1 INTRODUCTION TO MARLAP

1.1 Overview

Each year, hundreds of millions of dollars are spent on projects and programs that rely, to varying degrees, on radioanalytical data for decision-making. These decisions often have a significant impact on human health and the environment. Of critical importance to informed decision-making are data of known quality appropriate for its intended use. Making incorrect decisions due to data inadequacies, such as failing to remediate a radioactively contaminated site, necessitates the expenditure of additional resources, causes delays in project completions and, depending on the nature of the project, can result in the loss of public trust and confidence. The Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual addresses the need for a nationally consistent approach to producing radioanalytical laboratory data that meet a project's or program's data requirements. MARLAP provides guidance for the planning, implementation, and assessment phases of those projects that require the laboratory analysis of radionuclides. The guidance provided by MARLAP is both scientifically rigorous and flexible enough to be applied to a diversity of projects and programs. This guidance is intended for project planners, managers, and laboratory personnel.

MARLAP is divided into two main parts. Part I is primarily for project planners and managers and provides guidance on project planning with emphasis on analytical planning issues and analytical data requirements. Part I also provides guidance on preparing project plan documents and radioanalytical statements of work (SOWs), obtaining and evaluating radioanalytical laboratory services, data validation, and data quality assessment. Part I of MARLAP covers the entire life of a project that requires the laboratory analysis of radionuclides from the initial project planning phase to the assessment phase.

Part II of MARLAP is primarily for laboratory personnel and provides guidance in the relevant areas of radioanalytical laboratory work. Part II offers information on the laboratory analysis of radionuclides. It provides guidance on a variety of activities performed at radioanalytical laboratories including sample preparation, sample dissolution, chemical separations, instrument measurements, data reduction, etc. Note that Part II of the manual is not a compilation of analytical procedures. While the chapters in Part II do not contain detailed step-by-step instructions of how to perform certain laboratory tasks, they do provide information on many of the options available for these tasks, and discuss advantages and disadvantages of each.

MARLAP was developed collaboratively by the following Federal agencies: the Environmental Protection Agency (EPA), the Department of Energy (DOE), the Nuclear Regulatory Commission (NRC), the Department of Defense (DOD), the National Institute of Standards and

Technology (NIST), the United States Geological Survey (USGS), and the Food and Drug Administration (FDA). State participation in the development of MARLAP involved contributions from representatives from the Commonwealth of Kentucky and the State of California.

1.2 Purpose of the Manual

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 and needs. To attain this goal, MARLAP provides the necessary guidance for national consistency in radioanalytical work in the form of a performance-based approach for meeting a project's data requirements. In general terms, a performance-based approach to laboratory analytical work involves clearly defining the analytical data needs and requirements of a project in terms of measurable goals during the planning phase of a project. These project-specific analytical data needs and requirements then serve as measurement performance criteria for decisions as to exactly how the laboratory analysis will be conducted during the implementation phase of a project. They are used subsequently as criteria for evaluating analytical data during the assessment phase. Therefore, through a performance-based approach, MARLAP provides guidance in the planning, implementation and assessment phases for those projects that require the laboratory analysis of radionuclides. The manual focuses on activities performed at radioanalytical laboratories, as well as activities and issues that direct, affect, or can be used to evaluate activities performed at radioanalytical laboratories. The guidance in MARLAP is intended to help ensure the generation of radioanalytical data of known quality appropriate for its intended use.

Specific objectives of MARLAP include:

- Promoting a directed planning process for projects involving individuals from relevant disciplines including radiochemistry;
- Highlighting common radioanalytical planning issues;
- Providing a framework and information resource for using a performance-based approach for planning and conducting radioanalytical work;
- Providing guidance on linking project planning, implementation, and assessment;
- Providing guidance on obtaining and evaluating radioanalytical laboratory services;

- Providing guidance for evaluating radioanalytical laboratory data, i.e., data verification, data validation, and data quality assessment;

- Promoting high quality radioanalytical laboratory work; and

- Making collective knowledge and experience in radioanalytical work widely available.

As indicated by the list of objectives, MARLAP provides guidance to project planners, managers, and laboratory personnel for a range of activities for those projects and programs that require the laboratory analysis of radionuclides.

1.3 Use and Scope of the Manual

The guidance contained in MARLAP is for both governmental and private sectors. Users of MARLAP include project planners, project managers, laboratory personnel, regulators, auditors, inspectors, data evaluators, decision makers, and other end users of radioanalytical laboratory data.

Since MARLAP uses a performance-based approach to laboratory measurements, the guidance contained in the manual is applicable to a wide range of projects and activities that require radioanalytical laboratory measurements. Examples of data collection activities that MARLAP supports include:

- Site characterization activities;
- Site cleanup and compliance demonstration activities;
- License termination activities;
- Decommissioning of nuclear facilities;
- Remedial and removal actions;
- Effluent monitoring of licensed facilities;
- Environmental site monitoring;
- Background studies;
- Routine ambient monitoring; and
- Waste management activities.

MARLAP and the *Multi-Agency Radiation Survey and Site Investigation Manual* (MARSSIM, 2000) are complementary guidance documents in support of cleanup and decommissioning activities. MARSSIM provides guidance on how to plan and carry out a study to demonstrate that a site meets appropriate release criteria. It describes a methodology for planning, conducting, evaluating, and documenting environmental radiation surveys conducted to demonstrate compliance with cleanup criteria. MARLAP provides guidance and a framework for both project planners and laboratory personnel to ensure that radioanalytical data will meet the needs and requirements of cleanup and decommissioning activities.

While MARLAP is designed to support a wide range of projects, some topics are not specifically discussed in the manual. These include high-level waste, mixed waste, and medical applications involving radionuclides. While they are not specifically addressed, much of MARLAP's guidance may be applicable in these areas. Although the focus of the manual is to provide guidance for the planning, implementation, and assessment phases of those projects that require the laboratory analysis of radionuclides, much of the guidance on the planning and assessment phases can be applied wherever the measurement process is conducted, for example, in the field. In addition, MARLAP does not provide specific guidance on sampling design issues, sample collection, field measurements, laboratory quality assurance issues, or laboratory health and safety practices. However, a brief discussion of some aspects of these activities has been included in the manual because of the effect these activities often have on the laboratory analytical process.

1.4 Key MARLAP Concepts and Terminology

Some of the terms used in MARLAP were developed for the purpose of this manual, while others are commonly used terms that have been adopted by MARLAP. Where possible, every effort has been made to use terms and definitions from consensus-based organizations (e.g., International Organization for Standardization [ISO], American National Standards Institute [ANSI], American Society for Testing and Materials [ASTM], International Union of Pure and Applied Chemistry [IUPAC]).

The following sections are intended to familiarize the reader with the key terms and concepts used in MARLAP. In general, each term or concept is discussed individually in each section without emphasizing how these terms and concepts are linked. Section 1.5 ties these terms and concepts together to provide an overview of the MARLAP process.

1.4.1 Data Life Cycle

The data life cycle (EPA, 2000) approach provides a structured means of considering the major phases of projects that involve data collection activities (Figure 1.1). The three phases of the data life cycle are planning, implementation, and assessment. MARLAP provides information on all three phases for two major types of activities: those performed at radioanalytical laboratories and those that direct, affect, or evaluate activities performed at radioanalytical laboratories (such as project planning, development of plan documents, data verification and data validation). Consequently, MARLAP provides guidance for project planners, managers, and laboratory personnel.

DATA LIFE CYCLE		
	PROCESS	PROCESS OUTPUTS
Planning	Directed Planning Process	Development of Data Quality Objectives and Measurement Quality Objectives (Including Optimized Sampling and Analytical Design)
Planning	Plan Documents	Project Plan Documents Including Quality Assurance Project Plan (QAPP); Work Plan or Sampling and Analysis Plan (SAP); Data Validation Plan; Data Quality Assessment Plan
Planning	Contracting Services	Statement of Work (SOW) and Other Contractual Documents
Implementation	Sampling	Laboratory Samples ↓
Implementation	Analysis	Laboratory Analysis (Including QC Samples) Complete Data Package ↓
Assessment	Verification	Verified Data Data Verification Report ↓
Assessment	Validation	Validated Data Data Validation Report ↓
Assessment	Data Quality Assessment	Assessment Report
Data of Known Quality Appropriate for the Intended Use		

FIGURE 1.1 — The Data Life Cycle

One of the specific objectives of the MARLAP Manual is to provide guidance on, and to emphasize the importance of, establishing the proper linkages among the three phases of the data life cycle—planning, implementation and assessment—thereby resulting in an integrated and iterative process that accurately translates the expectations and requirements of data users into measurement performance criteria for data suppliers. From an analytical perspective, the integration of the three phases of the data life cycle is critical to ensure that the analytical data requirements defined during the planning phase serve as measurement performance criteria during the implementation phase and subsequently as criteria for data evaluation during the assessment phase. The proper linkages and integration of the three phases of the data life cycle should be established during the planning phase. Without the proper linkages and integration of the three phases, there is a significant likelihood that the analytical data will not meet a project's data requirements, and the data may be evaluated using criteria that have little relation to their intended use. Therefore, failure to integrate and adequately link the three phases of the data life cycle increases the likelihood of project cost escalation or project failure.

1.4.2 Directed Planning Process

MARLAP recommends the use of a directed or systematic planning process. A directed planning process is an approach for setting well-defined, achievable objectives and developing a cost-effective, technically sound sampling and analysis design that balances the data user's tolerance for uncertainty in the decision process with the resources available for obtaining data to support a decision. While MARLAP recommends and promotes the use of a directed planning process, it does not recommend or endorse any particular directed planning process. However, MARLAP employs many of the terms and concepts associated with the data quality objective (DQO) process (ASTM D5792, EPA, 2000). This was done to ensure consistent terminology throughout the manual, and also because many of the terms and concepts of this process are familiar to those engaged in environmental data collection activities.

1.4.3 Performance-Based Approach

MARLAP provides the necessary guidance for using a performance-based approach to meet a project's analytical data requirements. In a performance-based approach, the project-specific analytical data requirements that are determined during directed planning serve as measurement performance criteria for analytical selections and decisions. The project-specific analytical data requirements also are used for the initial, ongoing, and final evaluation of the laboratory's performance and the laboratory's data. MARLAP provides guidance for using a performance-based approach for all three phases—planning, implementation and assessment—of the data life cycle for those projects that require radioanalytical laboratory data. This involves not only using a performance-based approach for selecting an analytical protocol, but also using a performance-based approach for other project activities, such as developing acceptance criteria for laboratory quality control samples, laboratory evaluations, data verification, data validation, and data quality assessment.

There are three major steps or processes associated with a performance-based approach. The first is clearly and accurately defining the analytical data requirements for the project. This process is discussed in more detail in Section 1.4.9 of this chapter. The second involves using an organized, interactive process for selecting or developing analytical protocols to meet the specified analytical data requirements and for demonstrating the protocol's ability to meet the analytical data requirements. The last major activity involves using the analytical data requirements as measurement performance criteria for the ongoing and final evaluation of the laboratory data, which would include data verification, data validation, and data quality assessment. MARLAP provides guidance in all three of these areas. Within the constraints of other factors, such as cost, a performance-based approach allows for the use of any analytical protocol that meets the project's analytical data requirements. For all relevant project activities, the common theme of a performance-based approach is the use of project-specific analytical data requirements that are developed during project planning and serve as measurement performance criteria for selections, evaluations, and decision-making.

1.4.4 Analytical Process

Most environmental data collection efforts center around two major processes: the sampling process and the analytical process. MARLAP does not provide general guidance on the sampling process, except for brief discussions of certain activities that often affect the analytical process (field processing, preservation, etc.). The analytical (or measurement) process is a general term used by MARLAP to refer to a compilation of activities starting from the time a sample is collected and ending with the reporting of data. These activities typically include field sample preparation and preservation, sample receipt and inspection, laboratory sample preparation, sample dissolution, chemical separations, preparation of samples for instrument measurements, instrument measurements, data reduction, data reporting, and quality control of the process. Figure 1.2 illustrates the major components of an analytical process. It should be noted that a particular analytical process for a project may not include all of the activities listed. For example, if a project involves the analysis of tritium in drinking water, then the analytical process for the project will not include sample dissolution and the chemical separation of the radionuclide of concern. It is important to identify the relevant activities of the analytical process for a particular project early in the planning phase. Once the activities have been identified, the analytical requirements of the activities can be established, which will ultimately lead to defining how the activities will be accomplished through the selection or development of written procedures for the various activities.

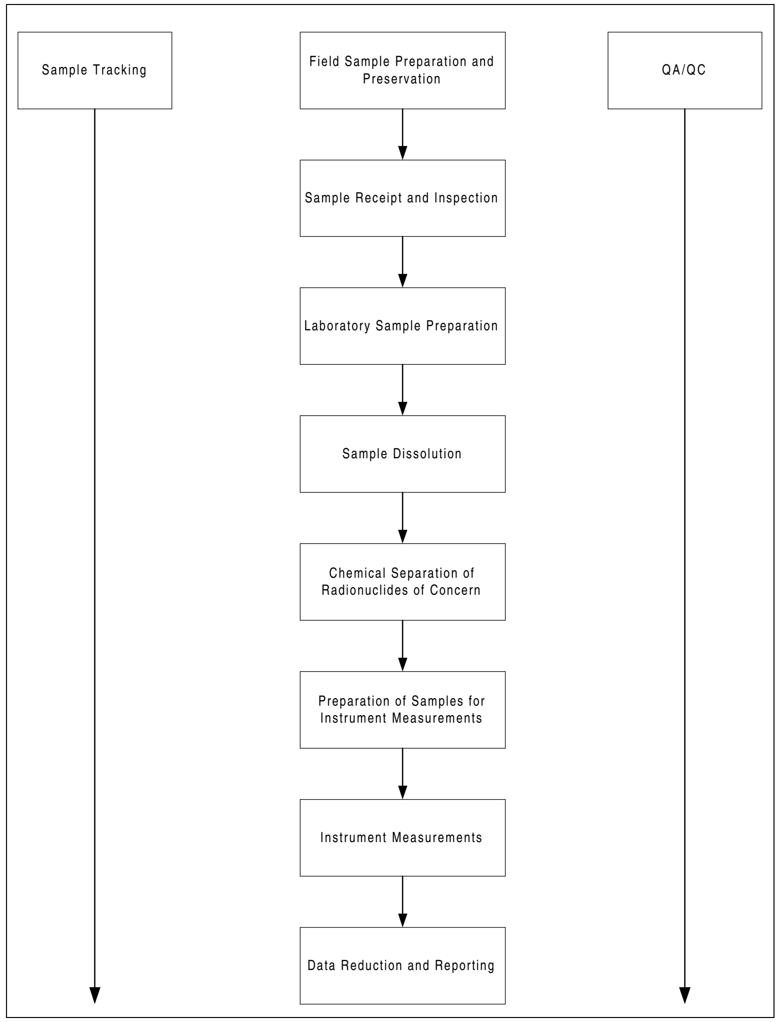

FIGURE 1.2 — Typical Components of an Analytical Process

The analytical process should not be confused with the written procedures necessary to perform the associated activities of the analytical process. The analytical process (i.e., the compilation of activities starting from the time a sample is collected and ending with the reporting of the data) should be performed according to written procedures

1.4.5 Analytical Protocol

MARLAP uses the term "analytical protocol" to refer to a compilation of specific procedures and methods that are performed in succession for a particular analytical process. For example, a protocol for the analysis of drinking water samples for tritium would be comprised of the set of procedures that describe the relevant activities, such as sample tracking, quality control, field sample preparation and preservation, sample receipt and inspection, laboratory sample preparation (if necessary), preparing the samples for counting, counting the samples, and data reduction and reporting. A written procedure may cover one or more of the activities, but it is unlikely that a single procedure will cover all of the activities of a given analytical process. It should be noted that with a performance-based approach, there may be a number of alternative protocols that might be appropriate analytical protocols for a particular analytical process. Selecting or developing an analytical protocol requires knowledge of the particular analytical process, as well as an understanding of the analytical data requirements developed during the project planning phase.

1.4.6 Analytical Method

A major component of an analytical protocol is the *analytical method,* which normally includes written procedures for sample digestion, chemical separation (if required) and counting. It is recognized that in many instances the analytical method may cover many of the activities of a particular analytical process. Therefore attention is naturally focused on the selection or development of an analytical method. However, many analytical methods do not address activities such as field preparation and preservation, certain aspects of laboratory preparation, laboratory subsampling, etc., which are often important activities within an analytical process. The analytical protocol is generally more inclusive of the activities that make up the analytical process than the analytical method. For this reason, MARLAP focuses on the selection, implementation, and assessment of analytical protocols that cover the entire analytical process for a particular project or program.

1.4.7 Uncertainty and Error

An important aspect of sampling and measurement is uncertainty. The term "uncertainty" has different shades of meaning in different contexts, but generally the word always refers to a lack of complete knowledge about something of interest. In the context of metrology (the science of measurement), the more specific term "measurement uncertainty" often will be used. "Uncertainty (of measurement)" is defined in the *Guide to the Expression of Uncertainty in Measurement* (ISO 1995—"GUM") as a "parameter, associated with the result of a measurement, that characterizes the dispersion of values that could reasonably be attributed to the measurand." The "measurand" is the quantity being measured. MARLAP recommends the terminology and methods of GUM for describing, evaluating, and reporting measurement uncertainty. The uncertainty of a measured value is typically expressed as an estimated standard deviation, called a "standard uncertainty" (or "one-sigma uncertainty"). The standard uncertainty of a calculated result usually is obtained by propagating the standard uncertainties of a number of other measured values, and in this case, the standard uncertainty is called a "combined standard uncertainty." The combined standard uncertainty may be multiplied by a specified factor called a "coverage factor" (e.g., 2 or 3) to obtain an "expanded uncertainty" (a "two-sigma" or "three-sigma" uncertainty), which describes an interval about the result that can be expected to contain the true value with a specified high probability. MARLAP recommends that either the combined standard uncertainty or an expended uncertainty be reported with every result. Chapter 19 discusses the terminology, notation, and methods of GUM in more detail and provides guidance for applying the concepts to radioanalytical measurements.

While measurement uncertainty is a parameter associated with an individual result and is calculated after a measurement is performed, MARLAP uses the term "method uncertainty" to refer to the predicted uncertainty of a measured value that likely would result from the analysis of a sample at a specified analyte concentration. Method uncertainty is a method performance characteristic much like the detection capability of a method. Reasonable values for both characteristics can be predicted for a particular method based on typical values for certain parameters and on information and assumptions about the samples to be analyzed. These predicted values can be used in the method selection process to identify the most appropriate method based on a project's data requirements. Chapter 3 provides MARLAP's recommendations for deriving analytical protocol selection criteria based on the required method uncertainty and other analytical requirements.

When a decision maker bases a decision on the results of measurements, the measurement uncertainties affect the probability of making a wrong decision. When sampling is involved, sampling statistics also contribute to the probability of a wrong decision. Since decision errors are possible, there is uncertainty in the decision-making process. MARLAP uses the terms "decision uncertainty" or "uncertainty of the decision" to refer to this type of uncertainty. Decision uncertainty is usually measured by the estimated probability of a decision error under specified assumptions. Appendix B discusses decision uncertainty further in the context of the DQO process.

A concept that should not be confused with uncertainty is error. In general, error refers to something that deviates from what is correct, right or true. In terms of measurements such as laboratory analyses, the difference between the measured result and the actual value of the measurand is the error of the measurement. Since the actual value of the measurand is generally not known, the measurement error cannot be determined. Therefore, the error of a measurement is primarily a theoretical concept with little practical use. However, the measurement uncertainty, which provides an estimated bound for the likely size of the measurement error, is very useful and plays a key role in MARLAP's performance-based approach.

1.4.8 Precision, Bias, and Accuracy

Analytical data requirements often have been described in terms of precision and bias. Precision is usually expressed as a standard deviation, which measures the dispersion of measured values about their mean. It is more natural to speak of imprecision, since larger values of the standard deviation indicate less precision and greater imprecision. Bias is a persistent difference between the measured result and the true value of the quantity being measured, which does not vary if the measurement is repeated. If the measurement process is in statistical control, then imprecision may be reduced by averaging the results of many independent measurements of the same quantity. Bias is unaffected by averaging.

A bias in a data set may be caused by measurement errors that occur in steps of the measurement process that are not repeated, such as the determination of a half-life. Imprecision may be caused by measurement errors in steps that are repeated many times, such as weighing, pipetting, and radiation counting. However, distinguishing between bias and imprecision is complicated by the fact that some steps in the process, such as instrument calibration or tracer preparation, are repeated at frequencies less than those of other steps, and the measurement errors in seldom repeated steps may affect large blocks of data. Consequently measurement errors that produce apparent biases in small data sets might produce apparent imprecision in larger data sets.

Because the same type of measurement error may produce either bias or imprecision, depending on one's point of view, the concept of measurement uncertainty, described in Section 1.3.7, treats all types of measurement error alike and combines estimates of their magnitudes into a single numerical parameter (i.e., combined standard uncertainty). The concepts of imprecision and bias are useful in context when a measurement process or a data set consisting of many measurement results is considered. When one considers only a single measurement result, the concept of measurement uncertainty tends to be more useful than the concepts of imprecision and bias. Therefore, it is probably best to consider imprecision and bias to be characteristics of the measurement process or of the data set, and to consider measurement uncertainty to be an aspect of each individual result.

Quality control samples are analyzed for the purpose of assessing imprecision and bias. Spiked samples and method blanks are typically used to assess bias, and duplicates are used to assess imprecision. Since a single measurement of a spike or blank cannot in principle distinguish between imprecision and bias, a reliable estimate of bias requires a data set that includes many such measurements.

Different authors have given the word *accuracy* different technical definitions, expressed in terms of bias and imprecision. MARLAP avoids all of these technical definitions and uses the term "accuracy" in its common, ordinary sense, which is consistent with its definition in the *International Vocabulary of Basic and General Terms in Metrology* (ISO, 1993). In MARLAP's terminology, the result of a measurement is "accurate" if it is close to the true value of the quantity being measured. Inaccurate results may be caused either by bias or imprecision in the measurement process.

While it is recognized that the terms bias, imprecision, and accuracy are commonly used in data collection activities, these terms are used somewhat sparingly in this manual. MARLAP emphasizes and provides guidance in the use of measurement uncertainty as a means of establishing analytical data requirements and in the evaluation of single measurement results.

1.4.9 Performance Objectives: Data Quality Objectives and Measurement Quality Objectives

One of the outputs of a directed planning process is DQOs for a project or program. DQOs are qualitative and quantitative statements that clarify the study objectives; define the most appropriate type of data to collect; determine the most appropriate conditions from which to collect the data; and specify tolerable limits on decision error rates (ASTM D5792; EPA, 2000). DQOs apply to all data collection activities associated with a project or program, including sampling and analysis. In particular, DQOs should encompass the "total uncertainty" resulting from all data collection activities, including analytical and sampling activities.

From an analytical perspective, a process of developing the analytical data requirements from the DQOs of a project is essential. These analytical data requirements serve as measurement performance criteria or objectives of the analytical process. MARLAP refers to these performance objectives as "measurement quality objectives" (MQOs). The MARLAP Manual provides guidance on developing the MQOs from the overall project DQOs (Chapter 3). MQOs can be viewed as the analytical portion of the DQOs and are therefore project-specific. MARLAP provides guidance on developing MQOs during project planning for select method performance characteristics, such as method uncertainty at a specified concentration; detection capability; quantification capability; specificity, or the capability of the method to measure the analyte of concern in the presence of interferences; range; ruggedness, etc. An MQO is a statement of a performance objective or requirement for a particular method performance characteristic. Like DQOs, MQOs can be quantitative and qualitative statements. An example of a quantitative MQO would be a statement of a required method uncertainty at a specified radionuclide concentration, such as the action level—i.e., "a method uncertainty of 3.7 Bq/kg (0.10 pCi/g) or less is required at the action level of 37 Bq/kg (1.0 pCi/g)." An example of a qualitative MQO would be a statement of the required specificity of the analytical protocol—the ability to analyze for the radionuclide of concern given the presence of interferences—i.e., "the protocol must be able to quantify the amount of ^{226}Ra present given high levels of ^{235}U in the samples."

The MQOs serve as measurement performance criteria for the selection or development of analytical protocols and for the initial evaluation of the analytical protocols. Once the analytical protocols have been selected and evaluated, the MQOs serve as criteria for the ongoing and final evaluation of the laboratory data, including data verification, data validation, and data quality assessment. In a performance-based approach, analytical protocols are either selected or rejected for a particular project, to a large measure, based on their ability or inability to achieve the stated MQOs. Once selected, the performance of the analytical protocols is evaluated using the project-specific MQOs.

1.4.10 Analytical Protocol Specifications

MARLAP uses the term "analytical protocol specifications" (APSs) to refer to the output of a directed planning process that contains the project's analytical data requirements in an organized concise form. In general, there will be an APS developed for each analysis type, and since most projects require that a number of different analyses be performed, several APSs will normally be developed for a particular project. These specifications serve as the basis for the evaluation and selection of the analytical protocols that will be used for a particular project. In accordance with a performance-based approach, the APSs contains only the minimum level of specificity required to meet the project's analytical data requirements without dictating exactly how the requirements are to be met. At a minimum, the APSs should indicate the analyte of interest, the matrix of concern, the type and frequency of quality control (QC) samples, and provide the required MQOs and any specific analytical process requirements, such as chain-of-custody for sample tracking.

Depending on the particular project, a number of specific analytical process requirements may be included. For example, if project or process knowledge indicates that the radionuclide of interest exists in a refractory form, then the APSs may require a fusion step for sample digestion. However, the level of specificity in the APSs should be limited to those requirements that are considered essential to meeting the project's analytical data requirements. In most instances, a particular APS document would be a one-page form (see Chapter 3, Figure 3.2). For a particular project, APSs would be developed for each analysis required.

Within the constraints of other factors, such as cost, MARLAP's performance-based approach allows the use of any analytical protocol that meets the requirements contained in the APSs. The requirements in the APSs, in particular the MQOs, are used for the selection and evaluation of the analytical protocols. Once the analytical protocols have been selected and evaluated, the APSs then serve as criteria for the ongoing and final evaluation of the laboratory data, including data verification, data validation, and data quality assessment.

1.4.11 The Assessment Phase

As noted, the MARLAP Manual provides guidance for the assessment phases for those projects that require the laboratory analysis of radionuclides. The guidance on the assessment phase of projects focuses on three major activities: data verification, data validation, and data quality assessment.

Data verification assures that laboratory conditions and operations were compliant with the statement of work and any appropriate project plan documents (e.g., Quality Assurance Project Plan), which may reference laboratory documents such as laboratory standard operating procedures) Verification compares the material delivered by the laboratory to these requirements (compliance) and checks for consistency and comparability of the data throughout the data package, correctness of calculations, and completeness of the results to ensure that all necessary documentation is available. The verification process produces a report identifying which requirements are not met. The verification report is used to determine payment for laboratory services and to identify problems that should be investigated during data validation. Verification works iteratively and interactively with the generator (i.e., laboratory) to assure receipt of all available, necessary data. Although the verification process identifies specific problems, the primary function should be to apply appropriate feedback resulting in corrective action improving the analytical services before the work is completed.

Validation addresses the reliability of the data. The validation process begins with a review of the verification report and laboratory data package to screen the areas of strength and weakness of the data set. The validator evaluates the data to determine the presence or absence of an analyte and the uncertainty of the measurement process for contaminants of concern. During validation, the technical reliability and the degree of confidence in reported analytical data are considered. Validation "flags" (i.e., qualifiers) are applied to data that do not meet the acceptance criteria established to assure data meet the needs of the project. The product of the validation process is a validation report noting all data sufficiently inconsistent with the validation acceptance criteria in the expert opinion of the validator. The appropriate data validation tests should be established during the project planning phase.

Data quality assessment (DQA), the third and final step of the assessment phase, is defined as the "scientific and statistical evaluation of data to determine if data are of the right type, quality, and quantity to support their intended use." DQA is more global in its purview than the previous verification and validation steps. DQA, in addition to reviewing the issues raised during verification and validation, may be the first opportunity to review other issues, such as field activities and their impact on data quality and usability. DQA should consider the combined impact of all project activities in making a data usability determination, which is documented in a DQA report.

1.5 The MARLAP Process

An overarching objective of the MARLAP Manual is to provide a framework and information for the selection, development, and evaluation of analytical protocols and the resulting laboratory data. The MARLAP process is a performance-based approach that develops APSs and uses these requirements as criteria for the analytical protocol selection, development and evaluation processes, and for the evaluation of the resulting laboratory data. This process, which spans the three phases of the data life cycle for a project—planning, implementation and assessment—is the basis for achieving MARLAP's basic goal of ensuring that radioanalytical data will meet a project's data requirements. A brief overview of this process, which is referred to as the MARLAP process and is the focus of Part I of the manual, is provided below.

The MARLAP process starts with a directed planning process. Within a directed planning process, key analytical issues based on the project's particular analytical processes are discussed and resolved. The resolution of these key analytical issues produces the APSs, which include the MQOs. The APSs are documented in project plan documents (e.g., Quality Assurance Project Plans, Sampling and Analysis Plans). A SOW is then developed that contains the APSs. The laboratories receiving the SOW respond with proposed analytical protocols based on the requirements of the APSs and provide evidence that the proposed protocols meet the performance criteria in the APSs. The proposed analytical protocols are initially evaluated by the project manager or designee to determine if they will meet the requirements in the APSs. If the proposed analytical protocols are accepted, the project plan documents are updated by the inclusion or referencing of the actual analytical protocols to be used. During analyses, resulting sample and QC data will be evaluated primarily using MQOs from the respective APSs. Once the analyses are completed, an evaluation of the data will be conducted, including data verification, data validation, and data quality assessment with the respective MQOs serving as criteria for evaluation. The role of the APSs (particularly the MQOs, which make up an essential part of the APSs) in the selection, development, and evaluation of the analytical protocols and the laboratory data is to provide a critical link between the three phases of the data life cycle of a project. This linkage helps to ensure that radioanalytical laboratory data will meet a project's data requirements, and that the data are of known quality appropriate for their intended use. The MARLAP process is illustrated in Figure 1.3. Although the diagram used to represent the MARLAP Process is presented in a linear fashion, it is important to note that the process is an iterative one, and there can be many variations on this stylized diagram.

1.6 Structure of the Manual

MARLAP is divided into two main parts. Part I provides guidance on implementing the MARLAP process as described in Section 1.5. This part of the manual focuses on the sequence of steps involved when using a performance-based approach for projects requiring radioanalytical laboratory work starting with a directed planning process and ending with DQA. Part I provides the overall guidance for using a performance-based approach for all three phases of a project. A more detailed overview of Part I is provided in Section 1.6.1.

Part II of the manual provides information on the laboratory analysis of radionuclides to support a performance-based approach. Part II provides guidance and information on the various activities performed at radioanalytical laboratories, such as sample preparation, sample dissolution, chemical separations, preparing sources for counting, nuclear counting, etc. Using the overall framework provided in Part I, the material in Part II can be used to assist project planners, managers, and laboratory personnel in the selection, development, evaluation, and implementation of analytical protocols for a particular project or program. A more detailed overview of Part II is provided in Section 1.6.2. In addition to Part I and Part II, MARLAP has several appendices that support both Part I and Part II of the manual. An overview of the appendices is provided in Section 1.6.3 of this chapter.

Because of the structure and size of the manual, most individuals will naturally focus on those chapters that provide guidance in areas directly related to their work. Therefore, to help ensure that key concepts are conveyed to the readers, there is some material is repeated, often in very similar or even the same language, throughout the manual.

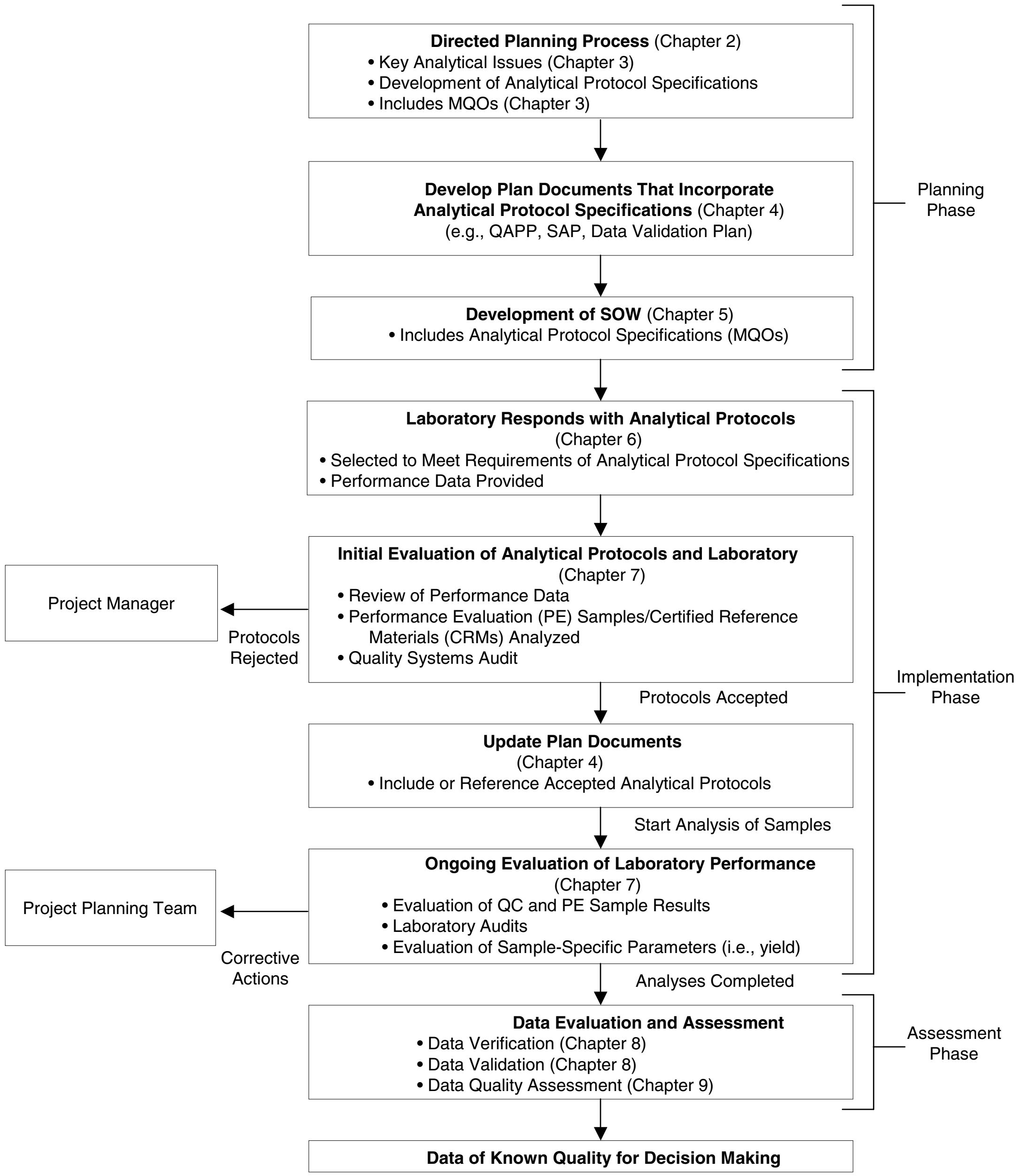

FIGURE 1.3 — The MARLAP Process

1.6.1 Overview of Part I

Part I begins with Chapter 2, *Project Planning Process,* which provides an overview of the directed planning process and discusses important analytical outputs of the planning process. Chapter 3, *Key Analytical Planning Issues and Developing APSs*, provides an overview of key analytical planning issues that need to be addressed during a directed planning process and provides guidance on developing APSs, which are outputs of the planning process. These outputs are incorporated into plan documents (e.g., work plans, quality assurance project plans, sampling and analysis plans), which are covered in Chapter 4, *Project Plan Documents*. Chapter 4 provides an overview of different types of project plan documents and provides guidance on the linkage between project planning and project plan documents. Information from the plan documents is then incorporated into a SOW, which is covered in Chapter 5, *Obtaining Laboratory Services*. Chapter 5 provides guidance on developing a SOW that incorporates the APSs. Chapter 6, *Selection and Application of an Analytical Method,* provides guidance on selecting or developing analytical protocols that will meet the MQOs and other requirements as outlined in the APSs. Chapter 7, *Evaluating Protocols and Laboratories,* provides guidance on the initial and ongoing evaluation of analytical protocols and also provides guidance on the overall evaluation of radioanalytical laboratories. Chapter 8, *Radiochemical Data Verification and Validation,* provides an overview of the data evaluation process, provides general guidelines for data verification and validation, and provides "tools" for data validation. The last chapter of Part I, Chapter 9, *Data Quality Assessment,* provides an overview of data quality assessment and provides guidance on linking data quality assessment and the planning process.

Figure 1.3, the MARLAP Process, illustrates the sequence of steps that make up the framework of a performance-based approach for the planning, implementation, and assessment phases of projects that require the laboratory analysis of radionuclides. The primary audience for Part I is project planners and managers. However, Chapter 6, *Selection and Application of an Analytical Method,* is intended primarily for laboratory personnel. This is because, under a performance-based approach, a laboratory would be able to use any analytical protocol that meets the analytical requirements as defined by the APSs. Other factors, such as cost, also will play a role in the selection of analytical protocols. While the primary audience for Part I is project planners and managers, other groups, such as laboratory personnel, can benefit from the guidance in Part I.

1.6.2 Overview of Part II

The chapters in Part II are intended to provide information on the laboratory analysis of radionuclides. The chapters provide information on many of the options available for analytical protocols, and discuss common advantages and disadvantages of each. The chapters highlight common analytical problems and ways to identify and correct them. The chapters also serve to educate the reader by providing a detailed explanation of the typical activities performed at a radioanalytical laboratory. Consistent with a performance-based approach, the chapters in Part II do not contain detailed step-by-step instructions on how to perform certain laboratory tasks, such as the digestion of a soil sample. The chapters do contain information and guidance intended to assist primarily laboratory personnel in deciding on the best approach for a particular laboratory task. For example, while the chapter on sample dissolution does not contain step-by-step instructions on how to dissolve a soil sample, it does provide information on acid digestion, fusion techniques, and microwave digestion, which is intended to help the reader select the most appropriate technique or approach for a particular project.

The primary audience for Part II is laboratory personnel and the chapters generally contain a significant amount of technical information. While the primary target audience is laboratory personnel, other groups, such as project planners and managers, can benefit from the guidance in Part II. Listed below are the chapters that make up Part II of the manual. It should be noted that Part II of the manual does not provide specific guidance for some laboratory activities that are common to all laboratories, such as laboratory quality assurance, and laboratory health and safety practices. This is primarily due to the fact that these activities are not unique to radioanalytical laboratories and considerable guidance in these areas already exists.

Chapter 10	Requirements When Collecting, Preserving, and Shipping Samples That Require Analytical Measurement
Chapter 11	Sample Receipt, Inspection and Tracking
Chapter 12	Laboratory Sample Preparation
Chapter 13	Sample Dissolution
Chapter 14	Separation Techniques
Chapter 15	Nuclear Counting Instrumentation
Chapter 16	Instrument Calibration and Source Preparation
Chapter 17	Nuclear Counting and Data Reduction and Reporting
Chapter 18	Laboratory Quality Control
Chapter 19	Measurement Statistics
Chapter 20	Waste Disposal

Chapters 10 through 17 provide information on the typical components of an analytical process in the order in which activities that make up an analytical process are normally performed. While not providing step-by-step procedures for activities such as sample preservation, sample digestion, nuclear counting, etc., the chapters do provide an overview of options available for the various activities and importantly, provide information on the appropriateness of the assorted options under a variety of conditions. Chapter 18, *Laboratory Quality Control,* provides guidance on monitoring key laboratory performance indicators as a means of determining if a laboratory's measurement processes are in control. The chapter also provides information on likely causes of excursions for select laboratory performance indicators, such as chemical yield, instrument background, quality control samples, etc. Chapter 19, *Measurement Statistics*, provides information on statistical principles and methods applicable to radioanalytical measurements, calibrations, data interpretation, and quality control. Topics covered in the chapter include detection and quantification, measurement uncertainty, and procedures for estimating uncertainty. Chapter 20, *Waste Disposal* provides an overview of many of the regulations for waste disposal and provides guidance for managing wastes in a radioanalytical laboratory.

1.6.3 Overview of the Appendices

MARLAP includes several appendices to both Part I and Part II of the manual to provide additional guidance on specific topics. Brief descriptions of the appendices are provided below.

- Appendix A, *Directed Planning Approaches,* provides an overview of a number of directed planning processes and discusses some common elements of the different approaches.

- Appendix B, *The Data Quality Objective Process,* provides an expanded discussion of the Data Quality Objectives Process including detailed guidance on setting up a "gray region" and establishing tolerable decision error rates.

- Appendix C, *Measurement Quality Objectives for Method Uncertainty and Detection and Quantification Capability,* provides the rationale and guidance for developing MQOs for select method performance characteristics.

- Appendix D, *Content of Project Plan Documents*, provides guidance on the appropriate content of plan documents.

- Appendix E, *Contracting Laboratory Services,* contains detailed guidance on contracting laboratory services.

- Appendix F, *Laboratory Subsampling,* provides information on improving and evaluating laboratory subsampling techniques.

- Appendix G, *Statistical Tables,* provides a compilation of statistical tables.

1.7 References

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