Designation: ~~C1128 – 18~~ 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 ~~establishing traceability of WRMs to certified reference materials~~ value assignment. When traceability to SI is desired for a WRM, it will be achieved by a defined, statistically sound characterization ~~process.~~ 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 ~~preparation~~production and characterization of certified reference materials (CRM). Refer to ISO 17034 and ISO Guide 35 for ~~those~~ 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
~~Select, Collect, Prepare, and Process Materials~~	~~7~~
Prepare and Process Materials	7
~~Packaging and Store Materials~~	~~8~~
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] 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.

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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
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 ~~100:2008~~100 Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections (2008))
JCGM ~~200:2012~~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:*

[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.
[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.

3.2.1 *certified reference material ~~(CRM)—~~(CRM), n—*reference ~~material, accompanied by a certificate,~~ material (RM) characterized by a metrologically valid procedure for one or more ~~of whose property values are certified by a procedure, which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty interval at a stated level of confidence.~~specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability. **~~adapted from~~ 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 *certifying body*—technically competent body (organization or firm, public or private) that issues a reference material certificate which provides the information detailed in ISO Guide 31.~~ **~~ISO Guide 30~~**

3.2.2 ~~*characterization—*~~*characterization, n—of a reference material,* determination of ~~one or more physical, chemical, biological, or technological property values that are relevant to its intended end use.~~the property values or attributes of a reference material, as part of the production process. **~~adapted from~~ ISO Guide 30**

3.2.3 *combined standard ~~uncertainty—~~uncertainty, n—*standard uncertainty that is obtained using the individual standard uncertainties associated with the input quantities in a measurement model. **JCGM ~~200:2012~~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—~~factor, n—*number larger than one by which a combined standard uncertainty is multiplied to obtain an expanded uncertainty. **JCGM ~~200:2012~~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—~~uncertainty, n—*product of a combined standard uncertainty and a coverage factor. **adapted from JCGM ~~200:2012~~200**

3.2.7 ~~*homogeneity—*~~*homogeneity, n—*~~condition of being of uniform structure or composition with respect to one or more specified properties.~~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.~~adapted from ISO Guide 30~~

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—~~uncertainty, n—*non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand. **adapted from JCGM ~~200:2012~~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—~~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 ~~200:2012~~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**

3.2.10.1 *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)—~~(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.~~adapted from ISO Guide 80~~

3.2.13 *reference material ~~(RM)—~~(RM), n—*~~material or substance~~ material, sufficiently homogeneous and stable with respect to one or more ~~of whose property values are sufficiently homogenous and well established~~ specified properties, which has been established to be fit for its intended use ~~such as the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.~~in a measurement process. **ISO Guide 30**

3.2.13.1 *Discussion—*
~~Reference material property values are assigned by a procedure (or procedures) which establish the traceability of those values.~~Standards used for calibration and for quality control are two types of reference materials.

~~3.2.11.2 *Discussion—*~~
~~A reference material may be referred to in this guide also as a calibration standard or a control standard.~~

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

3.2.15 *reference material ~~document—~~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—~~method, n—*~~thoroughly investigated method, clearly and exactly describing the necessary conditions and procedures, for the measurement of one or more property values~~ measurement method, that has been shown to have ~~accuracy~~ the appropriate trueness and precision ~~commensurate with~~ for its intended use and ~~that can therefore be used to assess the accuracy of other methods for the same measurement, particularly in permitting the characterization of an RM.~~has been officially defined as reference method by a competent body. **ISO Guide 30**

3.2.17 *~~stability—~~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.~~period of time. **ISO Guide 30**

3.2.18 *standard ~~uncertainty—~~uncertainty, n—*measurement uncertainty expressed as a standard deviation. **JCGM ~~200:2012~~200**

3.2.19 *uncertainty ~~budget—~~budget, n—*statement of a measurement uncertainty, of the components of that measurement uncertainty, and of their calculation and combination. **JCGM ~~200:2012~~200**

3.2.20 *uncertainty ~~interval—~~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)—~~(WRM), n—*reference material, which is not certified, but is characterized using defined, statistically sound characterization processes, and can be used routinely to ~~calibrate~~ calibrate, control, or verify measuring instruments or measuring systems. ~~**adapted from JCGM 200:2012**~~

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.

~~3.2.19.1 *Discussion—*~~

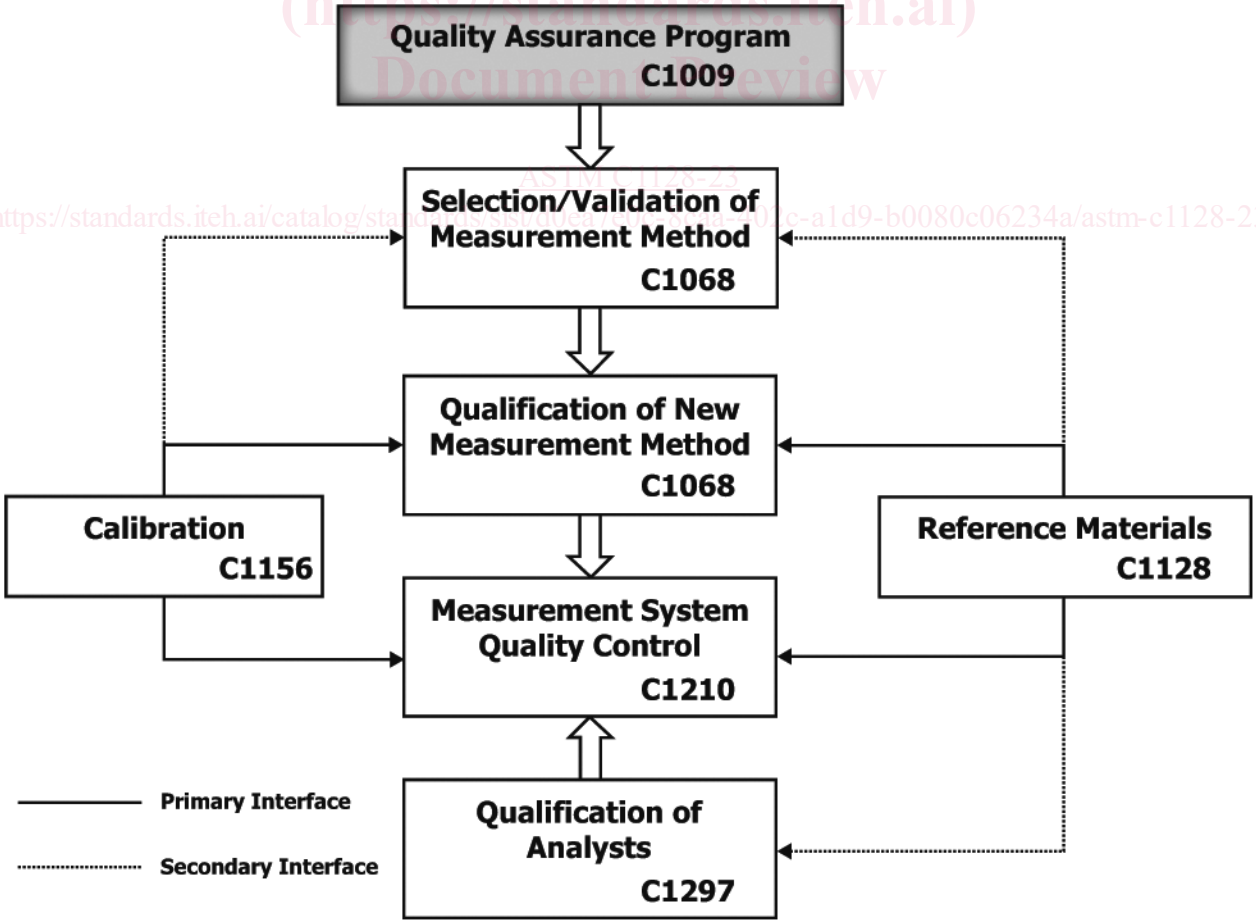

FIG. 1 ~~Quality Assurance~~Essential Elements of Analytical Laboratory ~~Data~~Quality Assurance System

~~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.~~

~~3.2.19.2 *Discussion*—~~

~~A WRM is usually prepared by a single laboratory for its own use as a calibration standard, as a control standard, or for the qualification of a measurement method (see Guide C1068) as indicated in Fig. 1.~~

~~3.2.19.3 *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~~—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 ~~for a stated purpose~~ (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.

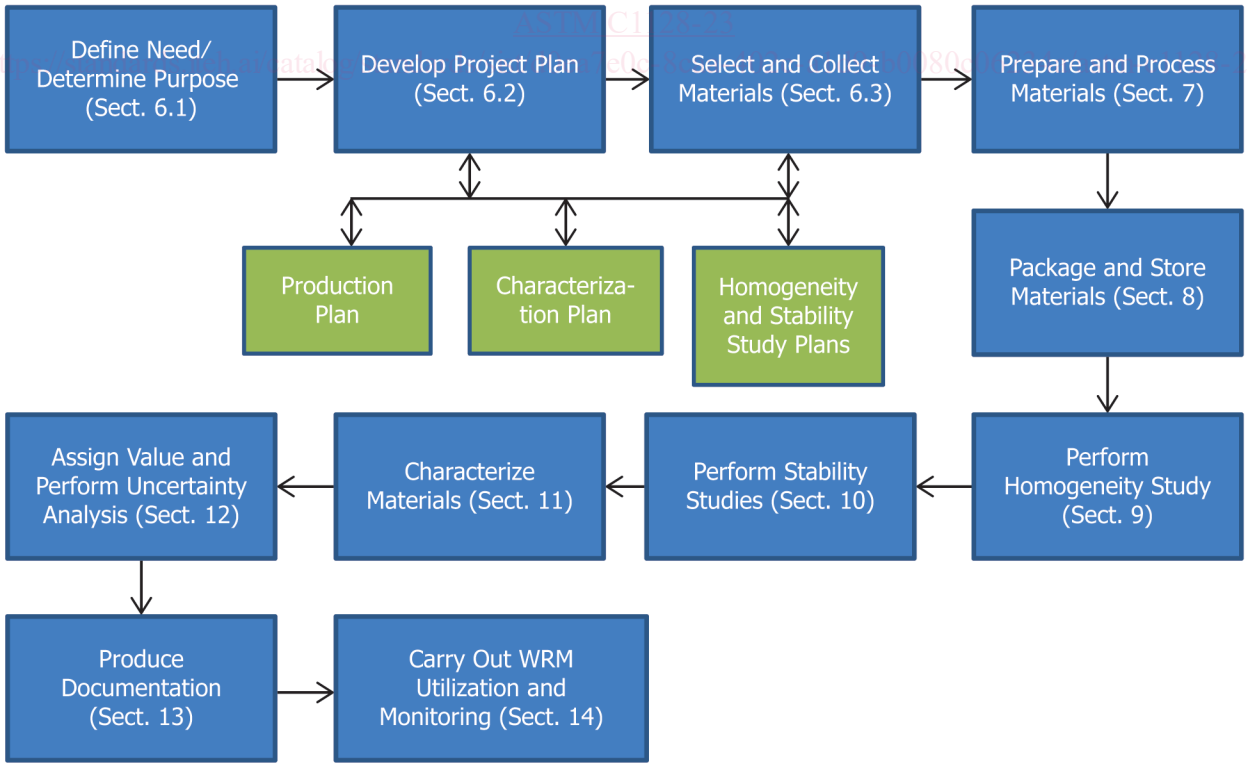

FIG. 2 General Process for Preparing Working Reference Materials

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.~~ CRM to provide metrological traceability. However, the preparation guidelines in ISO 17034 generally apply to ~~WRM.~~WRMs.

4.2.2.2 ~~WRMs provide~~ 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, ~~WRM~~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 ~~generally well characterized~~ 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, and they~~ 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, ~~producers~~production of WRMs should ~~comply~~ be in accordance with applicable requirements of ISO ~~17034, which are less stringent for WRMs than the requirements for producers of CRMs.~~ 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 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. Also, CRMs are often too expensive, or their supply is~~ 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

~~NOTE 1—Material from the planning modules in the 2016 IAEA Technical Meeting on the Preparation of Working Standards for Safeguards (1) have been used in this section.~~

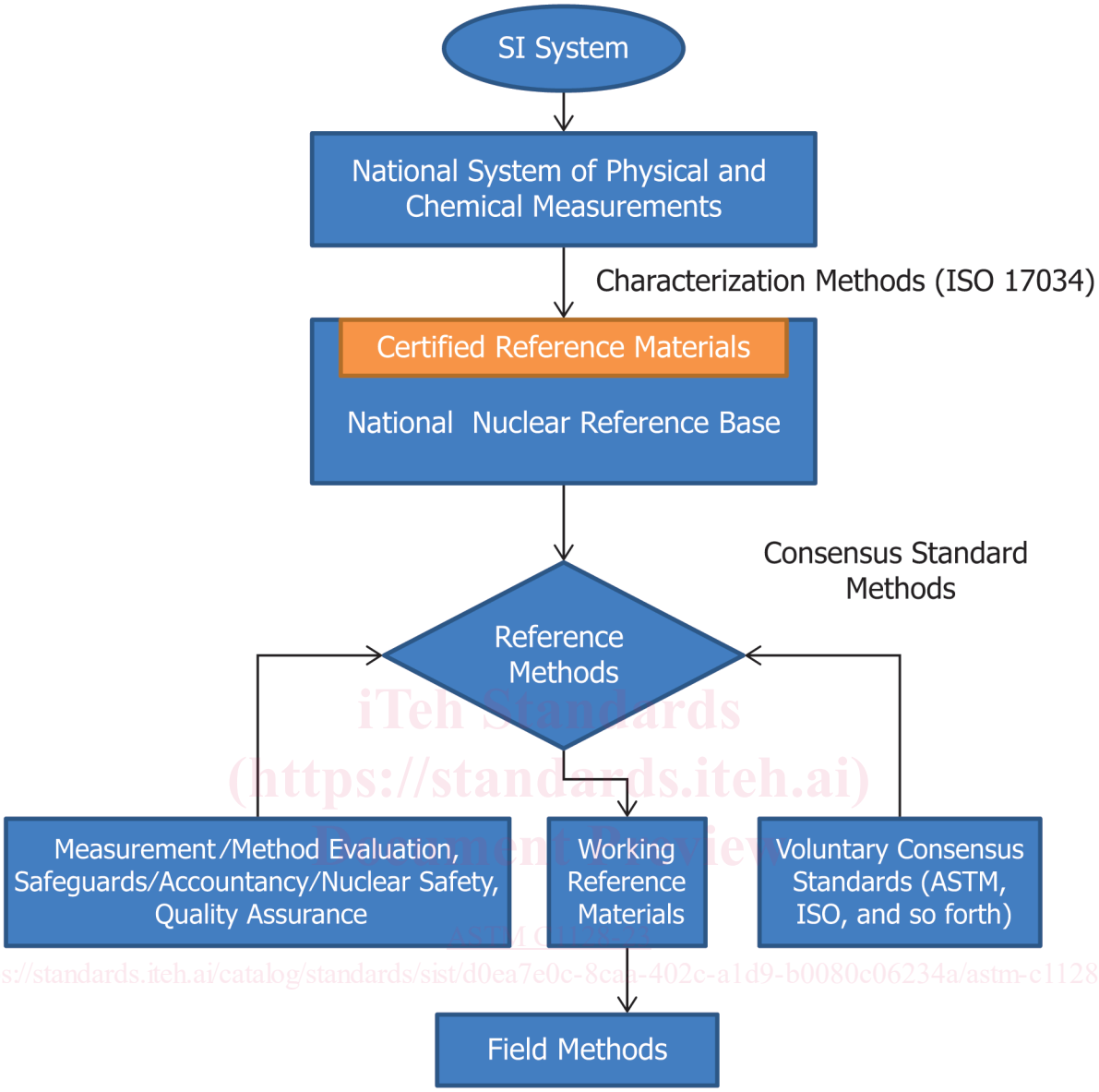

FIG. 3 Typical National Nuclear Measurement System

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 ~~the Scope:~~ 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:

(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 ~~*Define What is Needed*—Producing a WRM requires forethought to ensure the completed WRM meets the needs of the laboratory and its data users. A detailed plan that specifies needs and how the WRM will be produced should be prepared in advance of any work. The subjects discussed in this section should be considered and addressed as appropriate. Failure to properly define the needs can easily result in wasted time, funding, and materials, as well as requiring rework. Questions to ask in defining the need include, but are not necessarily limited to the following:~~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:

~~NOTE 2—Answers to some of the questions below may not be immediately available. An iterative process may be required to ask questions, collect information and then answer or re-answer one or more of the questions before proceeding to the project plan.~~

(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 ~~*How is the WRM Defined?*~~ The ~~definition should contain as much detail as possible to ensure that the final WRM meets the needs for the measurement and is fit for purpose. A fit for purpose checklist is included to assist in defining the principle attributes the need to have reference values. Questions to ask in defining the WRM include, but are not necessarily limited to the following:~~preparer of the WRM may document the basis for their make-versus-buy decision in their planning document.

~~*(1) What are the requirements of the project or customer?* What will the data be used for? What are the quality objectives for the project/customer? What information does the project/customer require?~~

(2) What Measurement Methods will the WRM Support? Will the WRM be used for a single method of analysis or for multiple methods? As an example, a WRM might be prepared for determination of uranium in uranium dioxide. If a WRM is also required for the isotopic abundance analysis of uranium, it might be possible to prepare and characterize the same WRM for isotopic abundance analysis as well.

NOTE 3—Careful consideration should be given to the preparation of multi-purpose WRMs, because they tend to be difficult to prepare and characterize.

(3) What are the Target Attributes (or properties) to be Characterized?

• Identify the target attributes for the WRM and their desired measurement uncertainties. This will be based on process knowledge of the measurement system and analytical quality control requirements for the samples. Examples of target attributes to consider may include concentration, matrix characteristics, particle size distribution, and isotopic composition.

• Acceptable measurement uncertainty for a given attribute is determined by the end use of the material (such as calibration or quality control) and the rigor and required precision to attain acceptable expanded uncertainty for the method as a whole (considering, for example, process control versus safeguards or accountancy).

• For example, to produce a WRM used for calibration of a measurement process, more care in preparation and rigor in characterization are required.

(4) Which analytical methods will be used to characterize the material? These will depend on how the material will ultimately be used and the acceptable expanded uncertainty, as well as the attributes of the WRM. Considering the methods used to characterize the material can also assist in the planning process (6.2.4).

(5) What are the Desired Chemical and Physical Properties? The WRM physical and chemical properties should match those of the samples as closely as possible, giving consideration for how the WRM will be used. The two largest sources of measurement uncertainty in an analytical protocol are the ones associated with sample preparation (digestion, separation, extraction, and so forth) and measurement of the analyte in that preparation. A WRM that more closely mimics the chemical and physical properties of the samples will provide a more accurate assessment of digestion procedures and the induced matrix effects and interferences for the analytes measured by a given measurement technique.

(6) What are the Impurity Limits or Concentrations Desired? This will be based on process knowledge for the measurement system and samples analyzed. Impurities which cause chemical or physical interferences in the measurement system will be important properties to consider for the WRM.

NOTE 4—Careful consideration should be given to the spiking and blending of impurities into a material. Artificially produced impurities may not match the chemical species of the samples and may give a false indication of the performance for sample preparations and measurements. Extra planning and preparation during production will have to be planned to match impurity species to those which are in the samples. Ideally to match the impurities of the WRM to a sample it is best to procure the raw material from an active process from which the samples are pulled for the analyses.

(7) What quantity is required? The quantity of WRM prepared will depend on such factors as the length of time required for its use, the quantity of material required for a measurement, the frequency of use, the amount of raw material available, and the WRM's anticipated for shelf life. Consideration will need to be given to the amount of WRM that will be needed for characterization (including for homogeneity and stability studies) and for archival purposes. Additional needs may develop during the use of a WRM such as the exchange of materials with another laboratory for an interlaboratory testing program. For this and other possible contingencies, the preparation of a quantity beyond the anticipated amount should be planned.

6.1.1.2 *What raw materials are available which meet the requirements?* See Section 6 for more information.

6.1.1.3 *What are the shelf life requirements?* When planning for shelf life, stability of the materials over time will also have to be considered in addition to degradation of container materials for the environmental conditions the units will be stored. Generally, materials used for standards in the nuclear fuels cycle are inherently stable but there are factors to consider for nuclear materials that will be sealed for long periods of time. These include buildup of gas and pressure within the container due to radiolysis or nuclear reactions from alpha decay with elements of the container walls. Also, change in mass for hydroscopic materials over time will need to be considered.

6.1.1.4 *How will the WRM be packaged and stored?* See Section 8 for more information. Depending on packaging requirements, equipment may be required for such things as sealing glass ampoules or packaging a WRM in a special atmosphere.

6.1.2 *Define When It Is Needed*—The need date will affect the amount of resources that need to be applied to preparing the WRM. It is based on the current and projected laboratory workload and existing supplies of WRM, if any.

6.1.3 *Define Collaborations and Resources*—A plan for collaborations and defining resources will ensure an efficient production campaign 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 outside resources for activities that the laboratory is not able to perform itself,

and so forth. An evaluation of any outside collaborators and their ability to meet performance requirements per the relevant ASTM and ISO standards is an important step in the planning process. Limitations on resources or collaborators can negatively impact the total time required to complete the project.

6.1.4 *Finalize the Scope*—The scope is finalized based on the considerations in 6.1.1 through 6.1.3. This may be an iterative process, balancing available resources with need dates.

6.1.5 *Document the Scope*—It is important to document the scope to ensure agreement and common understanding, and as the basis for the project plan. The degree of detail and complexity of documentation should be commensurate with the size and complexity of the project itself. An example of documentation is as follows:

6.1.5.1 *Summary statement* that identifies what analyte or analytes will be determined using the WRM; what matrix will be used; and uncertainty requirements (expressed as expanded uncertainty or combined standard uncertainty). The statement should also include an estimate of demand (how much will be used in a year or other appropriate time period). Traceability requirements should also be included.

6.1.5.2 *Estimated resource requirements,* including cost, personnel, use of laboratory equipment, and so forth. Availability of these resources should be verified.

6.1.5.3 *Estimated time* required to prepare the WRM. This time frame should not extend beyond the need date. If it does, consider options for reducing the estimated time.

6.3 *Develop the Project Plan—WRM Project Planning Documents*—Once the scope and available resources have been defined, a project plan should be developed for preparing the WRM. The project plan typically includes the following tasks: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.

NOTE 5—ISO Guide 35 provides additional guidance in aspects of developing the project plan.

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 ~~*Feasibility Studies*—These studies should be simple in scope. They are used in cases where sufficient knowledge or data~~ If sufficient information is not available ~~prior to execution of the plan and will ensure that the WRM produced will meet all requirements defined in the scope. Examples include the following.~~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:

~~6.2.1.1 Studies to determine if the material selected is fit for purpose according to the defined scope.~~

(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.

~~6.2.1.2 Studies to determine the best ways to process and prepare the raw material without compromising its desired property values.~~

~~6.2.1.3 Studies to test, improve, or verify that equipment, personnel, and measurement procedures meet standards for performance required for producing and characterizing the WRM to the specific requirements defined.~~

6.3.7 ~~*Homogeneity Assessment (Section 9)*—The project plan should include details for homogeneity studies. The results from the study are included in the measurand's expanded uncertainty.~~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 ~~*Stability (short and long term) Assessment and Monitoring (Section*~~ *Develop Plan for Production Execution:* ~~*10)*—The project plan should include details for stability studies. The results from the study are included in the measurand's expanded uncertainty.~~

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 ~~*Characterization Plan (Section*~~*Develop Plan for* ~~*11)*—~~*Characterization Execution:* ~~The project plan should include details for the characterization of the WRM. The plan should provide sufficient detail to ensure that the measurements will be performed as planned. The following are topics recommended to be addressed.~~

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 ~~*Traceability*—How traceability is established should be defined. An~~ 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. Consideration also~~ 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 ~~CRMs when available.~~a CRM when available. When designing an analysis plan consulting with a statistician experienced in analytical processes is recommended.

~~6.2.4.2 *Measurements*—Measurements should be made using procedures and protocols that are qualified and appropriately evaluated for analytical fitness for purpose in accordance with Guide C1068. Processing and measurement of blanks and controls should be included as appropriate to verify the validity of measurement procedures. Qualified and proficient operators should be used to operate analytical instrumentation. A feasibility study may be performed to validate that the methods used meet the established specifications and are otherwise fit for purpose when measuring the WRM aliquots.~~

~~6.2.4.3 *Design of Analysis Plan*—A well designed analytical plan will ensure the data generated during the characterization of the material and subdivisions used as WRMs in the laboratory will be sufficient to assign a measurand that is traceable to a certified property value and to allow for evaluations into matrix effects, interferences, and contaminations associated with the analytical procedures and measurements techniques used. When designing an analysis plan consulting with a statistician experience in analytical processes is recommended. In general, the material to be used for the production of a WRM is prepared in bulk taking into consideration homogeneity, stability, and the property values defined in the scope of work. An analysis plan for statistical sampling, preparation and analyses for both the processed bulk material and random samples population of WRM subdivisions should be described. The use of calibration standards, controls, and blanks should also be described. The analytical plan should identify any procedures that are required, such as for sampling and sample preparation. For further guidance see Section 11 of this guide and ISO 17034 which describes four basic approaches commonly used for characterization of reference materials and recommendations for when to use them.~~

~~NOTE 6—Defining the tolerance of the uncertainty interval during pre-planning is extremely important. The level of detail used in the statistical evaluation will depend on the number of measurements documented and performed according to the analysis plan. A small number of data points will limit the uncertainty analysis and could result in an expanded uncertainty which does not meet the needs of the end users. As mentioned above when designing an analysis plan, consulting with a statistician experienced in analytical processes is recommended.~~

~~6.2.4.4 *Reporting Sources of Uncertainties (Section 12)*—The analysis plan should provide for the generation of sufficient data sets to allow for an uncertainty analysis. The project should include a detailed uncertainty analysis of the data used to assign the final measurand for the WRM. All sources of uncertainty that could be a significant contributor to the propagated uncertainty of the assigned value should be included in the calculation and the uncertainty budget.~~

~~6.2.4.5 *Reporting of Results*—A template for reporting each measurement result from each laboratory should be included. Enough detail about the measurement should be included to allow for an evaluation of its performance and validity of the measured value. Laboratory measurements should be reported with an expanded uncertainty including the coverage factor or confidence interval applied. Calculating uncertainties in accordance with JCGM 100:2008 is recommended.~~

6.3.9.4 ~~*Assignment of Measurand*—~~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 ~~*Documentation and Records*—Useful information that should be retained and available to auditors, stakeholders, and users~~If any production or characterization tasks will be performed by subcontractors, the preparer of the WRM ~~are similar to those suggested for production of QCMs. See ISO Guide 80 for further information. Pertinent documents are records should be maintained for the lifetime of the WRM then a pre-determined length of time afterwards according to the laboratory's quality assurance program and record management procedures.~~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 a schedule for the project with sufficient level of detail.~~*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.

~~6.2.6 Identify or develop procedures required for the preparation activities.~~

~~6.2.7 Identify materials required (see Section 7) and, if they must be procured, the procurement requirements including lead time.~~

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~~ 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.

~~6.2.9 The project plan should include requirements for packaging and storage (see Section 8).~~

~~6.2.10 Applicable requirements of ISO 17034 and ISO/IEC 17025 should be addressed. If the laboratory has a quality system that is compliant with these standards, a reference to quality system documentation may be sufficient.~~

~~6.2.11 If any work will be performed by subcontractors, they should have the necessary competence to perform the work that is assigned to them.~~

~~NOTE 7—The amount of detail in the project plan, and the level of effort to prepare it, are 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.~~

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. ~~Select, Collect, Prepare,~~ Prepare and Process Materials

7.1 ~~*Select and Collect Materials:*~~ *Preparation:*

7.1.1 ~~*Selecting Materials*—~~The ~~selection of materials is an important part of planning. Proper selection is critical to achieving credible WRMs. Selection depends on availability (source), cost, chemical and physical properties, and stability or reproducible stoichiometry. The starting materials for the preparation of WRMs may already be in the desired form or may be other materials that are processed into those forms. In the former case, the starting material is process material. For example, a batch of uranium dioxide pellets, boron carbide powder, or plutonium nitrate solution might be taken directly from a process run, treated as necessary, characterized, and packaged as a WRM. 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~~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 ~~$_3O_8$~~) ~~to prepare a WRM or matrix material (see Appendix X2). Considerations for the selection of materials include, but are not necessarily limited to, the following:~~the materials involved. Various aspects of preparation are discussed in this section.

~~7.1.1.1 The material should meet appropriate requirements for initial purity and composition.~~

~~7.1.1.2 The material selected for production of a WRM must be as similar as possible to the sample material in chemical and physical properties, particularly in those that will affect the method of analysis. One way to achieve similarity in composition is to prepare the WRM by the same or similar process used to prepare the sample material.~~

~~7.1.1.3 Homogeneity should be considered based on how the WRM will be used. Usually, WRMs are used for calibration and measurement control. A common approach to producing a control standard is to take material from a batch of production material, treat it as necessary to ensure homogeneity, and establish initial measurement control limits by using the same method and conditions used for sample analysis. To produce a calibration standard, more care in preparation and rigor in characterization are required. See Section 9 for assessment of homogeneity.~~

~~7.1.1.4 The WRM composition must be sufficiently stable to make the preparation of the WRM cost effective, and the stability must be known well enough to establish a shelf life with a high degree of confidence. Given the presence of radioactive constituents in WRMs, it may be necessary to account for radioactive decay as a function of time. See Section 10 for assessment for stability.~~

~~7.1.1.5 Depending on historical data or process knowledge a feasibility study may be needed to determine if one or more of the criteria listed above for the selected material meets the requirements defined for the WRM.~~

7.1.2 ~~*Collect Materials*—When materials are not available in house, consideration should be given to cost, transportation logistics and lead time, and whether certification or other documentation of composition, or purity, or both, is required.~~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.