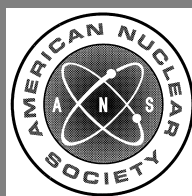


American Nuclear Society

**verification and validation of radiological
data for use in waste management
and environmental remediation**

an American National Standard



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**American National Standard
Verification and Validation of Radiological Data for
Use in Waste Management and Environmental Remediation**

Secretariat
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**American Nuclear Society
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Working Group ANS-41.5**

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Foreword

(This Foreword is not part of American National Standard “Verification and Validation of Radiological Data for Use in Waste Management and Environmental Remediation,” ANSI/ANS-41.5-2012.)

The American Nuclear Society (ANS) Nuclear Facilities Standards Committee (NFSC) is responsible for development of American National Standards Institute (ANSI) standards for nuclear facilities, including criteria and operations required for environmental remediation of nuclear facility sites that have become contaminated. The ANS Subcommittee on Decommissioning and Site Remediation Standards manages the development and maintenance of standards that address the cleanup of radioactive materials and radioactivity mixed with hazardous substances. This subcommittee has authorized a working group to develop a new ANSI/ANS standard, ANSI/ANS-41.5-2012, for verification and validation of data from radiological analysis supportive of waste management and environmental remediation. The verification process will be called compliance verification in this standard. Compliance verification may involve compliance to written analytical specifications (e.g., statement of work, contract, project plans) but may also involve compliance to programmatic or project-specific requirements. Therefore, this standard does not assume or require that the laboratory work be performed under contract.

This standard provides requirements and recommended practices for determining the validity of radioanalytical data for waste management and environmental remediation. These applications will include site characterization, waste acceptance, waste certification, waste treatment design, process control, litigation, and other applications as deemed necessary. This standard will provide a minimum set of checks and tests that will ensure a consistent approach for compliance verification and validation of data produced by any radioanalytical laboratory. This standard should eliminate many of the inconsistencies in the approaches, evaluation algorithms, parameters evaluated, and qualifiers used in existing site-specific data compliance verification and validation programs.

The requirements of this standard apply only to independent compliance verification and validation processes and should not be construed to apply to any actions taken by laboratories to internally generate or review data, including audits and performance evaluation studies. Other standards are available that provide quality and performance requirements for radioanalytical laboratories [e.g., ANSI N42.23-1996 (R2003), “Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories”]. However, this standard expects that certain laboratory quality control (QC) and programmatic quality assurance (QA) measures have taken place that feed data for review by the data verifiers and validators. The interface of these QA/QC measures with the compliance verification and validation process will be discussed in Sec. 3 of this standard. Since this standard does not place requirements on laboratories or on waste management and environmental remediation programs, the points of interface in Sec. 3 are necessarily listed as only recommendations, and all requirements are contained in Secs. 4 through 7 of this standard.

While this standard will provide a minimum set of checks and tests for compliance verification and validation of data, the acceptance criteria for the test and checks are intentionally not provided in most cases. This is because each waste management or environmental remediation program or project may have unique measurement quality objectives (MQOs) based on the intended use of the data. This standard has been developed with the assumption that a proper data quality objective (DQO) process has been used by the project to define the quality of data needed for the decision process and to develop corresponding MQOs of

accuracy, precision, sensitivity, selectivity, and representativeness to be met. Therefore, set limits for QC parameters and many other acceptance criteria will not be recommended in the standard, but rather the user will be referred to the limits established by the DQO process. This approach will allow data qualification to be based on how factors such as error, bias, lack of precision, lack of sensitivity, or lack of selectivity affect the decision process. The DQO process should also provide guidance for the frequency, percentage, and extent of data validation. This standard will incorporate an evaluation of data end use and action levels throughout the qualification process. This approach will prevent unnecessary rejection of data for minor quality problems.

This standard contains four appendices, which are informative and contain additional description of the relationship of the programmatic and laboratory QA/QC measures with the compliance verification and validation processes. This foreword is not part of the standard.

This standard might reference documents and other standards that have been superseded or withdrawn at the time the standard is applied. A statement has been included in Sec. 8, "References," that provides guidance on the use of references.

This standard does not incorporate the concepts of generating risk-informed insights, performance-based requirements, or a graded approach to QA. The user is advised that one or more of these techniques could enhance the application of this standard.

The working group would like to gratefully acknowledge the contributions by Jason C. Jang, who died prior to the completion of this standard.

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Verification and Validation of Radiological Data for Use in Waste Management and Environmental Remediation

1 Purpose and scope

1.1 Purpose

This standard¹⁾ specifies verification and validation criteria and processes for determining the validity of radioanalytical data for waste management and environmental remediation. These applications include site characterization, waste acceptance, waste certification, waste treatment design, process control, litigation, and other applications requiring data verification and validation. This standard provides a minimum set of checks and tests to be performed by a reviewer independent from the laboratory data generator. These checks and tests will ensure a consistent approach for independent verification and validation of data produced by any radioanalytical laboratory. This standard should eliminate many of the inconsistencies in the approaches, evaluation algorithms, parameters evaluated, and qualifiers used in existing site-specific data verification and validation programs.

1.2 Scope

This standard establishes criteria and processes for verification and validation of radioanalytical data for waste management and environmental remediation activities. It applies to the independent review of the data generation process for field measurements and radioanalytical laboratories. This standard sets the requirements for how the data are reviewed and qualified against the data quality requirements [e.g., measurement quality objectives (MQOs)] that are established by the project to meet their intended use. While this standard does not specifically address all nondestructive assays and in situ measurements, the general principles and some of the elements of

this standard may apply. This standard does not address nonradioassay measurement methods (e.g., inductively coupled plasma–mass spectroscopy, kinetic phosphorescence analysis, X-ray diffraction).

2 Acronyms and definitions

2.1 List of acronyms

ANSI: American National Standards Institute

APS: analytical protocol specification

CLP: contract laboratory program

COC: chain of custody

CSU: combined standard uncertainty

DER: duplicate error ratio

DL: decision level

DOE: U.S. Department of Energy

dpm: disintegrations per minute

DQA: data quality assessment

DQO: data quality objective

L_c : critical level

LCS: laboratory control sample

LCS %D: laboratory control sample percent difference

LLD: lower limit of detection

MARLAP: Multi-Agency Radiological Laboratory Analytical Protocols Manual

MDC: minimum detectable concentration

¹⁾The current standard, ANSI/ANS-41.5-2012, is hereinafter referred to as “this standard.”

MQO: measurement quality objective

MSD: matrix spike duplicate

MSS: matrix spike sample

MSS %D: matrix spike sample percent difference

NIST: National Institute of Standards and Technology

PE: performance evaluation

PT: performance testing

QA: quality assurance

QAPP: quality assurance project plan

QC: quality control

RDL: required detection level

RPD: relative percent difference

SAP: sampling and analysis plan

SOW: statement of work

TPU: total propagated uncertainty

2.2 Definitions

accuracy: A concept employed to describe the dispersion of measurements with respect to a known value. The result of a measurement is “accurate” if it is close to the true value of the quantity being measured. Inaccurate results may be caused by imprecision or bias in the measurement process.

action level: The numerical value that causes the decision maker to choose one of the alternative actions. The action level may be a derived concentration guideline level, background level, release criterion, regulatory decision limit, etc. The action level is often associated with a particular matrix/analyte combination. (Note that the action level is specified during the planning phase of a data collection activity; it is not calculated from the sampling data.)

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 analysis category but may include multiple target analytes.

analytical protocol specification (APS): The output of a project planning process that contains the project’s analytical data needs and requirements in an organized, concise form.

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. Also see “desk audit.”

background: Ambient signal response due to spurious electronic noise or incidental radiation in the vicinity of the detector system as recorded by measuring instruments that is independent of radioactivity contributed by the radionuclides being measured in the sample.

batch: A group of samples prepared at the same time, in the same location, using the same method, and by the same analyst.

bias: A fixed deviation from the true value that remains constant over replicated measurements within the statistical precision of the measurement. Synonyms are deterministic error, fixed error, and systematic error.

calibration: The set of operations or processes conducted under specified conditions that establish the relationship between values indicated by a measuring instrument or system and the corresponding known values. The term “calibration” refers to both the first calibration after the instrument is placed in use and to any recalibrations subsequently performed.

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 issued by a certifying body [e.g., National Institute of Standards and Technology (NIST), International Atomic Energy Agency].

combined standard uncertainty (CSU): The standard (1σ) uncertainty of a calculated result obtained by propagating the standard uncertainties of a number of input values of the

measurement process. The value is sometimes referred to as total propagated uncertainty (TPU).

compliance verification: Compliance verification is the process of determining whether the data are complete, correct, consistent, and in compliance with established standards or written analytical specifications [e.g., statement of work (SOW), contract, project plans]. The process of compliance verification is independent of validation. The compliance verification is conducted at various levels both internal and external to the data generator. The output of verification is a data set ready for data validation.

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

critical level (L_c): See “decision level.”

data quality assessment (DQA): The last phase of the data collection process, which consists of a scientific and statistical evaluation of the data set to assess its validity and usability. The focus of DQA is the evaluation of the data relative to their intended use.

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

decision level (DL) (also L_c): The minimum measured analyte quantity or concentration (a posteriori result) required to give a stated confidence that a positive amount of the analyte is present. For this standard, the stated confidence level will be assumed to be 95%. Correspondingly, the probability of a Type I error (probability of erroneously concluding a radionuclide is detected in a sample that is blank) is set at 0.05. However, other confidence levels may be established by the MQOs.

desk audit: An off-site or remote review of laboratory-submitted documents.

duplicate: A second aliquot of the sample (equally sized, prepared, and analyzed as part of the same batch) used to measure the overall precision of the sample measurement process beginning with laboratory subsampling of the field sample.

holding time: The elapsed time expressed in days from the date of collection (rather than receipt by the laboratory) of the sample until the date of analysis.

laboratory control sample (LCS) A standard material of known composition, or an artificial sample (created by fortification of a clean material similar in nature to the environmental sample), that is prepared and analyzed in the same manner as the environmental sample.

matrix spike sample (MSS): An aliquot or aliquant of a sample spiked with a known concentration of target analyte(s) prior to sample preparation. The recovery of the target analyte(s) from the MSS is used to determine the bias of the method in the specific sample matrix.

measurement quality objective (MQO): Quantitative or qualitative statements of performance objectives or requirements for a particular method performance characteristic such as the method uncertainty, detection capability, range, specificity, ruggedness, etc. The MQOs may be viewed as the analytical portion of the DQOs and are, therefore, project- or program-specific.

method blank: A prepared sample of a matrix as similar as practical to the associated samples that is free, to the extent possible, of the radionuclides of interest that is carried through the entire analytical process to evaluate potential contamination from the measurement process for determination of the DL and the MDC. The method blank may also be used to determine the standard deviation of the net blank.

minimum detectable concentration (MDC): The minimum quantity or concentration of a radionuclide required (a priori) to give a stated confidence that the measurement result would be above the DL (detected). For this standard the stated confidence level will be 95%. Correspondingly, the probability of a Type II error (probability of erroneously not concluding a radionuclide is detected in a sample that has the MDC quantity or concentration) is set at 0.05. For this standard the alpha (Type I) and beta (Type II) probabilities are both set at 0.05.

performance check: A check of the response (efficiency, energy, and/or background) of a detection system to determine if changes have occurred since the last time the system was calibrated.

performance criteria: The established level of quality (bias, precision, detection sensitivity, etc.) and operational commitments (turnaround times, reporting protocol, etc.) that are (a) agreed upon between the service laboratory and the customer (or intergovernmental agencies or intracompany entities) with written analytical specifications and (b) established by the service laboratory and documented within the operational or QA program manual of the laboratory.

performance testing (PT) sample: Reference materials or samples of known composition used to evaluate the performance of the laboratory.

precision: The degree of agreement or central tendency of repeated measurements of the same parameter. A measurement with small random uncertainties is said to have high precision.

quality assurance (QA): All those planned and systematic actions necessary to provide adequate confidence that an analysis, measurement, or surveillance program is of the type and quality needed and expected.

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

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.

quality control chart: A chart developed to evaluate the response of an instrument or process to predetermined, statistically based control limits. The predetermined statistical limits are not typically developed using the overall quality performance (bias and precision) parameters for an analytical technique.

radionuclide: An isotope of an element that is radioactive.

reference material (standard): A material or substance one or more properties of which are sufficiently well established (within specified uncertainty limits) to be used for the cal-

ibration of an apparatus, assessment of a measurement method, or assigning of values to materials.

replicate: One of multiple aliquots or aliquants of a sample taken during the first stage of the analytical process.

required detection level (RDL): The minimum detection capability for a method required by the MQOs and/or SOW.

sample: A single item or specimen from a larger whole or group taken for the purpose of estimating properties or composition of the larger whole or group.

sampling: The process of obtaining representative samples and/or measurements of a subset of a population.

shall, should, and may: 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.

standard deviation: The square root of the variance of a 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 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).

tolerance chart: A chart developed to evaluate the response of an instrument or process to a predetermined tolerance level as determined by an appropriate QC source. The predetermined tolerance level, typically expressed as a percentage, 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 as specified by the MQOs and is related to the instrument calibration.

total propagated uncertainty (TPU): see “combined standard uncertainty.”

traceability: Demonstrated linkage by means of an unbroken chain of comparisons of a measurement to nationally or internationally recognized standards or certifying body within specified uncertainty limits.

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

validation: A technically based analyte- and sample-specific evaluation process that extends beyond method or written analytical specification (e.g., SOW, contract, project plans) compliance, provides a level of confidence that an analyte is present or absent, and examines the uncertainty of the reported concentration of the analyte relative to the intended use of the data. Data validation is a systematic process, performed externally from the data generator, that applies a defined set of performance-based criteria to a body of data that may result in qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

validator: An individual that performs the validation process and meets the qualifications for validator defined in Sec. 7.

verifier: An individual that performs the compliance verification process and meets the qualifications for verifier defined in Sec. 7.

3 General principles

3.1 Data life cycle

The goal of the data collection process is to produce credible and cost-effective data to meet the needs of a particular project. The data life cycle provides a structured means of considering the major phases of projects that involve data collection activities. The three phases of the data life cycle are planning, implementation, and assessment. These three phases consist of a number of routinely performed activities including planning the project and developing project plan documents; writing a SOW; sampling; and conducting analyses, audits, performance evaluation (PE) studies, com-

pliance verification, data validation, and DQA. This standard has been developed with the assumption that a proper DQO process (see EPA/600/R-96/055 [1]²⁾) has been used by the project to define the quality of data needed for the decision process and to develop corresponding MQOs of accuracy, precision, sensitivity, selectivity, and representativeness to be met. Compliance verification and data validation are sometimes considered isolated processes, but the efficiency and success of the validation effort are heavily dependent on the completion of the preceding steps in the data collection process. Compliance verification compares the data to the requirements of the written analytical specifications (e.g., SOW, contract, project plans). Data validation evaluates the data produced and/or adequacy of the methods applied against the MQOs and other APSs developed during the planning process. DQA, the third and final step of the assessment phase, compares the data produced to the overall project DQOs. A detailed description of the data life cycle is provided in Appendix A.

3.2 Planning documents

Planning documents include items such as QAPP, data validation plan, work plan, and/or sampling and analysis plan (SAP). Specifications for format, content, and style of planning documents are outside the scope of this standard. Other guidance is available for the development of these plans. Brief descriptions are included in Appendix A. This standard anticipates that these project planning documents are generated to direct the analytical work performed by any type of laboratory. For example, “Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)” [2] (hereinafter referred to as MARLAP)—which was developed by representatives from eight federal agencies including the U.S. Environmental Protection Agency, the U.S. Department of Energy, the U.S. Department of Homeland Security, the U.S. Nuclear Regulatory Commission, the U.S. Department of Defense, the U.S. Food and Drug Administration, the U.S. Geological Survey, and NIST and provides guidance for any project or activity including site characterization, site cleanup, decommissioning, remedial and removal

²⁾ Numbers in brackets refer to corresponding numbers in Sec. 8, “References.”

actions, etc., that requires radioanalytical data for decision making—addresses the issue of documenting the laboratory analytical requirements. The following is stated in MARLAP, Vol. 1, Chapter 5, “Obtaining Laboratory Services”: “MARLAP recommends that technical specifications be prepared in writing in a single document, designated an SOW, for all radioanalytical services, regardless of whether the services are to be contracted out or performed by an Agency’s laboratory.”

3.3 Data validation plan

The data validation plan is either a stand-alone document or part of other planning documents. The contents of a typical data validation plan are provided in Appendix A.

3.4 Audit items germane to the validation process

When audits are conducted to assess a laboratory’s capability to meet proposed or written analytical specifications (e.g., SOW, contract, project plans), specific information gathered during the audit is anticipated by this standard to be used in the compliance verification and validation processes rather than having that information be provided within data packages. See Appendix A for a typical way of using this information. Desk audits may be included in the validation process if included in the validation plan. For details on a quality audit program, see MARLAP, Vol. 1, Chapter 7, “Evaluating Methods and Laboratories.”

3.5 Use of external PE program results

Various guidance documents (ANSI N42.22-1995 (R2002) [3], ANSI N42.23-1996 (R2003) [4], and Regulatory Guide 4.15 [5]) recommend that a laboratory participate in an external PE program. Most laboratory contracts, SOWs, or Project QAPPs require successful participation in a PE program.

A more detailed discussion of the use of this type of information is presented in Appendix A.

3.6 Compliance verification

Compliance verification is the systematic process of checking data for completeness, correctness, consistency, and compliance with written analytical specifications (e.g., SOW, contract, project plans). The compliance verification pro-

cess compares the laboratory data package to requirements associated with the project and produces reports that identify those requirements that were and were not met. These requirements are expected to be contained in the SOW or project planning documents (e.g., QAPP and SAP). The inputs to the compliance verification process include field data, PE sample results, audit information, and calibration data in addition to the analytical data package. The information expected to be contained in the analytical data package is discussed in Appendix A.

Compliance verification tools evaluate a data set against a standard or written analytical specifications. Problems identified through data compliance verification may be separated into categories of correctable problems and noncorrectable problems, which are defined below.

Correctable problems may be subdivided into two subcategories. The first subcategory contains those correctable problems relating to deficiencies in data packages that may be addressed by obtaining additional information from the laboratory. The second subcategory of correctable problems consists of those that may be solved by either re-preparation and/or reanalysis of a sample.

Noncorrectable problems are those for which data cannot be regenerated.

Assessment of analytical laboratory deliverables against the SOW or other planning document requirements is used as a real-time evaluation tool for general analytical laboratory operations. The result of compliance verification is a verified data package (raw data package and verification report or checklist) that is then passed to the validator for validation.

3.7 Validation

Validation is the process of examining a verified data package to provide a level of confidence in the reported analyte’s identification, concentration (including detectability), and associated measurement uncertainty. Validation is analyte- and sample-specific and extends beyond the method or written analytical specification (e.g., SOW, contract, project plans) compliance. Validation produces a data set with a limited number of qualifiers associated with