



Designation: C1128 – 23

Standard Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials¹

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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.

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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.

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 Laboratories³

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 homogeneous 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.