Analytical Methods Requirements . .	D-21
D3.5 Measurement/Data Acquisition (B5): Quality Control Requirements	D-23
D3.6 Measurement/Data Acquisition (B6): Instrument/Equipment Testing, Inspection, and Maintenance Requirements	D-24
D3.7 Measurement/Data Acquisition (B7): Instrument Calibration and Frequency	D-25
D3.8 Measurement/Data Acquisition (B8): Inspection/Acceptance Requirements for Supplies and Consumables	D-26
D3.9 Measurement/Data Acquisition (B9): Data Acquisition Requirements for Non- Direct Measurement Data	D-27
D3.10 Measurement/Data Acquisition (B10): Data Management	D-28
D4.0 Group C: Assessment/Oversight	D-29
D4.1 Assessment/Oversight (C1): Assessment and Response Actions	D-29
D4.2 Assessment/Oversight (C2): Reports To Management	D-30
D5.0 Group D: Data Validation and Usability	D-31
D5.1 Data Validation and Usability (D1): Verification and Validation Requirements	D-31
D5.2 Data Validation and Usability (D2): Verification and Validation Methods .	D-32
D5.2.1 Data Verification	D-32
D5.2.2 Data Validation	D-33
D5.3 Data Validation and Usability (D3): Reconciliation with Data Quality Objectives	D-34
D6.0 References	D-35
Appendix E: Contracting Laboratory Services	E-1
E.1 Introduction	E-1
E.2 Procurement of Services	E-5
E.2.1 Request for Approval of Proposed Procurement Action	E-6
E.2.2 Types of Procurement Mechanisms	E-6
E.3 Request for Proposals—The Solicitation	E-8

	<u>Page</u>
E.3.1 Market Research	E-9
E.3.2 Length of Contract	E-10
E.3.3 Subcontracts	E-10
E.4 Proposal Requirements	E-11
E.4.1 RFP and Contract Information	E-11
E.4.2 Personnel	E-14
E.4.3 Instrumentation	E-17
E.4.3.1 Type, Number, and Age of Laboratory Instruments	E-18
E.4.3.2 Service Contract	E-18
E.4.4 Narrative to Approach	E-18
E.4.4.1 Analytical Methods or Protocols	E-18
E.4.4.2 Meeting Contract Measurement Quality Objectives	E-19
E.4.4.3 Data Package	E-19
E.4.4.4 Schedule	E-19
E.4.4.5 Sample Storage and Disposal	E-20
E.4.5 Quality Manual	E-21
E.4.6 Licenses and Accreditations	E-22
E.4.7 Experience	E-22
E.4.7.1 Previous or Current Contracts	E-23
E.4.7.2 Quality of Performance	E-23
E.5 Proposal Evaluation and Scoring Procedures	E-23
E.5.1 Evaluation Committee	E-24
E.5.2 Ground Rules — Questions	E-24
E.5.3 Scoring/Evaluation Scheme	E-24
E.5.3.1 Review of Technical Proposal and Quality Manual	E-26
E.5.3.2 Review of Laboratory Accreditation	E-28
E.5.3.3 Review of Experience	E-28
E.5.4 Pre-Award Proficiency Samples	E-28
E.5.5 Pre-Award Audit	E-29
E.5.6 Comparison of Prices	E-33
E.5.7 Debriefing of Unsuccessful Vendors	E-34
E.6 The Award	E-34
E.7 For the Duration of the Contract	E-35
E.7.1 Managing a Contract	E-35
E.7.2 Responsibility of the Contractor	E-36
E.7.3 Responsibility of the Agency	E-36

Contents

	<u>Page</u>
E.7.4 Anomalies and Nonconformance	E-36
E.7.5 Laboratory Assessment	E-37
E.7.5.1 Performance and Quality Control Samples	E-37
E.7.5.2 Laboratory Performance Evaluation Programs	E-38
E.7.5.3 Laboratory Evaluations Performed During the Contract Period	E-39
E.8 Contract Completion	E-40
E.9 References	E-41
Appendix F Laboratory Subsampling	F-1
F.1 Introduction	F-1
F.2 Basic Concepts	F-2
F.3 Sources of Measurement Error	F-4
F.3.1 Sampling Bias	F-4
F.3.2 Fundamental Error	F-5
F.3.3 Grouping and Segregation Error	F-7
F.4 Implementation of the Particulate Sampling Theory	F-10
F.4.1 The Fundamental Variance	F-10
F.4.2 Scenario 1 – Natural Radioactive Minerals	F-11
F.4.3 Scenario 2 – Hot Particles	F-12
F.4.4 Scenario 3 – Particle Surface Contamination	F-14
F.5 Summary	F-16
F.6 References	F-17
G Statistical Tables	G-1

List of Figures

Figure 1.1 The Data Life Cycle	1-5
Figure 1.2 Typical Components of an Analytical Process	1-7
Figure 1.3 The MARLAP Process	1-16
Figure 3.1 Typical components of an analytical process	3-3
Figure 3.2 Analytical protocol specifications	3-28
Figure 3.3 Example analytical protocol specifications	3-29
Figure 6.1 Analytical process	6-3
Figure 6.2 Method application life cycle	6-6
Figure 6.3 Expanded Figure 6.2 addressing the laboratory's method evaluation process	6-7
Figure 7.1 Considerations for the initial evaluation of a laboratory	7-18
Figure 8.1 The Assessment Process	8-6
Figure 9.1 Using physical samples to measure a characteristic of the population representative of	9-11
Figure 9.2 Types of sampling and analytical errors.	9-18
Figure 10.1 Example of chain-of-custody record.	10-10
Figure 11.1 Overview of sample receipt, inspection, and tracking	11-2
Figure 12.1 Degree of error in laboratory sample preparation (Scwedt, 1997)	12-1
Figure 12.2 Laboratory Sample Preparation Flowchart (for Solid Samples)	12-13
Figure 14.1 Ethylenediaminetetraacetic Acid ⁽¹⁾ (EDTA)	14-22
Figure 14.2 Crown ethers	14-23
Figure 14.3 The behavior of elements in concentrated hydrochloric acid on cation-exchange resins	14-57
Figure 14.4 behavior of elements in concentrated hydrochloric acid on anion-exchange resins	14-58
Figure 14.5 The electrical double layer	14-86
Figure 15.1 Gamma-ray Interactions with Germanium	15-12

	<u>Page</u>
Figure 15.2 Gamma-ray Spectra of ^{60}Co	15-13
Figure 15.3 Energy Spectrum of ^{22}Na	15-15
Figure 15.4 Efficiency vs. Gamma-ray Energy	15-16
Figure 15.5 Standard Cryostat HPGe Background Spectrum	15-20
Figure 15.6 Low Background Cryostat HPGe Background Spectrum	15-20
Figure 15.7 NaI(Tl) Energy Spectrum of ^{137}Cs	15-26
Figure 15.8 HPGe Energy Spectrum of ^{137}Cs	15-27
Figure 15.9 Spectrum of ^{210}Pb , ^{210}Bi , and ^{210}Po	15-29
Figure 15.10 Range vs. Energy for Alpha Particles in Air	15-35
Figure 15.11 Range vs. Energy for Beta Particles in Air and Water	15-43
Figure 15.12 Beta Detector Efficiency Curve for ^{131}I vs. Weight	15-43
Figure 15.13 Beta-gamma coincidence efficiency curve for ^{129}I	15-55
Figure 17.1 Gamma-ray spectrum	17-9
Figure 17.2 Gamma-ray analysis sequence	17-11
Figure 17.3 Low-energy tailing	17-16
Figure 17.4 Photopeak baseline continuum	17-17
Figure 17.5 Photopeak baseline continuum-step function	17-18
Figure 17.6 Alpha spectrum	17-23
Figure 18.1 Control chart for daily counting of a standard reference source, with limits corrected for decay	18-7
Figure 18.2 Three general categories of blank changes	18-12
Figure 18.3 Failed performance indicator: replicates.	18-15
Figure 18.4 Failed performance indicator: chemical yield	18-23
Figure 19.1 A symmetric distribution	19-4
Figure 19.2 An asymmetric distribution	19-4
Figure 19.3 The critical value x_c and minimum detectable value x_D of the net state variable	19-20
Figure 19.4 Expected fraction of atoms remaining at time t	19-44
Figure 19.5 A normal distribution	19-85
Figure 19.6 A log-normal distribution	19-86
Figure 19.7 Chi-square distributions	19-88
Figure 19.8 The t -distribution with 3 degrees of freedom	19-89
Figure 19.9 A rectangular distribution	19-91

	<u>Page</u>
Figure 19.10 A trapezoidal distribution	19-91
Figure 19.11 An exponential distribution	19-92
Figure 19.12 Type I error rate for the Poisson-normal approximation ($t_B = t_S$)	19-113
Figure 19.13 Type I error rates for Formula A	19-115
Figure 19.14 Type I error rates for Formula B	19-116
Figure 19.15 Type I error rates for Formula C	19-118
Figure 19.16 Type I error rates for the Stapleton approximation	19-119
Figure 19.17 Type I error rates for the nonrandomized exact test	19-121
Figure 19.18 Example: Normal probability plot	19-154
Figure B1 Seven steps of the DQO process.	B-2
Figure B2(a) Decision performance goal diagram null hypothesis: the parameter exceeds the action level.	B-11
Figure B2(b) Decision performance goal diagram null hypothesis: the parameter is less than the action level	B-11
Figure B3 How Proximity to the action level determines what is an acceptable level of uncertainty.	B-13
Figure C.1 Required Analytical Standard Deviation (σ_{Req})	C-10
Figure E-1 General Sequence Initiating and Later Conducting Work with a Contract Laboratory	E-4

List of Tables

Table 2.1 Summary of the directed planning process and radioanalytical specialists participation 2-10

Table 3.1 Matrix-specific analytical planning issues 3-23

Table 4.1 Elements of project plan documents 4-7

Table 4.2 Crosswalk between project plan document elements and directed planning process 4-11

Table 6.1 Tiered method validation approach 6-28

Table 7.1 Cross reference of information available for method evaluation 7-4

Table 9.1 Summary of the DQA process 9-6

Table 10.1 Summary of sample preservation techniques 10-25

Table 11.1 Typical topics addressed in standard operating procedures related to sample receipt, inspection, and tracking 11-4

Table 12.1 Examples of volatile radionuclides 12-4

Table 12.2 Properties of sample container materials 12-5

Table 12.3 Examples of dry-ashing temperatures (platinum container) 12-23

Table 13.1 Common fusion fluxes 13-7

Table 13.2 Examples of acids used for wet ashing 13-14

Table 13.3 Standard reduction potentials of selected half-reactions at 25 °C 13-14

Table 14.1 Oxidation states of elements 14-9

Table 14.2 Stable oxidation states of selected elements 14-10

Table 14.3 Redox reagents for radionuclides 14-14

Table 14.5 Radioanalytical methods employing solvent extraction 14-35

Table 14.6 Radioanalytical methods employing extraction chromatography 14-36

Table 14.7 Elements separable by volatilization as certain species 14-41

Table 14.8 Typical functional groups of ion-exchange resins 14-54

	<u>Page</u>
Table 14.9 Common ion-exchange resins	14-55
Table 14.10 General solubility behavior of some cations of interest	14-63
Table 14.11 Summary of methods for utilizing precipitation from homogeneous solution	14-74
Table 14.12 Influence of precipitation conditions on the purity of precipitates	14-76
Table 14.13 Common coprecipitating agents for radionuclides	14-83
Table 14.14 Coprecipitation behavior of plutonium and neptunium	14-85
Table 14.15 General properties of common filter papers	14-89
Table 14.16 Atoms and mass of select radionuclides equivalent to 500 dpm	14-91
Table 14.17 Masking agents for ions of various metals	14-117
Table 14.18 Masking agents for anions and neutral molecules	14-119
Table 15.1 Typical percent gamma-ray efficiencies for a 55 percent high-purity germanium detector with various counting geometries	15-17
Table 15.2 Nuclides for gamma-ray spectrometer calibration	15-48
Table 16.1 Nuclides for alpha calibration	16-10
Table 16.2 Nuclides for beta calibration	16-15
Table 17.1 Units for data reporting	17-38
Table 18.1 Problems leading to loss of analytical control	18-3
Table 18.2a Certified Massic activities for natural radionuclides with a normal distribution of measurement results	18-20
Table 18.2b Certified Massic activities for anthropogenic radionuclides with a Weibull distribution of measurement results	18-20
Table 18.2c Uncertified Massic activities	18-20
Table 18.3 Instrument background evaluation	18-27
Table 18.4 Root cause analysis of performance check results	18-37
Table 18.5 Instrument calibration: example frequency and performance criteria	18-41
Table 18A.1 Bias-correction factor for the experimental standard deviation	18-60
Table 19.1 Applications of the uncertainty propagation formula	19-34
Table 19.2 Density of air-free water	19-60
Table 19.3 95% confidence interval for a Poisson mean	19-94
Table 19.4 Critical gross count (well-known blank)	19-111

Contents

	<u>Page</u>
Table 19.5 Bias factor for the experimental standard deviation	19-125
Table 19.6 Estimated and true values of SD (tB = tS)	19-131
Table 19.7 Input estimates and standard uncertainties	19-139
Table 20.1 Examples of laboratory-generated wastes	20-2
Table D1 QAPP groups and elements	D-2
Table D2 Comparison of project plan contents	D-3
Table D3 Content of the three elements that constitute the project description	D-9
Table E.1 Examples of procurement options to obtain materials or services	E-7
Table E.2 SOW checklists for the agency and proposer	E-13
Table E.3 Laboratory technical supervisory personnel listed by position title and examples for Table suggested minimum qualifications	E-15
Table E.4 Laboratory technical personnel listed by position title and examples for suggested minimum qualifications and examples of optional staff members	E-16
Table E.5 Laboratory technical staff listed by position title and examples for suggested minimum qualifications	E-16
Table E.6 Example of a proposal evaluation plan	E-26
Table G.1 Quantiles of the standard normal distribution	G-1
Table G.2 Quantiles of Student's t distribution	G-3
Table G.3 Quantiles of chi-square	G-5
Table G.4 Critical values for the nonrandomized exact test	G-7
Table G.5 Critical values of Filliben's statistic	G-11
Table G.6 Summary of probability distributions	G-12

ACRONYMS AND ABBREVIATIONS

Note: Bracketed numbers following each definition represent the first chapter in which the acronym appears.

ADC	analog to digital convertor	[18]
AEA	Atomic Energy Act	[20]
AL	action level	[C]
ANSI	American National Standards Institute	[1]
AOAC	Association of Official Analytical Chemists	[3]
APHA	American Public Health Association	[6]
APS	analytical protocol specification	[1]
ARARs	applicable or relevant and appropriate requirements (CERCLA/Superfund)	[D]
ASL	analytical support laboratory	[15]
ASQC	American Society for Quality Control	[2]
ASTM	American Society for Testing and Materials	[1]
ATD	alpha track detector	[10]
BOA	basic ordering agreement	[4]
CAA	Clean Air Act	[20]
CBD	<i>Commerce Business Daily</i>	[E]
CC	charcoal canisters	[10]
CEDE	committed effective dose equivalent	[2]
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (Superfund)	[2]
CFM	cubic feet per minute	[16]
CFR	<i>Code of Federal Regulations</i>	[20]
CL	central line (of a control chart)	[15]
CMPO	[octyl(phenyl)]-N,N-diisobutylcarbonylmethylphosphine oxide	[14]
CMST	Characterization, Monitoring, and Sensor Technology Program (DOE)	[A]
COC	chain of custody	[2]
COR	contracting officer's representative	[5]
cpm	counts per minute	[12]
cps	counts per second	[15]
CRM	continuous radon monitor	[10]
CRM	certified reference material	[18]
CWA	Clean Water Act	[20]
CWLM	continuous working level monitor	[10]

Acronyms and Abbreviations

DAAP	di-amyloxyphosphonate	[14]
DCGL	derived concentration guideline level	[2]
DIN	di-isopropylnaphthalene	[16]
DL	discrimination limit	[C]
DoD	U.S. Department of Defense	[1]
DOE	U.S. Department of Energy	[1]
DOELAP	DOE Lab Accreditation Program	[18]
DOT	U.S. Department of Transportation	[5]
DPM	disintegrations per minute	[12]
DPPP	dipentylpentylphosphonate	[14]
DQA	data quality assessment	[1]
DQI	data quality indicators	[3]
DQO	data quality objective	[1]
DTPA	diethylene triamine penta-acetic acid	[10]
DVB	divinylbenzene	[14]
EDD	electronic data deliverables	[17]
EDTA	ethylene diamine tetra acetic acid	[10]
EGTA	ethyleneglycol bis(2-aminoethylether)-tetraacetate	[14]
EPA	U.S. Environmental Protection Agency	[1]
ERPRIMS	Environmental Resources Program Management System (U.S. Air Force)	[17]
ESC	expedited site characterization	[A]
eV	electron volts	[15]
FAR	<i>Federal Acquisition Regulations</i>	[E]
FDA	U.S. Food and Drug Administration	[1]
FWHM	full width of a peak at half maximum	[8]
FWTM	full width of a peak at tenth maximum	[18]
GC	gas chromatography	[14]
GLPC	gas-liquid phase chromatography	[14]
GM	Geiger-Mueller detector	[11]
GUM	<i>Guide to the Expression of Uncertainty in Measurement</i>	[1]
HDBP	dibutylphosphoric acid	[14]
HDEHP	bis(2-ethylhexyl) phosphoric acid	[16]
HDEHP	diethylhexylphosphoric acid	[14]

HDPE high density polyethylene [10]
HPGe high-purity germanium [semiconductor] [15]
HPLC high-pressure liquid chromatography; high-performance liquid chromatography
[14]
HTRW hazardous, toxic and radioactive waste [10]

ICP-MS inductively coupled plasma-mass spectroscopy [14]
IPPD integrated product and process development [A]
ISO International Organization for Standardization [1]
IUPAC International Union of Pure and Applied Chemistry [1]

LAN local area network [17]
LBGR lower boundary of the gray region [B]
LCL lower control limit [18]
LCS laboratory control samples [3]
LDPE low density polyethylene [10]
LEGe low energy germanium [15]
LIMS Laboratory Information Management System [17]
LLD lower limit of detection [19]
LLRW low-level radioactive waste [20]
LLRWPA Low Level Radioactive Waste Policy Act [20]
LOMI low oxidation-state transition-metal ion [10]
LPC liquid partition chromatography; liquid-phase chromatography [14]
LS liquid scintillation [15]
LSC liquid scintillation counting [15]
LWL lower warning limit [18]

MAPEP Mixed Analyte Performance Evaluation Program [DOE] [5]
MARSSIM Multi-Agency Radiation Survey and Site Investigation Manual [1]
MCA multichannel analyzer [15]
MDA minimum detection analysis [15]; minimum detectable amount [7]
MDC minimum detectable concentration [3]
MDL method detection limit [19]
MDC minimum detectable concentration [2]
MIBK methyl isobutyl ketone [14]
MQC minimum quantifiable concentration [3]
MQO measurement quality objective [1]

Acronyms and Abbreviations

MS	matrix spike	[8]
MSD	matrix spike duplicate	[8]
MVRM	method validation reference material	[5]
NELAC	National Environmental Laboratory Accreditation Conference	[5]
NESHAP	National Emission Standards for Hazardous Air Pollutants	[12]
NIST	National Institute of Standards and Technology	[1]
NRC	U.S. Nuclear Regulatory Commission	[1]
NRIP	NIST Radiochemistry Intercomparison Program	[18]
NTA or NTTA	nitrilotriacetate	[14]
NTU	nephelometric turbidity units	[10]
NVLAP	National Voluntary Laboratory Accreditation Program (NIST)	[5]
OA	observational approach	[A]
OFHC	oxygen-free high-conductivity	[15]
OFPP	Office of Federal Procurement Policy	[E]
PARCC	precision, accuracy, representativeness, completeness, and comparability	[3]
PCB	polychlorinated biphenyl	[20]
PDF	probability density function	[19]
PE	performance evaluation	[5]
PFA	perfluoroalcoholoxil™	[13]
PIC	pressurized ionization chamber	[15]
PT	performance testing	[5]
PTFE	polytetrafluoroethylene	[12]
PUREX	plutonium uranium reduction extraction	[14]
PVC	polyvinyl chloride	[10]
QA	quality assurance	[2]
QAP	Quality Assessment Program (DOE)	[5]
QAPP	quality assurance project plan	[1]
QC	quality control	[1]
RCRA	Resource Conservation and Recovery Act	[15]
REE	rare earth elements	[13]
REGe	reverse-electrode germanium [semiconductor]	[15]
RFP	request for proposals	[5]

RFQ	request for quotations [E]
RMDC	required minimum detectable concentration [8]
ROI	regions of interest [17]
RPD	relative percent difference [7]
RPM	Remedial Project Manager [2]
RSD	relative standard deviation [19]
RSO	Radiation Safety Officer [11]
SA	spike activity [7]
SAFER	streamlined approach for environmental restoration (DOE) [2]
SAM	Site Assessment Manager [2]
SAP	sampling and analysis plan [1]
SI	international system of units [3]
SMO	sample management office [2]
SOP	standard operating procedure [4]
SOW	Statement of Work [1]
SQC	statistical quality control [15]
SR	unspiked sample result [7]
SRM	standard reference material [18]
SSR	spiked sample result [7]
TAT	turnaround time [7]
TBP	tributyl phosphate [14]
TC	to contain [glassware] [18]
TCLP	toxicity characteristic leaching procedure [13]
TD	to deliver [glassware] [18]
TEC	technical evaluation committee [5]
TEDE	total effective dose equivalent [2]
TES	technical evaluation sheet (USGS) [5]
TFM	tetrafluorometoxil™ [13]
TIOA	tri-iso-octylamine [14]
TLD	thermoluminescent dosimeter [10]
TOPO	trioctylphosphinic oxide [14]
TPO	Technical Project Officer [2]
TPP	technical project planning [2]
TPU	total propagated uncertainty [19]
TQM	Total Quality Management [A]

Acronyms and Abbreviations

TRUEX trans uranium extraction [14]
TSCA Toxic Substances Control Act [20]
TSDf treatment, storage, or disposal facility [20]
TTA thenoyltrifluoroacetone [14]

UBGR upper bound of the gray region [7]
UCL upper control limit [18]
USGS United States Geological Survey [1]
UWL upper warning limit [18]

V volts [15]

WCP waste certification plan [20]

XtGe extended-range germanium [semiconductor] [15]