

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

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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., bioassay, 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, radiometrology

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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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# 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

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

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.<sup>1</sup>

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.<sup>2</sup>

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.<sup>3</sup>

IEEE Std 1012-1986 (Reaff 1992), IEEE Standard for Software Verification and Validation Plans.<sup>4</sup>

NBS IR 89-4125, Operational And Technical Requirements For Laboratory Accreditation Program For Personnel Dosimetry Processors, July 1989.<sup>5</sup>

## 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]<sup>6</sup> are not defined in this standard.

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

<sup>2</sup>ASME publications are available from the American Society of Mechanical Engineers, 22 Law Drive, Fairfield, NJ 07007, USA.

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

<sup>4</sup>IEEE publications are available from the Institute of Electrical and Electronics Engineers, 445 Hoes Lane, P.O. Box 1331, Piscataway, NJ 08855-1331, USA.

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

<sup>6</sup>The numbers in brackets preceded by the letter B correspond to those of the bibliography in clause 6.

### 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  $i$ th 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.

**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.

**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  $\beta$  probability of nondetection while accepting a probability  $\alpha$  of erroneously detecting that radionuclide in an appropriate blank sample. For this standard, the  $\alpha$  and  $\beta$  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.

**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

**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  $i$ th 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.

**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

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.

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#### 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<sup>7</sup> 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

<sup>7</sup>Selected samples may be needed to establish homogeneity of the material.

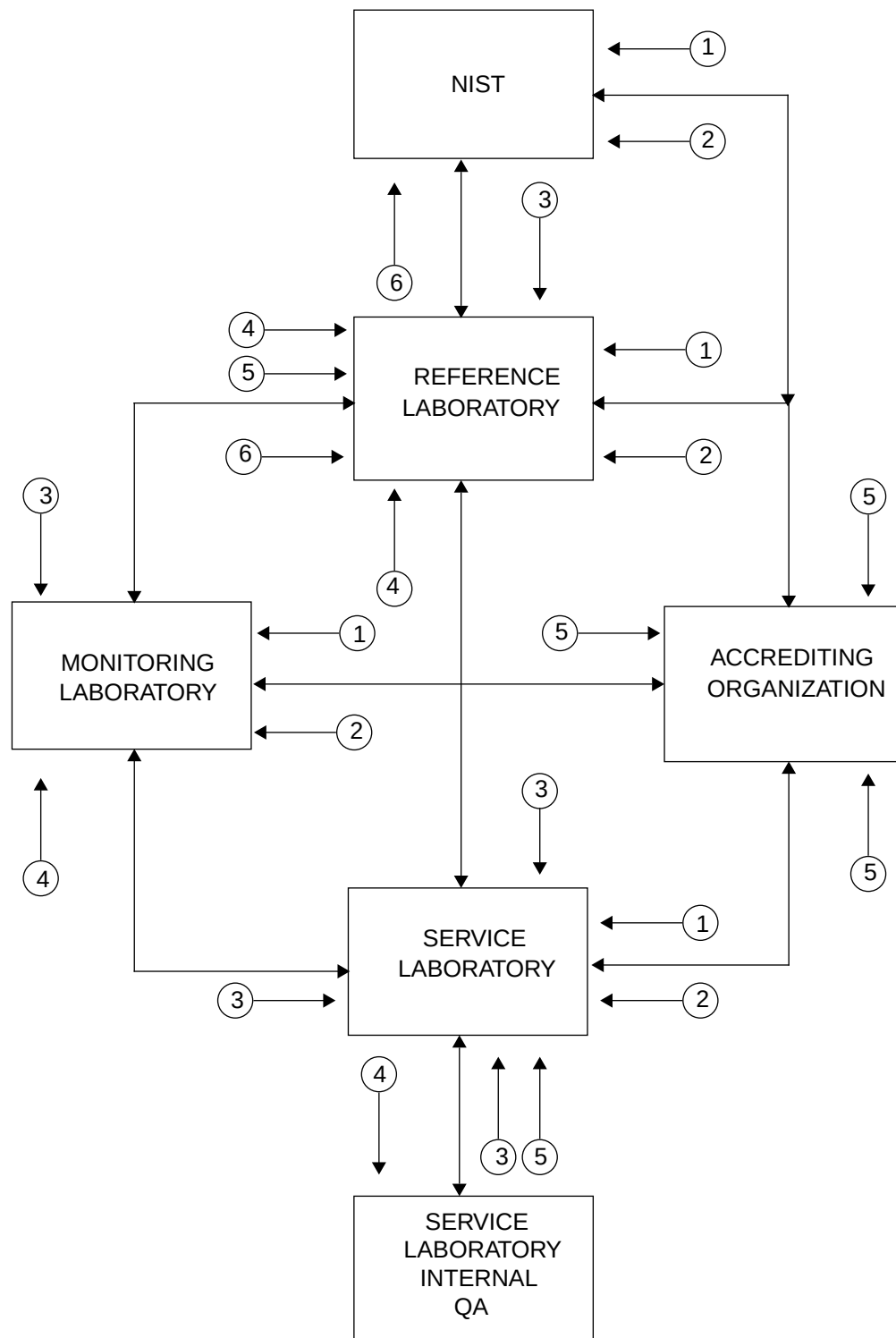


Figure 1—Conceptual diagram of the national performance testing program

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<sup>8</sup>) and from generic criteria developed previously for the third-party certification pro-

<sup>8</sup>Information on references can be found in clause 2.

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)<sup>9</sup> 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

<sup>9</sup>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.