Revision 1
February 1980

U.S. NUCLEAR REGULATORY COMMISSION

REGULATORY GUIDE

OFFICE OF STANDARDS DEVELOPMENT

REGULATORY GUIDE 5.58

CONSIDERATIONS FOR ESTABLISHING TRACEABILITY OF SPECIAL NUCLEAR MATERIAL ACCOUNTING MEASUREMENTS

A. INTRODUCTION

Part 70, "Domestic Licensing of Special Nuclear Material," of Title 10 of the Code of Federal Regulations requires that for approval to possess and use more than one effective kilogram of special nuclear material (SNM)[1] the licensee must provide an adequate material control and accounting system. Section 70.51, "Material Balance, Inventory, and Records Requirements," requires licensees to calculate material unaccounted for[2] (MUF) and the limit of error of the MUF[3] value (LEMUF) following each physical inventory and to compare the LEMUF with prescribed standards. Section 70.58, "Fundamental Nuclear Material Controls," requires licensees to maintain a program for the continuing determination of systematic and random measurement errors and for maintaining control of such errors within prescribed limits. Section 70.57, "Measurement Control Program for Special Nuclear Materials Control and Accounting," provides criteria for establishing and maintaining an acceptable measurement and control system.[4] Reference 1 describes the technical and administrative elements that are considered to be important in a measurement control program.

Implicit in the criteria stated in §70.57 is the requirement of traceability of all SNM control and accounting systems to the national standards of measurement as maintained by the National Bureau of Standards (NBS) by means of reference standards.

Reference standard is defined in §70.57(a)(3). *Traceability* is defined in §70.57(a)(4). These definitions are clarified as follows: *Traceability* means the ability to relate individual measurement results to the national standards of measurement through an unbroken chain of comparisons. *Reference standard* means a material, device, or instrument whose assigned value[5] is known relative to the national standards of measurement.

This guide presents conditions and procedural approaches acceptable to the NRC staff for establishing and maintaining traceability of SNM control and accounting measurements. No specific methods will be presented herein since the methodology to be used for any given measurement must be tailored to the needs and peculiarities of the relevant process material, reference standards, instrumentation, and circumstances. Rationales and pertinent analytical factors will be presented for consideration as to their applicability to the measurement at hand.

B. DISCUSSION

1. BACKGROUND

SNM measurements for control and accounting are performed on a great variety of material types and concentrations, with a diversity of measurement procedures, by a large number of licensees at all the various industrial, research and development, and academic facilities involved. Accurate, reliable measurements are necessary to achieve valid overall accountability. To this end, all measurement systems must be compatible with the national standards of measurement through the national measurement system (NMS). To obtain this necessary compatibility for any SNM measurement task, reference materials appropriate for each SNM type and measurement system may be required. Table 1 defines the various types of reference materials.

Traceability is a property of the overall measurement, including all Certified Reference Materials (CRMs), instruments, procedures, measurement conditions, techniques,

*Lines indicate substantive changes from previous issue.

[1] For definitions, see paragraphs 70.4(m) and (t) of 10 CFR Part 70.

[2] Currently called inventory difference (ID).

[3] Currently called the limit of error of the inventory difference (LEID).

[4] The listed regulations do not apply to special nuclear materials involved in the operation of a nuclear reactor or in waste disposal operations or used in sealed sources. See paragraphs 70.51(e), 70.57(b), and 70.58(a) of 10 CFR Part 70.

[5] The term "value" includes instrument response and other pertinent factors.

Table 1

TYPES OF REFERENCE MATERIALS

Type	*Definition*	*Example*
Reference Material (RM)	A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus or for the verification of a measurement method.* A generic class of characterized homogeneous materials produced in quantity and having one or more physical or chemical properties experimentally determined within stated measurement uncertainties. This term is recommended for use instead of "standard" or "standard material."	Any or all of the materials listed below.
Certified Reference Material (CRM)	RM accompanied by, or traceable to, a certificate stating the property value(s) concerned, [and its associated uncertainty,] issued by an organization, public or private, which is generally accepted as technically competent.*	Any primary or secondary certified reference material (see below).
Primary Certified Reference Material (PCRM)	A certified reference material of high purity possessing chemical stability or reproducible stoichiometry and generally used for the development/evaluation of reference methods and for the calibration of RMs. Primary certified reference materials are certified using the most accurate and reliable measurement methodologies available consistent with end-use requirements for the RM.	Standard Reference Materials of the National Bureau of Standards (NBS SRMs), materials of the International Atomic Energy Agency (IAEA) bearing the IAEA classification "S", and certified reference materials from the Department of Energy New Brunswick Laboratory.
Secondary Certified Reference Material (SCRM)	An RM characterized relative to a primary certified reference material generally used for development/evaluation of field measurement methods, for day-to-day intralaboratory quality assurance, or for interlaboratory comparison programs. SCRMs may be less pure or less stable than PCRMs, depending on their intended end use. Accuracy required of the certifying measurements also depends on intended end use.	Some Reference Materials available from the Department of Energy New Brunswick Laboratory. IAEA Reference Materials classification "R".
Working Reference Material (WRM)	An RM characterized relative to a primary or secondary certified reference material usually for use within a single laboratory or organization. WRMs are generally used to assess the level of performance of measurements on a frequent (e.g., daily) basis. WRMs are usually prepared from material typical of a given process. (Previously known as Working Calibration and Test Materials (WCTMs).)	Process stream materials and any RM prepared according to References 8, 9, 10, and 11 and related reports; IAEA's intercomparison exchange samples.

* This definition is that used by ISO Guide 6-1977(E) of the International Standards Organization.

and calculations employed. Each component of a measurement contributes to the uncertainty of the measurement result relative to national standards of measurement through the NMS. The NMS is composed of a number of components, including the NBS (which has the responsibility for maintaining the national standards of measurement), CRMs, national laboratories, calibration facilities, standards-writing groups, national standards, and the person making the ultimate measurement.

If the NBS, as the legal caretaker of the national standards of measurement for the United States, is viewed as an entity capable of making measurements without error, traceability can be defined as the ability to relate any measurement made by a local station (e.g., licensee) to the "correct" value as measured by the NBS. If it were possible for the NBS to make measurements on the same item or material as the local station, this relationship, and hence traceability, could be directly obtained. Since such direct comparisons are not ordinarily possible, an alternative means for achieving traceability must be employed. This necessary linkage of measurement results and their uncertainties to the NBS through the NMS may be achieved by:

a. Periodic measurements by the licensee of CRMs or Standard Reference Materials (SRMs). The measurement, *per se*, of an SRM or CRM without rigorous internal control of measurements does not provide the necessary linkage. Adequate and suitable reference materials, along with reliable measurement methods and good internal measurement assurance programs, are necessary to ensure accuracy (Ref. 1).

b. Periodic measurements of well-characterized process materials or synthesized artifacts that have been shown to be substantially stable and either being homogeneous or having small variability of known limits. The uncertainties associated with the values assigned to such process materials or artifacts are obtained by direct or indirect comparisons with Primary Certified Reference Materials (PCRMs).

c. Periodic submission of samples for comparative measurement by a facility having established traceability in the measurement involved, employing one or both of the above procedures, and involving only samples not subject to change in their measured values during storage or transit. ("Round-robin" sample exchanges between facilities can be useful in confirming or denying compatibility of results, but such exchanges do not of themselves constitute the establishment or maintenance of traceability.)

Valid assignment of an uncertainty value to any measurement result demands a thorough knowledge of all the observed or assigned uncertainties in the measurement system, including an understanding of the nature of the sources of these uncertainties, not just a statistical measure of their existence. It is not sufficient, for example, to derive a root-mean-square value for a succession of observed or assigned uncertainties (CRM, instrumental, and procedural) for which standard deviation values have been calculated by statistical methods for random events. To do so involves assumptions as to the randomness of these variances that may not be at all valid. The variances may, in fact, be due to a combination of systematic errors that appear to be randomly distributed over the long run but that are not at all random in their occurrence for a given analyst employing a given combination of standards, tools, and instruments. Thus, it is necessary to derive the uncertainty value of a measurement from methods that also involve a summation of the nonrandom (systematic) uncertainties, not from the mathematics of random events alone. *The valid determination of the uncertainty of a measurement relative to the NBS, and thus of the degree of traceability, is not a rigorous procedure but is the result of sound judgment based on thorough knowledge and understanding of all factors involved.*

Obviously, the effects of systematic error can be reduced if Reference Materials (RMs) are included at least once in every series of related measurements by a given analyst and combination of tools, instruments, and conditions. The calibration and correlation factors so obtained cannot be applied uncritically to successive measurements. It also follows that the applicability of any given RM to a series of measurements of process material should be examined critically both periodically and with every change or hint of change in the measurement characteristics of the process material.

It is doubtful that RMs can ever be exact representations of the material under measurement in any given instance, even for highly controlled process materials such as formed fuel pieces or uniform powdered oxide shown to be substantially uniform in both composition and measurement-affecting physical characteristics (e.g., density or shape for nondestructive assay (NDA) measurements). However, in most cases RMs that yield measurement uncertainties within the selected limits for the material in question can be achieved. Obviously, the errors resulting from mismatch of the RM with the measured material will be largest in heterogeneous matter such as waste materials, but in these cases the SNM concentrations normally will be low and the allowable limits of uncertainty correspondingly less stringent.

The important truth being stressed here is that *every measurement must be considered, in all aspects, as an individual determination subject to error from a variety of sources, none of which may be safely ignored.* The all-too-natural tendency to treat successive measurements as routine must be rigorously avoided. Test object and device RMs, in particular, tend to be mistakenly accepted as true and unvarying, but they may well be subject to changes in effective value (measured response) as well as unrepresentative of the samples unless wisely selected and carefully handled.

The characteristics required of CRMs include:

a. Sufficiently small and known uncertainties in the assigned values. (Normally, the uncertainties of the CRMs will contribute only a small fraction of the total uncertainty of the measurement.)

b. Predictability in the response produced in the measurement process. (Ideally, the measurement process will

respond to the RMs in the same way as to the item or material to be measured. If there is a difference in measurement response to the measured parameter arising from other measurement-affecting factors, these effects must be known and quantifiable.)

c. Adequate stability with respect to all measurement-affecting characteristics of the standard. (This is necessary to avoid systematic errors due to changes in such properties as density, concentration, shape, and distribution.)

d. Availability in quantities adequate for the intended applications.

It cannot be assumed that RMs will always remain wholly stable as seen by the measurement system employed, that working RMs will forever remain represesentative of the measured material for which they were prepared or selected, or that the measured material itself will remain unchanged in its measurement characteristics. Therefore, it is essential that these RMs, as well as the measurement instrumentation and procedures, be subject to a program of continuing confirmation of traceability. Many of the factors involved in such a program are discussed in Reference 2.[6]

2. MASS AND VOLUME MEASUREMENTS

The national systems of mass and volume measurements are so well established that RMs meeting the above criteria are readily available. Where necessary, the licensee can use the RMs to calibrate Working Reference Materials (WRMs) that more closely match the characteristics of the measured material in terms of mass, shape, and density in the case of mass measurements or are more easily adapted to the calibration of volume-measurement equipment.

Specific procedures for the use of mass and volume RMs for the calibration of measurement processes and equipment are given in the corresponding national standards (Refs. 3 and 4). Factors likely to affect uncertainty levels in inventory measurements of mass and volume are discussed in regulatory guides (Refs. 5, 6, and 7).

3. CHEMICAL ASSAY AND ISOTOPIC MEASUREMENTS

Methods for chemical analysis and isotopic measurement often are subject to systematic errors caused by the presence of interfering impurities, gross differences in the concentrations of the measured component(s) or of measurement-affecting matrix materials, and other compositional factors. Traceability in these measurements can be obtained only if such effects are recognized and either are eliminated by adjustment of the RM (or sample) composition or, in some cases, are compensated for by secondary measurements of the measurement-affecting variable component(s) and corresponding correction of the measured SNM value. The latter procedure involves additional sources of uncertainty and therefore should be employed only if it has a substantial economic or time advantage, if the interferences or biasing effects are small and limited in range, if the corrected method is reliable, and if the correction itself is verifiable and is regularly verified.

Systematic measurement calibration errors frequently arise and can be ascribed to improper use, handling, or treatment of reference materials. These errors are independent of the effect of impurities, concentration differences, etc., and are almost impossible to detect via an internal measurement control program. Interlaboratory measurement comparison programs where carefully characterized materials are used are means by which these systematic errors may be identified and corrective action initiated.

3.1 National Standards - Uses and Limitations

PCRMs generally are not recommended for use directly as WRMs, not only because of cost and required quantities but also because of differences in composition (or isotopic ratios) compared to the process materials to be measured. PCRMs are more often used to prepare RMs of composition and form matching the process material or to evaluate (and give traceability to) non-NBS but substantially identical material from which matching WRMs are then prepared. This is necessary because of both the wide diversity of process materials encountered and the very small number and variety of SNM PCRMs available. These RMs may be used directly as WRMs, if appropriate, or may be reserved for less frequent use in the calibration of suitable synthetic or process-material WRMs of like characteristics, as well as for verifying instrument response factors and other aspects of the measurement system. However, each level of subsidiary RMs adds another level of uncertainty to the overall uncertainty of the SNM measurement.

PCRMs can be used to "spike" process samples or WRMs to determine or verify the measurability of incremental changes at the working SNM level. However, because of possible "threshold" or "zero error" effects and nonlinearity or irregularity of measurement response with concentration, this process does not of itself establish traceability.

3.2 Working Reference Materials

WRMs that closely match the effective composition of process material, or a series of such WRMs that encompass the full range of variation therein, serve as the traceability link in most chemical analyses and isotopic measurements. The WRMs derive traceability through calibration relative to either PCRMs, Secondary Certified Reference Materials (SCRMs), or, more often, synthesized RMs containing either PCRMs or other material evaluated relative to the PCRM (see Section B.3.1 of this guide).

The characteristics required of a WRM are that it be chemically similar to the material to be measured (including interfering substances), that it be sufficiently stable to have a useful lifetime, and that it have sufficiently low uncertainty in its assigned value to meet the requirements of the measurement methods and of the accountability limits of error.

[6] Regulatory guides under development on measurement control programs for SNM accounting and on considerations for determining the systematic error and the random error of SNM accounting measurements will also discuss the factors involved in a program of continuing confirmation of traceability.

WRMs can be prepared (a) from process materials characteristic of the material to be measured or (b) by synthesis using known quantities of pure SNM. The former method offers the advantage that the WRM will include all the properties that can affect the measurement such as impurities, SNM concentration level, and chemical and physical form; it suffers from the disadvantage that the assigned value is determined by analyses subject to uncertainties that must be ascertained. The latter method involves preparations using PCRMs (not usually economical unless small amounts are used) or SCRMs with the appropriate combination of other materials to simulate the material to be measured. The advantages of the latter method include more accurate knowledge of the SNM content and better control of other variables such as the amount of impurities and the matrix composition. The chief disadvantage is that the synthesized WRM may not possess all the subtle measurement-affecting characteristics of the process material. Moreover, the preparation of synthesized WRMs may be substantially more costly than the analysis of WRMs prepared from process material. Detailed procedures for preparing plutonium and uranium WRMs are described in References 8, 9, 10, and 11.

The primary concern in the use of a WRM to establish traceability in SNM measurements is the validity of the assigned value and its uncertainty. Considerable care is necessary to ensure that the WRMs are prepared with a minimal increase in the uncertainty of the assigned value above that of the PCRM upon which the WRM value is based. If the assigned value of a WRM is to be determined by analysis, the use of more than one method of analysis is necessary to enhance confidence in the validity of the assigned value. The methods should respond differently to impurities and to other compositional variations. If the WRM has been synthesized from a PCRM or other reference materials, the composition and SNM content can be verified by subsequent analyses.

The composition of a WRM can change with time, e.g., changes in oxidation state, crystalline form, hydration, or adsorption. These changes and their effects on measurement are minimized by appropriate packaging and proper storage conditions. Additional assurance is attained by distributing premeasured amounts of the material into individual packets at the time of preparation, and these packets can be appropriately sized so that the entire packet is used for a single calibration or test. Even among such subsamples, there may be variability in SNM content, and this variability must be taken into account in determining the uncertainty of the assigned value.

3.3 Standard Laboratories and Sample Interchange

Traceability of chemical assay and isotopic analysis values also may be obtainable through comparative analyses of identical samples under parallel conditions. A comparative-measurement program may take either or both of two forms:

a. Periodic submission of process samples for analysis by a facility having demonstrated traceability in the desired measurement.

b. Interfacility interchange and measurement of well-characterized and representative materials with values assigned by a facility having demonstrated traceability in the measurement.

Round-robin programs in which representative samples are analyzed by a number of laboratories do not establish traceability but can only indicate interlaboratory agreement or differences, unless traceability of one or more of the samples in a set has been established as above.

The Safeguards Analytical Laboratory Evaluation (SALE) program as administered by the Department of Energy New Brunswick Laboratory (NBL) is an example of an acceptable comparative-measurement program.

4. NONDESTRUCTIVE ASSAY

Nondestructive assay (NDA) measurement methods are those that leave the measured material unchanged (e.g., gamma emission methods) or with no significant change (e.g., neutron activation) relative to its corresponding unmeasured state (Ref. 2). NDA offers the advantages that the same RM or the same sample can be measured repeatedly and yields valuable data on system uncertainties not otherwise obtained, that the measurement made does not consume process material, and that measurements can be made more frequently or in greater number, usually at a lesser unit cost than with destructive chemical methods. These advantages often yield better process and inventory control and enhanced statistical significance in the measurement data. However, like chemical measurement methods, NDA methods have many sources of interferences that may affect their accuracy and reliability. The interferences and their sources must be identified before valid traceability can be assured.

In nearly all NDA methods, the integrity and traceability of the measurements depend on the validity of the RMs by which the NDA system is calibrated. Calibrations generally are based on WRMs that are or are intended to be well characterized and representative of the process material or items to be measured. While the matching of RMs to process items, and consequent valid traceability, is not difficult to achieve for homogeneous materials of substantially constant composition (e.g., alloys) having fixed size and shape (e.g., machined pieces), such ideal conditions are not obtained for most SNM measurements. Many of the materials and items encountered are nonhomogeneous, nonconforming in distribution, size, or shape, and highly variable in type of material and composition. In order to ensure traceability of the measurement results through the NMS, variations in the physical characteristics and composition of process items and in their effects upon the response of the NDA measurement system must be evaluated and carefully considered in the selection or design of WRMs and measurement procedures (Refs. 12 and 13).

WRMs usually (a) are prepared from process materials that have been characterized by measurement methods whose uncertainties have been ascertained through the NMS (i.e.,

are traceable) or (b) are artifacts synthesized from well-characterized materials to replicate the process material.[7] However, calibration of the NDA method by means of such RMs does not automatically establish continuing traceability of all process item measurement results obtained by that method. The effects of small variations in the materials being assayed may lead to biased results even when the WRM and the material under assay were obtained from nominally the same process material. It therefore may be necessary either (a) to establish traceability of process item measurement results by comparing the NDA measurement results with those obtained by means of a reliable alternative measurement system of known traceability, e.g., by total dissolution and chemical analysis (see Section B.4.1) or (b) to establish adequate sample characterization to permit the selection of a similarly characterized WRM for method calibration (see Section B.4.2).

4.1 Traceability Assay by a Second Method

Any NDA method would be of little practical use if every measurement also required a confirmatory analysis. However, in cases in which there are a number of items or material samples of established similar characteristics, it is practical to establish traceability for a series of measurements by means of second-method evaluations of an appropriate proportion of randomly selected samples. If the correlation between the two methods is then found to be consistent, traceability is established for all NDA measurements on that lot of SNM and on other highly similar material.

For nominally uniform process or production material of which multiple subsamples can be obtained from a gross sample, the uniformity can be deduced from the distribution of the NDA measurement data. For thus characterized material, traceability can be established for all subsamples that approximate the mean[8] from the separate traceable second-method analysis of a few of the subsamples. Other like subsamples can then be selected as traceable WRMs whose assigned values are related to the separately analyzed subsamples through their respective NDA measurement results.

For subsample populations exhibiting a range of NDA values, especially where a destructive second-method analysis is used, the "twinning" method of sample selection may be employed. In this method, pairs of subsamples are matched by their NDA measurement values, and the matches are confirmed by NDA reruns. One member of each pair is evaluated by the traceable second-method analysis; the other member of that pair is then assigned the value determined for its twin and may serve thereafter as a traceable WRM for the measurement of that process material by that NDA method.

[7] The advantages stated for similarly derived WRMs (see Section B.3.2) also apply here.

[8] Subsamples whose measured values markedly deviate from the mean (i.e., "flyers") are not used for second-method analysis or for WRMs.

4.2 Characterization by a Second Method

If the process items or materials being measured are subject to non-SNM variations that affect the SNM measurement, it may be possible to employ one or more additional methods of analysis to measure these variations and thus to characterize process materials in terms of such analysis results. If the secondary analyses also are of an NDA method, they may often be performed routinely with the SNM measurements. In many cases, the results of secondary analyses may be used to derive simple corrections to the SNM measurement results. Correction also may be obtained and traceability preserved by the judicious modification of RMs so as to incorporate the same variable factors, i.e., so that they can produce the same relative effects in the SNM and non-SNM measurements as do the process variable(s).

Alternatively, it may be advantageous to prepare WRMs that span the normal range of variability of the measurement-affecting non-SNM parameter(s) (and also the SNM-concept range, if appropriate). These WRMs can then be characterized on the basis of their non-SNM measurement results or of some function(s) of SNM and non-SNM measurement results and can be assigned a correspondingly "characteristic figure." If this procedure can be carried out with adequate sensitivity and specificity relative to the interfering factors and within acceptable limits of uncertainty, the process material can be routinely characterized in like manner and the appropriate WRM selected on the basis of such characterization.

5. CONTINUING TRACEABILITY ASSURANCE

Initial or occasional demonstration that a laboratory has made measurements compatible with the NMS is not sufficient to support a claim of traceability. Measurement processes are by their nature dynamic. They are vulnerable to small changes in the skill and care with which they are performed. Deterioration in the reliability of their measurement results can be caused by (a) changes in personnel performance, (b) deterioration in or the development of defects in RMs, instrumentation, or other devices, or (c) variation in the environmental conditions under which the measurements are performed. The techniques discussed in preceding sections ensure traceability only if they are used within a continuing program of measurement control (Ref. 1).

C. REGULATORY POSITION

The measurement control program (Ref. 1) used by the licensee should include provisions to ensure that individual measurement results are traceable to the national standards of measurement through the national measurement system (NMS). RMs used to establish traceability of measurement results through the NMS should have assigned values whose uncertainties are known relative to the national standards of measurement. To meet this condition, the licensee should maintain a continuing program for calibrating each measurement process, using RMs that meet the criteria in the following paragraphs.

1. REFERENCE MATERIALS

1.1 The National Bureau of Standards

Devices and instruments calibrated by, and CRMs certified by, NBS along with reference material data supplied are acceptable RMs[9] for calibrating either methods or WRMs. However, it is very important that the licensee be able to demonstrate that the RMs are stable under the conditions for which they are used, that their validity has not been compromised, and that they meet the accuracy requirements of the intended applications.

1.2 Secondary Certified Reference and Working Reference Materials

SCRMs or WRMs that have been produced by the licensee or by a commercial supplier are acceptable provided their uncertainties relative to PCRMs are known.

A statement of uncertainty should be assigned to each RM based on an evaluation of the uncertainties of the calibration process. The statement should contain both the standard deviation and the estimated bounds of the systematic errors associated with the assigned value similar to the statistical information contained within the most recent NBS PCRM certificates.

1.2.1 RMs for Chemical and Isotopic Analyses

WRMs used for calibrating chemical assay and isotopic measurements may be prepared from standard reference materials (SRMs) supplied by NBS or from other well-characterized materials available to the industry. Such WRMs should be prepared under conditions that ensure high reliability and should be packaged and stored in a way that eliminates any potential for degradation of the WRM.

The assigned values of WRMs prepared from process materials should be determined by analysis, using two different methods whenever possible. A sufficient number of analyses should be done by both methods to allow a reliable estimate of the components of random variation that affect the measurement. If two methods are not available, as may be the case for isotopic analysis, it is recommended that a verification analysis be obtained from another laboratory.

The components of variance (random variation) of measurements used to assign a value to an RM should be known in advance. The statistical design of an RM characterization plan requires that measurement precision, etc., be known in order to calculate the number of measurements to be performed and the number of samples to be analyzed so that the desired uncertainty in the mean value assigned to the RM can be achieved. The maximum uncertainty permitted by the proposed end use of the RM must be an assumption that is factored into the characterization plan.

If WRMs are prepared from NBS SRMs or other PCRMs, they should be analyzed to verify that the makeup value is correct, i.e., that no mistakes have been made in their preparation. For this verification, at least five samples should be analyzed using the most reliable method available. Should the analytical results differ significantly from the makeup value, the WRM should not be used. Typical statistical and analytical procedures acceptable to the NRC staff for preparing WRMs are found in References 8, 9, 10, and 11.

Storage and packaging of WRMs should follow procedures designed to minimize any changes likely to affect the validity of the assigned values. Whenever practical, the WRM should be divided into small measured quantities at the time of preparation, and the quantities should be of appropriate size so that each entire unit is used for a single calibration or calibration test (Refs. 8, 9, 10, and 11).

1.2.2 Nondestructive Assay

RMs for NDA should be prepared from well-characterized materials whose SNM contents have been measured by methods that have been calibrated with CRMs or from synthetic materials of known SNM content. The NDA RMs should closely resemble in all key characteristics the process items to be measured by the system. Since destructive measurements ordinarily cannot be made on NDA RMs in order to verify makeup, as required for WRMs for chemical assay and isotopic analyses, RMs should be prepared in sets of at least three using procedures that guard against errors common to all members of the set. If all three RMs respond consistently to the NDA system, one RM could be used as the intended NDA RM, the second could be kept in reserve, and the third characterized using destructive chemical measurement techniques whenever possible. If destructive analysis is not possible, the consistency of the NDA system response to all the RMs in the set would provide a basis for judging the validity of the set of RMs. If one or more of the RMs in the set differs significantly from the expected response, no RMs from that set should be used. Statistical tests for this comparison can be found in References 8, 9, 10, and 11.

The design and fabrication of the RMs should take into account the measurement process parameters affecting the response of the system (Ref. 2), including:

a. SNM content,
b. Isotopic content,
c. Matrix material,
d. Density,
e. Container material and dimensions,
f. Self-absorption effects, and
g. Absorption and moderation effects.

Studies should be carried out in sufficient detail to identify the process item characteristics and the variations of the characteristics that can cause systematic error. The results of the studies should be used to establish reasonable bounds for the systematic errors.

NDA systems whose uncertainties relative to the national standards of measurement cannot be satisfactorily established

[9] International RMs and reference material such as IAEA RMs are included, if accepted by NBS.

directly through the calibration process should be tested by comparative analysis. This test should be done by periodically analyzing randomly selected process items with the NDA system in question and by another method with known uncertainty. The verification analysis can be done on samples obtained after reduction of the entire item to a homogeneous form. In some cases, verification analysis by small-sample NDA or by other NDA methods may be acceptable if the uncertainties of the verification method are known relative to the national standards of measurement.

2. MEASUREMENT ASSURANCE

The traceability of each measurement process through the NMS should be maintained by a continuing program of measurement assurance (Ref. 1). This program should include planned periodic verifications of the assigned values of all RMs used for calibrations.

2.1 Verification of Calibrations

A formal program fixing the frequency at which calibrations and calibration checks are performed should be established. The required frequencies are strongly dependent on system stability and should be determined for each case by using historical performance experience. Current performance of the measurement system based on measurement control program data may signal the need for more frequent verifications. Also, the effects of changes in process parameters such as composition of material or material flows should be evaluated when they occur to determine the need for new calibrations.

WRMs that are subject to deterioration should be recertified or replaced on a predetermined schedule. The frequency of recertification or replacement should be based on performance history. If the integrity of an RM is in doubt, it must be discarded or recalibrated.

2.2 Recertification or Replacement of CRMs

Objects, instruments, or materials calibrated by NBS or other authoritative laboratories and used as CRMs by the licensee should be monitored by intercomparisons with other CRMs to establish their continued validity. In any case, the values should be periodically recertified by the certifying agency or compared with other CRMs by the licensee in accordance with Table 2.

2.3 Interlaboratory Exchange Programs

The licensee should participate in interlaboratory exchange programs when such programs are relevant to the types of measurements performed and the materials analyzed in his laboratory. The values assigned to the materials that are to be analyzed in the interlaboratory exchange programs should be carefully and traceably certified so that any deviation that may occur can be readily identified and quantified.

Table 2

RECERTIFICATION OR REPLACEMENT INTERVALS FOR CRMs

Test Objects and Devices	*Maximum Period (Years)*
Mass	1
Length	5
Volumetric Provers	2
Thermometers and Thermocouples	2
Calorimetric Standards	2
Certified Reference Materials	
Because of the complex chemical/physical properties of chemical CRMs such as Pu metal, U_3O_8, U metal, UO_2, radioactive materials, etc., and the varied end uses to which they are put, a formal program of comparison or replacement frequency should be established. The required frequencies are strongly dependent on the system stability and should be determined for each CRM by historical performance experience.	

The data obtained through this participation and other comparative measurement data (such as shipper-receiver differences and inventory verification analyses) should be used to substantiate the uncertainty statements of his measurements.

When statistically significant deviations indicating lack of consistency in measurements occur in the results of the comparative measurements, the licensee should conduct an investigation. The investigation should identify the cause of the inconsistency and, if the cause is within his organization, the licensee should initiate corrective actions to remove the inconsistency. The investigation may involve a reevaluation of the measurement process and the CRMs to locate sources of bias or systematic error or a reevaluation of the measurement errors to determine if the stated uncertainties are correct.

3. RECORDS

The licensee should retain all records relevant to the uncertainty of each measurement process for 5 years [§70.51(e)(4)(iv) and (v); §70.57(b)(12)]. The records should include documents or certificates of CRMs, the measurement and statistical data used for assigning values to WRMs, and the calibration procedures used in preparing the WRMs.

REFERENCES

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