American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories

Sponsor

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The Institute of Electrical and Electronics Engineers, Inc.

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Abstract: A framework that can be used to create a national or an organizational NIST-traceable measurement quality assurance (MQA) program that will optimize the quality of radioassays performed by service laboratories is presented. This standard serves as a guide for MQA programs developed for specialized sectors of the radioassay laboratory community, i.e., bioasssay, routine environmental monitoring, environmental restoration and waste management, radiopharmaceuticals nuclear power radiochemistry, and other areas involved in radioassays.
Keywords: Laboratory, measurement quality assurance, quality, quality assurance, quality control, radiation instrumentation, radioactivity, radioassay laboratory, radioassays, radiometrolog

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Introduction

(This introduction is not a part of ANSI N42.23-1996, American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories.)

This standard is the responsibility of the Accredited Standards Committee on Radiation Instrumentation, N42. Committee N42 delegated development of this standard to Subcommittee N42.RM. Drafts were reviewed by Committee N42, Subcommittee N42.RM, and other interested parties, and the comments received were utilized in producing the standard as finally approved. The standard was approved by N42 letter ballot of 2 March 1995, closed 15 December 1995.

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Contents

CLAUSE PAGE

1. Overview ... 1

1.1 Introduction ... 1
1.2 Purpose ... 1
1.3 Scope ... 2

2. References ... 3

3. Glossary ... 3

3.1 Special word usage ... 4
3.2 Specific terms ... 4

4. National performance testing program ... 9

4.1 Organizational responsibilities ... 9
4.2 Accreditation ... 15
4.3 Test radionuclides and activity ranges ... 19

5. Quality assurance ... 20

5.1 QA program ... 21
5.2 Elements of a QA program ... 21
5.3 Sample process quality control ... 25

6. Bibliography ... 26

Annex A (normative) Operational requirements of a radioassay laboratory ... 27

ANNEX

A.1 Service laboratory operational criteria ... 27

A.2 Facility criteria ... 27

A.3 Waste management plan ... 29

A.4 Staff qualifications ... 29

A.5 Nuclear instrumentation ... 30

A.6 Laboratory performance criteria ... 32

A.7 Detection limit concepts ... 33

A.8 Reporting results by the service laboratory ... 38

A.9 Record retention by the service laboratory ... 39

A.10 Test selection ... 40

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ANNEX PAGE

A.11 Bibliography for Annex A 40

Annex B (normative) Examples of reference and monitoring laboratories, and existing measurement assurance programs 41

B.1 EPA laboratory intercomparison studies program 41

B.2 DOE QA programs 41

B.3 World Health Organization (WHO) International Reference Center for Radioactivity 45

B.4 IAEA Analytical Quality Control Service (AQCS) 45

B.5 Nuclear power utilities 46

B.6 Bibliography for Annex B 48

Annex C (informative) Guidance on QA investigations and corrective actions related to radioassay work 53

Annex D (informative) Data quality objectives applicable to radioassays 56

D.1 Project goals development process 56

D.2 Analytical process and data quality plan 56

D.3 QA program design 57

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American National Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories

1. Overview

1.1 Introduction

This standard is the basis for the creation of measurement quality assurance (MQA) programs that will optimize the quality of radioassays performed by service laboratories in the United States. Where quality performance testing criteria have been established for a specialized sector of the radioassay laboratory community, this document shall serve as an umbrella standard that provides the guidance relative to the organizational relationships and responsibilities of the accrediting organization and the reference, monitoring, and service laboratories.

A well-defined and implemented QA program provides the structures, policies, and responsibilities for the execution of quality control and assessments of operations to ensure success in meeting the data quality objectives of all projects in which radioassay is an integral part. A national program for testing, accrediting, and monitoring of all types of radioassay laboratories is outlined in this standard as a mechanism for achieving such a goal at the national level.

1.2 Purpose

The purpose of this standard is to provide

a) Guidance for a national program for testing, accrediting, and monitoring of all types of radioassay laboratories
b) Guidance for the minimum requirements necessary to maintain a viable service laboratory for the measurement of low and intermediate levels of radioactivity in all types of test media
c) Criteria for the establishment of monitoring and reference laboratories
d) Operational and QA criteria for service, monitoring, and reference radioassay laboratories
e) Guidance for the National Institute of Standards and Technology (NIST) and an accrediting organization to implement this standard

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This standard does not address quality assurance for the collection of samples requiring radioassays. It does not address directly the quality assurance necessary for radon-in-air measurements, although the principles provided in this standard should form the basis of QA programs for those laboratories that perform radioactivity measurements as part of their radon analyses.

1.3 Scope

This standard serves as a guide for MQA programs developed for specialized sectors of the radioassay community, that is: bioassay, routine environmental monitoring, environmental restoration and waste management, radiopharmaceuticals, and nuclear power chemistry. The standard also provides the means for establishing the more limited objectives that govern day-to-day operations of an individual laboratory, including mandated protocols, recommendations, and information to implement and maintain a radioassay QA program.

The overall utility of radioassay data results from the combination of defensible and traceable measurements, preceded by careful planning to determine data quality objectives (DQO). This standard addresses only those aspects of the user's program that deal with measurement-related issues. Guidance on the use of DQOs is provided in annex D.

This standard addresses

a) The operational framework of a national measurement assurance program
b) The roles of the accrediting organization; the National Institute of Standards and Technology (NIST); and the reference, monitoring, and service laboratories
c) The protocol for the preparation and distribution of test media by the monitoring or reference laboratories
d) The bases for calculating the required accuracy and precision parameters of radioassay measurements
e) The protocol for the evaluation and reporting of test results
f) The protocol for the assessment and evaluation of test results and reporting assessment findings by the accrediting organization
g) Guidance for determining the decision limit concentration and the *a priori* minimum detectable concentration for each radionuclide (see annex A)
h) The requirements for reporting radioassay results by service laboratories to the customer
i) Eleven key elements of a viable QA program applicable to the service, monitoring, and reference laboratories
j) The QA and QC programs for the service, monitoring, and reference laboratories

Since this is an umbrella MQA standard, no guidance or requirements have been established for the minimum performance criteria of precision, accuracy, or detection limits applicable to the routine operation of a radioassay laboratory. However, this standard requires that the service laboratory and its client (either from within the same company or governmental organization, or external) establish precision, bias, detection limit, and other quality performance specifications for each radionuclide within a preprocessing agreement or contract. These quality performance specifications shall be related to the data quality objectives requiring the radioassays. For radioassays requiring a quality measurement assurance program, this standard requires the use of a third-party monitoring laboratory to evaluate the ongoing capability of the service laboratory to meet pre-established, contractual performance specifications. If agreed by the client and service laboratory, the internal QA program of the service laboratory may function as a monitoring laboratory as long as the requirements for the monitoring laboratory as established in this standard are met.

This standard has outlined the minimum requirements necessary to maintain a viable measurement assurance program by a radioassay laboratory. As outlined in this standard, the testing of the reference, monitoring, and service laboratories to assay radionuclides specified in the various testing categories properly is for

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accreditation purposes only. This testing program has been developed to ensure that all radioassay laboratories have, at a minimum, radiation measurement instrumentation, representing each radioactive emission type, that has been calibrated with radioactive materials traceable to NIST. This testing is considered a minimum effort in the accreditation process. As stated previously, it is the responsibility of a person or organization requiring radioassays to incorporate detailed performance specifications for each radionuclide and matrix within a contract for the service laboratory. A monitoring laboratory shall be utilized to evaluate the compliance by the service laboratory with the performance specifications.

Several annexes have been developed to provide additional background information, clarifications, and, in some cases, requirements of the standard. Annex A is considered part of this standard and, as such, the requirements specified shall be followed. Annexes B, C, and D provide information and guidance to support the general concepts and recommendations outlined in this standard.

2. References

This standard shall be used in conjunction with the following publications. When the following standards are superseded by an approved revision, the revision shall apply.

ANSI N13.30-1996, American National Standard for Performance Criteria for Radiobioassay.[1]

ANSI N343-1978 (Reaff 1984), American National Standard Internal Dosimetry for Mixed Fission and Activation Products.

ANSI Z34.1-1993, American National Standard for Certification—Third-Party Certification Programs for Products, Processes, and Services.

ANSI/ASME N45.2.9-1979, Requirements for Collection, Storage and Maintenance of Quality Assurance Records for Nuclear Power Plants.[2]

ANSI/ASME NQA-1-1994, Quality Assurance Program Requirements for Nuclear Facilities.

ASTM E 29-93a, Standard Recommended Practice For Indicating Which Places Of Figures Are To Be Considered Significant In Specified Limiting Value.[3]

IEEE Std 1012-1986 (Reaff 1992), IEEE Standard for Software Verification and Validation Plans.[4]

NBS IR 89-4125, Operational And Technical Requirements For Laboratory Accreditation Program For Personnel Dosimetry Processors, July 1989.[5]

3. Glossary

The following terms are of a restricted nature for the purposes of this standard. Terms defined in the *Glossary of Terms in Nuclear Science and Technology* [B1][6] are not defined in this standard.

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[1]ANSI publications are available from the Sales Department, American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036, USA.

[2]ASME publications are available from the American Society of Mechanical Engineers, 22 Law Drive, Fairfield, NJ 07007, USA.

[3]ASTM publications are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA.

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[5]NBS publications are available from the Superintendent of Documents, US Government Printing Office, P.O. Box 37082, Washington, DC 20013-7082, USA.

[6]The numbers in brackets preceded by the letter B correspond to those of the bibliography in clause 6.

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3.1 Special word usage

The word “shall” is used to denote a requirement; the word “should” is used to denote a recommendation; and the word “may” is used to denote permission, neither a requirement nor a recommendation. To conform to this standard, all radioassays shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification shall be documented for deviations from recommendations.

3.2 Specific terms

3.2.1 accreditation: The systematic and objective determination of the competence of a laboratory to perform its services according to specific test methods/standards, made by a qualified accreditation body, and issuance of a certificate attesting to that competence by the body.

3.2.2 audit: A planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedures, instructions, drawings, and other applicable documents, and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

3.2.3 analyte: The particular radionuclide(s) to be determined in a sample of interest. As a matter of clarity when interpreting various clauses of this standard, a gamma-ray spectral analysis is considered one analyte.

3.2.4 assessment: A planned and documented activity performed to determine whether various elements within a quality management system are effective in achieving stated quality objectives.

3.2.5 accuracy: A concept employed to describe the dispersion of measurements with respect to a known value. A measurement with small systematic uncertainties is said to have high accuracy.

3.2.6 average relative bias (B_r): The average relative bias,B_r, for a test category is calculated from the individual relative biases, B_{ri}, and defined as

$$B_r = \frac{\sum_{i=1}^{N} B_{ri}}{N}$$

where

N is the maximum number of test samples measured by an individual service laboratory in a given test category. The sample size N shall be at least five.

i is the ith sample

3.2.7 background: Ambient signal response, recorded by measuring instruments, that is independent of radioactivity contributed by the radionuclides being measured in the sample.

3.2.8 bias: (a) The deviation of the expected value of a random variable from a corresponding stated (correct or known) value. (b) A fixed deviation from the true value that remains constant over replicated measurements within the statistical precision of the measurement. *Syn*: deterministic error, fixed error, systematic error.

3.2.9 bias statistic: An estimation of bias calculated from a finite sample of data using a specified formula.

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3.2.10 blind matrix spike: A matrix spike sample sent through normal processing wherein the processor knows that the sample is of quality control origin but does not know the nuclide or nuclide concentration. The terminology “single-blind matrix spike” is also commonly used.

3.2.11 blind quality control (QC) sample: Similar to a spike sample except that a blind sample is presented to the laboratory or analyst by whom the sample may be recognized as a QC sample but the quantity and identity of the analytes are unknown. *Syn*: single-blind QC sample.

3.2.12 blind replicate: A replicate sample unknown to the analyst.

3.2.13 blind replicate reference standard: A sample of known concentration prepared from a purchased standard reference material and submitted as a blind sample into the laboratory.

3.2.14 certified reference material: 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 that is used by a certifying body.

3.2.15 concentration: The quantity of radioactive material stated in terms of activity (or mass) per unit of volume or mass of a medium.

3.2.16 condition adverse to quality: An all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, and nonconformances. A significant condition adverse to quality is one that, if uncorrected, could have a serious effect on safety, quality, or operability.

3.2.17 data quality objective (DQO): The qualitative and quantitative statements that specify the quality of data required to support decisions for any process requiring radiochemical analysis (radioassay).

3.2.18 decision level concentration (DLC): Quantity of analyte at or above which an *a priori* decision is made that a positive quantity of the analyte is present. For this standard, the probability of a Type I error (probability of erroneously reporting a detectable nuclide in an appropriate blank or sample) is set at 0.05.

3.2.19 deficiency: A deviation from the accepted procedures, practices, or standards, or a defect in an item that could lead to degradation of quality.

3.2.20 double-blind matrix spike: A matrix spike sample sent through normal processing wherein the processor has no knowledge that the sample is of quality control origin.

3.2.21 double-blind quality control (QC) sample: Similar to a blind sample, except that the identity as well as the composition (quantity and identity of analytes) are unknown to the analyst.

3.2.22 flow down: Passing to a lower level or tier contractual requirements related to administrative, technical, or quality performance.

3.2.23 instrument quality control (QC) chart: A chart developed to evaluate the response of an instrument to predetermined, statistically based limits as determined by an appropriate QC source. The predetermined statistical limits are not typically developed with the overall quality performance (bias and precision) parameters for an analytical technique in mind.

3.2.24 instrument tolerance chart: A chart developed to evaluate the response of an instrument to a predetermined tolerance level as determined by an appropriate quality control source. The predetermined tolerance level, typically expressed as a percent, is set with the overall quality performance (bias and precision) parameters for an analytical technique in mind. For practical reasons, the response of most instruments is held in control to a tolerance not to exceed 3% and 5% as related to the initial instrument calibration.

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3.2.25 matrix spike: An aliquot of a sample that is spiked with a known concentration of the analyte of interest.

3.2.26 minimum detectable amount (MDA): The amount of a radionuclide that, if present in a sample, would be detected with a β probability of nondetection while accepting a probability α of erroneously detecting that radionuclide in an appropriate blank sample. For this standard, the α and β probabilities are both set at 0.05.

3.2.27 minimum detectable concentration (MDC): The MDA expressed in concentration units.

3.2.28 monitoring laboratory: An accredited laboratory that prepares and distributes test materials to a service laboratory for the purpose of monitoring the day-to-day operation of the service laboratory. The service laboratory may also be a monitoring laboratory providing third-party testing materials to another service laboratory or a special quality assurance function within the service laboratory not involved in the routine processing of samples.

3.2.29 nonblind testing: Testing of capabilities when the service laboratory is aware that they are being tested for conformance.

3.2.30 nonconformance: A deficiency in characteristic, documentation, or procedure that renders the quality of an item or service unacceptable or indeterminate. Any activity in the laboratory that adversely affects data quality can result in nonconformance.

3.2.31 nonpermanent records: Those records required to show evidence that an activity was performed in accordance with the applicable requirements but that need not be retained for the life of the project.

3.2.32 performance criteria: The established level of quality (bias, precision, detection sensitivity, etc.) and operational commitments (turnaround times, reporting protocol, etc.) that are

- Agreed upon between the service laboratory and the customer (or inter-governmental agencies or intra-company entities) within a formal contract, or
- Established by the service laboratory and documented within the operational or quality assurance program manual of the laboratory, or
- Both

3.2.33 permanent record: Records that shall be maintained for the lifetime of the project or as long as required by contract. These records may be forwarded to the customer for retention in their recordkeeping system upon request or at the end of the contract period. *Syn*: lifetime records.

3.2.34 precision: A concept employed to describe dispersion of measurements with respect to a measure of location or central tendency. A measurement with small random uncertainties is said to have high precision.

3.2.35 precision statistic: An estimator of precision calculated from a finite sample of data using a specified formula.

3.2.36 qualification (personnel): The characteristics or abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

3.2.37 qualified procedures: An approved and validated procedure that has been demonstrated to meet the specified requirement for its intended purpose.

3.2.38 quality assurance (QA): All those planned and systematic actions necessary to provide adequate confidence that an analysis, measurement, or surveillance program will perform satisfactorily in service.

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3.2.39 quality assurance (QA) program plan: A document that contains or references the quality assurance elements established for an activity, group of activities, a scientific investigation, or a project and describes how conformance with such requirements is to be assured for the activities.

3.2.40 quality control (QC): Those actions that control and measure the attributes of the analytical process, standards, reagents, measurement equipment, components, system, or facility according to predetermined quality requirements.

3.2.41 quality control (QC) chart: *See*: instrument QC chart.

3.2.42 reagent blank: A volume of demineralized water for liquid samples carried through the entire analytical procedure. The volume or weight of the blank shall be approximately equal to the volume or weight of the sample processed.

3.2.43 reference laboratory: A laboratory responsible for the national testing program for a sector of the radioassay community. A reference laboratory is authorized to prepare testing media by adding known amounts of radioactive material for distribution to service laboratories. The reference laboratory is responsible for evaluating the performance of the service laboratories in terms of accuracy and precision.

3.2.44 reference material (standard): 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 assigning values to materials.

3.2.45 relative bias (B_{ri}): The relative bias statistic for the *i*th measurement in a category with respect to the "true or expected" value (value of the spike known by comparison with or derivation from a standard reference material) is defined as

$$B_{ri} = \frac{(A_i - A_{ai})}{A_{ai}}$$

where

A_i is the *i*th measurement in a category being tested, not necessarily a replicate, but possibly a different quantity of spike for each measurement

A_{ai} is the stated quantity in the test sample, as defined by the spike

In order to avoid the expense of a large number of replicates at each radioactivity level in each category, the relative bias (which may be obtained at differing quantity levels) for that test category is calculated from *N* individual relative bias (B_{ri}) values and is defined as

$$B_r = \bar{B}_{ri} = \frac{\sum_{i=1}^{N} B_{ri}}{N}$$

where

B_r is the relative bias

N is the maximum number of test samples measured by an individual service laboratory in a given test category. The sample size N shall be at least five.

i is the *i*th sample

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3.2.46 relative precision (S_A): For the purposes of this standard, the relative dispersion of the values of B_{ri} from its mean B_r, defined as

$$S_A = \sqrt{\frac{\sum_{i=1}^{N} (B_{ri} - B_r)^2}{N-1}}$$

where

B_r is the relative bias

B_{ri} is the individual relative bias

N is the maximum number of test samples measured by an individual service laboratory in a given test category. The sample size N should be at least five.

i is the ith sample

3.2.47 replicate: One of multiple aliquants of a sample.

3.2.48 routine measurements: Radioassays performed on samples by established, validated, verified, and controlled procedures.

3.2.49 screening measurements: Measurements made to detect radioactive material under routine conditions, but not used to quantify the amount of a given radionuclide.

3.2.50 service laboratory: A laboratory, either internal to an agency (or company) or commercially contracted, that performs radioassay measurements for the purpose of providing analytical results, exclusive of the purpose of monitoring or testing. *Syn*: processing laboratory.

3.2.51 single-blind quality control (QC) sample: *See*: blind QC sample.

3.2.52 standard deviation: The square root of the variance of a random variable. For this application, the variance is a measure of the variation of the observations within a measurement set. The standard deviation is often estimated using a set of measurements of the random variable. The standard deviation has the same units as the measured quantity, and therefore is particularly convenient when describing the variability of the measured quantity. This parameter may also be expressed as a relative standard deviation (i.e., as a percentage of the measured quantity).

3.2.53 standard reference material (SRM): Material characterized by the National Institute of Standards and Technology (NIST) for the activity of radionuclides and issued with a certificate that gives the results of the characterization.

3.2.54 surveillance: The act of monitoring or observing whether an item or activity conforms to specific requirements. A surveillance is less extensive than an audit and concentrates on a single activity or item. It is usually conducted more frequently than an audit. Reports are issued to cognizant personnel or groups with a request for corrective action if required.

3.2.55 tolerance chart: *See*: instrument tolerance chart.

3.2.56 traceability: Demonstrated lineage of measurement process quality to the national physical standards.

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3.2.57 traceable reference material or standard: A NIST prepared standard reference material or a sample of known activity concentration prepared from a NIST traceable reference material (derived standard material). *See also*: certified reference material.

3.2.58 unbiased: A measurement of a random variable is called unbiased if the expected value of the measurement is equal to the stated value of the property being measured.

4. National performance testing program

This clause describes the respective roles of the organizations depicted in figure 1 that will provide independent verification of the performance of each service laboratory: NIST; the national reference laboratory(ies); site- or contract-specific monitoring laboratories; and the national accrediting organization(s). Figure 1 does not necessarily indicate a hierarchic structure, but rather a framework within which to describe organizational responsibilities as well as test-sample, information, and data flow.

For the purpose of this standard, testing is broadly defined to include any verification organization that furnishes performance evaluation samples to a service laboratory and/or evaluates the effectiveness of the operational and QA programs of a service laboratory. A distinction is drawn between a reference laboratory, which is responsible for a national testing program, and a monitoring laboratory, whose scope is limited to those QA/QC functions required to assure the quality of measurements at a specific site or within a specific contractual arrangement. While it is anticipated that there will be only a few national reference laboratories, there may be many monitoring laboratories per category within the radioassay community, each with its set of analytical requirements for the service laboratory(ies) that it monitors. Accreditation of any laboratory shall be conducted by an independent accrediting organization that shall evaluate the performance testing and other QA/QC records of the laboratory against criteria developed specifically for each type of laboratory. The accrediting organization shall be the National Voluntary Laboratory Accreditation Program (NVLAP) or a similar private or industrial accreditation program, such as the American Association for Laboratory Accreditation (A2LA).

Accreditation of each laboratory shall be limited to the analyte and matrix type specified, and each laboratory shall clearly represent those capabilities for which it is accredited.

4.1 Organizational responsibilities

The core group of organizations (NIST, reference laboratories, internal quality assurance) of the recommended national performance testing program (see figure 1) begins with a structure similar to that of the voluntary program that is now functioning. Specific responsibilities are placed on the core organizations, however, to ensure national intercomparability of radioassay data. The additional organizations in figure 1, a monitoring laboratory and an accrediting organization, are included to formalize, on a national scale, some practices that are now used by parts of the radioassay community. Each monitoring laboratory shall develop a performance-testing program specific to the analytical requirements of a particular user group (e.g., agency, company, or site). The accrediting organization shall develop assessment criteria and perform assessments appropriate to the laboratory being assessed (reference, monitoring, and service laboratories).

Through the combination of integrated and comprehensive MQA strategies, the quality and integrity of the measurements or services of a laboratory can be assured. Of these strategies, major emphasis shall be placed on

a) Strong internal QA programs
b) Active and thorough onsite expert evaluations
c) Strict adherence to the operational criteria
d) Laboratory evaluation by “blind” testing from interlaboratory and intralaboratory QC samples

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This combination of checks will assure that production processing will remain in control. Although periodic product evaluation remains a necessity, its frequency can be minimized when the production process remains in control.

The national performance testing program does not relieve a laboratory of its responsibilities to provide a strong internal QA program, one that provides for an adequate operational environment, personnel qualifications and training, procedures manuals, instrumentation, calibration, data reduction, record systems, and data reporting. The internal QA program also assures stable operations between proficiency evaluations. These responsibilities can only be fulfilled by an independent and proactive internal QA program within each laboratory that is maintained as part of the core program depicted in figure 1. The requirements for every laboratory's internal QA program are described in clause 5. Although figure 1 shows an internal QA program only for service laboratories, it shall be understood that a strong internal QA program is required for the monitoring and reference laboratories, as well as for NIST.

The following discussion (including figure 1 and throughout this standard) uses the following key. Keyed points are shown in parentheses throughout this standard.

KEY

(1) Assessment criteria, assessments (NIST, reference laboratory (RL), monitoring laboratory (ML), and service laboratory (SL), each under different criteria)
(2) Accreditation reports and certificates
(3) Test samples (traceable, nontraceable, blank, split, or replicate)
(4) Test results
(5) Performance or monitoring reports
(6) Test samples for verification

A laboratory shall first calibrate its measurement systems using NIST traceable reference standards when available, obtained from a reference laboratory (NIST, in the case of a reference laboratory) (3). Periodic proficiency evaluations (PE) shall be performed subsequently to demonstrate measurement consistency with the national physical standards and to test the capabilities of the laboratory to verify its own ability to produce high-quality products (3). Two testing strategies shall be available:

a) Samples of known concentrations of radionuclides of undisclosed values are obtained from a NIST-traceable laboratory ("blinds") (3)
b) The laboratory assays its own material[7] and sends a sample aliquant(s) to the next higher laboratory for conformational analysis (6)

For a service laboratory, either a monitoring or a reference laboratory shall provide the necessary performance evaluations. For a monitoring laboratory, PE testing should function through a reference laboratory, while the reference laboratory(ies) should be served by NIST. In both strategies, analyses shall be performed, comparisons made between the radioassay value obtained by the service laboratory and that obtained by its reference or monitoring laboratory, and PE reports generated and forwarded to the appropriate organizations (4, 5). The laboratory shall be notified of its performance through a report. For the purpose of accreditation, the reference, monitoring, and service laboratories shall each be tested to ensure a degree of traceability for instrument calibrations to the basic radiation emission types for common media (2).

The objective of periodic onsite evaluations is to assure the technical and scientific soundness of the methods and processes of the laboratory. Technical experts and representatives from an "accrediting organization" shall review and evaluate the analytical and service procedures of the laboratory (including validation of all key steps, as well as product and/or service verification), documentation, QA program, and proficiency test

[7]Selected samples may be needed to establish homogeneity of the material.

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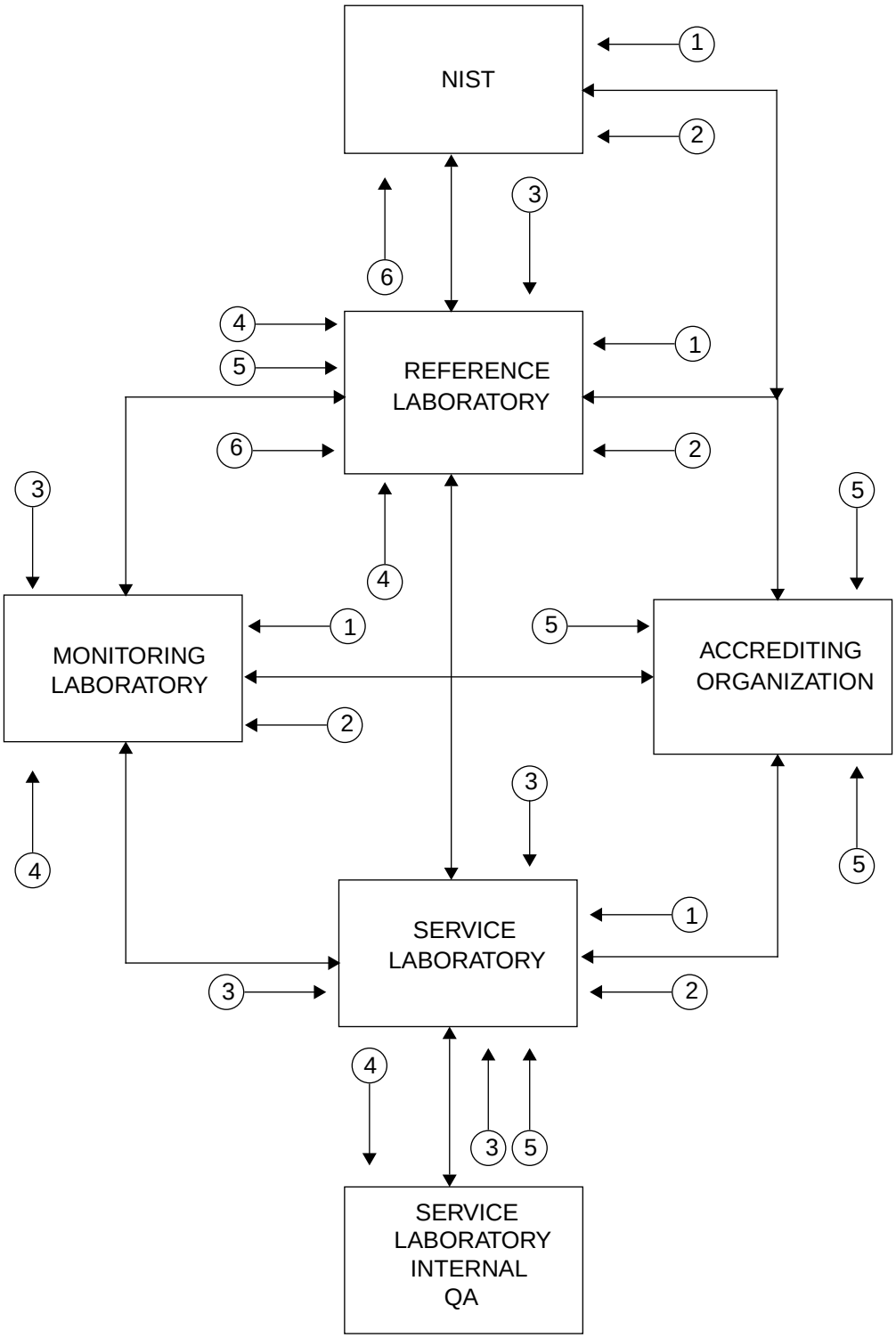


Figure 1—Conceptual diagram of the national performance testing program

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results to determine if the operations meet the operational criteria (1). The expert evaluations assure that the processes to be used by the laboratory are capable of yielding products that meet technical and quality specifications. A laboratory shall limit its claim to accreditation solely to those radionuclides and media for which it has been certified.

4.1.1 National Institute of Standards and Technology

The role of NIST is greatly expanded through the implementation of this standard. All laboratories performing radioassays shall maintain traceability (either directly or through the secondary testing laboratories) to NIST. Also, NIST shall be directly involved in an accreditation program patterned along the lines, or as a part, of the National Voluntary Laboratory Accreditation Program (NVLAP), or an equivalent private accreditation program.

NIST shall prepare and distribute to the reference laboratory(ies) those standard reference materials (SRMs) required by the community of radioassay laboratories to be accredited within this national performance testing program (3). NIST shall conduct tests with the reference laboratory to verify whether it meets the measurement performance criteria of the accreditation to ensure traceability to the national radioactivity measurement standard (3, 6). NIST shall determine if the reference laboratory meets testing requirements and shall report its findings to the accrediting organization as outlined in the following list (5). In the event of a nonagreement, NIST shall act as an official technical expert to the accrediting organization and assist in the resolution of technical disputes. A complete set of tests shall be conducted with each reference laboratory over a maximum of a four-year period for radionuclides to be used by the reference laboratory in discharging its responsibilities within this standard. The number of test and verification samples shared between NIST and each reference laboratory shall be established by agreement of NIST, the reference laboratory, and the accrediting organization; the number of samples should represent a minimum of 5% of each reference laboratory's production limit by analyte.

All test samples shall contain sufficient radioactivity such that counting statistics do not contribute significantly to the uncertainty. Reports of traceability shall be issued by NIST to the reference laboratory and the accrediting organization upon acceptable completion of a test. The report of measurement traceability shall include

a) The name and address of the reference laboratory
b) Identification of the test radionuclide
c) The certified activity concentration and total propagated uncertainty
d) The reference time (date of sample preparation or collection)
e) The characteristics of the test sample and radionuclide(s), i.e., half-life, interferences, acid/base concentration, and carrier concentration
f) The reported value and total uncertainty from the reference laboratory
g) The composition of the reference laboratory's random and systematic uncertainty and the method for their combination
h) The percent difference between the values of NIST and the reference laboratory
i) An evaluation and conclusion of the success by the reference laboratory in meeting the traceability criteria for acceptability

4.1.2 Accrediting organization

A key element mandated by this standard is the development of a method for accrediting service laboratories. The responsibilities of an accrediting organization within the framework of this standard will be drawn, at least in part, from existing NVLAP programs, such as for accreditation of personnel dosimetry processors (see NBS IR 89-4125[8]) and from generic criteria developed previously for the third-party certification pro-

[8]Information on references can be found in clause 2.

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gram (see ANSI Z34.1-1993). Specific responsibilities shall be developed cooperatively by NIST and representatives of user/vendor/regulatory organizations.

Written consensus assessment criteria and checklists shall be developed by the accrediting organization, NIST, the reference and service laboratories, and other appropriate participants (1). Accreditation test samples (discussed in 4.3) shall be provided by the reference laboratory(ies) and test results provided to the accrediting organization (3, 4). Expert assessment teams shall assess the performance of the reference and service laboratories by reviewing internal analytical and QA procedures and documentation, and evaluating performance on test samples. The assessment teams shall provide assessment reports to the reference laboratory and provide documentation to the accreditation organization (5). Both the reference laboratory and the accrediting organization shall provide technical and QA expertise to the service laboratory as needed to improve laboratory performance. The accrediting organization shall provide reports (certificates) of accreditation for those radionuclides and media for which the laboratory qualifies.

The coordination services of the accrediting organization shall include, but will not necessarily be limited to

a) Developing criteria and protocols for evaluating the overall performance of a laboratory and specifically for evaluating its QA programs (1)
b) Organizing and conducting the assessment of the capabilities and QA programs of a laboratory (1)
c) Evaluating the QA/QC performance of a laboratory and determining adequacy for accreditation (1)
d) Reporting its findings for each assessment to the reference or monitoring laboratory as appropriate (2)
e) Issuing a certificate of accreditation or a letter describing deficiencies to the laboratory (as appropriate)
f) Providing for technical advice to the laboratory in order to improve operational and QA programs and program plans
g) Evaluating the accuracy and reliability of reference methods, including statistical design and performance capability (ruggedness testing)[9] during interlaboratory comparisons
h) Issuing annual reports to include summaries of all assessments, performance evaluations by analyte and matrix type, and performance evaluations without anonymity (2)
i) Maintaining a list of accredited laboratories for distribution (laboratories electing to withdraw voluntarily shall not be kept on the distribution list)
j) Maintaining a database of performance evaluation results, onsite assessments, and corrective actions

4.1.3 Reference laboratory

The service and monitoring laboratories shall demonstrate measurement traceability for each test category or specific radionuclide through the reference laboratory. The reference laboratory shall define and develop traceability protocols and provide for the implementation of such protocols as required by each service laboratory for its accreditation program. Such functions shall include, but may not necessarily be limited to

a) Traceability to the national physical standard
b) The provision of test materials to be used for the evaluation of methods or to serve as a basis for the validation or accreditation of measurement capabilities (3)
c) The capability to analyze and verify all test materials it prepares for performance evaluations
d) The performance of intercomparison studies for the purpose of establishing comparability of measuring methods and instruments
e) The evaluation of performance test results, including evaluation of the accuracy and reliability of service laboratory performance

[9]A statistical design refers to the statistical evaluation of the capability of a laboratory. Ruggedness testing refers to the determination of the limitations or boundary conditions of a technique, i.e., the extent to which a radioassay procedure is valid when tested with varying chemical, matrix, and radioactive interferences, as well as the level of analyte.

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f) The development and maintenance of measuring instruments and devices to serve as secondary references where standard reference materials or standard reference data are not available or practical
g) The development of measuring methods and instruments as needed for the independent verification of reference materials or of a reference method for which a standard reference material would not be appropriate (6)
h) The provision of technical advice for QA programs and program plans
i) The evaluation of the integrity, stability, and accuracy of performance evaluation samples prepared for or by monitoring laboratories (5)
j) Liaison with NIST
k) Training

The reference laboratory shall provide blind test samples, including appropriate standards, duplicates, and blanks, to the service and monitoring laboratories for performance-testing purposes (3). The test sample media shall consist of water and filter paper.[10] The water test sample(s) shall be preserved in a manner in which the loss of activity to the walls of the container is minimal. Care shall be exercised that the method of stabilization or preservation does not cause mobilization or loss of radionuclides through chemical interactions. Uniform distribution of radionuclides on the filter media shall be ensured through periodic testing (6). The reference laboratory shall indicate the test category for each sample and may indicate the radionuclide and order-of-magnitude concentration range.

The reference laboratory, upon request, shall also provide performance evaluation samples (analyte and matrix) for the purpose of programmatic and accreditation needs.

4.1.3.1 Measurement protocol

The reference laboratory shall instruct the service laboratory to determine the amount of the radionuclide in a minimum of five samples or sample aliquants. The service laboratory shall be advised of any other procedures necessary to ensure that the tests will allow the minimum performance criteria of this standard to be met. The service laboratory shall be instructed to use the analytical instrumentation normally employed for analysis of the radionuclide under consideration.

4.1.3.2 Reporting protocol

The service laboratory shall be required by the reference laboratory to report the following information for each sample:

a) Identification of sample
b) Background and/or appropriate blank used for the respective measurements
c) Measured amount of activity, corrected for decay
d) Counting time used for the measurement
e) Identification and brief description of apparatus and equipment, including measuring and test equipment, used in analysis (manufacturer, model number and special features)
f) Identification and summary of the analytical procedures used to prepare the sample for analysis
g) Estimate of the total propagated (systematic and random) uncertainty (68% confidence level)

4.1.4 Monitoring laboratory

Each monitoring laboratory shall implement specific measurement traceability protocols defined by the specific accreditation criteria. A monitoring laboratory may also serve separately as a service laboratory when performing service-related measurements, but it becomes a monitoring laboratory when it generates test and

[10]The intent of performance testing in the accreditation process is to ensure that the calibrations of radiation detection systems at all radioassay laboratories become linked to NIST. Since most radioassay laboratories typically have calibration geometries for water and filter mounts (for precipitates), these matrices were selected for testing purposes.

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blind samples or analyzes matrix duplicates of samples sent to a service laboratory by the contractor to evaluate the performance of other service laboratories (3). An example of a monitoring laboratory might be one of the environmental measurements laboratories at a US Department of Energy (DOE) site (see annex B), which also serves at that site as a monitor of radiochemical analyses performed by other laboratories on the site or by offsite contractor laboratories. Another example of a monitoring laboratory might be the environmental laboratory of a nuclear utility operator that provides to staff at the nuclear power plant (NPP) sites of the utility the blind spikes, blanks, or duplicates that are interspersed among the actual environmental samples sent to a contractor service laboratory (3). A utility that does not have an extensive environmental analytical capability could arrange with a third-party service laboratory (another utility or a commercial laboratory) to monitor the performance of its principal environmental services laboratory. In this case, the third-party laboratory would be serving as a monitoring laboratory. The main function of the monitoring laboratory of providing test matrices to the service laboratory shall also be augmented by analyzing matrix duplicates of samples sent to a service laboratory by a contractor. Quality performance criteria for accuracy and precision shall be pre-established and demonstrated by the monitoring laboratory for such augmented functions as third-party testing of sample matrices.

The services of a monitoring laboratory shall include, but may not necessarily be limited to

a) Traceability to the national physical standard
b) The development of protocols specific to the contractual or site-specific requirements of the organization that the monitoring laboratory serves
c) The provision of test materials to be used for the evaluation of service laboratory performance (3)
d) The capability to analyze all test materials if prepared for performance evaluations or third-party sample verification matrices
e) The review of performance evaluation results
f) The evaluation of the accuracy and reliability of service laboratory performance as required by the specifications outlined in the contractual agreement between the client and the service and monitoring laboratories (5)
g) The provision of advice to the contracting agency for QA/QC performance of a service laboratory
h) The generation of reports to the accrediting organization and service laboratory
i) The conduct of periodic onsite assessments

The monitoring laboratory shall develop a protocol for evaluating the routine performance of any service laboratory performing radioassays of samples. The protocol shall address the performance criteria specified within the contractual agreement between the customer and the service laboratory. The protocol shall include but not be limited to the following items:

— Distribution and instructions
— Measurement protocol
— Reporting protocol
— Evaluation of laboratory performance, including accuracy, precision, and detection limit for each analyte and matrix
— Frequency of testing
— Certification of activity

4.2 Accreditation

4.2.1 Accreditation criteria

The accrediting organization, in conjunction with NIST, the accredited reference and monitoring laboratories, and representatives of service laboratories and client agencies or corporations, shall develop the criteria under which all laboratory accreditation processes shall be conducted. The accreditation criteria shall include, but not necessarily be limited to, those given in clauses 4 and 5 of this standard. Criteria shall be

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included for reviewing applications for accreditation; for the conduct of assessments; for approval or rejection of accreditation; and for periodic review of the reference, monitoring and service laboratories' performance (including reissuance or rejection of accreditation). Criteria shall also be developed to validate the protocols developed by the reference laboratory for evaluating the performance of the monitoring and service laboratories.[11]

The mean value for the measured activity of a minimum of five test samples from the reference laboratory shall be within 5% of the NIST value of the activity, and the standard deviation of the test set (see 4.3) shall be less than 5%. All test samples shall contain sufficient radioactivity such that Poisson counting statistics do not contribute significantly to the uncertainty. Reports of traceability (see 4.1.1) shall be issued by NIST to the reference laboratory and the accrediting organization upon acceptable completion of a test. Bias and precision criteria for evaluation of accreditation shall be identical to those in 4.2.1.7 and 4.2.1.8.

4.2.1.1 QA policy and organization

The QA program provides the structure, policies, and responsibility for the execution of QC and QA operations to ensure the accrediting organization that defined standards and quality of a stated confidence level are met. The QC program of any laboratory ensures the maintenance of the controlled analytical process. The QA program incorporates all the necessary elements to ensure that the QC system is functioning effectively.

Implementing the QA program is based on

a) Documentation of all aspects of the program
b) Validation and statistical control
c) Periodic verification and inspection

The QA policy of a laboratory shall include a commitment to a comprehensive QA program including documentation, validation, statistical control, and periodic verification and inspection. Management of the reference laboratory shall make a clear policy statement of such a commitment to quality.

Authority, responsibility, and reporting relationships shall be clearly defined and appropriately documented at each organizational level. QA functions shall be carried out by an independent section of the organization with reporting responsibilities to an upper level of management.

4.2.1.2 Elements of a QA program

The elements of a QA program for all laboratories are provided in clause 5.

4.2.1.3 Accreditation assessments

The minimal program shall consist of yearly internal and external assessments. The assessors shall be well-versed in the fundamentals of quality assurance and have the necessary technical background to detect inconsistencies and errors in the documentation, the application of protocol, and data analysis. The assessment team shall consist of at least three members: one member shall be a representative of NIST, preferably the technical project monitor; one shall be from a group of peers, and one shall be a representative of the accrediting organization.[12] Criteria for accreditation team workings and actions shall be established by the accrediting organization, with special consideration given to resolution of disputed evaluations among team members.

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[11]Validate means the applicability of the protocols for the intended purpose of evaluating the capabilities of the monitoring and service laboratories.

[12]A mechanism shall be developed to prohibit, when appropriate, commercial competitors from being part of an assessment team.

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The assessment report shall be documented and made available by the accrediting organization to NIST and the assessed laboratory. Corrective actions shall be initiated by the assessed laboratory and concluded in a timely fashion. A report shall be made available to appropriate organizations on completion of corrective actions.

4.2.1.4 Intercomparisons between laboratories

Interlaboratory comparisons are useful as a tool to evaluate the analytical system for undetected biases. Intercomparisons are especially important when there are no national standards of appropriate reference materials. When analytical biases are identified, the laboratory shall use the means at its disposal to resolve the discrepancy promptly, i.e., direct traceability analysis by NIST in the case of reference laboratories or resolution by an appropriate reference laboratory in the case of monitoring or service laboratories.

4.2.1.5 Frequency of testing

A participating reference, monitoring, or service laboratory shall be retested at least annually per category. In addition, a laboratory shall be retested at the request of the laboratory, provided that the laboratory provides documentation that such a retest is necessary due to significant alterations in its methods of analysis.

4.2.1.6 Evaluation of laboratory performance

The reference laboratory shall determine for each test category whether the results reported by the monitoring or service laboratory meet the performance criteria of this standard. The results of this evaluation shall then be reported to the accrediting organization for a decision to confer or deny accreditation.

4.2.1.7 Relative bias and bias performance criterion

For the accreditation program, the acceptable maximum average relative bias (see clause 3), B_r, is set at 5% for measurements having a Poisson random uncertainty (68% confidence level) of not more than 1%. The reference laboratory shall provide to the service laboratory samples having sufficient radioactivity to ensure that a 1% (68% confidence level) random (counting) uncertainty is achieved within normal processing times.

For tests performed by the monitoring laboratory, B_r shall be established within the contractual agreement wherein the spiked concentration, A_{ai}, is at least 20 times the desired MDC for any given radionuclide. Test samples having analyte concentrations less than 20 times the desired MDC should be analyzed for purposes of determining the value of the standard deviation of the net count rate and relative precision, S_A, at the MDC limit and other factors used in determining the MDC of a given analytical procedure. However, performance by the monitoring laboratory on test samples having concentrations less than 20 times the desired MDC should not be expected to meet the performance criteria for B_r based on one single set of measurements. It should be noted that testing a methodology at three times the MDC of the service laboratory is very valuable in determining if background or contaminating interferences create a bias in the results.

4.2.1.8 Relative precision and precision performance criterion

For the accreditation program, the minimum acceptable relative precision is set at 5% for measurements having a Poisson uncertainty (68% confidence level) of less than, or equal to, 1%. The reference laboratory shall provide samples having sufficient radioactivity to the service laboratory so that the random Poisson measurement uncertainty of 1% is achievable within normal processing times.

For tests performed by the monitoring laboratory, the relative precision statistic, S_A, (see clause 3) shall be established within the contractual agreement with the service laboratory and shall be determined when the spiked concentration, A_{ai}, is 20 times the desired MDC for any given radionuclide. Performance on test sam-

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ples having analyte concentrations of less than 20 times the desired MDC is not expected to meet the requirements for the statistic S_A.

4.2.1.9 Performance evaluation reporting requirements

The reference laboratory shall submit annually to the accrediting organization a report that includes the following:

a) Results of NIST traceability checks, sample verification analyses, and intercomparison studies
b) Recommendations from and action items in response to internal and external QA assessments

4.2.2 Reference laboratory accreditation

The accrediting organization and the reference laboratory shall jointly develop necessary program plans. The accrediting organization shall provide the reference laboratory with a mutually acceptable directive outlining the objectives of the task, the time period for accomplishment of the objectives, and guidance on the funding level for the task. The reference laboratory shall provide quarterly summaries of all tasks initiated under this agreement to the accrediting organization.

4.2.3 Monitoring laboratory accreditation

This subclause sets forth the minimum technical standards to be followed by any laboratory whose function is to monitor the routine performance of service laboratories performing radiochemical analyses. This subclause is intentionally designed for a double-blind program; i.e., control reference samples are to be completely indistinguishable in coding sequence and in appearance from the accompanying environmental samples. Depending on specific requirements, a simple blind program may be substituted.

4.2.3.1 Traceability to the National Institute of Standards and Technology

Traceability of each monitoring laboratory will be as a secondary reference to NIST through one of the reference laboratories. A monitoring laboratory shall conduct tests with at least one of the reference laboratories to ascertain whether the monitoring laboratory is traceable to the national radioactivity measurement systems within the accuracies set forth in the following paragraph. The reference laboratory shall determine if the monitoring laboratory is traceable and report its findings to the accrediting organization (5). A complete set of tests are performed over a four-year period for radionuclides to be used by the monitoring laboratory in the administration of its user measurement assurance programs. The number of test samples shared between the reference and each monitoring laboratory shall represent a minimum of 5% of the production level, by analyte, of the monitoring laboratory. The monitoring laboratory shall prepare and analyze samples and send a set of representative samples to the reference laboratory for analysis (6), or the reference laboratory shall prepare test samples for the monitoring laboratory to analyze (3).

The mean value for the measured activity of a minimum of five test samples from the monitoring laboratory shall be within 5% of the reference laboratory value of the activity, and that the standard deviation of the test set shall be less than 5%. All test samples shall contain sufficient radioactivity such that counting statistics do not contribute significantly to the uncertainty. Reports of traceability shall be issued to the monitoring laboratory and to the accrediting organization upon acceptable completion of a test. The reports of measurement traceability shall include the information provided by NIST to the reference laboratory (see 4.1.1).

4.2.4 Service laboratory accreditation

This subclause sets forth the minimum technical standards to be followed by a reference laboratory and the accrediting organization (see figure 1) in carrying out a program to accredit the ability of each service laboratory to conform to the accuracy and QA provisions of this standard. Performance evaluations are intentionally designed as a blind testing program, which is necessary to determine in a consistent manner the inherent

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measurement capabilities of radioassay service laboratories. This protocol should be used for the initial assessment of the performance of a service laboratory. Subsequent annual assessments shall also evaluate the internal QA assessments of a service laboratory, its performance on samples provided by monitoring laboratories, and previous onsite assessments.

4.2.4.1 Application for accreditation

A service laboratory shall initiate the accreditation process by written request for accreditation to the national accrediting organization. The accrediting organization shall conduct a pre-accreditation technical assessment of the internal operational and QA programs and manuals of the applicant. Once the applicant's laboratory meets the pre-accreditation assessment criteria, the appropriate reference laboratory shall issue a generic set of test samples to the applicant service laboratory for processing. The accrediting organization shall prepare certificates of accreditation for issuance to those service laboratories that fulfill the accreditation criteria requirements. The accrediting organization shall develop a system by which client (service laboratory user) organizations can obtain information concerning accredited service laboratories.

4.2.4.2 Test selection

Each service laboratory shall have the option of being tested (see 4.3), evaluated, and accredited as follows:

a) *For an entire measurement category*
   If the service laboratory elects this option, the testing laboratory has the option of selecting the radionuclide(s) to be used in the testing, and the service laboratory shall be evaluated for the entire category.
b) *For the measurement of a specific radionuclide*
   If the service laboratory so elects, it may designate a specific radionuclide to be used in the testing. In this event, the evaluation of the test results shall be for that radionuclide only and not for the entire category. Accreditation shall be limited solely to the radionuclide(s) tested, and issued certificates shall so stipulate.

4.3 Test radionuclides and activity ranges

The ability of each reference laboratory to perform accurate analyses shall be affirmed by performance evaluations as described in 4.2. Radionuclide concentrations and media shall be prepared by NIST or the reference laboratory according to the criteria provided in the following subclauses.

The activity of each test sample within a test category shall be such that the analysis will provide, at most, a 1% Poisson counting uncertainty (68% confidence level) while employing a reasonable counting time and a convenient counting geometry.

4.3.1 Photon-emitting nuclides

Each laboratory shall identify and quantify radionuclides by photon spectroscopy. The radionuclide(s) to be incorporated in the test sample shall be chosen from the table on page 20 of radionuclides ranked by photon energy.

4.3.2 Beta-particle-emitting nuclides

Each laboratory shall measure the activity of known beta-particle-emitting radionuclides in separate test media that are otherwise free of radionuclides. The radionuclide(s) to be incorporated in the test sample shall be chosen from the table on page 20 of radionuclides as related to beta particle energy ranges.

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Nuclide	Principal photon energy (keV)
^{241}Am	59.5
^{57}Co	122.1
^{134}Cs	604.7, 795.8
^{137}Cs	661.6
^{58}Co	810.7
^{54}Mn	834.8
^{60}Co	1173.2, 1332.5

Category	Radionuclide
I. Mean beta particle energy less than 100 keV	^{3}H
II. Mean beta particle energy more than 100 keV	^{90}Sr/^{90}Y in equilibrium

4.3.3 Alpha-particle-emitting nuclides

Each laboratory shall measure the activity of the alpha-particle-emitting radionuclide in the test sample media.

The radionuclide for testing shall be one of the following:

Category	Radionuclide
Alpha-particle measurements	^{230}Th, ^{239}Pu

5. Quality assurance

This clause discusses the basic requirements and guidance for establishing and executing a QA program for all laboratories (see the references in clause 2 and the bibliography in clause 6). The requirements of the program apply to activities that could affect the quality of the analyses. These activities include

a) The policy statement by management relative to quality assurance

b) Establishment of an appropriate QA program

c) Performance of functions to attain quality objectives

d) Verification that activities affecting quality have been correctly performed

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5.1 QA program

The QA program is the laboratory's specific policies and requirements, plans and procedures, and the implementation and maintenance thereof, established to ensure achievement of established performance criteria. The program shall provide for the planning and accomplishment of activities affecting quality. The program shall provide for indoctrination and training of personnel performing activities affecting quality to assure that suitable proficiency is achieved and maintained. Management shall regularly assess the adequacy of the program to assure its effective implementation.

5.2 Elements of a QA program

The QA program shall include, at a minimum, the elements in 5.2.1 through 5.2.11.

5.2.1 Organization

An organizational document, which defines the specific lines of authority and communication for laboratory management, and job descriptions outlining the responsibilities of management and staff, shall be maintained. A QA program plan shall be prepared and implemented and shall be maintained to remain current.

Person(s) responsible (e.g., a QA officer) for assuring that an appropriate QA program has been established and for verifying that activities affecting quality have been correctly performed shall have sufficient authority, access, and organizational freedom to

a) Perform assessments to assure compliance
b) Initiate corrective action requests
c) Assure that further analyses are not performed until the proper disposition of all nonconformances, deficiencies, or unsatisfactory conditions have occurred
d) Verify implementation of corrective actions

Such persons shall have direct access to responsible management at a level where appropriate action can be effected. Such persons shall report to a management level such that required authority and organizational freedom are provided, including independence from financial and schedular considerations.

5.2.2 Design control

This element shall describe the flow path of the operational function of the laboratory organization. Applicable operational functions and their relationship to other functions within the laboratory environment shall be documented. This documentation may include, but not be limited to, the definition, description, documentation, and performance of the following:

a) Sample receipt, storage, preservation, and control
b) Sample log-in
c) Sample analysis within specified holding times and measurement
d) Data verification and processing (including data deficiency reports)
e) Reporting
f) Records management

5.2.3 Procurement control

Written instructions and/or procedures shall be prepared for documenting and controlling procurement of equipment, supplies, and services that have an impact on quality. For example, procurement of the following items may have an impact on quality:

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a) Reference material (radioactive standards)
b) Reagents and supplies
c) Analytical instruments
d) Measurement and test equipment (e.g., volumetric labware, analytical pipettes, balances, thermometers, pH meters)
e) Computer software

Upon receipt of a quality-related order item, it shall be inspected to verify that the item received satisfies the description and requirements provided on the purchase order. If any requirement is not satisfied, the item shall be marked as nonconforming and segregated to prevent inadvertent use of the item.

Provisions for subcontracting of services that may have an impact on quality shall include

a) Transmittal (flow down) of applicable QA requirements to subcontractor(s)
b) Right of access for assessment/surveillance purposes
c) Notification to client that subcontracting is being performed as part of processing client work

5.2.4 Instructions and procedures

Written instructions and/or procedures covering the administrative, operational, and quality activities of the laboratory shall be established and implemented. Standard operating procedures include but are not limited to

a) Calibration and operation of laboratory instruments and equipment
b) Analytical methods and methods validation
c) Data verification
d) Implementation of interlaboratory and intralaboratory QC programs
e) Software quality assurance (verification and validation)
f) Preventative maintenance program for instruments and equipment
g) Training and qualification of laboratory personnel
h) Nonconformance (deficiencies) and corrective actions (see annex C for an example of documentation of nonconformance during processing)
i) Procurement of quality-related items
j) Sample receipt and control
k) Radioactive material handling and waste disposal
l) QA procedures
m) Instructions relative to the chain-of-custody of samples during laboratory processing [including as a minimum the signature or cross-referenced initials of the analysts (handwritten or electronic) at each stage of the sample process]
n) Sample preparation and storage
o) Disposal of sample extracts
p) Records retention
q) Document control

5.2.5 Document control

The preparation, issue, and change of documents that specify quality requirements or prescribe activities affecting quality shall be controlled to assure that appropriate documents are being employed. Such documents, including changes thereto, shall be reviewed for adequacy and approved for release by authorized personnel.

The control system shall be documented and shall provide for

a) Identification of documents to be controlled, including revision level and their specified distribution

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b) Identification of assignment of responsibility for preparing, reviewing, approving, and issuing documents
c) Review of documents for adequacy, completeness, and correctness prior to approval and issuance

Major changes shall be subject to the same level of review and approval as the original level of review and approval. Minor changes may receive less rigorous review prior to approval.

5.2.6 Identification and control

Controls shall be established to assure that only correct and accepted items (including computer software) are used or installed.

Where specified, items having a limited shelf life shall be identified and controlled to preclude use of any item whose shelf life has expired.

Provisions shall be made for the control of item identification consistent with the planned duration and conditions of use. Identification and control measures shall be used to assure that samples are identifiable at all steps of the analytical process and are traceable from their source to the resultant report.

5.2.7 Validation and verification

Conformance of an item or activity to specified requirements shall be planned and executed. Procedure or method validation shall be performed with matrix spikes prior to the commencement of processing samples in order to ensure that the analyte of interest is properly measured without significant interferences and to within pre-established, acceptable quality performance levels. Software verification and validation shall be performed with known results to ensure that calculational and data manipulation programs perform properly. Detailed guidance on software verification and validation can be found in IEEE Std 1012-1986. Data verification begins with the processing of data and continues through the review of the data and reporting of analytical results. Data review begins with a reviewer, who is independent of the data acquisition and processing, verifying that data has been correctly calculated and that appropriate and complete entries have been made. The review continues through the review and approval and verification that reported analytical results correspond to the data acquired and processed.

5.2.8 Control of instruments, measuring, and test equipment

Performance criteria and controls shall be established for instruments or test equipment. Performance of the measurement equipment shall be checked and evaluated at regular intervals while the equipment is in use. These checks shall be sufficient to demonstrate that the measurement equipment is properly calibrated and that all components are functioning properly. Measurements shall include instrument background and response to known sources of activity. The number of instrument QC measurements shall represent at least 5% of the total number of measurements by analyte.

Items that do not conform to specified requirements shall be managed to prevent inadvertent use. Procedures shall provide for identification, documentation, evaluation, and segregation and disposition of a nonconforming item.

Instrument control records shall, as a minimum, identify

a) Item tested, including model and serial numbers
b) Date of last calibration and next calibration due date
c) Applicable calibration certificates
d) Tester (person performing the calibration or test) and data recorder if applicable
e) Type of observation
f) Results and acceptability

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g) Acceptable control or tolerance limits
h) Action taken when any deviations are noted

5.2.9 Corrective actions

Corrective actions shall include provisions to identify promptly and to correct conditions out of compliance with the established performance criteria of the laboratory. Corrective actions shall be documented and reported to appropriate levels of management. Results for samples processed using out-of-compliance processes or system(s) since the last verified condition of compliance shall be considered potentially deficient. Appropriate deficiency report forms shall be issued and notifications provided.

In the case of a significant condition adverse to quality, the cause of the condition shall be determined and corrective action taken to preclude recurrence. The identification, cause, and corrective action for significant conditions adverse to quality shall be documented and reported to appropriate levels of management; follow-up action shall be taken to verify implementation of this corrective action. Management of the organization or activity shall provide for investigations of adverse findings, including notification of the appropriate organization in writing of action taken or planned.

Nonconforming items will be managed to prevent their inadvertent use.

Conditions adverse to quality shall be tracked with the proposed and actual dates that the corrective actions are completed and verified. Proposed dates for completion of corrective/prevention action may be extended with the approval of the QA officer.

5.2.9.1 QA records

Records (hard copy and/or electronic) that furnish documentary evidence of quality shall be specified, prepared, and maintained. Records shall be legible, identifiable, and retrievable. Records shall be protected against damage, deterioration, or loss. Requirements and responsibilities for record transmittal, distribution, retention, and maintenance shall be established and documented.

The records shall be stored in predetermined locations that meet the requirements of applicable standards, codes, and regulatory agencies. Measures shall be established to preclude the entry of unauthorized personnel into the storage area. These measures shall guard against larceny and vandalism.

Prior to record destruction, the records shall be reviewed to determine if the retention time should be extended.

5.2.10 Assessments

Management self-assessments are to be established and implemented. These periodic assessments shall focus on the integrated QA program and shall identify management barriers that hinder the organization from achieving its objectives. Results shall be documented. Management shall take prompt action on the recommendations resulting from the assessment process. Follow-up assessments shall include an evaluation of the effectiveness of management actions.

Internal audits and surveillance shall be planned, scheduled, and performed to verify compliance with all aspects of the QA program and to determine its effectiveness. These audits shall be performed in accordance with written procedures or checklists by personnel who do not have direct responsibility for performing the activities being audited. Results shall be documented and reported to and reviewed by responsible management. Follow-up action shall be taken where indicated.

QA audits or surveillance shall be scheduled in a manner to provide coverage and coordination with ongoing QA program activities. They shall be scheduled at a frequency commensurate with the status and importance

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of the activity. The audit/surveillance schedule shall be reviewed periodically and revised as necessary to assure that coverage is maintained to remain current. Regularly scheduled audits/surveillance shall be supplemented when necessary to provide adequate coverage.

The auditing organization shall develop and document an audit/surveillance plan. This plan shall identify the scope, requirements, personnel, applicable documents, schedule, and written procedures or check-lists.

The report shall be signed and issued by the team leader, and it shall include the following information, as appropriate:

a) Description of the scope
b) Identification of the auditor(s)
c) Identification of person(s) contacted during audit/surveillance activities
d) Summary of results, including a statement on the effectiveness of the QA program elements
e) Description of each reported adverse audit or surveillance finding in sufficient detail to enable corrective action to be taken by the audited organization

5.3 Sample process quality control

The laboratory shall have written QC procedures to verify that the quality of measurements comply with specified performance requirements. The QC procedures shall require

a) Use of radionuclide reference standards
b) Performance checks of measurement systems
c) Intralaboratory analyses (e.g., known quantities, replicates, and blanks)
d) Participation in at least one interlaboratory intercomparison program through an accredited monitoring laboratory as defined in this standard
e) Computational checks
f) Review of procedures, specifications, and operating logs
g) Observation of operations and evaluation of QC data
h) Documentation of conformance to the performance criteria of this standard
i) A method for evaluating QC data to ensure the long-term consistency of analytical results

5.3.1 Internal QC samples

QC samples, as described in the following list, shall be included with routine measurements for analysis. Unless specifically addressed within the laboratory/client contract, the level of processing of internal QC samples shall constitute, on the average, a minimum of 10% of the total processing load or throughput by analyte. The sum of the subsequent five categories of internal QC samples shall add to 10% of the processing load and not be limited to one type or category.

a) *Reagent blank:* The laboratory shall analyze reagent blanks to verify that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Results of reagent blank analyses shall be maintained with the corresponding analytical data.
b) *Replicate:* The laboratory shall perform replicate or blind replicate analyses to evaluate the precision of an analysis.
c) *Matrix spike:* The laboratory shall analyze matrix spikes, blind matrix spikes, and double-blind matrix spikes to evaluate the effect of the sample matrix upon the analytical methodology.
d) *Traceable reference material:* The laboratory shall use traceable reference materials to evaluate the accuracy and precision quality performance criteria of a procedure.
e) *Blind traceable reference material:* The laboratory shall use blind traceable reference material to evaluate the accuracy and precision quality performance criteria of a procedure or an analyst.

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5.3.2 QA reporting requirements

The results of QC samples shall be made available and reviewed with the appropriate laboratory staff and management within seven days of the sample analysis and prior to the reporting of results to the client. Further, the laboratory shall prepare and submit, on at least an annual basis, a summary of the QA program to the appropriate organization(s).

The report shall include, as a minimum, the following:

a) Results of management assessments, audits, and surveillance conducted at the laboratory, describing all findings, violations, observations, recommendations, action requests, and planned corrective actions taken

b) Nonconformance items reported for each calendar year describing the nonconformance item, the cause for the nonconformance, and the appropriate actions taken to resolve the nonconformance

c) Compilation of all internal and monitoring laboratory QC results, including

   1) The sample identification, test category, media, and the date of analysis

   2) QC samples

      i) The result and its associated uncertainty

      ii) The known value and its associated uncertainty

      iii) The calculated bias and precision statistics for the QC sample(s), as well as a coded pass/fail notation for performance criteria evaluations

   3) The results and their associated uncertainty for each replicate sample

d) Results of validation of new procedures against specified performance criteria

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Annex A

(normative)

Operational requirements of a radioassay laboratory

Annex A is considered an integral part of this standard and, therefore, the requirements set forth in this annex are considered essential and shall be followed.

Annex A provides operational requirements for the radioassay laboratory. Topics include facility design, contamination control, organization, training and proficiency examination, nuclear instrument selection, use and control, performance criteria, results data use and interpretation, and results reports.

A.1 Service laboratory operational criteria

The analytical results of service laboratories may provide the basis for a variety of health, economic and regulatory or other administrative decisions. The data from these laboratories shall be documented as sufficiently reliable to permit such decisions. Documentation of sufficiency of data requires the service laboratory to participate in one or more externally administrated intercomparison programs to the extent commensurate with the analytical workload of the laboratory. External intercomparison samples should serve to augment properly designed internal QC programs. The combined monitoring laboratory, external third-party, and internal QA programs shall represent a minimum (average over a year) of 10% of the total sample processing work load by analyte.

A.2 Facility criteria

The analysis of samples should be conducted in a laboratory facility where security and integrity of the samples and analytical data are provided. Separate work areas for wet chemistry operations and for the nuclear instrumentation/equipment critical to valid measurements of radioactive contaminants are necessary.

A.2.1 General laboratory area

Laboratory safety shall be an integral and deliberate effort in laboratory design and operation. The facilities shall be secured during off times.

Laboratory space, including benches and hoods, shall be sufficient to meet analytical processing requirements. Spatial requirements will vary according to the type of analytical technique and safety requirements. Recommended facility services shall include, but are not limited to

a) Sinks with hot and cold running water
b) Appropriate number and location of eye washes and /or safety showers
c) Source of distilled or deionized water and consideration given to a reserve tank to store the water, allowing for short-lived radon daughters to decay
d) Gas supply (natural gas, propane cylinder, etc.)
e) Vacuum source
f) Source of clean compressed air and consideration given to a storage tank, allowing air to stand and radon daughters decay
g) Exhaust hoods with appropriate filters and flow rates to perform required analyses

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h) Designated storage area for gas cylinders away from the environment when possible to prevent corrosion, overheating (i.e., exceeding 50 °C), or freezing
i) Storage area, which should not be above 50 °C, and the immediate storage area should be free of electrical circuits
j) Adequate electrical system, including both 115 V_{ac} and 220 V_{ac} service
k) Laboratory ventilation system, which should be designed to move air positively from lower activity to higher activity areas
l) An adequate storage area for routine laboratory supplies (e.g., glassware, containers etc.)
m) Laboratory lighting sufficient to allow detailed work and to provide comfort to the employees

A.2.2 Sample receipt and storage area

The facility should maintain a designated area for the receipt and screening of routine samples to assure radioactivity levels are at the anticipated levels, as well as a separate designated secure area for short- and long-term storage of samples, with due consideration given to chain of custody. The storage area has to be sufficiently spacious to organize and isolate samples properly according to radioactivity content for periodic inventory and for retrieval of samples for a length of time according to needs or contractual agreement. Separate sample receipt areas shall be maintained for samples of known high levels of radioactivity and for low-level or environmental-level samples. Pre-determined “known” radioactive material should be received in the designated “Radioactive Material Receipt Area.”

A.2.3 Chemical storage area

Chemicals and reagents shall be stored in a designated area, isolated from routine traffic. Storage facilities shall conform to all applicable federal, state and local regulations.

A.2.4 Shop area

A designated area is recommended for performing routine maintenance on standard laboratory equipment and components, and for manufacturing of unique laboratory related items.

A.2.5 Nuclear instrumentation areas

The nuclear instrumentation room has to be independent of rooms in which samples and standards are being prepared or in which other types of chemical analyses are being performed. All instruments shall be properly grounded, and a regulated power supply, either external or internal, shall be available to each instrument. The following items should be considered in the design or operation of the nuclear instrumentation room.

a) The air temperature should not exceed 27 °C, and the temperature variation under normal operating conditions should not exceed 3 °C.
b) The relative humidity shall be kept between 35–70%.
c) A regulated power supply system shall be used to assure the maximum protection of counting equipment, computer hardware, and data (an uninterruptable power system may be necessary to meet certain data quality objectives).
d) The facilities should have an approved fire protection system, which has been designed to consider damage to electronic and computer systems during fire retardation.
e) Sufficient radiation shielding (room and/or detector) should be provided to maintain a consistent radiation background at a level to permit accurate, sensitive analyses. Consideration should be given to building materials, sealants, air supply, etc. to minimize background. Nuclear instrumentation rooms dedicated to environmental measurements should not be located in close proximity to a potential source of contamination (e.g., onsite nuclear electric power stations) unless the counting

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room is located in a hardened facility. Floor loading due to heavy shielding may limit nuclear instrumentation rooms to the lowest level of a building.

f) Air ventilation of the rooms should be sufficient to exchange the air five to ten times every hour and maintain the room at a positive pressure of at least 5 mm of H_2O.

g) Radio frequency (RF) shielding of the electronic components and various cables may be required, depending on the source and levels of emissions. Although desktop computers meet the RF emission regulations of the US Federal Communications Commission, the levels may interfere with the proper operation of various nuclear instrumentation systems. Conformation of nuclear instrumentation system performance specifications is highly recommended if electronic devices emitting RF shall be located in the same general area.

A.2.6 Contamination control

Laboratory design and the actual implementation of the analytical programs should address situations or conditions necessary for the use, storage, and disposal of high-level radioactivity samples or sources. Laboratory and program designs shall be established to minimize interactions between higher and lower radioactivity areas as well as common utilization of equipment, nuclear instrumentation, and facilities. Specially controlled accessible facilities or areas should be considered for the receipt of radioactive materials, the preparation of calibration standards, and the storage of radioactive standards or waste.

A.2.7 Record storage area

A record storage area consistent with the requirements of 5.2.9 shall be maintained.

A.3 Waste management plan

An effective waste management plan that meets all applicable federal, state, and local requirements shall be prepared. The plan shall establish the policies and guidelines for effective management and disposal of waste. The policies and guidelines shall apply to all personnel who generate, handle, manage, and/or dispose of radioactive, hazardous, and/or mixed waste.

A.4 Staff qualifications

A.4.1 Education and experience

The following are recommended minimum standards for the technical supervisor:

— *Academic training:* Minimum of bachelor's degree in a science discipline or its equivalent with a minimum training to include a semester of analytical or inorganic chemistry.
— *Experience:* Minimum of five years of experience in radiochemistry (includes nuclear instrumentation) measurements.

The following are recommended minimum standards for the analyst position:

— *Academic training:* Minimum of high school diploma or its equivalent.
— *Experience:* The analyst shall demonstrate acceptable skills through the successful participation in the analysis of applicable proficiency test samples. However, a minimum of six months of on-the-job training, under direct supervision of a qualified analyst, may be required to become totally familiar with all laboratory policies and procedures.

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The following are recommended minimum standards for the QA officer:

— *Academic training:* Minimum of bachelor's degree in a science discipline or its equivalent with a demonstrable training in QA principles and statistics.
— *Experience:* Minimum of five years of experience in radiochemistry/radioassay measurements.

A.4.2 Training

The technical supervisor or designee shall be responsible for the training of each analyst. In most cases, the training can be done internally by the technical supervisor, especially for those procedures that the laboratory performs on a routine basis. However, consideration should also be given to establishing informal agreements with other laboratories or organizations in instances where the method to be learned is complex and unfamiliar to the supervisor. Training records for each analyst shall be maintained. These records shall contain a short description of the training received and a signed statement by the instructor stating that the employee can perform the analysis in a satisfactory manner. The tests used to determine proficiency are discussed in A.4.3.

A.4.3 Proficiency examinations

It shall be the responsibility of the technical supervisor to conduct periodic and planned proficiency examinations to test the ability of the analysts receiving training in a particular analytical procedure. The examination shall consist of the actual analysis of samples containing the radionuclide of interest. The examination may include, at the option of the supervisor, a written section on theory as well as the laboratory portion. The samples used for analysis should contain the radionuclide in concentrations known to the supervisor but not to the analyst. The supervisor shall decide whether the results from the analyst demonstrate acceptable performance as per the established laboratory performance criteria for the analysis. The performance shall be documented in the training record of the employee.

A.5 Nuclear instrumentation

Virtually all analyses in a radioassay laboratory eventually require the measurement of alpha, beta, or gamma radiation. In general, each type of radiation is analyzed with a different type of highly specialized equipment, all of which can detect a low-level signal, amplify it, process it, and store or record it as digital information [AB8], [AB9].[13]

A.5.1 Calibration

Instrument calibration should be performed as needed with only NIST traceable standards. An instrument QC/tolerance chart shall be prepared, as well as an instrument reliability test performed, at the time of the initial calibration. Recalibration should only be necessary in the event that the instrument/system has malfunctioned and the repaired equipment has responded to a QC test in a fashion that the tolerance level of the QC chart has been exceeded, i.e., the operating or response characteristics of the instrument/system have changed more than the tolerance limits permit. Only well-defined standards should be used, and care shall be taken to prepare homogeneous standards in accurately defined geometries having exact radiometric characteristics of the sample matrix/geometry (or correctable parameters). Replicate standards, multiple measurements, and good counting statistics should be stressed as important for every geometry used.

[13]These numbers refer to the bibliography in A.11.

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A.5.2 Instrument quality control

The elements discussed in the following subclauses are necessary to ensure proper instrument performance.

A.5.2.1 Instrument response check sources

In order to ensure that an instrument responds in the same manner subsequent to its calibration, an instrument response check is used in conjunction with an instrument QC chart. It is important to emphasize that the QC chart and the response of an instrument QC check source is directly linked to the calibration of the instrument. Therefore, if an instrument response check source becomes damaged or changes its characteristics, a new instrument QC chart can only be established through the use of a new source, after verification that the instrument calibration has not changed. Otherwise, the instrument shall be recalibrated and a new instrument control chart established.

Instrument response check sources should provide adequate counting statistics (less than a 1% Poisson statistic uncertainty) over the time period for which the source is to be counted. However, the source cannot be so radioactive as to cause pulse pileups, dead time that is significantly different from that to be expected from routine samples, or gain shift in the case of pulse height analyzer (PHA) systems. For example, a check source for a PHA system, equipped with a modern analog-digital converter (ADC), should not exceed a total count rate of about 1000 counts per second. Any source to be used as a check source should be either sealed or encapsulated to prevent loss of the source and contamination of the counting system. The check source-to-detector geometry has to be known and held constant. The geometry is extremely important because small changes can have large effects that overshadow normal statistical counting variations. Because alpha and beta sources are subject to leakage after a few years, they should be replaced periodically even though the nuclide may have a long half-life. Also, some alpha and beta sources should be surveyed (smeared) routinely for possible leakage.

A.5.2.2 Instrument control/tolerance charts

For the purpose of this standard, a distinction shall be made between an instrument quality control chart (referred to as a QC chart) and an instrument tolerance chart (referred to as a tolerance chart). The distinction lies in the fact that the development and use of a QC chart has a statistical basis related to the QC source measurement process, whereas a tolerance chart and its interpretation does not. A QC chart is used to determine if the response of the instrument has changed statistically; the magnitude of the statistical response change may or may not be significant when compared to the required precision and accuracy criteria for the overall analytical technique. A tolerance chart does not have a statistical basis related to the QC source measurement process, but instead is based upon maintaining a change in instrument response to a tolerance level judged acceptable to meet overall quality requirements for the technique. However, a tolerance level can never be more restrictive than what is statistically possible. Therefore, a statistical evaluation of the instrument QC process has to be performed to ensure reasonable tolerance limits are achievable. For most applications, it is recommended that both QC and tolerance charts be used in concert. The QC chart shall determine statistical changes in the instrument response, and the tolerance chart establishes the limit of the instrument response change that can be tolerated as related to the overall quality requirements for the technique.

The intent of instrument quality control is to ensure that the calibration response of the instrument is maintained to within specified tolerance limits. Although a radioactive QC check source is employed to determine the response of an instrument during an evaluation, care has to be taken not to introduce uncertainties associated with the QC source measurement process itself that could mask true instrument response changes. Such QC source measurement uncertainties include those associated with random Poisson counting statistics, QC source positioning, and radioactive impurities in the QC source material. These uncertainties should be kept to a minimum (less than 1.0% collectively if practical) in order to isolate true changes in the instrument response.

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A control chart ([AB3], [AB7], and [AB12]) is a graph prepared by plotting time on the abscissa and the count, count rate, or efficiency on the ordinate. Lines are drawn parallel to the time axis at values (corrected for decay if necessary) for the "mean" count rate (or the other mentioned parameters) and for specified statistical limits, typically values of ±2 and ±3 standard deviations of the measurement set. Tolerance limits for the instrument response change should be commensurate with the quality performance criteria for the measurement technique or sample analysis. However, the tolerance limits cannot be more restrictive than three times the measured standard deviation (Poisson plus systematic uncertainty) of the distribution of the control chart data set. Due to the random nature of radioactive decay, it is recommended that the Poisson uncertainty statistic of an individual measurement be equal to or less than 1% of the measurement, and the overall tolerance limit of the instrument response change should not exceed 20% of the overall accuracy performance specification for the technique. For example, the Poisson statistical uncertainty of a single QC measurement may be ±0.1%, the one standard deviation of the total uncertainty (Poisson plus systematic) of 20 QC measurements may be ±0.5%, the tolerance level for the instrument established at ±3.0%, and the overall analytical technique performance criterion for accuracy set at ±15%.

The "mean" count rate (or the other mentioned parameters under evaluation) is generally determined by averaging at least 20 measurements taken over a time period for which the Poisson counting uncertainty is at most 1%. The measurement data set should include system or instrument response variations (such as source positioning errors, temperature fluctuations, radon background fluctuations, etc.), in addition to the normal Poisson counting statistics. For longer background measurements, a series of at least 10 single background measurements should be sufficient to prepare instrument background control charts.

QC and tolerance charts shall be interpreted objectively. For a control chart, if a QC measurement occurs outside the expected three standard deviations statistical range, instrument service may or may not be needed. If the response of the instrument is not outside the established tolerance limits, the instrument may still be used. At this time, depending upon the magnitude of the instrument response change, the operator may want to initiate an investigation into the root cause of the response change. To determine whether the outlier measurement was statistically significant, the analyst should run a series of repeated measurements and apply another statistical test, such as a Chi-square test, to determine whether the variation was excessive.

Trends from control charts may show other information. For example, if regular measurements of the check source are moving in one direction, one can infer that some system variable is changing. This variation may not always require instrument service; instead, re-evaluation of the values of the standard deviation and other related limits of the control chart may be necessary. If the control chart has to be re-established, the calibration of the instrument or system has to be re-established or re-verified prior to the construction of a new control chart.

A.5.2.3 Monitoring of instrument background

Since laboratory instrumentation typically operates over several orders of magnitude of sample activity, careful control of detector contamination and routine monitoring of instrument background shall be an integral part of the control program of each laboratory. Any instabilities or trends observed in instrument background are one of the most specific indicators of performance inadequacy for low-activity samples. A routine program for monitoring of background is particularly important, since all test samples specified for use under this standard are of sufficient activity that background is not significant in the total recorded count, and any deficiencies in the ability to measure low activity samples would be overlooked.

A.6 Laboratory performance criteria

The performance requirements of the service laboratory may vary depending upon the analytical processing specifications of the client/agency contracting the services. The degree of sophistication of analytical processing is dependent on the intent of the overall program design data quality objectives. Various programs

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such as gross contamination surveys, environmental monitoring, and detailed scientific or parametric testing programs may have specific needs. Program designs may vary from those that only require imprecise screening measurements to determine the extent of contamination to those for detailed research and development studies or tests that incorporate experimental designs requiring constant precision of a high degree for each measurement. Therefore, it is incumbent upon both the contracting entity and the service laboratory to establish the accuracy, precision, and detection limit performance criteria within any binding contract prior to the initiation of laboratory processing. In addition, items related to any QA element discussed in clause 5 should also be incorporated into a performance requirement within the agreed-upon contract. The monitoring laboratory shall be used to evaluate the performance of the laboratory with respect to the established criteria.

A.7 Detection limit concepts

Detection limit concepts (critical level, lower limit of detection, MDC, etc.) are probably the most misunderstood and misapplied notions in radiation statistics. ([AB4], [AB5], [AB6], [AB10], and [AB11] describe these and other related concepts.) In particular, this standard finds the following common practices to be especially harmful and/or misleading:

a) The estimation of a “sample specific MDC”
b) The use of an estimated MDC to censor or otherwise qualify the results of individual measurements

Such mistaken use of detection limit concepts has occurred (and persists today) due to widespread misunderstanding of the statistical foundations that underlie detection limit notions. Such misunderstanding may be attributed, at least in part, to an unfortunate lack of guidance in much of the technical literature to the proper use of these ideas. In this clause, this standard attempts to clarify these issues.

Each laboratory shall establish detection limits using the concepts of this clause, and in doing so should use the guidance provided in the following subclauses. It should be noted that the guidance developed or presented in A.7.4 through A.7.6 is the most basic and simplified equations that are typically quoted and utilized. However, it is recommended that the more detailed and expansive equations for the more sophisticated applications, as found in ANSI N13.30-1996, [AB4], [AB5], [AB6], [AB10], and [AB11], be used when deemed appropriate.

A.7.1 Valid uses of the *a priori* MDC

The concept of the *a priori* MDC involves consideration of the probability of two types of mistaken conclusions:

a) Concluding that net radiation is emitted from a background-level sample (false positive/Type I error)
b) Failing to detect net radiation from a sample that is above background level by a specified amount (false negatives/Type II error)

In principle, it is necessary to specify both nominal measurement conditions (background, including all types of interferences, sample mass or volume, chemical yield, detector efficiency, etc.) and acceptable prob abilities α and β for Type I and Type II error, respectively, in order for the MDC to be uniquely determined. More detail concerning the manner in which the MDC may be estimated is given in the following list. The crucial consideration is summarized as “the MDC, by definition, is a concept that addresses the capabilities of a measurement process under a given set of nominal experimental conditions.” Therefore, the proper use of the *a priori* MDC lies in, for example:

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a) Comparing different measurement techniques or alternative implementations of a given technique (under the stated nominal conditions)

b) Determining whether a proposed measurement method is appropriate for a given set of sample characteristics (including estimates of the nominal levels of interferences anticipated in the batch) and client expectations

c) Judging whether certain regulatory limits are satisfied by the proposed measurement process as applied to a batch of sample measurements

A.7.2 Improper uses of the *a priori* MDC concept

Since the *a priori* MDC is defined only in terms of given nominal measurement conditions, this concept is misapplied whenever it is used to make judgments concerning any individual measurement. Thus, for example, the following uses of the MDC concept (these are discussed in more detail in A.7.3 and A.7.7) are ill-founded:

a) Comparing the results of an individual sample measurement to the MDC in order to assess the "worth" or "accuracy" of the measured value

b) Determining a "sample-specific MDC"

c) Censoring the results of an individual measurement due to its being "less than MDC"

A.7.3 Interpretation of individual measurement results

Radioactivity measurements are always subject to uncertainty. In addition to Poisson counting uncertainties, which are always present, there are often additional sources of uncertainty, such as those associated with detector calibration, sample preparation, interfering species that alter the count rate observed, etc. In some circumstances, these ancillary sources of uncertainty may be larger than the relative magnitude of the counting uncertainty. Since it is impossible to eliminate such sources of uncertainty, the activity or concentration reported should always be accompanied by an estimate of total propagated uncertainty bounds for each result. It is very important that the error analysis performed for the measurement be as thorough as possible since the confidence interval estimated for each result is the best information available for that individual measurement. In particular, once the measurement is made, there is no valid information gained by comparing the result of the measurement to the nominal *a priori* MDC or any other form of a nominal *a priori* detection limit. Nominal *a priori* MDC in this context means "typical" based on *a priori* typical parameters, not the actual parameters associated with the sample measurement under evaluation. This standard recommends the discontinuance of the widespread practice of such comparisons and/or using them as the basis for decisions regarding any individual measurement.

For the purpose of having a laboratory interpret whether an individual sample measurement is different from its representative appropriate blank, it is recommended that the laboratory compare the net count or count rate of the measurement with a decision level calculated using the sample-specific "appropriate" blank. The "appropriate" blank should include measurement interferences from impurities (elevated compton continuum, channel crosstalk from higher energy particles measured by liquid scintillation of alpha spectrometers, etc.,) that are not typically known *a priori* or included in the nominal *a priori* decision limit. This "true" decision level is different from the nominal *a priori* decision limit in that it truly represents the appropriate blank at the time of measurement. For some measurement processes, the determination of the "true" appropriate blank for each sample may be impractical. However, every effort should be taken to assess properly the parameters of the appropriate blank. As indicated in A.8, a service laboratory is required to report the estimate of the counting uncertainty and the value of the calculated decision level (in consistent units) for each sample measurement.

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A.7.4 *A priori* decision-level count rate and decision-level concentration[14]

Suppose that a sample containing no analyte is repeatedly counted for the same length of time as the appropriate blank (or detector background if appropriate) measurement and the net count rate recorded for each such count. If the number of gross counts obtained is "large" (more than approximately 70 counts is sufficient), then the net count rate is approximately normally distributed with a mean of 0 and a standard deviation, S_o, of $S_o = S_B \sqrt{2}$, where S_B is the standard deviation of the background gross count rate. It follows then that the net count rate observed when counting a sample containing no analyte will exceed $1.645 \times S_o$ during only 5% of such counts, i.e., the 95% upper confidence limit for the net count rate is $1.645 \times S_o$. This standard defines this confidence limit as the "decision-level count rate" (*DLR*); thus

$$DLR = 1.645 \times S_o \tag{1}$$

In principle, the decision whether a sample (for which the activity is being measured) emits radiation above the appropriate blank background level should be based solely upon whether the net count rate observed for that sample exceeds this *DLR*. This detection criterion results in a Type I error of $\alpha = 0.05$, i.e., false positive detection will occur with a 5% frequency.

Example:

Suppose that the average background count rate for a certain instrument and appropriate sample matrix blank is 60 counts per minute (min^{-1}) and that 5 min counting times are used. Suppose, additionally, that a sample containing no analyte is counted. Then the number of background counts that may occur has a mean of 300 and standard deviation of $\sqrt{300}$ and the sample gross counts follow the same distribution. The standard deviation (S_n) of the net count obtained by subtraction of background from the gross count is formed from the sum of (equal, in this case) components from gross (S_g^2) and background (S_b^2) counts and can be determined from the expression

$$S_n = \sqrt{(S_g)^2 + (S_b)^2}$$

Thus, the number of net counts that may occur has a mean of 0 and standard deviation of $\sqrt{600}$, so the observed net counts will exceed 40 (i.e., $1.645 \times \sqrt{600}$) during only 5% of such measurements. Therefore, the decision level count rate is

$$DLR = 40/5 = 8 \text{ min}^{-1}$$

For the purpose of this standard, the decision level activity and concentrations are defined as

$$DLA = \frac{DLR}{K} \tag{2}$$

and

$$DLC = \frac{DLR}{V \times Y \times I \times K} \tag{3}$$

where

[14]The development of the decision-level and minimum detectable count rates assumes a minimum background of 70 counts. Refer to [AB5] and [AB6] for additional information and general equation applicability for background counts of less than 70 counts. In addition, it should be noted that the equations provided assume equal counting intervals for the sample and background.

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DLA	is the decision level activity
DLC	is the decision level concentration
K	is the detector calibration factor (min^{-1}/Bq or s^{-1}/Bq)
V	is the sample mass or volume
Y	is the chemical yield
I	is the decay or ingrowth factor (correction for radioactive decay or ingrowth to reference date)

A.7.5 *A priori* minimum detectable count rate

Type II error is associated with failure to detect net activity when, in fact, such activity is present in the sample. It is clearly impossible to determine the probability (β) of committing a Type II error without specifying the activity of a sample since, on the one hand, it is very unlikely that a sample with an extremely high activity will yield a net count rate less than the *DLR*, but very possible that net activities barely above background level may go undetected on the other.

This standard defines the minimum detectable count rate (*MDR*) as the mean (expected) count rate of samples having a 5% probability of escaping detection. More specifically, if the mean net count rate for a sample (as determined, in principle, from the results of a very large number of repeated measurements) is the *MDR* value, then there is a 5% likelihood that the observed net count rate in a single counting of the sample will be less than the *DLR*. (It is important to note that the decision point for detectability lies at the *DLR*—the *MDR* is *not* the threshold for detection.) Assuming, as previously, that the count rate distributions are approximately Gaussian, one finds that the minimum detectable count rate is given by

$$MDR = \frac{2.71}{T} + 2 \times DLR \tag{4}$$

$$MDR = \frac{2.71}{T} + 3.29 \times S_0$$

$$MDR = \frac{2.71}{T} + 4.65 \times S_B \text{ for paired observations}$$

where

T	is the sample counting time
DLR	is the decision-level count rate defined in A.7.4
S_B	is the standard deviation of the background (or appropriate blank) count rate for the sample counting time *T* (paired observations)
S_0	is the standard deviation of the net count rate of a sample containing no analyte

It should be noted that the equations in this subclause, and A.7.4 and A.7.6, are the most basic and simplified equations that are typically quoted and utilized. However, it is recommended that the more detailed and expansive equations for the more sophisticated applications, as found in ANSI N13.30-1996, [AB4], [AB5], [AB6], [AB10], and [AB11], be used when deemed appropriate.

Example:

Again assuming, as in the previous example, a background count rate of 60 min^{-1} and a 5 min counting time, one obtains:

$$MDR = \frac{2.71}{T} + 2(8) = 16.5 \text{ min}^{-1}$$

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In summary, if the decision-level count rate is taken to be 8 net counts per minute, then

— The probability of incorrectly concluding that radioactivity above the level of an appropriate blank is present when counting a sample with no analyte is 5% (Type I error), and
— The probability of incorrectly concluding that no net radioactivity is present when counting a sample yielding, on the average, 16.5 net min^{-1} is also 5% (Type II error).

A.7.6 *A priori* minimum detectable activity and concentration

The *MDR* is expressed in net counts per minute (or second), but an equivalent form of detection limit that is stated in activity or concentration units is frequently of interest. This standard recommends that the terms "minimum detectable activity" (*MDA*) and "minimum detectable concentration" (*MDC*) be used to describe these parameters. These parameters should be estimated by converting the *MDR* into activity or concentration units, typically by use of equations such as the following:

$$MDR = \frac{MDR}{K} \tag{5}$$

and

$$MDC = \frac{MDR}{V \times Y \times I \times K} \tag{6}$$

where

K is the detector calibration factor (min^{-1}/Bq or s^{-1}/Bq)
V is the sample mass or volume
Y is the chemical yield
I is the decay or ingrowth factor (correction for radioactive decay or ingrowth to reference date)

A.7.7 *A priori* and *a posteriori* concepts and information

This standard recognizes all detection limits (*DLR*, *MDR*, *MDA*, *MDC*, etc.) as *a priori* (before the measurement) concepts. However, the estimation of these quantities requires specification of nominal values of a number of parameters (background count rate, count time, estimated interferences, chemical recoveries, decay times, etc.) and, of course, it may be necessary to revise these nominal assessments at various times. This is particularly true for the appropriate blank count rate, since, by definition, the true appropriate blank for a measurement process includes estimates of the nominal levels of any interferences that may be present in a sample batch.

In a number of situations, regulatory limits or contract specifications may require that the measurement process meet or exceed certain *MDC* limits for the sample batch of interest. Such a requirement may demand that the nominal background count rate and other parameters involved in the estimation of the detection limit be assessed for each sample or sample batch. Since these determinations require that some preliminary measurements be made, one finds that the assessment of *a priori* detection limit parameters for future measurements may require the knowledge of *a posteriori* information concerning the nominal characteristics of the sample batch gained from preliminary measurements.

Again, however, this standard finds no value in making such nominal assessments on an individual sample basis, that is, this standard finds no merit in the estimation of a "sample-specific *MDC*." Indeed, even if the "true" *MDC* were somehow known, a sample precisely at the *MDC* limit of concentration would have a (50% likelihood of being measured below the *MDC* limit. The question of whether the sample contains net

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activity is best answered by comparing the measurement result to the decision level or considering the confidence interval for the measurement result, not by comparing the result to the estimated *MDA* or *MDC*.

A.7.8 Ancillary recommendations

This standard offers the following recommendations as natural extensions to the discussion of detection limit parameters in A.7.7.

a) The *MDC* or "less than *MDC*" should never be recorded in the database in lieu of a concentration measured to be "less than *MDC*" since this practice, in addition to providing an indefensible report of the individual measurement, creates an uncorrectable bias in the long-term trends determined from a database containing such censored data
b) Similarly, the *MDC* should never be reported to the client in lieu of low-level measurements
c) The measured activity or concentration should be reported with its associated total propagated uncertainty estimate but without comparison to the estimated *a priori MDA* or *MDC*
d) The total uncertainty propagation algorithm for each measurement process should be developed as fully as possible since the confidence interval it produces is the best information available concerning an individual measurement and its "accuracy," "worth," etc.

A.8 Reporting results by the service laboratory

Unless otherwise stated in a contractual agreement, service laboratory analytical results for each sample shall be reported to the customer and shall include the following items:

— Sample identifying code or information
— Reference date and time for the reported results (e.g., collection date)
— Identification of the specific measurement procedure
— Identification of radionuclide(s) specified by the customer and other radionuclides detected
— Calculated concentration or activity value (whether negative, positive, or zero) using the appropriate blank for each nuclide
— Estimates of the counting uncertainty and total propagated uncertainty (which contains counting and other random and systematic uncertainties)
— Decision level (in units consistent with the counting uncertainty) and nominal *a priori MDC* for each nuclide analyzed
— Identification of the individual responsible for the report

An estimate of the total propagated uncertainty of the reported result shall contain contributions from all parameters of the analytical process.

For data included in written reports

— Uncertainty estimates should be rounded to two significant digits
— Concentration values should be rounded to the same decimal place as the uncertainty estimate
— *A priori MDC* and decision-level values should be rounded to no more than two significant digits (provided the original data will support this significance limit)

The following information should be available and reported to the customer upon request:

— Total volume or weight of the sample submitted and analyzed
— Identification of specific measurement processes and analyst
— Specific analytical parameters, i.e.: chemical yields, count times, decay factors, efficiency of detection, etc.

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— Date, time and place of sampling
— Receipt information
— QC data demonstrating the quality of the measurement process
— Definition of reporting conventions such as “detection limit”

For data retained in electronic storage media, no rounding or truncation of negative values should take place.

For data to be averaged, no truncation or zeroing of negative values should take place.

In addition to or in place of a results report as outlined in this subclause, the client may request that a data package be supplied for data validation purposes. The content of the data package should be established by mutual agreement between the service laboratory and the client prior to initiating any radioassay program. If the data package is required, the client should request it at the time of submission of the samples for which data validation will be performed.

A.9 Record retention by the service laboratory

Laboratory records are classified as “permanent” or “nonpermanent” and shall be retained in accordance with their classification unless otherwise stated in a contractual agreement. Both types of records and their required retention times are discussed in the following paragraphs.

The following is an example list of “permanent” QA records that shall be retained for the life of the project or facility:

— Laboratory procedures and revisions to procedures
— Laboratory data calculation work sheets
— QC sample cross-check work sheets and results
— Nonconformance reports
— Audit/surveillance reports and responses
— Gamma-ray spectra or other instrument printouts
— Documentation of computer programs written to the laboratory data base
— Instrument log books
— Field sheets or sample analysis request forms
— Sample receipt log books (chain of custody)
— Instrument calibration records and data
— Radioactive standard calibration certificates, log books, and disposal records
— Results of analysis of internal QC samples
— Personnel training record;
— Job description and responsibilities
— Procedure testing and verification

The following is an example list of records classified as “nonpermanent” and that need only to be retained for a minimum of seven years and not the lifetime of the project or nuclear plant:

— Audit plans and logs
— Nonconformance logs
— Surveillance plans and logs

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A.10 Test selection

Each service laboratory shall perform all of the analyses requested for that batch of samples, test as well as actual. The contract terms shall include payment for test samples as if they were actual samples. Penalties may be negotiated as appropriate to each contract, including requirements for repeat analysis of test samples. The US DOE Environmental Measurements Laboratory program (see annex B) for monitoring of contractor performance is suggested as a model for inclusion in the contract of analyte selection, repeat analyses, and penalties.

Onsite programs, or those within the same organization (e.g., several laboratories within a DOE facility), may not be able to invoke contractual clauses. However, the "client" within the organization should prepare written specifications based on the guidance provided within this standard and should ensure that an appropriate QA program (including internal and external QC programs) is established to assess the performance of the internal laboratory so that oversight is maintained by the cognizant organizational management. The Savannah River Site and Los Alamos National Laboratory programs (see annex B) are suggested as models for a QA monitoring laboratory within a large facility.

A.11 Bibliography for Annex A

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[AB3] ASTM Special Technical Publication 15D, *ASTM Manual on Presentation of Data and Control Chart Analysis*, 1976.

[AB4] Currie, L. A., "Detection in Analytical Chemistry; Importance, Theory, and Practice," in *American Chemical Society Symposium Series 361*, 1988.

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[AB9] NCRP 58 (1985), "A Handbook of Radioactivity Measurement Procedures," Second Edition.

[AB10] NUREG-1156, Accuracy and Detection Limits for Bioassay Measurements in Radiation Protection, Statistical Considerations.

[AB11] NUREG/CR-4007, Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, Sept. 1984.

[AB12] US Department of Health, Education, and Welfare, Public Health Service # 999-RH15, "Quality Control of Radioactivity-Counting Systems."

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Annex B

(normative)

Examples of reference and monitoring laboratories, and existing measurement assurance programs

The following summaries are provided as examples of programs that now serve as “reference laboratories” (see 4.1.3 and 4.2.2) or as “monitoring laboratories” (see 4.1.4 and 4.2.3), which meet some of the objectives of this standard. See clause 4 and figure 1 for functional descriptions of these two types of QA laboratories.

B.1 EPA laboratory intercomparison studies program

The US Environmental Protection Agency (EPA) has, since October 1962, operated a laboratory intercomparison program [BB3].[15] At first, the program was a service for its own laboratories (then the Public Health Service), which were performing radioanalysis of fallout from atmospheric nuclear weapons tests. For the past 15 years or so, the program has served as the main US resource for performance testing of federal, state, utility, and commercial laboratories that perform radiochemical analyses of environmental samples. The EPA program services a large, mostly voluntary clientele, numbering some 330 laboratories as of 1991. The EPA makes available a variety of radionuclides, concentrations, and matrices; the protocol provides for test samples at a minimum frequency of two per year for some and up to as many as six per year for others. Sample distributions are spaced across the year to smooth the work effort committed to intercomparisons. The EPA reference laboratory (Las Vegas) uses radionuclide standards traceable to NIST, which in turn conforms to the International Reference System maintained at the Bureau International des Poids et Measures (BIPM). Test samples are synthesized by blending the solution standards into various natural or synthetic matrices. Each participating laboratory is expected to perform three independent determinations for each radionuclide included in an intercomparison. The reported data are analyzed to determine the mean value and standard deviation for each laboratory result and to calculate for all laboratories the normalized range, normalized deviation, sample standard deviation, and the grand average. Each participating laboratory receives a complete summary report, with results coded for anonymity.

B.2 DOE QA programs

The US Department of Energy (DOE) uses a variety of QA programs, both external and internal, to evaluate potential sources of bias or uncertainty in the environmental data generated by the contractors at its various facilities. The discussion in the following subclauses highlights some of the QA programs operated by DOE.

B.2.1 Quality Assessment Program (QAP)

The DOE’s Environmental Measurements Laboratory (EML) in New York has since 1976 operated the QAP as an intercomparison program for environmental measurements performed by contractors and subcontractors of DOE’s Office of Environmental Health and Safety and Office of Environmental Restoration and Waste Management. It is believed that this program is the only DOE QA program that functions agency-wide, and so it differs significantly from the QA programs that each DOE contractor uses to monitor its own subcontractors. The QAP program uses nonblind samples with sufficient activity to test the accuracy of each of the contractor laboratories; there were 110 participants in 1993.

[15]These numbers refer to the bibliography in B.6.

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Both synthetic and natural matrix reference materials are used. Filters are spotted with solution standards to simulate distributed air particulates on the filter, and solution standards are mixed into deionized water or pond water to prepare the synthetic intercomparison samples. Soil, vegetation, bone, and animal tissue samples are collected, processed, and analyzed by EML staff for use as natural matrix intercomparison samples; the EML inventory of natural matrix reference materials as of 1984 consisted of eight soils, ten vegetation, and three tissue samples [BB12].

A set of six samples, consisting of two air filters, bone ash or animal tissue, soil, vegetation, and water, is sent semi-annually to each participating laboratory. The radionuclide concentrations are varied from test to test by altering the spike concentration or by blending various combinations of the natural reference materials; samples are checked for homogeneity before they are shipped. A single result for each radionuclide in each sample is reported to EML within three months of receipt of samples. Results are compared principally as ratios to the EML value, although the final report includes reported concentration values as well. A preliminary report is available to each participating laboratory via email within three days of the reporting deadline, and a final printed report follows within a month. The results for each participating laboratory can be attributed directly, because an identification list is provided in each report. DOE field offices are responsible for reviewing the performance of each participating laboratory. An annual workshop was held during the early years of the QAP to evaluate the program and make adjustments; however, despite a consensus that the workshops were valuable, that effort has ceased due to funding limitations.

B.2.2 Internal laboratory QA programs

Most of the DOE facilities operate QA programs to monitor both the laboratories functioning at each facility as well as offsite contractor laboratories. In this manner, each of the DOE laboratories discussed in the following subclauses serves a role much like the one proposed for a “monitoring laboratory.” The following program summaries are provided as examples of the types of programs that others may choose to incorporate into their own QA programs.

B.2.2.1 EML analytical services contractor QC program

Laboratories performing environmental measurements under the DOE’s Office of Energy Research contracts in support of EML’s programs are monitored using double-blind samples. Approximately 15–20% of the samples sent to a contractor laboratory consist of reference, split, or blank samples encoded into the sequence used for the actual samples and disguised to ensure that they are visually indistinguishable from the actual samples. Deposition (rain water) and air filter controls are spiked and are disguised appropriately with dirt or dust. Bone, milk, and food (vegetation) controls are prepared by blending from stocks of ashed reference materials (all actual samples of these types are submitted to contractors as ash). Soil and sediment controls are blended from stocks of dried and homogenized reference materials similar in appearance to the actual samples. Reference materials are normally obtained from NIST or the International Atomic Energy Agency (IAEA). Splits may be prepared coincidentally in a batch or, if decay permits, the splits may be interspersed through several batches over time. The combination of double-blind test samples, known activity levels in some controls, and split samples for other controls permits EML staff to evaluate bias and precision of the analytical effort of the contractor laboratory for each batch of samples with assurance that the bias and precision exhibited for the control samples are representative of that for the real samples in that batch. Contractual payments are predicated on contractor performance on the QA samples submitted with each batch.

B.2.2.2 Los Alamos National Laboratory (LANL)

The philosophy and objectives of the LANL QA program are described in a series of annual reports [BB6], the most recent available being that for 1990. Specific procedures to accomplish the objectives are published separately [BB7]. The analytical chemistry activities of the Health and Environmental Chemistry Group at LANL are diverse and encompass industrial hygiene, environmental surveillance and compliance, bioassay,

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industrial waste-water, and U.S. Transuranium Registry (USTR) tissue studies. The Group's mission for the Laboratory is to provide qualitative and quantitative analytical data to health physicists, industrial hygienists, environmental surveillance scientists, environmental compliance managers, epidemiologists, waste managers, and waste treatment plant operators. One of the fundamental responsibilities of the Group's management is to maintain a continuing program to ensure the reliability and validity of analytical laboratory data. The QA program addresses small, single investigator projects through large-scale facilities monitoring programs requiring year-to-year continuity. The operational philosophy is that a QA program should apply to all aspects of sample and information management. The program begins at the conception of a project, follows through collection and storage of samples before analysis, includes all phases of chemical and physical analyses, extends through interpretation and data entry into the Group's laboratory information management system (LIMS), and culminates with the final reporting of results.

Two concepts are used in the QA program to provide confidence in each analytical result reported by the laboratory: quality control, the mechanism established to control errors; and quality assessment, the system used to verify that the entire analytical process is operating within limits considered acceptable. Consistent use of good laboratory practice, standard operating procedures and protocols, qualified personnel, reliable and well-maintained equipment, appropriate calibrations and standards, and close supervision of all operations are primary components of the QC system. Quality assessment describes the techniques used to assess the quality of the analytical process, principally through the use of a system of control charts, which are plots of multiple data points from the same or similar samples versus time. The charts are used to determine if the system is in a state of statistical control and to visualize and monitor the relative variability of repetitive data. The control charts are also used with reference materials and spiked samples to assess the accuracy of measurements.

The LANL approach is based firmly on the concept of traceability through concurrent analysis of matrix-based certified QA materials. Laboratory policy is to maintain traceability to NIST. Traceability is not achievable in all cases, because NIST has limited resources for certification of matrix-based standard reference materials (SRMS) for as many elements and compounds as are needed at LANL. This restraint has lead LANL in two directions. First, a large library of reference materials has been obtained from other national and international agencies [EPA, U.S. Geological Survey (USGS), DOE EML, Canadian Geological Survey (CGS), Canadian Certified Reference Materials Project (CCRMP), IAEA, Centre de Recherces Petrographiques et Oeochimiques, and the Japanese Ecological Survey] in addition to the available NIST biological and environmental SRMS. Secondary QC materials are prepared in-house. Second, literature data are compiled on the reference materials so that consensus values for constituent concentrations that are not agency certified may be developed. Over 300 reference materials are available, some of which have certified or consensus values for as many as 80 constituents. Two data bases, SRMS and REFS, are used to permit random collection of literature data on reference materials. At any time, this collection may be sorted as a function of any logical combination of parameters and printed for determination of a consensus value.

Concurrent analysis of reference materials of similar matrix is performed with every batch of samples. In this fashion, both precision and accuracy may be assessed, and traceability to a national standards laboratory is maintained for every batch. Because the compositional properties of these reference materials simulate those of the sample routinely measured, their correct measurements can imply correct measurement of routine samples. If sufficient quantities of reference materials are not available, the laboratory develops as many of its own secondary materials, traceable to agency reference materials, as possible. Participation in intercomparison studies is a necessary, but not sufficient, component of this QA program, because the external samples appear too infrequently to ensure that day-to-day control is maintained. LANL strives to run a minimum of 10% of all analyses as matrix-based QA reference materials, although for some determinations this level of quality assurance has proved to be prohibitively expensive. Details of how the QA program is implemented are provided in [BB6].

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B.2.2.3 Savannah River Site (SRS)

In 1988, a comprehensive QA Improvement Program [BB11] was established for the radiological environmental monitoring programs at the Savannah River Site. The primary objective of quality assurance is to ensure that data generated by the environmental monitoring program at the site are accurate, precise, and defensible. The SRS QA Program is built into the routine environmental monitoring activities. To implement an effective QA program, the SRS Environmental Monitoring Section (EMS) focused on the following elements: developing specific QA activities for all EMS work groups; requiring that all EMS personnel receive QA training through courses tailored for the SRS Environmental Monitoring program; instituting a "Quality Indicators" program that routinely highlights the progress of key QA activities within each EMS work group, a customized data tracking system and statistical data validation program, and a newly developed Quality Assessment Program (QAP) for monitoring the performance of subcontract laboratories.

In the area of environmental sample collections, a new QC environmental collection team was given responsibility for special routine evaluation of sample collection sites with respect to identification and implementation of QA enhancement. This evaluation program allows analysts to identify redundant sampling equipment at key sampling locations, maintain a more aggressive preventive maintenance program, and use field control samples to measure sampling techniques. Other specific QA activities that enhance the overall program include internal audits, control charts, and control samples. A focus on the standard operating procedures (SOP) programs in environmental monitoring work groups has also been developed.

All EMS personnel complete QA training that is custom-designed for the group. The training emphasizes the theory and routine application of nationally recognized QC procedures and QA/QC concepts specific to their job duties. Special emphasis was made on the regulations that govern SRS environmental programs including DOE Order 5700.6B/NQA-1, EPA-600/8-83-024 (QAMS-004/80), and EPA-600/4-83-004 (QAMS-005/80) (see [BB1], [BB4], [BB5], and [BB13]).

To achieve a total quality environment, a system called "Quality Indicators" was developed to focus and evaluate the SRS QA program. The concept of quality indicators includes development of a set of measurable indicators for each work group and visually displaying and monitoring these indicators against a quality goal.

EMS uses a QA/QC data tracking system that was developed in-house. This system automatically retrieves and reports QA/QC sample data in near real time for immediate and constant surveillance of the quality of work performed by the analysts in the environmental chemistry laboratories. The system compares values to historical data and control limits, and performs a trend analysis that is required by DOE orders. Routine samples are automatically flagged, and a trend analysis report is produced if control limits are exceeded. This system reduces the response time needed to assess any potential environmental contamination from days or weeks to hours.

EMS also uses a data validation computer program to review radioactive concentrations of both effluent and environmental samples. The results are compared, via site-specific computer software, against historic levels, established limits, and control locations. Statistical tools including goodness of fit tests, control charting, probability plotting, and numerical and graphical summaries are used to alert management to potential changes in the environmental baseline and to monitor sample results for regulatory purposes.

The QAP for monitoring subcontract laboratories performance consists of three types of evaluations. First is a performance evaluation, where samples consisting of QC standards and duplicate, blind, and split samples are sent to each subcontract laboratory quarterly. Second, a statistical evaluation is performed using the newly developed mean relative difference (MRD) statistic. This statistic evaluates the difference between lab duplicate and blind samples for "intra"-laboratory quality assurance/quality control and the difference between split samples for "inter"-laboratory quality assurance/quality control. Finally a performance evaluation is routinely conducted, which involves an onsite audit.

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The QAP for subcontract laboratories has resulted in more productive laboratory audits. Continuous monitoring of MRD statistics and QC standards proficiency results has allowed previously unidentified problems in a subcontract laboratory to be identified prior to scheduled QA/QC audits.

This Savannah River Site EMS QA program, which is built on statistical foundations and laboratory training, has led to improved overall performance of EMS and each of its subcontract laboratories. EMS can now continually monitor its in-house and subcontract laboratories performance and assure that the reported data are both accurate and defensible.

B.3 World Health Organization (WHO) International Reference Center for Radioactivity

The WHO International Reference Center (IRC) for Radioactivity has, since 1970, operated an international QA program [BB14]. Synthetic samples or natural matrix samples are collected, processed, and shipped by IRC staff at the laboratory of the Service Central de Protection Contre les Rayonments Ionisants (Le Vesinet, France). Usually, two intercomparison samples are sent annually, although not necessarily at the same time. Results are analyzed against the known value in the case of synthetic samples, or against the mean (outliers excluded) for natural-matrix samples.

Reports to participating laboratories include results for all participants coded for anonymity, with comparative tables and bar graphs for each laboratory; the IRC laboratory value is usually provided as a reference, even for natural-matrix samples. Reports are usually mailed four to six months after sample shipment (one or two months after the data-reporting deadline).

Scheduling is somewhat irregular; a water sample has been sent annually, a milk sample approximately every other year, and a soil, vegetation, or animal flesh/bone specimen as the “filler” sample in the set of samples dispensed at four two-year intervals. Standards and blanks are available with most intercomparison sets, so a participating laboratory can update its standardization at the time of processing. Thus, this program serves as a means of upgrading capability as much as it does for intercomparison of results. Typically 20 to 30 laboratories participate in each intercomparison, although for some of the more difficult samples as few as 10 laboratories (out of 20 to 30 requesting) will report results.

B.4 IAEA Analytical Quality Control Service (AQCS)

The IAEA provides its AQCS program for the primary purpose of assisting laboratories to check and improve the quality of their work [BB10]. Natural matrix materials are used both as known reference sources and as unknowns for intercomparison purposes. Typically, once a particular sample is characterized by intercomparison, it is later offered by the AQCS program as a reference material. There is no charge for participating in an intercomparison study, but reference materials have to be purchased. Three analytical results per radionuclide per sample have been accepted, although six analyses per radionuclide per sample are requested. Data reports include a brief description of the analytical method used by each participating laboratory. The mean and standard deviations are calculated for the results from each participating laboratory, and the mean is compared against the grand average for purposes of intercomparison (outliers are excluded). Ultimately, the grand average value becomes the reference value when that sample is offered as a reference material. The IAEA laboratories at Siebersdorf, Austria, and at Monaco offer standards that conform with the BIPM; however, the standard reference materials that are calibrated through intercomparison are linked to the BIPM only to the extent that the participating laboratories are themselves in conformance with BIPM. Summary reports from AQCS to participating laboratories often becomes available only several months to a year after distribution of the intercomparison samples. Intercomparison results are summarized by laboratory code in order to maintain anonymity.

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The sporadic scheduling of intercomparisons for samples of greatest interest and the slowness of feedback tend to make the AQCS program of lesser value than the others as a quality assessment program. On the other hand, the wide variety of natural-matrix samples and radionuclides make this a valuable program for testing or developing new analytical methods.

B.5 Nuclear power utilities

B.5.1 Yankee Atomic Environmental Laboratory

The measurement QA program at the Yankee Atomic Electric Company's Environmental Laboratory (YAEL) has been designed to accommodate the various analytical processing and monitoring laboratory functions performed for its sponsoring utilities. At the present time, YAEL provides radiochemical processing and QA services for six New England nuclear power utilities: radioassays of samples from each utility's radiological environmental monitoring program (REMP); radioactive effluent measurements (10-CFR Part 50) and radioactive waste stream profiles (10-CFR Part 61); and quarterly QA programs for plant (radio)chemistry and whole body counting (in vivo bioassay) functions. Due to the complexity of its operation, the YAEL currently functions as both a monitoring and a service laboratory.

There are two QA committees to oversee the quality of the services of the laboratory; the Laboratory Quality Control Audit Committee (LQCAC), which is comprised of a senior-level technical representation of the sponsoring utilities, and the Laboratory Quality Assurance Review Committee (LQARC), which is an internal committee comprised of technical management and the QA officer of the Laboratory. The LQARC meets quarterly to review the status of the internal and external QA programs, to establish QA policies and performance criteria for acceptance by the LQCAC, and to review and resolve audit findings. The LQCAC meets semiannually and performs an annual detailed technical audit of all Laboratory processes. Additional QA audits and program reviews are conducted internally by YAEC's Quality Assurance Department and externally by the various regulatory agencies, such as the US Nuclear Regulatory Commission and the US Federal Emergency Management Agency.

The performance evaluation portions of YAEL's measurement assurance program for its radiochemical processing and QC testing functions are illustrated in figures B.1 and B.2. The laboratory maintains NIST traceability through its active participation in four national programs—the Nuclear Energy Institute (NEI)-NIST Nuclear Industry Measurement Assurance Program established in 1986, the NRC/NIST traceability program, and the EPA/NIST traceability program (see B.1), and a third-party commercially available quarterly measurement assurance program for specific media and nuclides. The NEI/NIST program [BB8] is the principal method of traceability for YAEL's various activities. This program establishes and maintains indirect traceability via bimonthly performance testing of single-blind (known nuclides of unknown quantities and interferences) samples of various media (typically liquid, gases, and air filter matrices) and radionuclides. The indirect traceability testing schedule is established annually by the Steering Committee of the NEI/NIST program, a committee comprised of a representative of each sponsoring member or utility. Direct traceability, wherein YAEL sends prepared samples whose radioactive content has been quantified by YAEL to NIST for verification, can also be conducted on a limited basis. Since the number of radionuclides and media are limited, this program has a limited applicability to 10-CFR Part 50 and 10-CFR Part 61 effluent and waste stream processing measurement assurance programs.

The NRC maintains a measurement assurance program [BB8] as part of its official inspection program of the effluent sampling and monitoring program at the utility. Gaseous and liquid effluent samples at the YAEL's sponsoring utilities are analyzed by the NRC, and analytical results are compared against regulatory performance criteria scaled for the various radionuclides and media. The plants also submit aliquots of the split liquid samples to YAEL for radionuclide processing as part of this NRC measurement assurance program. Intercomparison feedback to the utility occurs through an official inspection report, usually within several weeks of the inspection. The NRC maintains both a direct and indirect traceability program with NIST and

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the Radiological and Environmental Sciences Laboratory at Idaho Falls, ID under an interagency contract ([BB2] and [BB9]).

In addition to the NRC intercomparison program, the YAEL also participates in a commercially available, quarterly QA program for the pure beta-emitting radionuclides of ^{89}Sr, ^{90}Sr, and ^{55}Fe in simulated liquid effluent samples. The samples are prepared to include interfering activation and fission product nuclides in ratios typically found in nuclear plant operations.

The Laboratory maintains a QA program plan that outlines annual performance evaluation and internal audit schedules. With the exception of reagent and media blanks and instrumentation background checks, the YAEL submits, on the average, a sufficient number of process QC samples corresponding to about 5% of the anticipated sample processing load. The QA staff prepare any additional internal process QC samples for media and nuclides not included in the external QA programs, i.e., service laboratory internal QA functions. Feedback of individual QC test results are provided to the analytical processing staff in a timely fashion. Updates of graphical trending of processing nuclides occurs monthly, and compilation of all QA results occurs semiannually. Corrective action requests for results not meeting established YAEL quality performance criteria for accuracy and precision are issued to management by the QA officer concomitantly with the individual results feedback. In addition to the internal MQA, the sponsoring utilities maintain an external MAP by submitting double-blind duplicate or replicate samples for processing.

The YAEL functions as a monitoring laboratory by administrating quarterly QA programs for the sponsoring utilities in which test media are prepared and verified by laboratory staff. These programs were established based on charters developed and agreed upon by the participants and have quality performance criteria for accuracy and precision and implementation protocols. Gaseous, liquid, air particulate filter, and charcoal media are spiked with various traceable radionuclides, verified of content and distributed to the Chemistry Departments at the power plants. Tabular and graphical quarterly compilations of coded results (for anonymity) from program participants are provided for intercomparitive purposes. For the collaborative in-vivo QA program, organs spiked with traceable radionuclides are prepared, loaded into a reference phantom, and distributed to the utilities for evaluation.

B.5.2 Houston Lighting and Power

The Houston Lighting and Power (HL&P) Radiological Laboratory (RL) is an off-site radioanalytical and QC laboratory supporting the South Texas Project Electric Generating Station (STPEGS). The RL is responsible for the STPEGS Radiological Environmental Monitoring Program (REMP) and the Interlaboratory Radioassay Measurement Assurance Program (MAP) along with other analytical/technical services. As a result of the RL's complex relationship with onsite laboratories, it functioned in the past as a service, monitoring, and reference laboratory. Currently, the analytical functions of the RL are limited to the service and monitoring functions.

The STPEGS consists of two 1250 MW pressurized water reactors, which went online in 1988 and 1989. Onsite radioassay laboratories include two Chemistry Laboratories, two Health Physics Laboratories and one Whole Body Counting Laboratory. Organizationally, the RL is part of the same operations organization as the site laboratories for which it provides QA services (e.g., MAP). The RL, however, operates with the necessary autonomy and authority, and with full management support of its QA charter. Further assurance of program effectiveness is provided by approved administrative and technical procedures that define the relationships between the RL, higher level laboratories (e.g., NIST, EPA) and the onsite STPEGS laboratories.

There are two primary QA assessment processes that review the RL:

a) The Nuclear Assurance Department, which performs routine task surveillances and an annual comprehensive audit that includes the input of a technical specialist

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b) A comprehensive quarterly internal audit process, which includes a summary report to management regarding the status of the RL

Additional QA assessments are performed less frequently by outside organizations such as the American Nuclear Insurers (ANI), the Institute of Nuclear Power Operators (INPO), and the NRC.

The analytical capabilities of the RL are continuously being evaluated via a comprehensive internal QA program. Figure B.3 illustrates the performance evaluation mechanisms associated with the RL. The RL maintains NIST measurement traceability via the NEI/NIST MAP, and therefore that program is regarded as the principal method of NIST traceability. Other sources of measurement assurance are the EPA Interlaboratory Comparison Program and a collective group consisting of interutility QC programs and vendor supplied SRMs, including matrix equivalent SRMs. Figure B.3 shows that onsite laboratories participate in the NRC Confirmatory Measurements Program. As a STPEGS affiliated laboratory, the RL could also be tested by the NRC. However, to date such an evaluation has not taken place. Currently the STPEGS MAP provides the most comprehensive and demonstrable performance evaluation of onsite measurement capabilities. This is accomplished by quarterly tests of media and nuclide(s), which annually consist of approximately 20 analysis categories (e.g., ^{131}I in lapel charcoal, ^{89}Sr and ^{90}Sr in AP filter). The reported results are evaluated for accuracy and precision, and a final report documenting the test is sent to the participating laboratories, management, and records.

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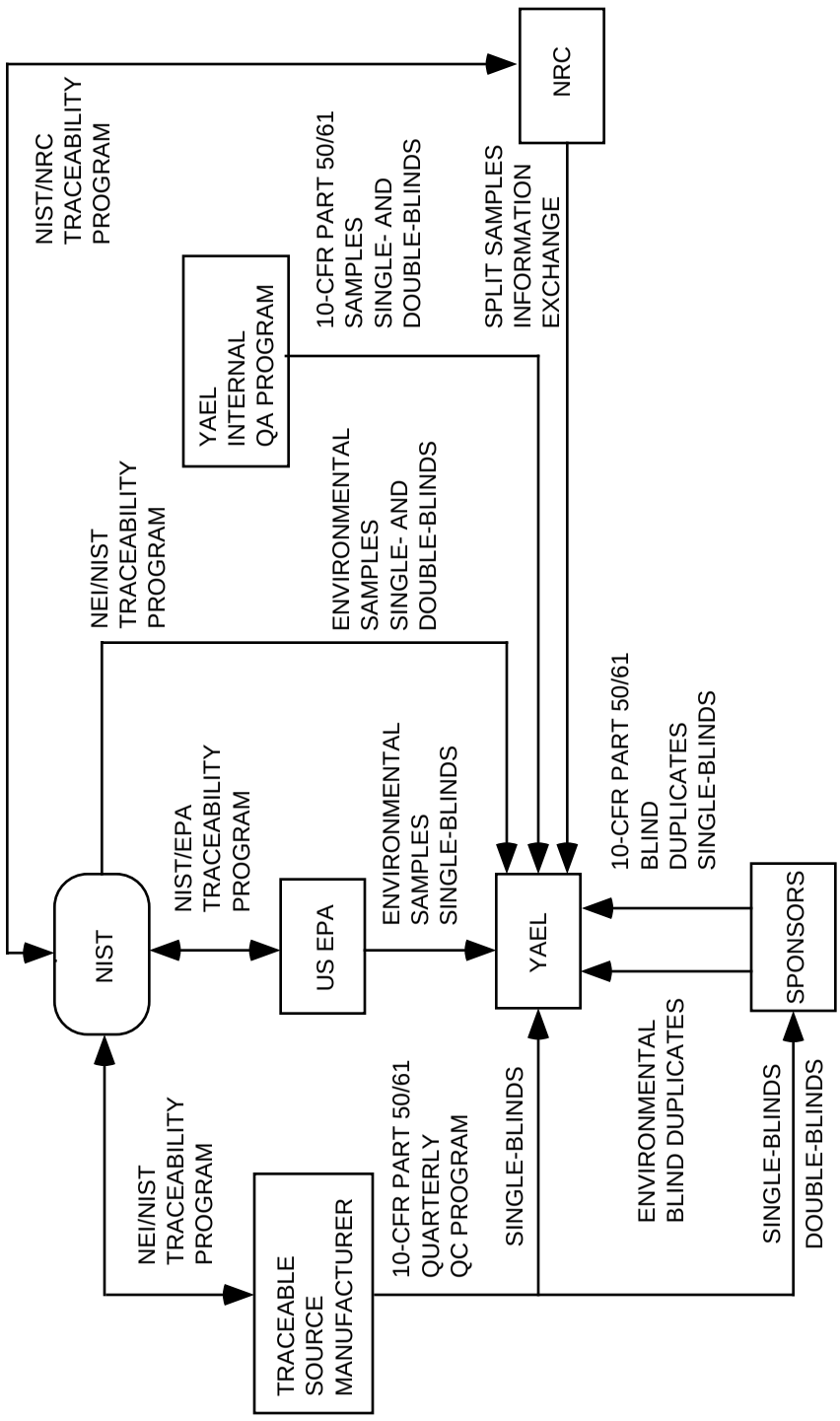


Figure B1—Yankee Atomic Environmental Laboratory (YAEL): NIST traceability and measurement assurance programs for radioassay functions

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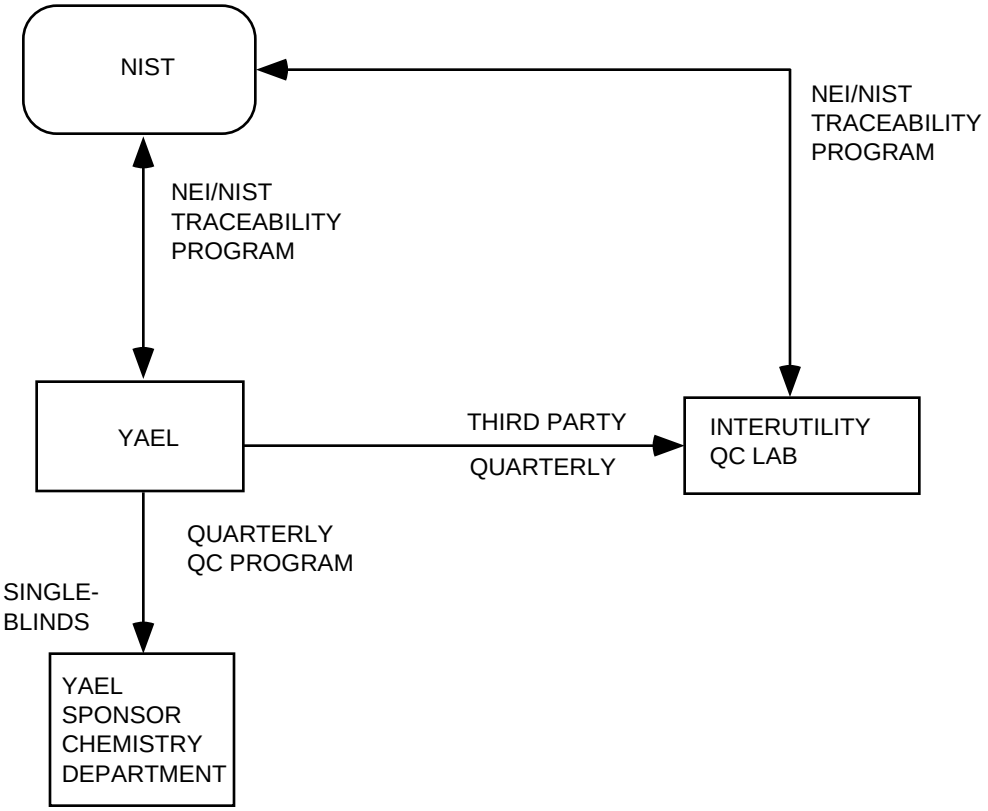


Figure B2—Yankee Atomic Environmental Laboratory: NIST traceability for the preparation of radioactive performance evaluation materials

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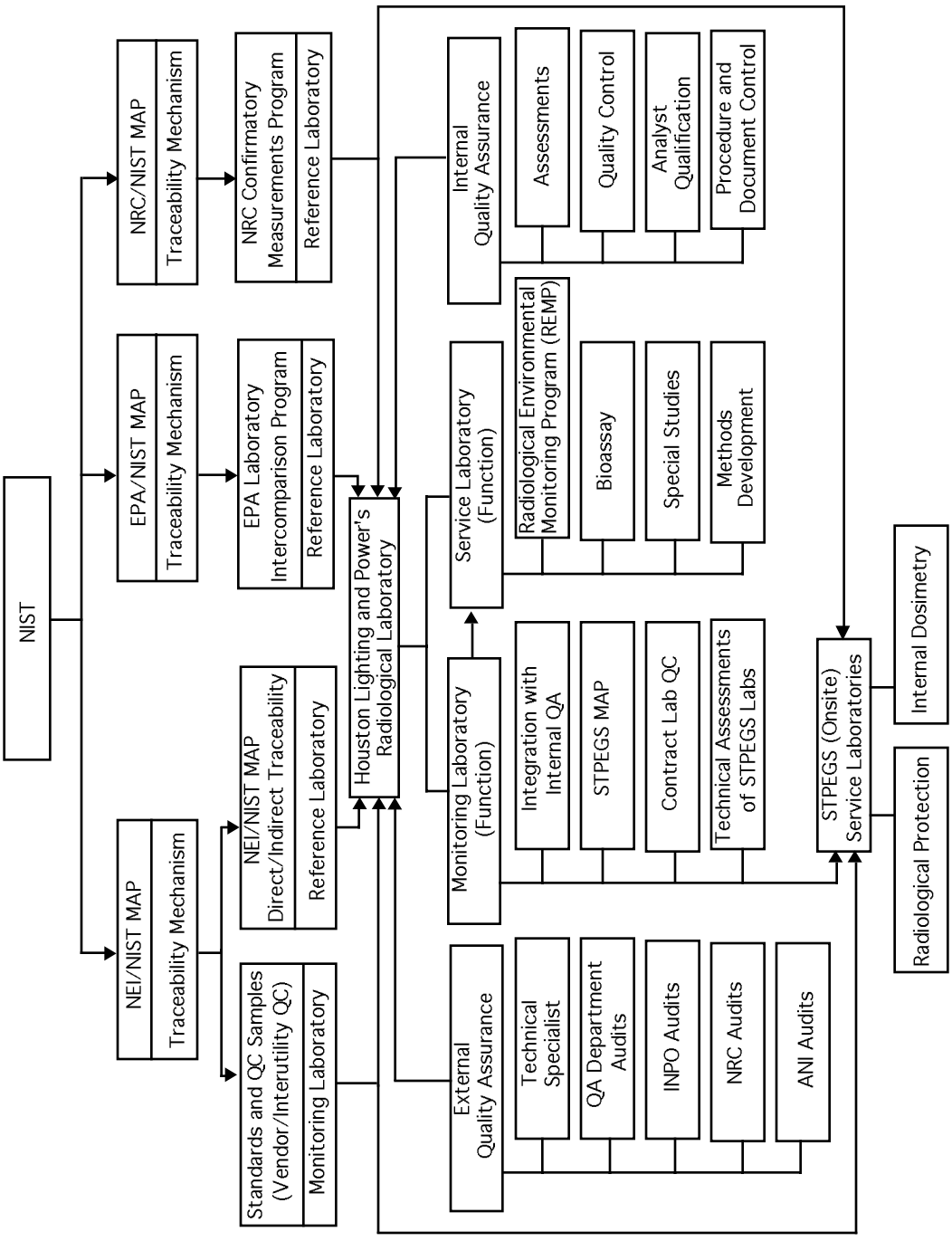


Figure B3—Houston Lighting and Power Company's radioassay measurement assurance program

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Annex C

(informative)

Guidance on QA investigations and corrective actions related to radioassay work

Investigations and corrective actions are essential elements of any QA program. According to ASME NQA-1b-1991, corrective actions consist of five basic elements.

a) Identification and documentation of conditions adverse to quality
b) Classification of conditions adverse to quality—impact on reliability, regulatory compliance, etc.
c) Cause analysis—determine and document root cause of significant conditions adverse to quality
d) Corrections—determine and implement actions necessary to correct conditions adverse to quality
e) Follow-up—verify and monitor that the corrective actions are effective

These basic elements have been evaluated, interpreted, and expanded upon as to their applicability to radioassay processes. The following are elements of an investigation and corrective action program that would be applicable to radioassay laboratories:

— Problem description
— Cause of the deficiency (root cause analysis)
— Investigation to determine if problem is generic and may be prevalent in other radioassay processes
— Corrective action to prevent recurrence
— Management review of the problem and actions taken
— Personnel accountability
— Management commitment

The essential elements for “root cause” investigations related to radioassay work include the following:

— Review all paperwork, workbooks, or worksheets and validate the following for appropriateness: radioassay procedure, equipment usage and settings, detector calibrations, efficiencies and backgrounds, chemical reagents and carriers, and calibration/tracer solutions.
— Verify all calculations, equations, and software impacting data.
— Verify radiochemical recovery, proper nuclear instrumentation background, or reagent blank subtraction. Are these parameters within the expected range based on historical data for the technique, analyst, and instrumentation? Does the reagent blank contain radionuclide or chemical interferences? If so, is the reagent blank activity or chemical interference due to cross contamination, impurities in the radiotracer, or the reagents? Can the reagent blank interferences be subtracted from the sample or batch of samples of concern?
— If radionuclide interferences are possible due to the sample matrix, verify insufficient decontamination steps by analyzing the final matrix for other nuclides; e.g., review the total alpha spectrum for possible interfering peaks or counts in the region of interest (ROI), and analyze final matrix of beta emitting nuclides [liquid scintillation (LS) vials or planchets] on a gamma spectrometry unit to reach appropriate sensitivity based on the ratio of the beta to gamma efficiencies. If applicable, review the total beta LS spectrum for possible interferences into the ROI.
— Review previous QC data and trends for the nuclide and matrix. Have there been repetitive problems or is there a progressive trend?
— Review all personal or processing reports/logs for noted processing difficulties. Note any difficulties consistent with trends for the technique, matrix, or analyst. Personnel factors should include, but limited to, qualification status and the analyst’s level of attention to detail.

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— Review scheduled proficiency tests for the analyst/nuclear instrumentation staff. Were the results marginal, just meeting performance criteria?
— For long-lived nuclides, re-count the final matrix to verify the original result. This count should be longer than the first if the nuclide concentration is near the MDC. The results should overlap at two times the total propagated uncertainty (TPU) range.

Interim corrective actions may be necessary when the “root cause” of a problem cannot be immediately found and a detailed procedure analysis has to be performed. For example, if the gravimetric recovery of a technique is giving erroneous results, the laboratory may want to switch to a radiotracer to determine a radiometric recovery. Use of the gravimetric recovery data would be discontinued until the root cause of the gravimetric recovery problem was determined. This would be considered an interim corrective action, since the root cause has not been fully determined and the discovery/investigation process will require an extensive parametric evaluation.

The proper implementation of a corrective action to a root cause results in

— A solution that prevents recurrence
— A solution that is within the control of the lab
— A solution that allows the lab to meet other quality, production, or economic goals

The form shown in figure C1 is an example of a mechanism that may be used to identify and resolve analytical deficiencies observed on any type of sample, actual or quality control. The deficiency report can be generated by anyone along the chain-of-custody in the laboratory, from sample receipt to final data report, and follows the sample data sheets throughout the laboratory. If a paper data collection and reporting system is used, the form should be of a highly visible color such as fluorescent yellow or pink. If a computerized data system is used, the appropriate deficiency(ies) and resolution(s) can be keyed to appear with each data set, but again should be made highly visible to the analysts, reporters, and data recipients.

The keyed numbers in this example are out of positional sequence, because as previously unconsidered deficiencies were brought to light, they were added in time sequence to avoid confusion for earlier data sets. A laboratory starting anew with this type of deficiency identification system may choose to develop a more organized key to deficiencies.

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SAMPLE STATUS AND QUALITY CONTROL RECORD

Sample # _____ Analysis _____

Indicate any deficiency in the processing of this sample by circling the appropriate number. Further explanation may be entered under Comments. Forward to your supervisor for disposition. The supervisor should attach this form to the deficient analysis worksheets and forward to the appropriate group for correction of the deficiency. If a result has been calculated by computer, return the computer analysis report slip with the deficiency noted, to data processing. If worksheets are removed from the data processing file, they must be replaced with a Data Removal Sheet.

DEFICIENCY

1 sample exhausted
2 lost in transit
3 leaked in transit
38 delayed receipt
4 insufficient sample
6 insufficient information
34 improperly collected
37 suspected mislabeling
9 sample loss suspected
15 undesignated collection problem
7 lab accident
20 processing difficulty
19 questionable purity
13 low yield (< 40%)
14 high yield (> 95%)
36 carrier aliquoting
35 tracer aliquoting
25 poor mounting
32 sample aliquoting
18 undesignated lab problem
33 cross-contamination
23 counter malfunction
26 poor plating
31 improper geometry
8 short count
28 poor resolution
24 excessive quenching
11 efficiency out-of-date
12 background out-of-date
17 undesignated counting problem
27 analysis deleted
10 poor statistics
29 data transcription error
30 calculation error
22 computer problem
21 replicate nonagreement
5 results differ from expected value
16 undesignated data processing problem
39 not reported
40 disaster

COMMENTS (use reverse side if more space is needed)

_____
_____
_____
_____

DISPOSITION

_____ Check calculation
_____ Check for counter malfunction
_____ Recount
_____ Repeat chemistry

_____
_____

Initiated by _____
Date initiated _____

ROUTING

1. _____
2. _____
3. _____

Figure C1—Sample status and QC record

Annex D

(informative)

Data quality objectives applicable to radioassays

Goals and expectations are established for all radioassay projects. However, many times the goals and expectations are ill conceived and not communicated to the analyst successfully. The resulting costly analyses will likely produce data that will not re-analyze or abandonment of the project altogether. For resource conservation, timely project completion, and attainment of project goals, it is imperative that the project issues are identified, evaluated, and discussed and a clear consensus understanding be established by the project participants (e.g., regulator, management, project coordinator, sample collectors, analyst, data reviewers, and budget analyst). The team also needs to be intimately involved in designing the project goals and expectations, planning the analytical process, establishing criteria for the quality of resulting data, and designing a program that will assure the data quality.[18] In order to establish effective radioassay quality objectives, consideration should be given to the following subclauses.

D.1 Project goals development process

Develop the project goals according to the following design parameters, facts, elements, or condiserations:

a) Collect, review, and evaluate existing information
b) Evaluate relevant regulations/guidance for impact on the project (e.g., action limits)
c) Identify variables
d) Establish the scope of project
e) Establish objectives
f) Identify obstacles and develop strategies to negotiate the obstacles
g) Establish project priorities
h) Establish project expectations
i) Establish rules for judging the progress and success of the project
j) Establish confidence (uncertainty) limit for key indicators of project progress and success
k) Design data collection
l) Prepare work plan
m) Re-evaluate progress, action plan, and decision tree periodically to optimize program

D.2 Analytical process and data quality plan

Develop the data quality and analytical process plan according to the following:

a) Establish purpose/objective/scope/priorities of analytical operations
b) Establish management structure and responsibilities for analytic program and project, samples, and data
c) Identify and prioritize target analytes and matrices
d) Establish acceptable levels of precision, bias, and analytical uncertainties
e) Establish sampling, sample preservation, subsampling, and storage criteria
f) Establish number of samples and rate that samples will be provided to the laboratory
g) Establish method selection and validation criteria

[18]Blacker, S. M. and Maney, J. P., “The System DQO Planning Process,” *Environmental Testing and Analysis*, July/Aug. 1993, pp. 46–50.

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h) Establish reporting format and requirements
i) Establish requirements for defensibility of project data
j) Develop a cost management program

D.3 QA program design

Develop a QA program according to the following design parameters:

a) Design a MQA program that envelops the dynamic range of the analytical requirements,
b) Design the onsite assessment process
c) Design the performance evaluation testing program to include
    1) Objective of performance evaluation testing
        i) Generic testing (calibrations)
        ii) Project specific testing (analyte, concentration, interferences, matrices, method/sensitivity)
    2) Tiered performance evaluation testing for traceability
        i) Testing mechanism
        ii) Blind testing
        iii) Conformational analyses testing
    3) Quality assurance/quality control of the test materials

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