Designation: C 1128 – 01

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

This standard is issued under the fixed designation C 1128; 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 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:

	Section
Planning	6
Preparation	7
Packaging and Storage	8
Characterization	9
Statistical Analysis	10
Documentation	11

1.3 The values stated in SI units are to be regarded as the 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:*

C 859 Terminology Relating to Nuclear Materials[2]

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry[2]

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards in the Nuclear Industry[2]

2.2 *ISO Standards:*

ISO Guide to the Expression of Uncertainty in Measurement[3]

ISO 17025 General Requirements for the Competence of Calibration and Testing Laboratories[3]

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials[3]

3. Terminology [4]

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *certified reference material (CRM)*[5]—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)*[5]—a material or substance one or more properties of which are sufficiently well established to 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)*[5]—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 C 1068) 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

[1] This guide is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.8 on Quality Assurance Applications.

Current edition approved June 10, 2001. Published September 2001. Originally published as C 1128 – 89. Last previous edition C 1128 – 95.

[2] *Annual Book of ASTM Standards*, Vol 12.01.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] See C 859 for other terms specific to the nuclear fuel cycle.

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

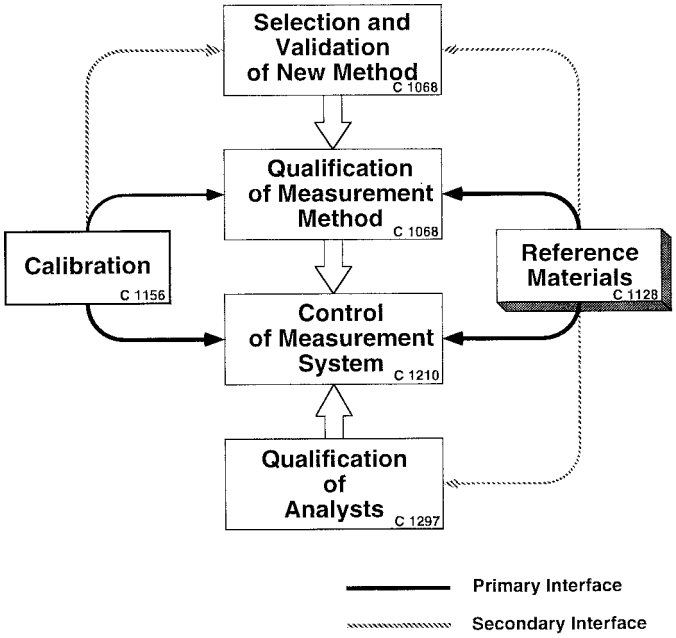

FIG. 1 Quality Assurance of Analytical Laboratory Data

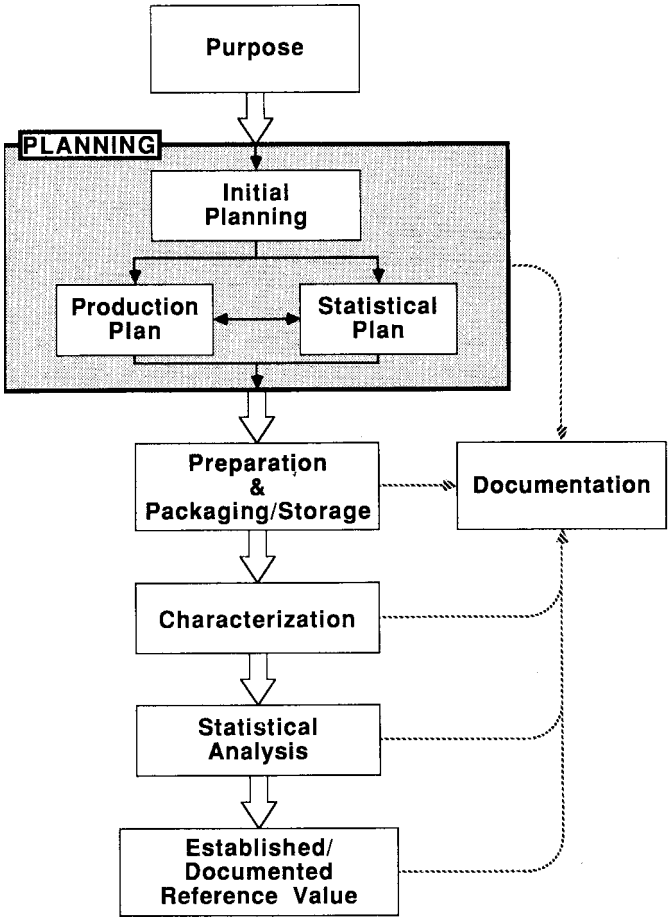

FIG. 2 Producing a Working Reference Material

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 rigor used to achieve those characteristics is not usually as stringent as for 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 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 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 credibility of the completed WRM. 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, i.e., 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 actual preparation, a

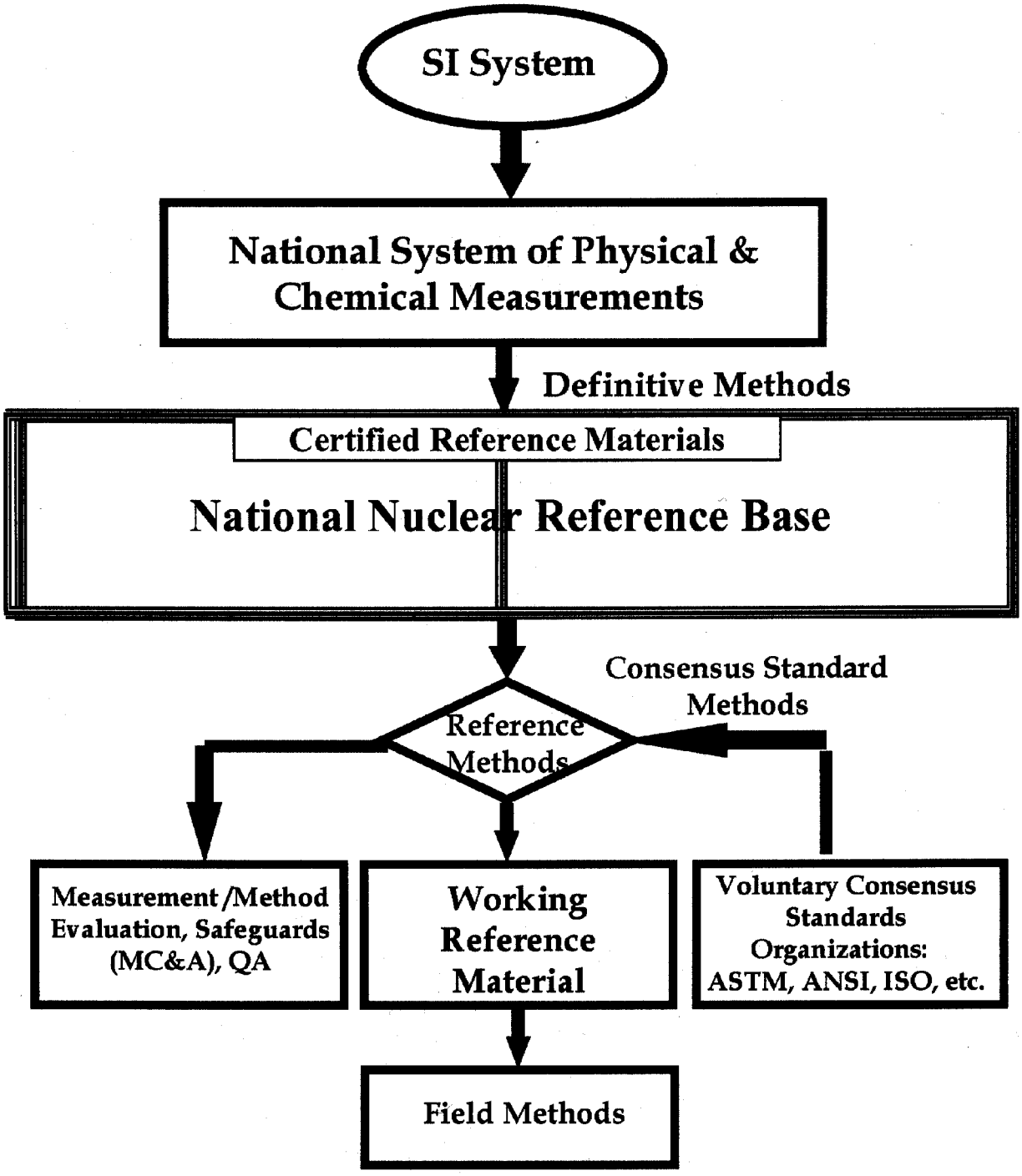

FIG. 3 United States Nuclear Measurement System

production plan and a statistical analysis plan will be developed.

6.2 *Initial Planning*:

6.2.1 *Application of WRM*—A WRM can be prepared for a single method of analysis or for several methods. For example, one might be prepared for the determination of uranium in uranium dioxide. If a standard is also required for the isotopic analysis of uranium, it might be possible to prepare and characterize that WRM for isotopic analysis as well. During the preparation of a WRM for the determination of a major constituent, it might be possible to add desired impurities and to establish values for those impurities. Careful consideration should be given to the preparation of multi-purpose WRMs, however, because they tend to be difficult to prepare and characterize.