



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

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 destructive analysis of nuclear fuel cycle materials. Guidance is provided for establishing traceability of WRMs to certified reference materials by a defined characterization process. The guidance provided is generic; it is not specific for a given material.

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

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1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

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](#)
[C1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry](#)

2.2 ISO Standards:³

- [ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories³](#)
- [ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials³](#)
- [ISO Guide 34 General Requirements for the Competence of Reference Material Producers](#)

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 200:2008 International Vocabulary of Metrology—Basic and General Concepts and Associated Terms \(VIM\) \(ISO/IEC Guide 99\)](#)

3. Terminology⁵

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *certified reference material (CRM)*⁶—a reference material with one or more property values that are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body (as defined by ISO Guide 30). A certifying body is a technically competent body (organization or firm, public or private) that issues a reference material certificate (as defined by ISO Guide 30). A reference material certificate is a document certifying one or more property values for a certified reference material, stating that the necessary procedures have been carried out to establish their validity (as defined by ISO Guide 30).

3.1.2 *reference material (RM)*⁶—a material or substance one or more properties of which are sufficiently well established to

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 Feb. 1, 2015. Published February 2015. Originally approved in 1989. Last previous edition approved in 2008 as C1128 – 01 (2008). DOI: 10.1520/C1128-15.

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 See C859 for other terms specific to the nuclear fuel cycle.

⁶ It is important that a well defined uncertainty in the stated value(s) be given in the certificate.

be used for the calibration of an apparatus, the assessment of a measurement method, or assigning values to materials (as defined by ISO Guide 30). A reference material may be referred to in this guide also as a standard, such as calibration standard or control standard.

3.1.3 *working reference material (WRM)*⁶—a RM 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.

4. Summary of Guide

4.1 This guide covers the preparation of WRMs from nuclear fuel cycle materials. 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. Because this guide is generic, requirements and detailed information for specific nuclear materials are not given. A flow diagram to illustrate an approach to producing WRMs is given in Fig. 2.

5. Significance and Use

5.1 Certified reference materials (CRMs) prepared from nuclear materials are generally of high purity, possessing chemical stability or reproducible stoichiometry. Usually they are certified using the most unbiased and precise measurement methods available, often with more than one laboratory being involved in making certification measurements. CRMs are generally used on a national or international level, and they are at the top of the metrological hierarchy of reference materials. A graphical representation of a 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

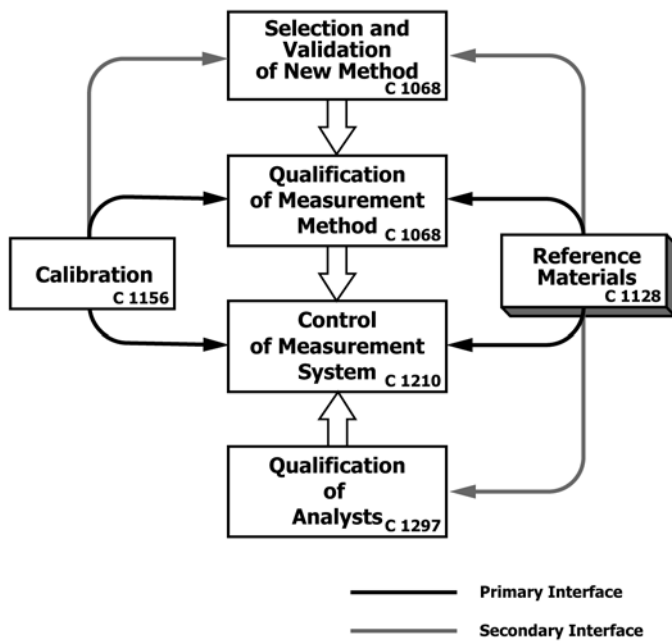


FIG. 1 Quality Assurance of Analytical Laboratory Data

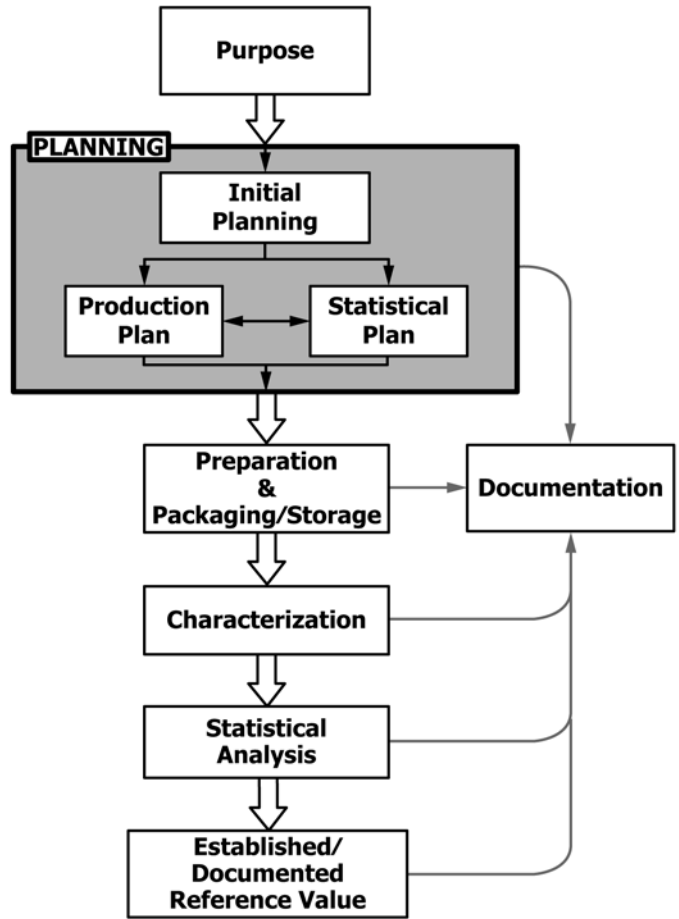


FIG. 2 Producing a Working Reference Material

rigor used to achieve those characteristics is not usually as stringent as for CRMs. Similarly, producers of WRMs should comply with applicable requirements of ISO Guide 34, which are less stringent for WRMs than the requirements for producers of CRMs. Where possible, CRMs are often used to calibrate the methods used for establishing the concentration values (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 materials, particularly for control and accountability purposes, CRMs are sometimes 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 WRMs from nuclear materials. Also, CRMs are often too expensive, or their supply is 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 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, and radioactivity are

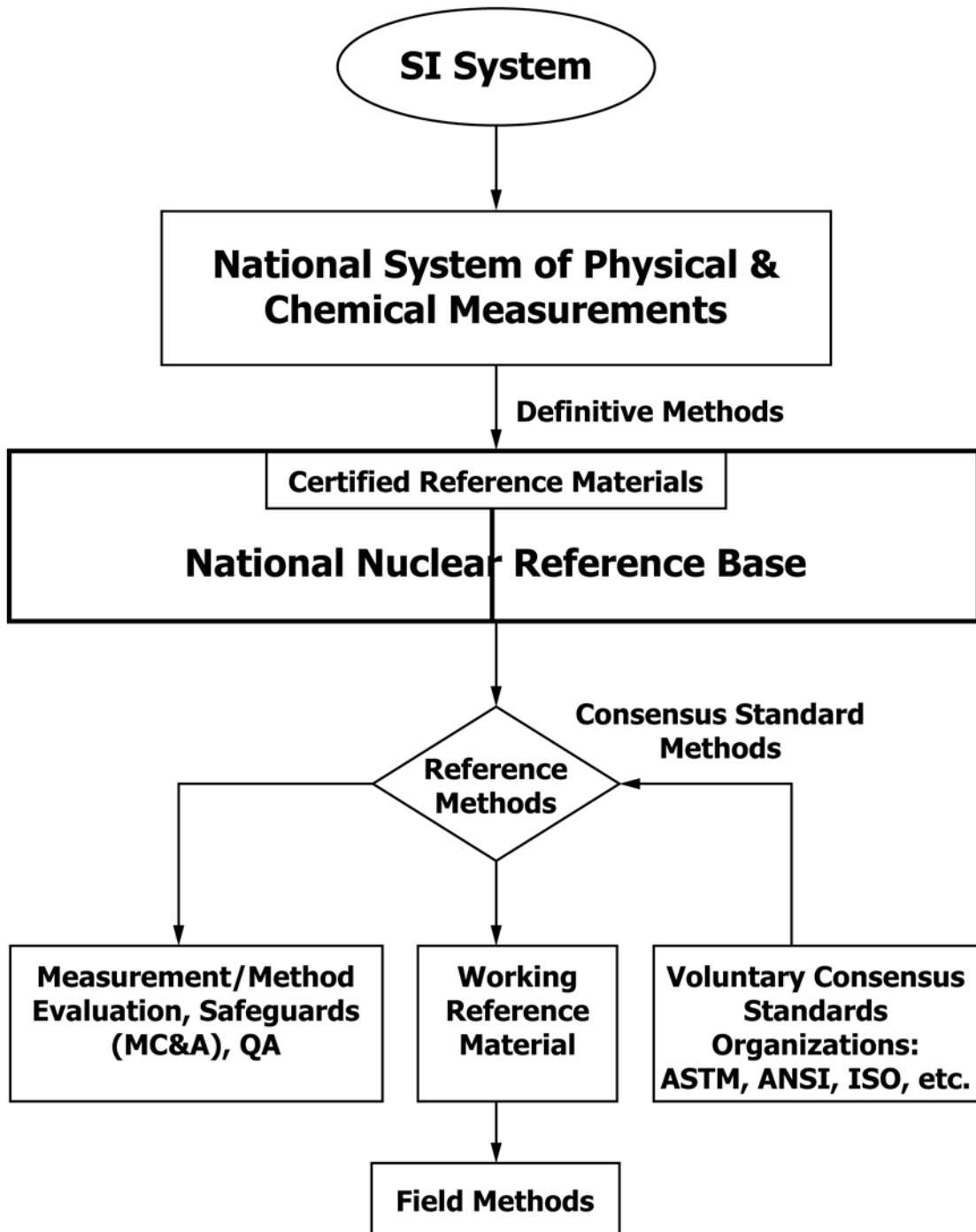


FIG. 3 National Nuclear Measurement System

factors involved, with all three factors being involved with some materials. Those preparing WRMs from nuclear materials must be aware of how these factors affect preparation, as well as being aware of the other criteria governing the preparation of reliable WRMs.

6. Planning

6.1 Producing a WRM requires forethought to ensure the completed WRM meets the needs of the laboratory and its data users. Planning also ensures that the necessary resources are

available. Time, funding, and materials can be wasted easily without thorough planning. Planning should include developing an outline or general scheme for preparing the WRM. The intended use of the WRM, the sources available for obtaining needed materials, and the equipment required are some areas of planning that should be considered. These considerations and others, that is, initial planning, a production plan, and a statistical plan (see Fig. 2), are discussed in this section. Initial planning generally starts with the application or need for a WRM and the quantity needed. As planning progresses into the