1. QUALITY ASSURANCE

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1. QUALITY ASSURANCE

1.1 OVERVIEW

The Management of the Environmental Measurements Laboratory (EML) is fully committed to the maintenance of an effective quality assurance (QA) program in order that all work carried out by EML will be of high quality. The EML Institutional Quality Assurance Program Plan (EM-6-2) describes the QA procedures used at the Laboratory. Guidelines for the design of the EML Institutional Quality Assurance Program Plan are provided for by: DOE Order 5700.6C, "Quality Assurance;" DOE-ER-STD-6001-92, "DOE Standard: Implementation Guide for Quality Assurance Programs for Basic and Applied Research;" and Chicago Operations Office Order CH 5700.6C, "Quality Assurance." The EML QA program has been designed to conform to those guidelines.

The management system of the QA program at EML includes the entire staff, from the Laboratory Director, through the Deputy Director and the Division Directors to the project leaders and first line supervisors, and finally to the scientist or engineer in the laboratory or in the field. The QA Officer oversees the functioning of the Institutional QA Program (see Section 1.5).

An important aspect of the EML QA program is the written documentation of QA and quality control (QC) procedures that are used in the performance of research projects. A Project QA Plan is prepared for each project that is contained in a field work proposal, describing or referencing the procedures used during the project and the quality of results from these procedures that is required for the successful performance of the project. Twice each year, the Division Director or Program Coordinator who oversees a project submits to the EML QA Officer a brief evaluation of the current level of quality of performance on that project. The QA Officer selects a few of these projects and makes a detailed examination of the evidence of quality that was used to evaluate them. The documentation of the quality of the work is described in this section, and in subsequent sections of this Manual. Under normal circumstances, all research reports contain a section in which the QA procedures used and the results obtained are discussed. When they believe that it will be beneficial, Division Directors may require the submission of

special QA reports for specific projects within their divisions. QA reports can be used during assessments of a project by the Division Director or by the QA Officer to assure that QA requirements are being met. For a QA program to be effective, corrective action must always be taken when substandard results are detected, and subsequent follow-up audits must be made to verify that any problems have been solved. The Division Directors oversee the planning, performance, and documentation of any needed remedial actions required as the result of an audit.

The most important requirement for the success of the QA program is the commitment in the Laboratory that our goal is to always perform high quality work. This requires both a degree of dedication of the staff and an absolute honesty in data preparation.

1.2 SCOPE

The policies, procedures, guidelines, and implementation practices at EML with regard to its QA program are presented in this section. Also, outlined are the responsibilities of EML personnel to ensure the quality of the data at all phases of the project from planning to the reporting of the data.

1.3 DEFINITIONS

Three definitions related to QA practices are given below. A more complete list may be found in Section 1.11.

QA involves all those planned and systematic actions necessary to provide adequate confidence that a facility, structure, system, or component will perform satisfactorily and safely in service.

QC, which is included within QA, comprises all those actions necessary to control and verify the features and characteristics of a material, process, product, or service to specified requirements.

Audit/Appraisal is a planned and documented activity performed in accordance with procedures to determine, by examination and evaluation of objective evidence, the adequacy of and extent to which applicable elements of the QA program have been developed, documented, and effectively implemented in accordance with specified requirements.

1.4 STAFF QUALIFICATIONS

The first step in establishing a QA program is the securing of personnel who are competent to perform the technical procedures, and training them so that they are thoroughly familiar with the instruments and procedures that they will use. This is as important as are written QC procedures and record keeping. Technical personnel should understand the nature of the physical and chemical properties that affect the measurement procedures that they perform, so that they may recognize and interpret any deviations from the expected behavior. When technical personnel are to perform a procedure in which they are not experienced, they are first trained, then tested in order to demonstrate that they can perform the procedure with an acceptable level of uncertainty.

1.5 STAFF RESPONSIBILITIES

1.5.1 INTRODUCTION

All individuals and all levels of management at EML have responsibilities within the QA program. Although the Laboratory Director has overall supervision of and responsibility for the program, and the QA Officer oversees its functioning, the Deputy Director, Division Directors, first line supervisors, project leaders, field workers and laboratory workers are responsible for the quality of the work that is performed under their control. The specific responsibilities that are described below are applicable in most instances. However, each research project has its own individual characteristics, and Division Directors may reassign specific responsibilities within particular projects to attain the most efficient organization.

1.5.2 RESPONSIBILITIES OF FIELD WORKERS AND LABORATORY WORKERS

The field workers and laboratory workers are responsible for the quality of their performance of assigned tasks. They must be thoroughly familiar with the QA aspects of the procedures that they follow. For the most part, these procedures are described in later sections of this Manual. All personnel must carefully adhere to those procedures, must report to their supervisors any problems that caused deviation from these procedures or results from standard procedures or expected results, and must undertake to correct identified problems. They are responsible for the prevention of the loss of quality during handling of the equipment and materials that they use in their work, and for the routine maintenance, calibration and the general care of their equipment. They maintain records of instrument calibrations and of the results of the QC procedures that they perform.

1.5.3 RESPONSIBILITIES OF FIRST LINE SUPERVISORS

First line supervisors ensure that the personnel under their supervision are aware of, and fulfill their responsibilities for the QA aspects of the tasks that they perform. They ensure that only authorized personnel perform the research operations, that proper care is taken of equipment and materials to avoid loss of quality, and that approved sampling and measurement procedures (most of which are described in Sections 2, 3, and 4) are used. First line supervisors are responsible for the specification and supervision of appropriate QA procedures to be performed by the personnel under their supervision, and they periodically review the procedures that are in use to ensure their adequacy for determining the quality of the results that will be obtained from current and future operations. They see that logbooks and other records, such as calibrations and the results of QC procedures, are maintained properly, that appropriate information is recorded, and that any changes that are made in documents that give instructions or describe procedures are reviewed and approved. They test the validity of measurement data that they report, and ensure that corrective actions are taken when problems arise, and that follow-up verification is obtained of the success of those actions.

First line supervisors ensure that measuring and test equipment and materials are traceable to national standards or to equivalent calibration standards, and that they are

cared for properly by those who are using them. They implement documented QA procedures to ensure that the quality of items and services procured to accomplish tasks under their supervision meets appropriate QA criteria, and they ensure that documents needed to verify the quality of these items and services are retained in a recoverable form. They design and implement documented QA procedures to cover the calibration and continuing operation of such equipment and the confirmation of the suitability of such supplies. They ensure that defective equipment is not used until it has been repaired, and that records are kept of the repair of equipment.

1.5.4 RESPONSIBILITIES OF INDIVIDUAL PROJECT LEADERS

Project leaders must participate in the preparation of the Project QA Plan, in which are specified the level of quality required for each phase of their research projects, and the QC methods to be used to ensure that the required levels of quality are attained. They must continually evaluate the specified levels of uncertainty and the levels that are actually being attained, and they must maintain retrievable records that furnish evidence that shows whether the quality of data produced by their project conforms to the requirements of the project. Project leaders ensure that the field measurements, sample collection and handling, laboratory analysis, data analysis and reporting of results are performed properly to prevent the loss of the quality of the data. They must be actively involved in monitoring the quality of all aspects of the work that is being done on their projects, and they review and approve documents and changes in documents that describe the QA requirements of work done on these projects. They must seek to obtain corrective actions if these are needed, and must initiate and provide follow-up verification of any such corrective actions.

When equipment, supplies or services are purchased for use on their projects, project leaders must design and implement documented QA procedures to cover the calibration and operation of the equipment and the confirmation of the suitability of the supplies or services. Project leaders are responsible for determining that documents needed to verify the quality of such equipment, supplies or services are retained in a recoverable form. They must also continually monitor the quality of equipment, supplies and services that are actually in use on their projects. When contractors are hired to perform some aspects

of a research project, the project leader must see that the contract documents include specifications of the quality of the work that must be done by the contractor, and must

design and implement a documented QA program to cover the contractor's performance under the contract.

Project leaders have the overall responsibility for the quality of the final products from their projects, and especially for the quality of the data and interpretations published under their projects. They ensure that measurement data that they have received are validated and that inaccurate information is corrected before the research results are submitted for publication. They are responsible for the proper care and protection of measurement data that they have received. Project leaders normally prepare the progress reports and the final publications of project accomplishments and they must verify the accuracy of the published data, and must include a QA section within these reports. The project leader is responsible for the timely submission of QA reports if these are required for the project. The frequency and content of these reports are to be specified by the appropriate Division Director. Normally such reports should list the data from QC measurements, and should include a discussion of the adequacy of the QC results in light of the project requirements. This discussion should include a review of corrective measures taken and of the results obtained for any problems discussed in the previous report, and it should describe any corrective measures that are being taken for current problems. The project leader must see that retrievable records that furnish evidence of the quality of the work done on the project are specified, prepared and maintained, and must be sure that any and all documentation required for possible QA audits is available.

1.5.5 RESPONSIBILITIES OF DIVISION DIRECTORS AND PROGRAM COORDINATORS

Division Directors and Program Coordinators provide ongoing supervision of QA for work within their divisions and programs. They develop specific QA objectives, involving documented procedures, for the laboratory worker and field worker, communicate these objectives to the workers, and then ensure that these documented operating procedures and protocols are followed, and that the desired quality of work is maintained. They periodically review the QA procedures that are in use to ensure their

adequacy for determining the quality of the results that will be obtained from current and future operations. Division Directors and Program Coordinators must approve any changes in documents that detail procedures that are used within their divisions. They also review the procedures used to select items and services procured for their divisions and programs, and they ensure that QA requirements for these items and services are being met and are properly documented.

The Division Directors and Program Coordinators have the ultimate responsibility for the quality of the products delivered by each of the projects within their divisions and programs, and must be actively involved in ensuring that the required quality of work is achieved in all aspects of these projects. They must be active in the review, criticism, and correction of the work of persons who are working on their projects. Twice a year, Division Directors and Program Coordinators supply the EML QA Officer with brief written evaluations of each project for which they are responsible. Division Directors and Program Coordinators must ensure that any QA reports that they require for a project are submitted on time and are complete. They oversee the procedures used in the peer review of reports originating in their division, and they ensure that these procedures are rigorous enough to detect and eliminate faulty data and interpretations. They ensure that all research reports undergo peer review within EML before publication in EML reports or submission to scientific journals. They encourage project leaders to submit reports of their research to peer-reviewed scientific journals whenever this is feasible. Division Directors and Program Coordinators perform QA reviews of projects under their supervision semiannually, and ensure that any needed corrective actions are taken.

1.5.6 RESPONSIBILITIES OF THE QA OFFICER

The EML QA Officer keeps informed about and investigates any problems that arise in the quality of the work that is performed at the Laboratory, and keeps the Laboratory Director informed. Twice a year the QA Officer receives brief evaluations from the Division Directors and Program Coordinators of the quality of work that is being performed on the projects that are under their supervision. The QA Officer selects a few of these projects for more detailed investigation, choosing some at random, others based upon their importance to the Laboratory, and others because of their quality history. The QA Officer maintains written records of the semiannual evaluations submitted by the

Division Directors and Program Coordinators and of any subsequent investigations, as well as of any other QA appraisals or investigations performed at the Laboratory. The QA Officer also ensures that the EML Institutional Quality Assurance Program Plan is kept current.

1.6 FIELD MEASUREMENTS AND SAMPLING

1.6.1 INTRODUCTION

Some measurements of environmental parameters, such as the intensity and composition of environmental radiation, are performed in the field, while others, such as the concentration of specific radionuclides in samples of soil or vegetation, are performed in the laboratory on samples that were collected in the field. In either situation the procedures used in the field work must be performed correctly if the measurements are to produce valid results. There are many potential pitfalls involved in this initial step, and precautions must be taken to avoid them to ensure the validity of the final data. Before sampling is begun, all project personnel should be informed of the procedures to be used, so that it is certain that the material and the amount of material to be sampled or the property to be measured are appropriate at all phases of the project.

1.6.2 TECHNIQUES

A field measurement or a collected sample must be representative of the parameter or material that is to be analyzed. However, obtaining a representative measurement or sample of environmental parameters or materials is often not straightforward. Most environmental parameters and materials vary with location and time. For example, cosmic-ray intensities vary with time, and various organs of plants and animals differ in their contents of organic and inorganic constituents. Soil typically contains particles of various sizes, and the chemical composition and surface reactivity of these particles often vary as a function of particle size. Water bodies, such as lakes and the ocean, are commonly stratified, with variations in physical and chemical compositions from one layer to another, and usually have a surface film in which many reactive chemical constituents are strongly concentrated. The atmosphere also is stratified, and air parcels

differ from place to place in their concentrations of trace gases and suspended particles. Thus, even at a single site, the composition of air will vary with time, and especially as the wind shifts. The intensity of radiation from radionuclides within the soil varies from location to location. When possible, statistical tests should be applied to determine the number of observations or samples that are needed to achieve the required level of uncertainty.

Before measurements of environmental radiation are begun, the appropriateness of the measurement location must be ascertained. If radiation from the soil is to be measured, the representativeness of the soil at the measurement location must be checked. It must be verified that there are no objects in the vicinity of the radiation detector that can affect the readings that the detector gives.

If samples are to be collected, before sampling is begun the researcher must define clearly the material that is to be sampled to be certain that the sample will be representative of that material. If vegetation is to be sampled, will the sample include the roots of the plant, and if so, must all soil that is clinging to the roots be meticulously removed? If a sample of the atmospheric aerosol that is representative of air over a large region is to be collected, does wind direction matter, at what height above ground is the sample to be taken, and is there danger that nearby structures or activities will affect the composition of the sample? The sample should be representative of the complete material, but it must not be contaminated by extraneous materials. The sampling process itself can affect the validity of the sample. Thus, a rain gauge may distort the flow of winds in its vicinity and cause less rain to fall into its opening than would fall onto the same area of open soil. A water sampler lowered into a lake may carry water from a near surface layer into a lower layer that is being sampled. Therefore, the details of sampling and of sample handling must be considered carefully before sampling is performed in order that the entire research effort not be jeopardized by the careless omission of some needed safeguard.

Because the intensity of environmental radiation and the composition and physical characteristics of environmental materials often vary with location over short distances, and may vary significantly with time, it is usually advisable to do replicate measurements or sampling. The analysis of the data from such measurements or samples can provide a measure of the random error of the entire operation. When a material that is being sampled is clearly heterogeneous, as are many soils and biological materials, replicate

samples should be taken. When the intensity of radiation or the composition of the material is expected to change significantly with time, it is usually advisable to take a series of measurements or samples over the period of interest. This replicate or serial measurement or sampling can yield a more useful picture of the characteristics of interest than would data for a single measurement or sample. The measurement or sampling interval and frequency are often dictated by the research objectives of the project. If portions of samples are to be retained in storage, this should be considered in planning how much material is to be collected, and the treatment it is to receive.

1.6.3 PREPARATION AND STORAGE OF SAMPLES

Many types of measurements of environmental materials, such as vegetation, water, and atmospheric particles, require that a sample of the material be collected and returned to the laboratory for analysis. These samples may require some form of pretreatment before they can be analyzed. For example, vegetation samples may be dried or ashed, and water samples may be filtered. Where possible, groups of samples that are expected to contain high concentrations of an analyte are processed independently from those with low concentrations to minimize the possibility of cross contamination. Pretreatment normally alters the physical state of the sample, and also sometimes alters its chemical state. In planning the pretreatment, the researcher considers the nature of the measurement that is to be made and the state that the sample must be in to undergo that measurement. The possibility should also be considered that the results obtained from the sample might lead to a desire to reanalyze it for the same property, or to analyze it for some other property. Hence, it will often be desirable to collect more sample than will be processed, and to process more sample than will be used in the analysis (if the analytical process is such as to consume the sample).

Another consideration in planning and in carrying out the pretreatment of samples is the reference state of the samples that will be used in reporting the results of the measurements. For example, samples of vegetation, of soil or of sediment are usually weighed before being analyzed, and the results are reported with reference to unit weight and/or the unit area from which the samples were collected. Normally these types of samples are weighed directly after collection (wet weight), dried and weighed again (dry weight) before they are analyzed. Under this protocol, the researcher must ensure that all samples are dried to the same extent to report results per unit dry weight. But this

restriction is lifted if results are reported per unit weight or per unit area of sample collection.

Decisions concerning the pretreatment process to be used can be complicated if the process is such that it may alter the property of the sample that is to be measured. For example, if a volatile component, such as iodine, is to be measured in vegetation, the pretreatment of the vegetation sample must not be such as to volatilize that component.

Project leaders are responsible for their own samples until the project is complete. Upon completion of a project, any portions of samples that are to be saved should be entered into and stored in an archival system.

1.6.4 CODING AND RECORD KEEPING

At the time of sample collection or of field measurement, samples and resulting data sets are normally given code numbers that serve to identify them during subsequent analytical, calculation and data reporting stages. Sometimes a coding system separate from that used for field measurements or sample collection is used for the analysis stage, either for the convenience of the analysts or to aid in ensuring the "blindness" of the QC samples or measurements. A number of factors enter into the designing of these codes. There is always an advantage in keeping the codes as simple as possible to minimize the probability that errors will be made in transferring data. However, the code should be distinctive enough to distinguish each set of samples and data from other, unrelated samples and data that pass through the laboratory. Care must be taken to mark samples and field data clearly to minimize the possibility of misreading of labels and notes. Especially during the sampling or field measurement phase, it is often desirable to use codes that contain information on the site and/or time of sampling or measurement, or upon the nature of the sample or measurement.

In designing protocols for recording field measurements or sampling and subsequent analytical data, the researcher must consider whether the recorded data will be sufficiently complete for the possible future uses that may be made of the final analytical results. Normally, much more detailed information is available during the field measurement or sampling and analytical phases than is expected to be of importance for the interpretation

of the results, and only the information that appears to be immediately useful is recorded. At times, as the final data have been studied, it has become evident that some of the unrecorded information might have been useful in interpreting those results. To avoid the possible loss of useful information, the researcher should make a habit of keeping detailed field and laboratory notes, either in a bound notebook or another recoverable medium. Researchers should think through as many possibilities as they can based on their experience before designing the systems and codes of data recording.

1.7 CHEMICAL AND RADIOCHEMICAL ANALYSES

1.7.1 INTRODUCTION

The EML QA program for chemical and radiochemical analyses has long been well-defined and carefully followed. QC measures are an integral part of each analytical procedure, and quantitative estimates of analytical uncertainties are made and reported routinely.

1.7.2 APPROVED PROCEDURES

There are often several chemical or radiochemical techniques that may be used to accomplish a particular analysis. In most instances, past experience at EML or amongst the scientific community generally has shown that one of these techniques is to be preferred to the others, at least for a particular type of sample and with the concentration of the constituent of interest falling within a specific range. In these instances, the particular analytical procedure to be used is specified in Section 4 of this Manual. The details of these procedures are documented in those sections, and they are updated periodically. At times it may be necessary for the analyst to modify a procedure when working with a particular set of samples. Such modifications must be noted so that the exact procedures that were used can be identified if questions arise at a later time. In order to have a written record of all pertinent information relating to the analysis of all samples, analysts should record their daily progress or results in a laboratory notebook or other recoverable medium.

1.7.3 QC SAMPLES

Whenever possible, the project leader, as part of the external QC program, should submit QC samples to the analyst along with routine samples in such a way that the analyst does not know which of the samples are the QC samples. These external QC samples, which usually include duplicate and blank samples, should test sample collection and preparation as well as sample analysis whenever this is possible. In addition, analysts are expected to run internal QC samples that will indicate to them whether the analytical procedures are in control. Both the external and internal QC samples should be prepared in such a way as to duplicate the chemical matrix of the routine samples, insofar as this is practical. The QC samples that are routinely used consist of five basic types: blank samples, replicate samples, reference materials, control samples and "spiked" samples. Special definitions of these terms related to QA are defined in the Appendix.

Blank samples are analyzed to give a measure of any contamination of the sample that is occurring during the course of the collection, preparation or analysis. The analyst commonly introduces blank samples into the sample stream. Often these are "reagent blanks" that are prepared by starting with deionized water or with an empty sample container and going through all of the normal procedures involved in the analysis; i.e., adding all of the reagents at the proper points. Whenever possible, the matrix of blank samples should be the same as that of the samples being analyzed. The data for the routine samples are usually corrected by subtracting from their measured values the value of the blank. It must be remembered, however, that blank measurements of only the analytical processes cannot be used to evaluate contamination that occurs during the collection and preparation of the sample.

Replicate samples are obtained sometimes by repeating the collection as well as the analysis of samples, but often only replicate aliquots of the same laboratory sample are analyzed. Repeated sampling of a heterogeneous solid environmental material, such as soil, may not yield truly replicate samples. If such materials are taken into solution before being measured, replicate subsamples of the solution are often analyzed. Commonly, individual samples are measured more than once, and for nondestructive techniques, such

as gamma-ray spectroscopy on whole samples, replicate measurements may involve exactly the same sample, renumbered and resubmitted for analysis.

The third type of QC sample, the reference material or standard reference material, is used primarily to calibrate the measurement method or apparatus.

The concentration of the control sample, or check standard, has an overall analytical uncertainty that is known well enough for this control sample to be used in place of a standard reference material to insure that the measurement method is in control. Such control samples should have the same matrix and the same range of concentrations as do the routine samples when this is possible.

Spiked samples, the fifth type of QC sample, are prepared by adding a known amount of the constituent of interest to blank samples or to samples that have already been analyzed, to provide samples with known concentrations. They may be used to estimate chemical yields of analytical processes. Where the presence of other constituents in a sample may be affecting the response of an instrument to the constituent of interest, multiple spiking may be done. In this process, a series of samples are spiked by the addition of increasing amounts of the constituent of interest. The measured values are inspected to determine whether the instrument yielded a linear response to the increasing concentrations, and whether the rate of increase of the response was that expected from the differences in concentration between samples.

1.8 INSTRUMENTAL ANALYSES

1.8.1 INTRODUCTION

Virtually all measurements performed at EML require the use of an electronic instrument to provide quantitative data. This is true of measurements of radiation, of radioactive materials and of non-nuclear measurements. Thus, it is of crucial importance to the quality of the work done at EML that all analysts be thoroughly familiar with the proper procedures to be followed in calibrating, operating and caring for the instruments

that they must use. Usually this requires that the analyst understand at least the general scientific principles that are the basis of the measurement.

1.8.2 INSTRUMENTS

To maximize the quality of the research data produced at EML, every effort is made to keep abreast of the state of the art in instrumentation used in environmental research and, where it is financially possible, to obtain the best available instruments. To keep instruments performing efficiently, a schedule of preventive maintenance is followed where appropriate. A record of instrument performance is maintained, and any modifications made in the instruments, whether permanently or for a particular project, are documented. Any such modifications in any instrument must conform to the safety standards and practices that are specified in the EML Safety Manual.

1.8.3 CALIBRATIONS

Provisions have been made for the periodic, quantitative assessment of the performance of most instruments used by EML. For many instruments, calibration standards are available. These standards are measured to obtain a curve that relates the intensity of the signal from the instrument to the concentration of the substance or the intensity of the property being measured. In other instances, this calibration consists of a one point check using a single standard reference instrument, source, material or sample. For the quality of the measurements to be optimized, the analyst must use the appropriate standards, calibration procedures and frequency of calibration, and must keep a record of the traceability of the standardization.

1.8.4 BACKGROUND EVALUATIONS

Commonly, instruments will provide a background signal that may be the result of minor fluctuations in an electrical field. Also, phenomena or materials that produce effects that are similar to the effects produced by the parameter or substance of interest

may occur in the environment of the detector and may produce a signal from the detector. It is then necessary to measure this background signal so that the data from routine measurements may be corrected for its presence. Even when the signal from the measured parameter or samples is high relative to the background and the background correction is small, the background signal should be measured regularly to be sure that it has not changed and that the instrument has not become defective or contaminated. The analyst must be sure that the procedures that are in use to measure background are adequate in nature and are performed with the needed frequency. A record must be kept of the measured backgrounds and this record should be analyzed statistically so that data may be properly corrected, and also so that variations resulting from instrument problems or from contamination can be detected and eliminated.

1.8.5 CHECKS OF THE STABILITY OF THE INSTRUMENT

The analyst must always be on guard against any instability of the instruments that are being used to produce analytical data. All electronic components are subject to variations because of changes in environmental factors, such as temperature and humidity, and to degradation over time, and even new equipment may contain weak components. The records of instrument calibrations and of background measurements, including control charts, are the main data base used to judge the stability of an instrument. The occasional remeasurement of a set of samples that showed a range of instrumental response may be useful in providing another check on the performance of an instrument.

1.9 DATA REDUCTION, STORAGE, AND REPORTING

1.9.1 INTRODUCTION

The quality of the data reported by EML depends not only upon the care with which sampling and analysis are performed, but also upon the care with which calculations of the resulting data are performed, and upon the manner in which the data are presented in reports. A key aspect of a QA program is maintaining records that document every step of the process that leads to the data that ultimately are reported.

1.9.2 FIELD AND LABORATORY RECORDS

A measurement is useful if it is representative of the environmental material or parameter that is under study. The field notes taken during measurement or sampling normally provide a basis for judging the representativeness of a sample or a field measurement. Similarly, the laboratory notes made during the analysis of a sample serve as a basis for judging the quality of the analysis, indicating whether any problems arose during the analytical procedures that might have adversely affected their outcome. For this reason, every effort should be made during field measurement or sampling and during sample preparation and analysis to record all aspects of the procedure that might reasonably be expected to affect the outcome of the analysis. As a general rule, every possibly relevant variable that is amenable to quantification should be recorded, even if

only by a check mark on a form. These records may be needed not only by the person who is writing them, and not only for the time period during which they are being written, but in many instances they may be needed by other persons and at some future time. It is important, therefore, that the notes be both legible and clear in meaning, so that others who read them will be able to reconstruct the events that are referred to.

It is often convenient to preserve electronically the data records from those instruments which provide digital electronic output signals. When this is done, great care must be taken to ensure that blocks of data are thoroughly annotated to identify the time and location and all other important circumstances concerning the measurements. Often the best way to do this is to organize blocks of data into files with distinctive file names, and to keep a logbook with the supporting information for each file. Alternatively, if electronic editing capabilities are available, comments can be entered into the data file itself.

1.9.3 DATA REDUCTION AND STORAGE

When a new project is initiated, the project leader and appropriate Division Directors should discuss the policies to be followed concerning the manner and length of time of storage of data that will be produced during that project. There are types of data, such as those related to possible legal actions involving the U.S. Government, that should be stored indefinitely. When data are stored electronically, backup files must be prepared to eliminate the possibility that the data may inadvertently be lost. When it begins to become evident that a medium on which data are stored, such as a particular type of magnetic disk or tape, is in danger of becoming obsolete, and there is thus a danger that the data will become irretrievable, the data should be copied to a better medium.

Whenever data are to be transferred or stored, an appropriate control procedure should be established to minimize the danger that human error will compromise the quality of the data. Data reduction is normally accomplished electronically using computer programs, both to avoid the drudgery of repeated "hand" calculations and to avoid calculation errors. For small numbers of unusual measurements or samples, however, hand calculations may be more efficient than writing a computer program of limited

applicability. When unfamiliar calculations are performed, whether by hand or by using computer programs, care must be taken to be sure that the logic built into the calculation is correct. Calculations are often checked by performing them on data that lead to a known result.

1.9.4 DATA REPORTING

When data are reported, an estimate of their uncertainty must be given. Contained below are the guidelines and definitions of the terms that are used at EML for reporting the errors and uncertainties of data. The meaning of reported uncertainties must be indicated either by stating exactly what they represent or by describing how they were calculated, because a simple $X \pm Y$ statement may be interpreted in any one of a number of ways. The statement of uncertainty should include estimates of all significant sources of error involved, whether these result from the field measurement or sampling phase, the analysis phase or the data reduction phase, if they will affect the final result within the number of significant figures reported.

When data are reported, the reporting format must be commensurate with their expected use. Tables of data allow the full presentation of values and of their estimated uncertainties. Graphical presentation typically allows better visualization of the data. With both tabular and graphical presentations, it is important to assume that nothing will be immediately obvious to the reader, and the column headings or legends must include all information that is necessary in order to understand the data that are presented.

When data are presented, it is important to report only the appropriate number of significant figures. Usually the data should be carried to additional figures during preliminary calculations, and then the final result should be rounded off to the proper number of significant figures. When the tables are printed by a computer, the format that is used may result in too many decimal places being reported for some samples. If this happens, the table should be edited to limit all data to the appropriate number of figures. One approach to determining the number of significant figures to be reported (Sanderson et al., 1980) first determines the number of significant figures in the uncertainty, and rounds the reported value to the same decimal place as the uncertainty. In this approach, it is assumed that the rounded-off standard deviation reported should not differ from the

calculated value of the standard deviation by more than 20%. For example, if the calculated standard deviation is 0.1635, it should be reported as 0.16, which differs from the original by only 2%. Rounding off to 0.2 should be avoided because it differs from the original by over 22%. The value of the measurement is then rounded such that its last significant figure will be in the same decimal place as that of the error.

When data are presented in memoranda or internal EML reports, it is usually both possible and desirable to include a complete discussion of the QA data that are pertinent to the measurements. The information to be presented may include, in addition to the uncertainties, various statistical tests and indications of analytical sensitivity, such as the lower limit of detection (LLD), the instrumental detection limit (IDL), the method detection limit (MDL), the limit of detection (LOD) (Currie, 1988), the specificity (identification), and purity (absence of contaminant). The specificity and purity may be estimated from the resolution of a signal or from the goodness of fit to known quantities, such as energy, wavelength or rate of radioactive decay. Reporting the results for QC samples, replicates and blank samples also provides important information about the quality of data. If a discussion of QA cannot be included in a journal or symposium publication, normally a separate, supporting QA report should be prepared.

Terms such as "below detection limits" should not be reported in place of the actual analytical results obtained. Reporting results as "less than" some minimum detectable level also results in some loss of statistical information, and may lead to erroneous interpretations. When low activities or concentrations are measured, the actual results obtained, including any negative values, normally should be reported along with the associated overall uncertainties and the measures of analytical sensitivity that are mentioned above. An interpretation of negative values can be included in the text. Waite et al. (1980) and Gilbert and Kinnison (1981) have discussed techniques that may be followed in averaging data sets that contain "less than" values.

A number of terms have been used by the scientific community to describe data quality, but these terms are often given different meanings by different individuals. The definitions that follow are the preferred usage for the purposes of this document and have been taken from Croarkin (1985), Taylor (1985, 1987), and Taylor and Oppermann (1986).

Error - The difference between the true value and the measured value of a quantity or parameter.

Uncertainty - The range of values within which the true value is estimated to lie. It is a best estimate of possible inaccuracy due to both random and systematic errors.

Random Errors - Errors that vary in a nonreproducible way around the limitingmean. These errors can be treated statistically by use of thelaws of probability.

Systematic Errors - Errors that are reproducible and tend to bias a result in one direction. Their causes can be assigned, at least in principle, and they can have both constant and variable components. Generally, these errors cannot be treated statistically.

A statement of uncertainty assigns credible limits to the reported value, stating to what extent that value may differ from its true value. The uncertainty of a measured value can be defined by a statistically determined confidence interval for the random error and by an estimate of the bounds for systematic error; they should be stated separately. When appropriate, they may also be combined into a single range to describe the overall uncertainty. Since there are a variety of methods suggested in the literature for combining random and systematic errors, the particular method used must be explicitly stated. One accepted practice (Croarkin, 1985) is to combine in quadrature, systematic errors that are known to be independent, to add linearly systematic errors that may not be independent, and to combine systematic and random errors linearly. The confidence level chosen must be stated whenever a confidence interval is reported.

Systematic errors which can be determined by calculation or by experiment should be eliminated by an appropriate correction. Estimating the magnitude of some systematic errors may require scientific judgement on the part of the experimenter. All significant sources of error should be identified and reported.

1.10 MATERIALS AND PROCEDURES

1.10.1 INTRODUCTION

Certain materials and procedures must be used during routine analyses for the successful performance of the EML QA program. These include standard instruments, reference materials, and standard reference materials that are used to calibrate instruments or to test the quality of measurements. They also include the QC procedures that are used during the performance of routine analyses and data validation procedures that are used to check the reasonableness of data. In addition, whenever it is possible, EML takes part in intralaboratory comparisons, using more than one operator, instrument or technique, and in interlaboratory comparisons with other government or private laboratories that are making comparable measurements.

1.10.2 STANDARDS AND REFERENCE MATERIALS LIBRARY

EML divisions may maintain a library of standard and reference materials that are needed for calibrating the analytical instruments in that division and for use in the internal QC program in that division. Where possible, any sample matrix that is routinely analyzed should be represented among these reference materials. The division staff maintains records to document the history of acquisition and use of those materials. The Division Director, or a designated subordinate, is responsible for keeping this library complete and current.

1.10.3 DATA VALIDATION

For many analytical processes, control charts or other QC records are continually updated to enable the operator to determine whether these processes are under control. The operator enters data for blank samples, background measurements and standard or control samples into the records, and onto the appropriate charts. The records and charts are reviewed and evaluated to determine whether the process is in control and the data are acceptable, or if corrective actions must be taken. Chemical yields, rates of

radioactive decay, energy calibrations, and other operating characteristics must be evaluated routinely and unusual results must be flagged for immediate investigation.

1.10.4 INTRALABORATORY COMPARISONS

Whenever it is possible, attempts are made to verify that no systematic error is introduced into the results of analytical procedures because of differences in techniques among analysts, or because of differences in performance between supposedly equivalent instruments. Analyses of selected samples are repeated using different analysts or different instruments, and the results are compared to insure that variations are within the expected range. In addition, where it is possible, measurements are made or samples are analyzed using more than one technique, to be sure that the technique that is routinely in use gives results that are consistent with results from other approved techniques.

1.10.5 INTERLABORATORY COMPARISONS

It is a policy at EML to take part in as many interlaboratory comparisons of measurement and analytical techniques as is practical. The EML staff has taken the initiative in organizing many such intercomparisons, as for example for thermoluminescent detectors (TLDs) and radon detectors, and EML has participated in many intercomparisons sponsored by other government or private laboratories or organizations, as in the chemical analysis of precipitation. In addition, EML has provided reference measurements for Department of Energy contractors in the analysis of radionuclides in environmental samples.

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1.11 APPENDIX

DEFINITIONS OF QA TERMS USED IN THE CHEMICAL LABORATORY

Some terms concerning standards and calibration are defined by Taylor (1985):

A standard is "a substance or material, the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property of another. In chemical measurements, it often describes a solution or substance, commonly prepared by the analyst, to establish a calibration curve or the analytical response function of an instrument."

A primary standard is "a substance or artifact, the value of which can be accepted (within specific limits) without question when used to establish the value of the same or related property of another material."

A check standard is "in physical calibration, an artifact measured periodically, the results of which typically are plotted on a control chart to evaluate the measurement process."

Standardization, in analytical chemistry, is "the assignment of a compositional value to one standard on the basis of another standard."

A standard reference material is "a reference material distributed and certified by the National Institute of Standards and Technology."

A certified reference material (CRM) is "a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body."

A certified value is "the value that appears in a certificate as the best estimate of the value for a property of a reference material."

A reference material (RM) is "a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for the assignment of values to materials."

Traceability is "the ability to trace the source of uncertainty of a measurement or a measured value."

Calibration is the "comparison of a measurement standard or instrument with another standard or instrument to report or eliminate by adjustment any variation (deviation) in the accuracy of the item being compared."

A calibrant is "a substance used to calibrate or to establish the analytical response of a measurement system."

Intercalibration is "the process, procedures, and activities used to ensure that the several laboratories engaged in a monitoring program can produce compatible data. When compatible data outputs are achieved and this situation is maintained, the laboratories can be said to be intercalibrated."

A standard method is "a method (or procedure) of test developed by a standards-writing organization, based on consensus opinion or other criteria, and often evaluated for its reliability by a collaborative testing procedure."

A reference method is "a method which has been specified as capable, by virtue of recognized accuracy, of providing primary reference data."

Two useful terms defined by the International Institute of Technology (IOLM, 1976) are:

Repeatability of measurements - "The closeness of the agreement between the results of successive measurements of the same quantity carried out by the same method, by the same observer, with the same measuring instruments, in the same laboratory, at quite short intervals of time."

Reproducibility of measurements - "The closeness of the agreement between the results of measurements of the same quantity, where the individual measurements are made:

- by different methods, with different measuring instruments,
- by different observers, in different laboratories,
- after intervals of time that are quite long compared with the duration of a single measurement, and
- under different normal conditions of use of the instruments employed."

"The term reproducibility is also used when some of the factors listed above are different in the individual measurements; these factors should be specified in detail in each particular case."

Some additional terms that are of use in discussing QA are defined by Taylor (1985):

A technique is "a physical or chemical principle utilized separately or in combination with other techniques to determine the composition (analysis) of materials."

A method is "an assemblage of measurement techniques and the order in which they are used."

A procedure is "a set of systematic instructions for using a method of measurement or of sampling or of the steps or operations associated with such."

A protocol is "a procedure specified to be used when performing a measurement or related operation, as a condition to obtain results that could be acceptable to the specifier."

An analyte is "the specific component measured in a chemical analysis; also called analate."

A sample is "a portion of a population or lot. It may consist of an individual or groups of individuals. It may refer to objects, materials, or to measurements, conceivable as part of a larger group that could have been considered."

An increment is "an individual portion of material collected by a single operation of a sampling device, from parts of a lot separated in time or space. Increments may be either tested individually or combined (composited) and tested as a unit."

A composite sample is "a sample composed of two or more increments selected to represent a population of interest."

A blind sample is "a sample submitted for analysis whose composition is known to the submitter but unknown to the analyst. A blind sample thus is one way to test proficiency of a measurement process."[1]

Double blind refers to "a sample, known by the submitter but submitted to an analyst in such a way that neither its composition nor its identification as a check sample are known to the latter."*

A blank is "the measured value obtained when a specified component of a sample is not present during the measurement. In such a case, the measured value/signal for the component is believed to be due to artifacts, hence should be deducted from a measured value to give a net value due to the component contained in a sample. The blank measurement must be made so that the correction process is valid."[2]

A split sample is "a replicate portion or subsample of a total sample obtained in such a manner that it is not believed to differ significantly from other portions of the same sample."

A duplicate sample is "a second sample randomly selected from a population of interest (see also split sample) to assist in the evaluation of sample variance."

A replicate is "a counterpart of another, usually referring to an analytical sample or a measurement. It is the general case for which duplicate is the special case consisting of two samples or measurements."

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[1] *These definitions of "blind sample" and "double blind" are applicable to certain uses in quality assurance, but these terms are also commonly used with other, somewhat different meanings.*

[2] *This definition of "blank" is applicable to certain uses in QA, but this term is also commonly used with other, somewhat different meanings.*

A duplicate measurement is "a second measurement made on the same (or identical) sample of material to assist in the evaluation of measurement variance."

A control sample is "a material of known composition that is analyzed concurrently with test samples to evaluate a measurement process (see also Check Standard)."

The limiting mean is "the value approached by the average as the number of measurements, made by a stable measurement process, increases indefinitely."

An outlier is "a value which appears to deviate markedly from that for other members of the sample in which it occurs."

The coefficient of variation is "the standard deviation divided by the value of the parameter measured."

The relative standard deviation is "the coefficient of variation, expressed as a percentage."

Validation is "the process by which a sample, measurement method, or a piece of data is deemed to be useful for a specific purpose."

A control chart is "a graphical plot of test results with respect to time or sequence of measurement together with limits within which they are expected to lie when the system is in a state of statistical control."

The control limits are "the limits shown on a control chart beyond which it is highly improbable that a point could lie while the system remains in a state of statistical control."

The warning limits are "the limits shown on a control chart within which most of the test results are expected to lie (within a 95% probability) while the system is in a state of statistical control."

The terms error, uncertainty, random errors and systematic errors, all of which concern data quality, are defined in Section 1.9.4, Data Reporting.

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