Designation: C1128 – 18

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 preparation, packaging, and storage; requirements for characterization; homogeneity and stability considerations; and establishing traceability of WRMs to certified reference materials by a defined, statistically sound characterization process. 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 and characterization of certified reference materials (CRM). Refer to ISO 17034 and Guide 35 for those 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
Packaging and Store 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. 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 the 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 the Determination of Impurities in Plutonium Metal: Acid Digestion and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Analysis

[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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[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.

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

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 Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections (2008))
JCGM 200:2012 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)*—reference material, accompanied by a certificate, 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. **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**

3.2.3 *characterization*—determination of one or more physical, chemical, biological, or technological property values that are relevant to its intended end use. **adapted from ISO Guide 30**

3.2.4 *combined standard uncertainty*—standard uncertainty that is obtained using the individual standard uncertainties associated with the input quantities in a measurement model. **JCGM 200:2012**

3.2.4.1 *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.

3.2.5 *coverage factor*—number larger than one by which a combined standard uncertainty is multiplied to obtain an expanded uncertainty. **JCGM 200:2012**

3.2.6 *expanded uncertainty*—product of a combined standard uncertainty and a coverage factor. **adapted from JCGM 200:2012**

3.2.7 *homogeneity*—condition of being of uniform structure or composition with respect to one or more specified properties. **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**

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

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*—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**

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

3.2.10.1 *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**

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

3.2.10.3 *Discussion*—QCMs have assigned values that are indicative, and do not require characterization by metrologically valid procedures. As such, QCMs cannot be expected to

[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.

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**

3.2.11 *reference material (RM)*—material or substance one or more of whose property values are sufficiently homogenous and well established for its intended use such as the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. **ISO Guide 30**

3.2.11.1 *Discussion*—Reference material property values are assigned by a procedure (or procedures) which establish the traceability of those values.

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

3.2.12 *reference material certificate*—document accompanying a certified reference material stating one or more property values and their uncertainties, and confirming that the necessary procedures have been carried out to ensure their validity and traceability. **ISO Guide 30**

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

3.2.14 *reference method*—thoroughly investigated method, clearly and exactly describing the necessary conditions and procedures, for the measurement of one or more property values that has been shown to have accuracy and precision commensurate with 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. **ISO Guide 30**

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

3.2.16 *standard uncertainty*—measurement uncertainty expressed as a standard deviation. **JCGM 200:2012**

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

3.2.18 *uncertainty interval*—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**

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

3.2.19.1 *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.

3.2.19.2 *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*—degree to which a WRM, when used 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 *qualification*—process of determining WRM to be fit for purpose.

3.3.2.1 *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.

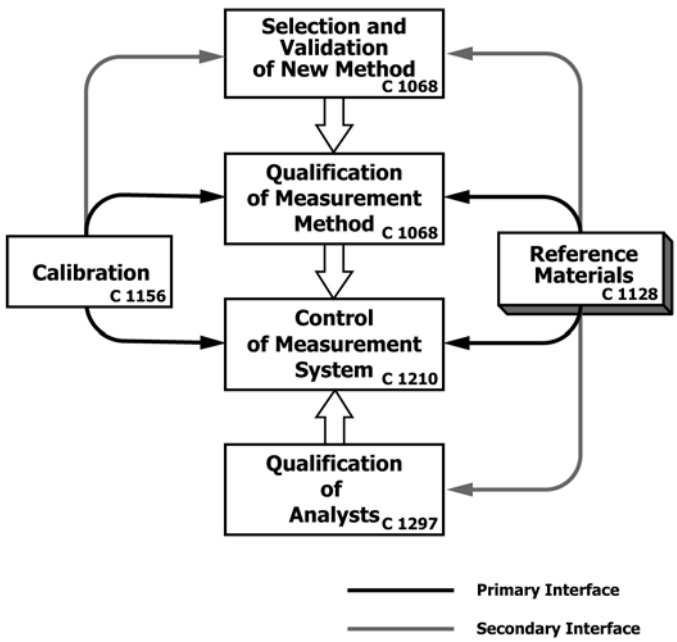

FIG. 1 Quality Assurance of Analytical Laboratory Data