Designation: C1128 – 23

Standard Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials[1]

This standard is issued under the fixed designation C1128; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the preparation and characterization of working reference materials (WRM) that are produced by a laboratory for its own use in the analysis of nuclear fuel cycle materials. Guidance is provided for proper planning, preparation, packaging, and storage; requirements for characterization; homogeneity and stability considerations; and value assignment. When traceability to SI is desired for a WRM, it will be achieved by a defined, statistically sound characterization process that is traceable to a certified value on a certified reference materials. While the guidance provided is generic for nuclear fuel cycle materials, detailed examples for some materials are provided in the appendixes.

1.2 This guide does not apply to the production and characterization of certified reference materials (CRM). Refer to ISO 17034 and ISO Guide 35 for guidance on reference material production, characterization, certification, sale, and distribution requirements.

1.3 The information provided by this guide is found in the following sections:

	Section
Perform WRM Planning	6
Prepare and Process Materials	7
Packaging and Storage of Materials	8
Perform Homogeneity Study	9
Perform Stability Studies	10
Characterize Materials	11
Perform Uncertainty Analysis	12
Produce Documentation	13
Carry Out WRM Utilization and Monitoring	14

1.4 The values stated in SI units are to be regarded as standard. The non-SI units of molar, *M*, and normal, *N*, are also regarded as standard. Any non-SI units of measurement shown in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1108 Test Method for Plutonium by Controlled-Potential Coulometry

C1165 Test Method for Determining Plutonium by Controlled-Potential Coulometry in H_2SO_4 at a Platinum Working Electrode

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within Nuclear Industry

C1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium

C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

C1625 Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry

[1] This guide is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.08 on Quality Assurance, Statistical Applications, and Reference Materials.

Current edition approved Jan. 1, 2023. Published February 2023. Originally approved in 1989. Last previous edition approved in 2018 as C1128 – 18. DOI: 10.1520/C1128-23.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C1637 Test Method for Determination of Impurities in Plutonium Materials—Acid Digestion and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Analysis
C1672 Test Method for Determination of Uranium or Plutonium Isotopic Composition or Concentration by the Total Evaporation Method Using a Thermal Ionization Mass Spectrometer
C1689 Practice for Subsampling of Uranium Hexafluoride
D1193 Specification for Reagent Water
D8293 Guide for Evaluating and Expressing the Uncertainty of Radiochemical Measurements

2.2 *ISO Standards:*[3]
ISO 10576–1 Statistical Methods – Guidelines for the Evaluation of Conformity with Specified Requirements – Part 1: General Principles
ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories[3]
ISO 17034 General Requirements for the Competence of Reference Material Producers
ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials
ISO Guide 31 Reference Materials – Contents of Certificates, Labels and Accompanying Documentation
ISO Guide 33 Reference Materials – Good Practice in Using Reference Materials
ISO Guide 35 Reference Materials – General and Statistical Principles for Certification
ISO Guide 80 Guidance for the In-House Preparation of Quality Control Materials (QCMs)

2.3 *Joint Committee for Guides in Metrology:*[4]
JCGM 100 Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections (2008))
JCGM 200 International Vocabulary of Metrology—Basic and General Concepts and Associated Terms (VIM) (ISO/IEC Guide 99)

2.4 *IAEA Documents:*[5]
IAEA-TECDOC-1350 Development and Use of Reference Materials and Quality Control Materials

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

3.2 *Definitions:*

3.2.1 *certified reference material (CRM), n*—reference material (RM) characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability. **ISO Guide 30**

3.2.1.1 *Discussion*—The uncertainty interval for each certified property of the material is usually constructed as the interval between the certified property value minus the expanded uncertainty (see 3.2.6) of the value, and the property value plus the expanded uncertainty. For the expanded uncertainty, its coverage factor is chosen so as to provide the stated level of confidence. For example, to provide a stated level of 95 % confidence, the coverage factor is usually approximately 2.

3.2.2 *characterization, n—of a reference material*, determination of the property values or attributes of a reference material, as part of the production process. **ISO Guide 30**

3.2.3 *combined standard uncertainty, n*—standard uncertainty that is obtained using the individual standard uncertainties associated with the input quantities in a measurement model. **JCGM 200**

3.2.3.1 *Discussion*—The combined standard uncertainty is the combination of one or more individual components of uncertainty that make up the uncertainty budget for an attribute. It is understood to be the equivalent of one times the standard deviation.

3.2.4 *coverage factor, n*—number larger than one by which a combined standard uncertainty is multiplied to obtain an expanded uncertainty. **JCGM 200**

3.2.5 *commutability, n*—property of a reference material that relates to agreement between measurement results from a reference material and measurement results from the sample material for the given application.

3.2.6 *expanded uncertainty, n*—product of a combined standard uncertainty and a coverage factor. **adapted from JCGM 200**

3.2.7 *homogeneity, n*—uniformity of a specified property value throughout a defined portion of a reference material (RM). **ISO Guide 30**

3.2.7.1 *Discussion*—A reference material is said to be homogenous with respect to a specified property if the property value, as determined by tests on samples of specified size, is found to lie within the specified uncertainty interval.

3.2.7.2 *Discussion*—The ‘defined portion’ may be, for example, an RM batch or a single unit within the batch. **ISO Guide 30**

3.2.8 *measurement uncertainty, n*—non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand. **adapted from JCGM 200**

3.2.8.1 *Discussion*—Measurement uncertainty may be expressed as either standard uncertainty or expanded uncertainty and any expression should indicate which form is being used.

3.2.9 *metrological traceability, n*—property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty. **JCGM 200**

3.2.10 *period of validity, n—of a reference material*, time interval during which the producer of the reference material warrants its stability. **ISO Guide 30**

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.
[4] Available from Bureau International des Poids et Mesures, Pavillon de Breteuil, F-92312 Sèvres Cedex, France, www.bipm.org.
[5] Available from International Atomic Energy Agency (IAEA), Vienna International Centre, PO Box 100, 1400 Vienna, Austria, www.iaea.org.

3.2.10.1 *Discussion*—In nondestructive analysis (NDA), period of validity is frequently referred to as "working life."

3.2.11 *property value, n—of a reference material*, value corresponding to a quantity representing a physical, chemical or biological property of an RM. **ISO Guide 30**

3.2.11.1 *Discussion*—The terms "property value" and "quantity value" may be used interchangeably.

3.2.12 *quality control material (QCM), n*—material used routinely to assess the precision of test procedures. **ISO Guide 80**

3.2.12.1 *Discussion*—Such materials are variously referred to in the open literature as "in-house reference materials," "quality control samples," "check samples," "set up samples," and so forth. **ISO Guide 80**

3.2.12.2 *Discussion*—QCMs are used to demonstrate that a procedure is under statistical control.

3.2.12.3 *Discussion*—QCMs have assigned values that are indicative, and do not require characterization by metrologically valid procedures. As such, QCMs cannot be expected to establish metrological traceability or trueness of a measurement result. QCMs should always be sufficiently homogeneous and stable with respect to the properties of interest.

3.2.13 *reference material (RM), n*—material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process. **ISO Guide 30**

3.2.13.1 *Discussion*—Standards used for calibration and for quality control are two types of reference materials.

3.2.14 *reference material certificate, n*—document containing the essential information for the use of a CRM, confirming that the necessary procedures have been carried out to ensure the validity and metrological traceability of the stated property values. **ISO Guide 30**

3.2.15 *reference material document, n*—document containing all the information that is essential for using any reference material. **adapted from ISO 17034**

3.2.15.1 *Discussion*—Adapted from the definition of "product information sheet" in ISO Guide 30.

3.2.16 *reference method, n*—measurement method, that has been shown to have the appropriate trueness and precision for its intended use and has been officially defined as reference method by a competent body. **ISO Guide 30**

3.2.17 *stability, n*—ability of a reference material, when stored under specified conditions, to maintain a stated property value within specified limits for a specified period of time. **ISO Guide 30**

3.2.18 *standard uncertainty, n*—measurement uncertainty expressed as a standard deviation. **JCGM 200**

3.2.19 *uncertainty budget, n*—statement of a measurement uncertainty, of the components of that measurement uncertainty, and of their calculation and combination. **JCGM 200**

3.2.20 *uncertainty interval, n*—interval derived from the actual measurement of the characteristic and its uncertainty, covering the values that could reasonably be attributed to this characteristic. **ISO 10576–1**

3.2.20.1 *Discussion*—An uncertainty interval is expressed in terms of probability of α or Type 1 error (for example, 0.05 or 5 %), while a confidence interval is expressed in terms of (1 – α); for example, 0.95 or 95 %.

3.2.21 *working reference material (WRM), n*—reference material, which is not certified, but is characterized using defined, statistically sound characterization processes, and can be used routinely to calibrate, control, or verify measuring instruments or measuring systems.

3.2.21.1 *Discussion*—Adapted from definition of "working measurement standard" in JCGM 200.

3.2.21.2 *Discussion*—WRMs are distinct from QCMs (as defined herein and in ISO Guide 80) in that WRMs are RMs and provide metrological traceability to a CRM or other stated reference.

3.2.21.3 *Discussion*—A WRM is prepared by a laboratory for its own use for calibration or for quality control, or for the validation of a measurement method (see Guide C1068) as indicated in Fig. 1.

3.2.21.4 *Discussion*—The definition of "quality control material" in IAEA-TECDOC-1350 is similar to the definition of WRM in this guide, and is as follows: "Material used for the purposes of internal quality control and subjected to the same part of the same measurement procedure as that used for test materials." IAEA-TECDOC-1350 anticipates these materials to provide metrological traceability, unlike QCMs as defined in ISO Guide 80.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *fitness for purpose, n*—degree to which a WRM, when used as intended in a measurement process, enables a user to make technically and administratively correct decisions (adapted from Guide C1068).

3.3.2 *project plan, n*—document which specifies what a WRM project needs to accomplish and how the WRM will be produced and characterized.

3.3.3 *qualification*—process of determining WRM to be fit for purpose.

3.3.3.1 *Discussion*—The process of qualification does not result in a certificate such as for a CRM. The degree of rigor applied in the qualification process is based on the intended use of the WRM.

4. Summary of Guide

4.1 This guide covers the preparation of WRMs from nuclear fuel cycle materials. Examples of these materials are compounds and metal of uranium and plutonium, absorber materials such as boron carbide, and cladding materials such as zirconium and stainless steel. The criteria governing the preparation of reliable WRMs are identified and discussed. While the guidance provided is generic for nuclear fuel cycle materials, detailed examples for some materials are provided in the appendixes. A flow diagram to illustrate an approach to producing WRMs is given in Fig. 2.

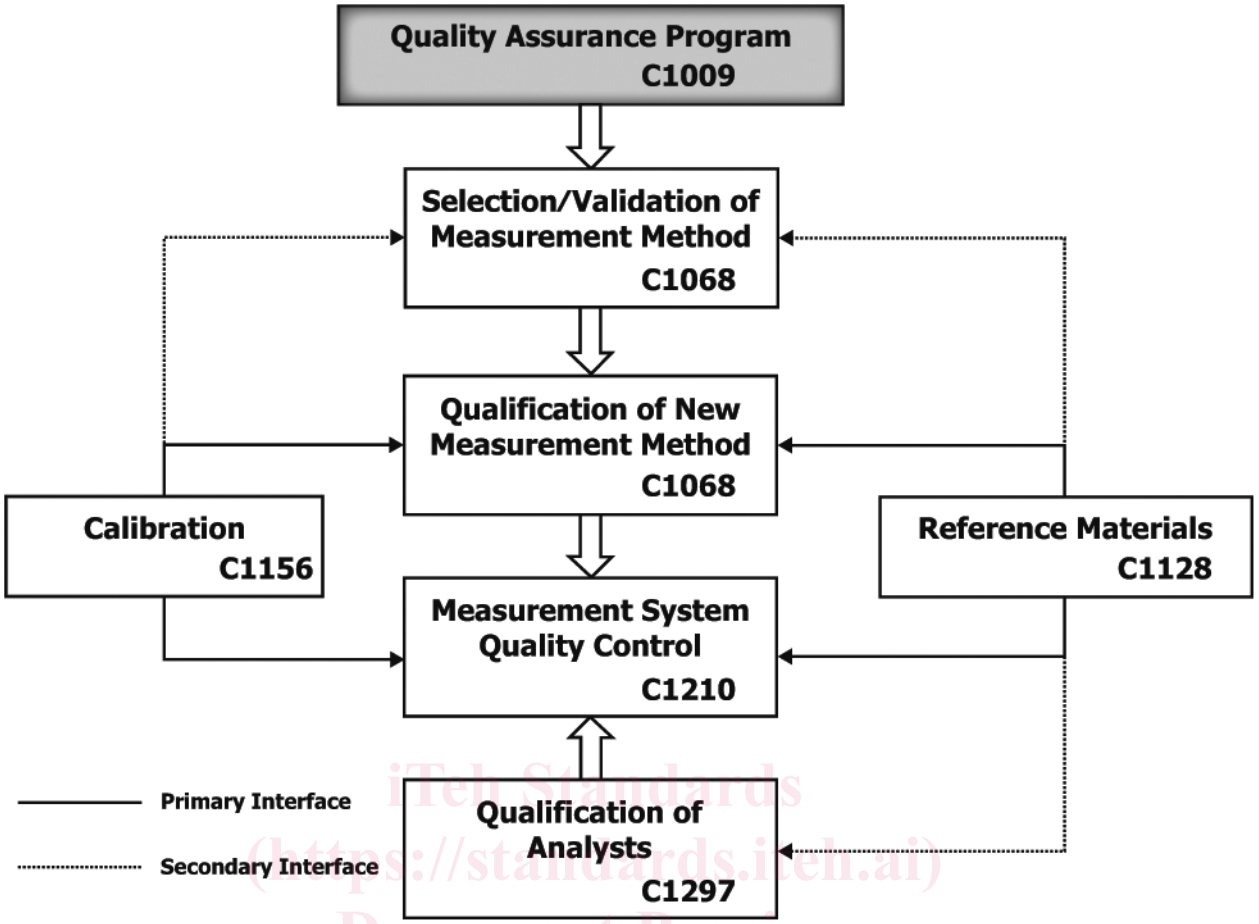

FIG. 1 Essential Elements of Analytical Laboratory Quality Assurance System

4.2 This guide distinguishes between three categories of reference materials:

4.2.1 *Certified reference materials (CRM),* as described in ISO 17034 and ISO Guides 30, 33, and 35.

4.2.2 *Working reference materials (WRM),* which are the focus of this guide. It is important to note that:

4.2.2.1 WRMs are not certified as described in ISO 17034, and they are typically produced using a CRM to provide metrological traceability. However, the preparation guidelines in ISO 17034 generally apply to WRMs.

4.2.2.2 The process of WRM qualification provides metrological traceability to SI (see 13.1) through a CRM, or a stated reference, and may be used for calibration. As such, WRMs are generally equivalent to what IAEA-TECDOC-1350 refers to as "quality control materials."

4.2.3 *Quality control materials (QCM),* as defined in ISO Guide 80, are produced by a laboratory with limited characterization to only provide an indication of its relevant property values for statistical control of a measurement system. A QCM is not certified and cannot be expected to provide metrological traceability. QCMs may not be used for calibration, but are typically utilized by a laboratory for a limited scope of usage. Examples of uses include: preparation of control charts, instrument performance checks, and the determination of operator variability.

5. Significance and Use

5.1 Certified reference materials (CRMs) prepared from nuclear materials are well characterized, traceable, and sufficiently homogenous and stable for their intended use. Usually they are certified using the most unbiased and precise measurement methods available, often with more than one laboratory being used on a national or international level. CRMs are at the top of the metrological hierarchy of reference materials. A graphical representation of a typical national nuclear measurement system is shown in Fig. 3.

5.2 Working reference materials (WRMs) need to have quality characteristics that are similar to CRMs, although the rigor used to achieve those characteristics is not usually as stringent as for CRMs. Similarly, production of WRMs should be in accordance with applicable requirements of ISO 17034. Where possible, CRMs are typically used to calibrate the methods used for establishing reference values assigned to WRMs, thus providing traceability to CRMs as required by ISO/IEC 17025. A WRM is normally prepared for a specific application.

5.3 Because of the importance of having highly reliable measurement data from nuclear material analysis, particularly for material control and accountability purposes, CRMs are used for calibration when available. However, CRMs prepared

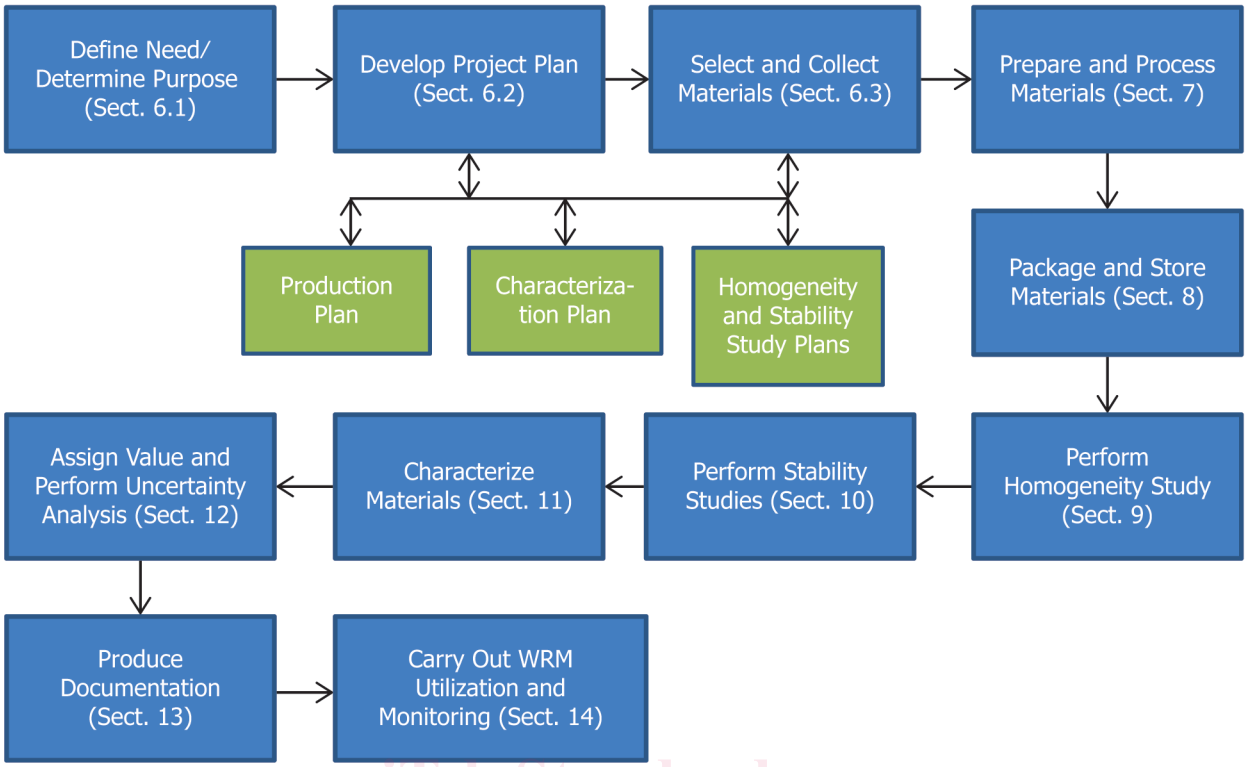

FIG. 2 General Process for Preparing Working Reference Materials

from nuclear materials are not always available for specific applications. Thus, there may be a need for a laboratory to prepare nuclear material WRMs to meet specific needs; for example, to match the matrix in process samples. In such cases, a WRM can be tailored to meet specific needs of a process or laboratory. Also, CRM supply may be too limited for use in the quantities needed for long-term, routine use. When properly prepared, WRMs will serve equally well as CRMs for most applications, and using WRMs will help preserve supplies of CRMs.

5.4 Difficulties may be encountered in the preparation of RMs from nuclear materials because of the chemical and physical properties of the materials. Chemical instabilities, problems in ensuring stoichiometry, homogeneity, and radioactivity are among the factors to be considered, with all three factors being involved with some materials. Those preparing WRMs from nuclear materials need to be aware of how these factors may affect preparation, as well as being aware of the other criteria governing the preparation of reliable WRMs.

5.5 While use of WRMs provides benefits for the laboratory, it is important to observe the distinction between WRMs, which are prepared by a laboratory for use by that laboratory (or, in some cases, an affiliated satellite laboratory or production facility served by the laboratory), and CRMs which provide certificates of analysis (in accordance with ISO Guide 31) and can be offered for sale.

6. Perform WRM Planning

6.1 *General:*

6.1.1 The WRM planning process involves the following elements:

6.1.1.1 Identifying the scope of the project, including all needs and requirements for WRM production, characterization, and value assignment (see 6.2 and Appendix X1).

6.1.1.2 Developing a detailed WRM project plan (6.3) that addresses all of the needs and requirements identified; and

6.1.1.3 Performing the selection and collection of materials that will be used to produce the WRM units (6.4).

6.1.2 The amount of detail in the project plan, and the level of effort to prepare it, should be based on the size and complexity of the WRM preparation task. In some cases the project plan can be brief as long as it contains the necessary information for the project to be completed successfully. If the project is to produce a WRM that has been produced previously, much of the needed information may already be available, and the planning process streamlined accordingly.

6.2 *Define WRM Needs and Requirements / Determine Purpose*—Producing a WRM requires forethought to ensure the completed WRM meets the needs of its end-users and stakeholders and the requirements for its application. A detailed project plan that specifies needs and how the WRM will be produced should be prepared and approved in advance of any work. The subjects discussed in this section should be considered and addressed as appropriate. Failure to properly define all needs and requirements can result in wasted time, funding, and materials, as well as requiring rework.

6.2.1 Needs and requirements for the WRM should be identified and documented. These can include the following:

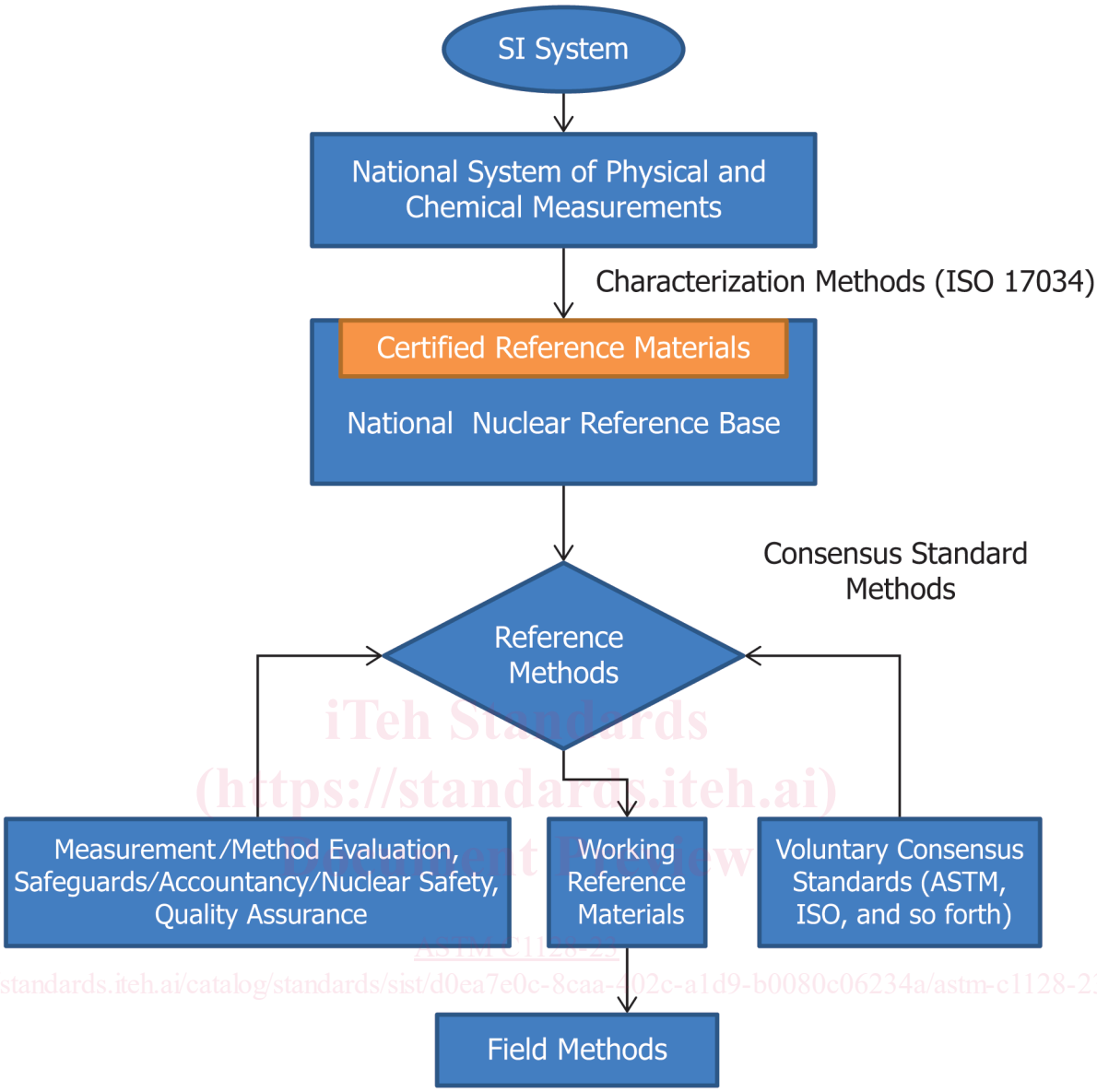

FIG. 3 Typical National Nuclear Measurement System

(1) Key elements of the WRM project purpose and scope;
(2) Quantity of WRMs needed;
(3) Intended purpose(s) of the WRM;
(4) Physical and chemical properties of the WRM;
(5) Important aspects and limitations of end-user's measurement method(s);
(6) Measurand(s) level and uncertainty;
(7) Data quality objectives;
(8) Fitness for purpose considerations;
(9) Criteria for material selection and collection;
(10) Requirements for producing stable and homogeneous WRM units;
(11) Criteria for container selection;
(12) Sampling criteria;
(13) Environmental controls during production and characterization;
(14) Selection and validation of characterization method(s);
(15) Control of measurement equipment calibration and traceability to the SI;
(16) Control of unit storage;
(17) Guidance on value assignment and uncertainty calculation;
(18) Period of validity; and
(19) Post-production stability monitoring.

6.2.1.1 Appendix X1 provides a template containing questions and statements that may be used to aid in documenting the WRM project plan requirements.

6.2.2 The planning process should follow the concept of defining needs and requirements, then documenting how the selected material will be used to produce stable and homogenous WRM units that will then be characterized. The project

plans should outline the process that will be used for value assignment and calculating the expanded uncertainty and the uncertainty budget.

6.2.3 The producer of a WRM should consider whether a commercially-available reference material is fit for purpose and could be used instead of producing a WRM. If a commercial RM does not meet one or more of the following conditions for the planned end-user application, then a WRM should be prepared:

(1) An existing, available RM does not match the needs of the end-users and the requirements of each measurement method;

(2) An existing RM is not available in sufficient quantity to meet usage needs;

(3) An existing RM is not available at an acceptable cost compared to producing a WRM;

(4) Another justification as determined by the preparer of the proposed WRM.

6.2.3.1 The preparer of the WRM may document the basis for their make-versus-buy decision in their planning document.

6.3 *Develop WRM Project Planning Documents*—The WRM producer is responsible for ensuring that all aspects of the production and characterization are well planned and documented in sufficient detail to ensure that all identified needs and requirements are implemented effectively.

6.3.1 The planning process should include the follow activities, which are adapted from a similar list in ISO 17034:

(1) Perform material selection, including any sampling and verification of identify of the material;

(2) Select inner-most container that is durable, long-lasting, resistant to damage from radioactive decay, and compatible with the chemical properties of the WRM;

(3) Maintain suitable environments for production (and subsequent characterization);

(4) Define material processing;

(5) Define acceptance criteria for measurand levels and their uncertainties;

(6) Specify acceptance criteria for, and assessment of, homogeneity, including sampling;

(7) Specify acceptance criteria for, and assessment/monitoring of, stability, including sampling;

(8) Design and organize appropriate characterization, including sampling;

(9) Select appropriate measurement procedures;

(10) Validate measurement procedures;

(11) Verify and calibrate measuring equipment;

(12) Establish metrological traceability for measurement result(s), as appropriate;

(13) Assess commutability, when applicable;

(14) Assign property value(s);

(15) Calculate expanded uncertainty(ies) and uncertainty budget(s);

(16) Issue WRM documents for production, characterization, and value assignment, as well as documents addressing safety and regulatory compliance;

(17) Ensure appropriate labeling and packaging of WRM including appropriate hazard communication labeling;

(18) Ensure appropriate control of transportation, if required for production, characterization, or storage;

(19) Ensure adequate storage facilities and conditions; and

(20) Ensure post-production stability monitoring, if applicable.

NOTE 1—ISO Guide 35 provides additional guidance on aspects of developing a project plan.

6.3.2 Activities and milestones should be scheduled and tracked to completion by the project leader or management.

6.3.3 All tasks should be assigned to experienced, trained, and qualified staff.

6.3.4 One individual may be assigned to perform or lead more than one aspect of the WRM production, characterization, and value assignment.

6.3.5 The WRM project plan should:

(1) Identify WRM needs and requirements;

(2) Identify WRM production execution plan;

(3) Identify WRM characterization execution plan, including value assignment;

(4) Identify work control documents and procedures that will be needed to implement the execution plans and ensure that they are complete and available when needed;

(5) Develop plans for performing any required homogeneity and stability studies; and

(6) Guide the material selection and collection process.

6.3.6 If sufficient information is not available to perform or to complete the project plan, it may be necessary to perform feasibility studies on specific aspects of the WRM project such as:

(1) Studies to determine if the material selected is fit for purpose according to the defined scope;

(2) Studies to determine the best ways to process and prepare the selected materials without compromising desired property values; or

(3) Studies to test, improve, or verify that equipment, personnel, and measurement procedures meet standards for performance required for producing or characterizing the WRM, or both, to the specific requirements. Feasibility studies should be simple in scope.

6.3.7 Planning should include defining resources needed to ensure efficient production and characterization campaigns to contain costs and minimize delays. This includes financial resources, availability of qualified personnel to prepare the WRM, availability of instrumentation and equipment required for characterization, homogeneity and stability studies, equipment required for packaging the WRM, availability of any outside resources for activities that the laboratory is not able to perform itself, and so forth.

6.3.7.1 The WRM producer is responsible for evaluating any outside collaborators and their ability to meet performance requirements in accordance with relevant ASTM and ISO standards during the planning process. Limitations on resources or collaborators can negatively impact WRM quality and the time required to complete the project.

6.3.8 *Develop Plan for Production Execution:*

6.3.8.1 The execution plan for production should provide a step-by-step summary of processes and procedures that will be

performed on the selected material(s). The plan should document all aspect of material handling, including preparation steps to ensure homogeneity, stability, and any specific material characteristics as well as the packaging of WRM units and their subsequent storage and distribution for characterization. The plan should be written in a manner that illustrates how all identified production requirements will be satisfied.

6.3.9 *Develop Plan for Characterization Execution:*

6.3.9.1 The characterization plan should document which measurement method will be used for each measurand to be determined. The plan should provide a step-by-step summary of process and procedures that will be performed on the selected units of the WRM. Quality assurance requirements including QC standards, blank, and measurement sequence should be described in sufficient detail to illustrate that characterization requirements and data quality objectives will be satisfied. The characterization plan should document the following aspects:

(1) Random selection of units; sampling parameters including minimum sample size;

(2) Sample dissolution, dilution, treatment, and analytical preparation;

(3) Measurement protocols;

(4) Data collection and reporting; and

(5) The calculation of quantity values, expanded uncertainties, and uncertainty budgets.

6.3.9.2 The plan should be written in a manner that illustrates how all identified characterization requirements will be satisfied.

6.3.9.3 The analysis scheme should be developed that will directly link the traceability of measurements made on the WRM aliquots to a CRM or other established reference, assuming traceability to the SI is required. Consideration also should needs to be given to the calibrations and controls when using common laboratory equipment such as analytical balances or calibrated labware. Corrections made to the measurements for interferences or contaminations should also be validated using a CRM when available. When designing an analysis plan consulting with a statistician experienced in analytical processes is recommended.

6.3.9.4 How the final measurand is calculated and assigned as well as the calculation of the expanded uncertainty should be included in the project plan.

6.3.9.5 Procedures that will be used for production or characterization activities should be identified and developed if needed.

6.3.9.6 Materials and equipment should be identified. If any items must be procured, the procurement requirements and purchase orders must be generated with sufficient lead time.

6.3.9.7 The project plan should include requirements for packaging and storage (see Section 8).

6.3.9.8 If any production or characterization tasks will be performed by subcontractors, the preparer of the WRM is responsible for verifying that they have the necessary technical competence and an appropriate quality assurance program to perform the task assigned to them in a manner consistent with the WRM planning requirements.

6.3.10 *Develop Plan for Homogeneity and Stability Studies:*

6.3.10.1 *Homogeneity Assessment (Section 9)*—The project plan should include details for homogeneity studies when needed. The results from the study are included when calculating expanded uncertainties for the assigned values.

6.3.10.2 *Stability (Short and long term) Assessment and Monitoring (Section 10)*—The project plan should include details for stability studies when needed. The results from the study are included when calculating the expanded uncertainties for the assigned values.

6.3.11 *Environmental, Safety, and Health*—Identify and assess environmental, safety, and health concerns. These may include the following:

6.3.11.1 *Chemical and Radiological Hazards*—Performance of a hazard analysis to identify activity-based hazards and the administrative and engineered controls required to minimize them.

6.3.11.2 *Radiolysis*—Assessment of the risk of radiolysis (generation of hydrogen, oxygen, or both) due to radioactive decay of actinides in solution.

6.3.11.3 *Criticality*—Assessment for the risk of criticality when handling and storing materials with fissile content.

6.3.11.4 *Waste Management*—Assessment of the waste that will be generated and how it will be disposed.

6.3.11.5 *Laboratory Safety Basis*—Review of the activity against the safety limits within which the laboratory is required to operate.

6.3.11.6 *Industrial Hygiene*—Ensure that applicable requirements of the international Globally Harmonized System for labeling of packages and communication of hazards using Safety Data Sheet are implemented.

6.3.11.7 *Transportation of Radioactive Materials*—Ensure that applicable transportation regulations (for example, Department of Transportation regulations in the United States) are followed for shipping of selected materials, WRM units, and samples for characterization.

6.4 *Select and Collect Materials*—Selection of materials is an important part of planning that should be based on identified requirements.

6.4.1 Proper material selection is critical to achieving credible, stable, and homogenous WRMs. The planning process will evaluate the importance and relative significance of material availability (source), cost, chemical and physical properties, and stoichiometry. The starting materials for the preparation of WRMs may already be in the desired chemical and physical form or it may need to be processed. In the former case, the starting material may be a process material. For example, a batch of uranium dioxide pellets, boron carbide powder, or plutonium nitrate solution might be taken directly from a nuclear material process, treated as necessary, packaged as a WRM, and then characterized. In the latter case, various approaches are used to produce the form desired. For example, high-purity uranium hexafluoride might be dissolved and the solution converted to urano-uranic oxide (U_3O_8) to prepare a pure or matrix matched WRM (see Appendix X3).

NOTE 2—Consideration for environmental, safety, and health factors when handling the bulk material is an important aspect in the selection process. An activity-based hazard analysis of the chemical and radiological hazards associated with the work will assist in establishing the controls

required to do the work safely.

7. Prepare and Process Materials

7.1 *Preparation:*

7.1.1 The objective of preparation is to make physical and chemical manipulations so as to produce a homogeneous and stable material in the form required for a WRM. For a given WRM, the physical and chemical manipulations that will be used depend on the starting material(s), the WRM form required, and the physical and chemical properties of the materials involved. Various aspects of preparation are discussed in this section.

7.1.2 The form of the WRM can be any stable state of the element of interest or a somewhat unstable state whose stoichiometry is easily reproducible. The forms most commonly used for nuclear materials have been oxides as powder or pellets, metal, and nitrate solutions.

7.2 *Processing*—Processing of the material includes preparation and subdividing into usable WRM aliquots.

7.2.1 A preparation procedure should be written using a scheme for preparing the WRM developed during the planning stage. The procedure should include the necessary steps for making the required chemical and physical manipulations, and it should include requirements for recording data generated during preparation. If the reference value will be calculated based on process or make-up parameters (weights, volumes, and so forth), write the procedure accordingly to minimize the possibilities of losing any material during processing. Information for preparation and dissolution of uranium and plutonium materials can be found in Practices C1347 and C1168, respectively. Procedures to illustrate the preparation of two WRM solutions are given in Appendix X4.

7.2.2 *Subdividing*—During the creation of the WRM aliquots it must be ensured that homogeneity of the bulk material is maintained. Key factors to consider when developing a plan to subdivide the bulk material are listed below.

7.2.2.1 Liquids should be thoroughly homogenized prior to being subdivided, and regularly stirred or agitated to ensure that the solution remains homogenous while it is subdivided. If filtration is required, it should be done before subdividing.

7.2.2.2 The separation of finer particulates from the bulk material when subdividing solids and powders should be considered and assessed if deemed to be significant.

7.2.2.3 Subdividing of the bulk material should be performed as quickly as possible.

8. Packaging and Storage of Materials

8.1 *Packaging*—Once preparation is complete, the WRM is packaged for use. A frequent practice is to divide the WRM into essentially equal portions or units, each of which represents enough material for a one-time use. If a WRM is sufficiently stable, it could be divided into larger portions for multiple uses over a short time duration. There is a risk here, however, because each time a container is opened there is a potential for loss of WRM integrity (for example, from contamination or evaporation). The key to packaging is to contain the WRM portions in such a manner as to preserve their integrity for the life of the WRM (see Section 6). A technique sometimes used for solutions is to evaporate each weighed portion to near-dryness in its packaging container, giving a weighed amount of the element of interest for a one-time use. Various aspects of packaging are discussed in this section. A procedure to illustrate packaging a WRM solution is given in Appendix X4.

8.1.1 *Container*—It is important that the container material be chemically compatible with the WRM matrix and that the material will not contribute to the contamination of the WRM. To avoid contamination, containers are often specially cleaned before packaging. When radioactive material such as plutonium is involved, the primary container is often packaged in a secondary or outer container to protect against radioactive contamination. For long term storage, considerations must also be given for the degradation of container materials due to various causes (such as pressurization, radiolysis, radiolitic aging, chemical degradation, and so forth).

8.1.2 *Addition to Container*—The manner of adding WRM to containers depends on the nature of the material (for example, whether the WRM is solid, liquid, or gas), the type of container, and whether the weight of each WRM portion is required. It is exceedingly important that the WRM be delivered into each container without any part of the material adhering to the neck or top of the container (or outside of the container), particularly when solution is added to glass ampoules that will be heat sealed. Special apparatus is sometimes used for delivery to the containers (see Fig. 4 as an example). When a WRM is to be apportioned by weight, it is usually added to tared containers, which are reweighed after addition. When radioactive material is involved, special care is required to keep the outsides of the containers free of contamination. Each container should be surveyed after addition, and those contaminated should be discarded.

8.1.3 *Cover Gas*—With some materials, stability is enhanced by packaging the WRM in an inert gas or dry air. A common way to do this is to package in a glove box containing the atmosphere desired. The materials most often packaged in an inert and dry atmosphere or simply in dry air are the oxides, particularly powders. This is done to ensure stability and integrity, even when an oxide is basically stable. When a special atmosphere is used, care must be taken to ensure that containers will not lose the atmosphere over the shelf life of the WRM.

8.1.4 *Sealing Containers*—If a special atmosphere is used as discussed in 8.1.3, the method of sealing the containers is important. For screw cap containers, sealing the caps with a sealant over the cap is one way. Using glass ampoules that are heat sealed is another approach (a procedure for sealing glass ampoules is given in Appendix X4). Glass ampoules are commonly used for solutions to avoid loss of integrity through evaporation. When simply closing a vial or bottle with a screw cap is satisfactory, a cap liner that provides a reasonably air-tight seal should be used.

8.1.5 *Labeling*—Each WRM container should be labeled for identification. Individual identification of each container or unit is recommended, and required if each unit needs to be uniquely identifiable (for example, by a characteristic that affects the use of the WRM, such as the net weight of the

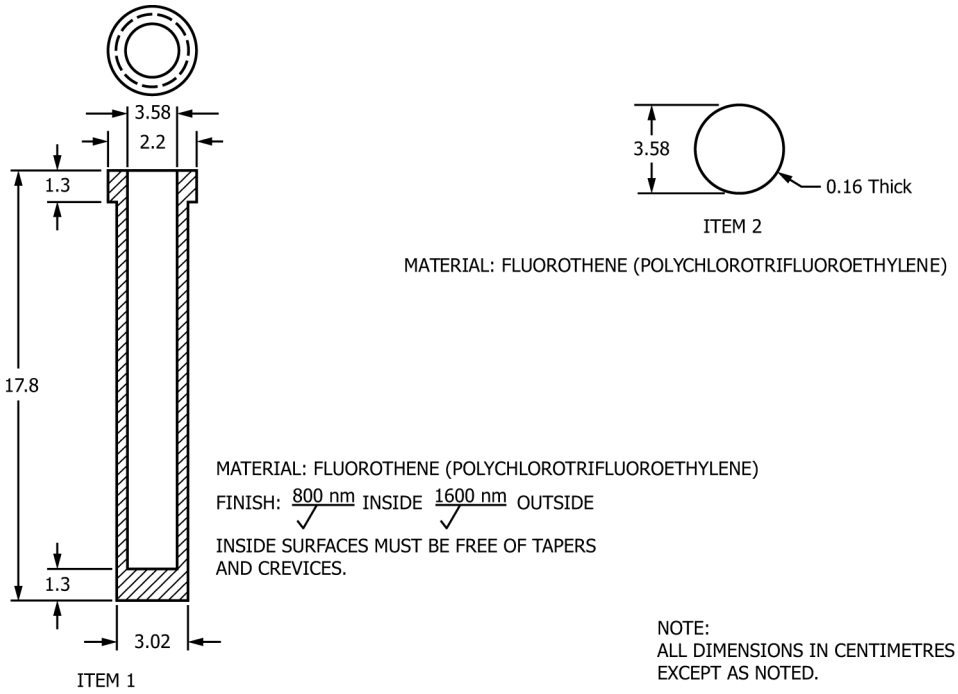

FIG. 4 Example of a Polychlorotrifluoroethylene (Fluorothene) Knockout Tube (used for uranium hexafluoride; see Practice C1689 for additional details)

WRM). As a minimum, information on a label must provide traceability to the WRM. It should have the date of preparation and must have shelf life information indicated on the label. It is essential that labels be firmly attached to the containers and that their markings be non-smearing and non-fading. Bar-code labeling may be desirable since more information can be added in a smaller space.

8.2 *Storage*—Although a major purpose of packaging is to preserve the integrity of WRMs, attention should also be given to how and where the packaged WRMs are stored. Exposure over time to large fluctuations in temperature, or to above-ambient temperatures, could adversely affect the container seals and the WRMs themselves. Exposure to conditions that would damage or destroy labels, or even damage containers, should be avoided.

8.3 *Transportation*—If the WRM is to be transported from one facility (such as a primary laboratory) to another (such as a satellite laboratory or a production facility), packaging needs to be sufficient for maintaining integrity, radiological control and safety, and applicable regulatory requirements.

9. Perform Homogeneity Study

9.1 *Summary*—WRMs are often subdivided from a large batch into individual units. The purpose of the homogeneity study is to assess the contribution of components of the uncertainty budget to the overall WRM expanded uncertainty due to heterogeneity between units. Special care should be taken when preparing and packaging the WRM to minimize heterogeneity in the certified value.

9.2 If subdivision of the WRM is planned, consideration should also be given in the planning phase to the need to evaluate homogeneity of bulk material before subdividing, as well as after subdividing.

9.3 *Acceptance Criteria*—The standard uncertainty of the between-unit homogeneity study (S_h) contributes to the overall combined standard uncertainty of the WRM assigned value. In some cases, the magnitude of S_h can be negligible in comparison to the batch characterization standard uncertainty (S_m) while at other times it may be equal to S_m. Acceptance criteria for the contribution of inhomogeneity to the overall combined standard uncertainty should be established based on fitness for purpose.

9.4 *Homogeneity Study:*

9.4.1 *Sampling*—The number of units selected for sampling should be economically feasible while still maintaining representation of the whole batch. Stratified random sampling should be employed for selection of the units. A minimum of two replicates should be measured in a random sequence in order to differentiate between a trend in unit filling sequence as opposed to analytical drift. The measurement sequence must be planned to detect analytical drift, otherwise a trend in filling sequence may be offset. For example, the first replicate measurement sequence can be measured in a random order and the second replicate measurement sequence could be a reverse of the first.

9.4.2 *Measurement Method*—The analytical method used for measurement requires good repeatability, otherwise S_h may be underestimated. Ideally, the uncertainty associated with method variability, ($s_r/\sqrt{n}$; where s_r is the repeatability standard deviation and n is the number of replicates) should be less than one-third of S_y. An increase in n, the number of replicates, can

make this achievable. If this is not possible, an increase in the between-unit homogeneity uncertainty estimate may result.

9.4.3 *Between-unit Homogeneity*—A between-unit homogeneity study should be performed when a batch of units has been prepared. Certainly, solids such as powders and ores are more heterogeneous in nature than solutions, however in all cases, homogeneity should be determined.

9.4.4 *Within-unit Homogeneity*—Within-unit homogeneity does not contribute to the WRM expanded uncertainty but rather acts as a study to determine the minimum amount of WRM in which the effect on the repeatability of measurement is negligible. Design of the experiment involves increasing the sample size and determining when the uncertainty of the measured property has sufficiently decreased.

9.5 *Statistical Evaluation:*

9.5.1 *Graphical Representation*—Data should be plotted into graphical form so a visual inspection can be performed. The graphical representation may indicate a trend in filling sequence or outliers, or both, among other things. Theoretically, the plot should approximate a trend line with a slope of zero. If there is a trend in the filling sequence, it should be reflected in the determination of S_h as discussed in 9.5.3.

9.5.2 *ANOVA*—If n > 1, analysis of variances (ANOVA) can be used to determine if a difference exists among the means of the WRM homogeneity study. The ANOVA data analysis add-on, available in commercial spreadsheet programs, is an excellent tool to use for this purpose.

9.5.3 *Determination of S_h*—Assuming the same number of replicates per subgroup, no drift in the unit filling sequence, and the data follows a normal distribution, S_h can be evaluated by Eq 1:

$$S_h = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (1)$$

where:

MS_{among} = mean of squares among units (calculated easily with ANOVA software),
MS_{within} = mean of squares within units (calculated easily with ANOVA software), and
n = number of replicates measured per bottle.

A trend in the filling sequence results in a rectangular distribution and must be accounted for using Eq 2

$$S_h = \frac{\max - \min}{2\sqrt{3}} \quad (2)$$

where:

max = maximum value in the filling sequence, and
min = minimum value in the filling sequence.

For studies in which good repeatability cannot be achieved, or a sufficient number of replicates cannot be sampled, several references are available to assist in the calculation of S_h **(1-3)**.

10. Perform Stability Studies

10.1 *Importance of Stability*—The stability of a WRM is an important parameter to consider when assessing the appropriateness of a reference material for an intended use. Over time, due to changes in environmental conditions, interactions with storage containers, and so forth, the WRM characteristics may change. Indeed, almost all materials "degrade" over time, some very slowly and others much more dramatically. Depending on the WRM, the stability of one or several parameters may be important. The extent to which these changes occur, and to what properties of the material they occur, must be understood. Then, this information can be compared to the intended use of the material in order to determine the suitability of the WRM over time.

10.2 *Parameters of Stability:*

10.2.1 *Parameter Selection*—Prior to preparing the WRM, the user should carefully determine the stability parameter or parameters that are important to the application of the WRM. The parameter can be the concentration of the element, the oxidation state, the chemical form, pH, and so forth. By carefully considering which parameter or parameters are critical to the application, the user will be able to devise an appropriate study to test the stability of the WRM. This ensures that the stability of the material is appropriate for the application, and can save time and resources by avoiding studies that are not necessary for the application.

10.2.2 *Acceptance Criteria*—The combined standard uncertainty of the stability parameter (or parameters) contributes to the overall combined standard uncertainty of the WRM assigned value. In some cases, the magnitude of this combined standard uncertainty can be negligible in comparison to the batch characterization standard uncertainty (S_m) while at other times it may be equal to S_m. It is ultimately the discretion of WRM users to establish the acceptance criteria for the contribution of instability to the overall combined standard uncertainty based upon a "fit-for-purpose" evaluation.

10.2.3 *Duration of Stability*—Certifying bodies are required to state shelf lives for their CRMs in order to inform the user about the expected lifetime of the CRM. For WRMs, the user should also formally document the shelf life of the material after stability tests have been performed (if necessary) to assess the degradation effect in time. Depending on the duration of intended use, the stability parameter may be evaluated over time under storage conditions and after repeated usage. Due to the time and resources required to prepare some WRMs used in the nuclear fuel cycle, recertification by the user after the expiration date according to the need of the user is a possibility. Depending on the use of the material, in-house re-characterization is often appropriate, but for some situations a third party re-characterization may be needed. The need for new stability studies on the re-characterized material should be evaluated, and justification for or against new studies should be documented. In some cases, if the material was used for instrument control, then the control charts data for a method can be used to justify the WRMs continued stability over time beyond the original expiration date.

10.3 *Conditions of Use*—The stability study will determine the impact of environmental and operational conditions on the WRM. It defines the storage conditions of the material (temperature, humidity, pressure, absence or presence of light, container material, matrix, acidity, and so forth) and the conditions for use of the material in order to meet the required performance of the material. It will define the duration of