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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 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
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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 a Quality Assurance Program for Analytical Chemistry 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 Guide to the Expression of Uncertainty in Measurement
- ISO 17025 General Requirements for the Competence of Calibration and Testing Laboratories³
- ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials³

3. Terminology⁴

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 C1068) as indicated in Fig. 1.

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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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁴ See C859 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.

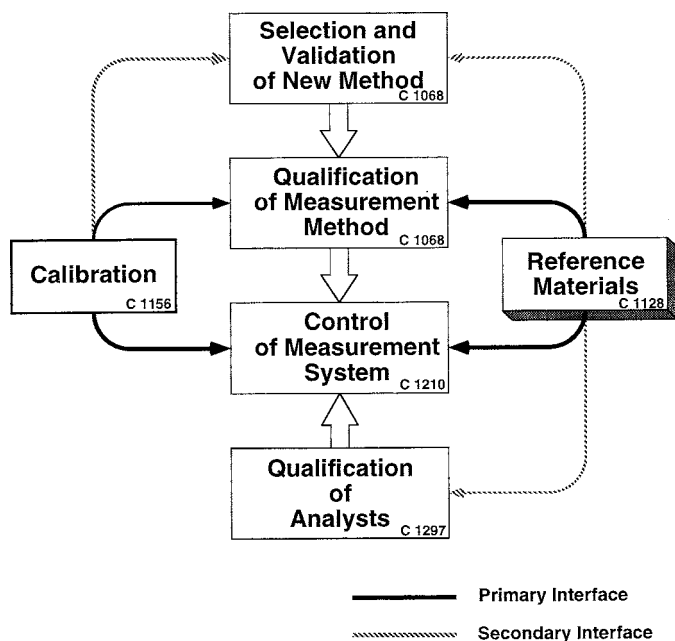


FIG. 1 Quality Assurance of Analytical Laboratory Data

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 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

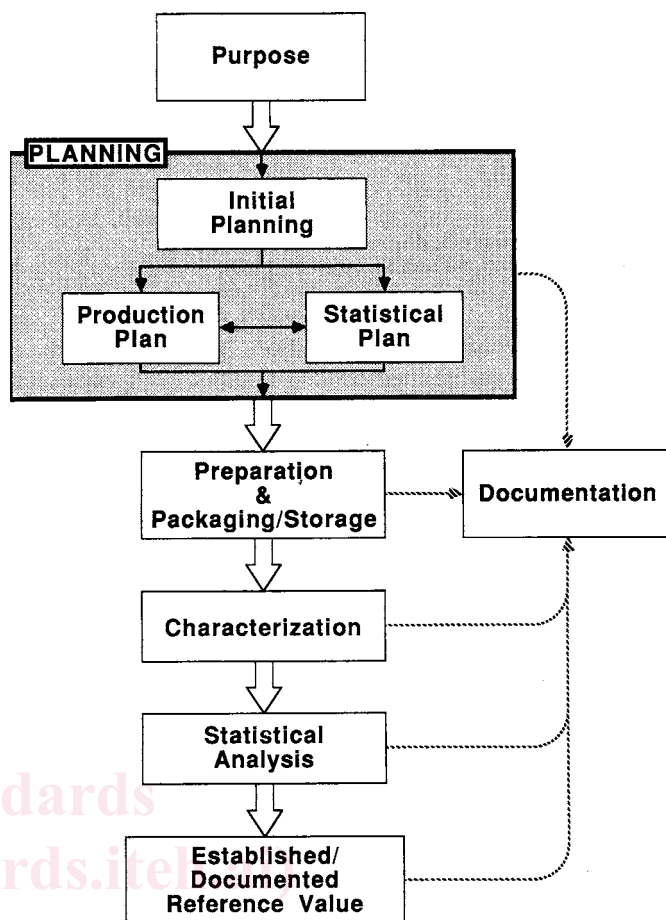


FIG. 2 Producing a Working Reference Material

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,

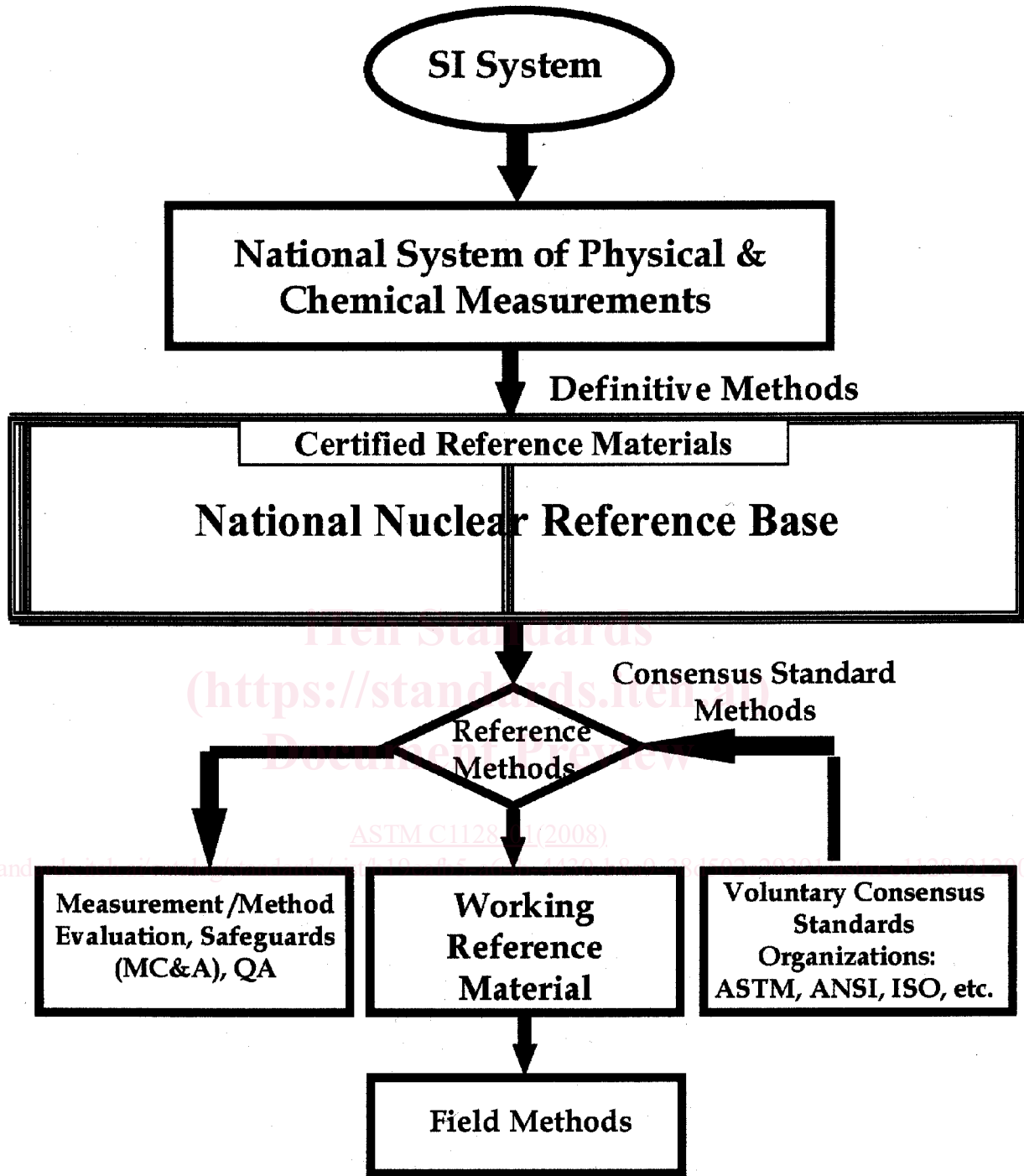


FIG. 3 United States Nuclear Measurement System

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 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.

6.2.2 Quantity—The quantity of WRM prepared will depend on such factors as the length of time required for its use, the frequency of use, the amount of material available, and the WRM's anticipated shelf life. Consideration should be given to the amount of WRM that will be needed for characterization and for archival purposes. 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 over the anticipated amount should be planned.

6.3 Production Plan—An outline that specifies how the WRM will be produced should be prepared during planning. The subjects discussed in 6.2 and in this section should be considered and addressed if appropriate. A preparation procedure should be written and included as a part of the production plan (see 7.4). The production plan must be integrated with the statistical plan (see 6.4).

6.3.1 Materials—The selection of materials is an important part of planning because proper selection is critical to achieving credible WRMs. Selection depends on availability (source), cost, chemical and physical properties, and stability or reproducible stoichiometry. The material selected for 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 material by the same or similar process used to prepare the sample material. Probably the most important criterion for selection is stability. 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. Somewhat unstable materials whose stoichiometries can be reproduced easily can be used for WRMs.

6.3.2 Equipment—Generally, standard laboratory equipment will be involved in preparing a WRM. Analytical setups and instrumentation will be required, possibly to analyze starting materials for impurities and other constituents and certainly to analyze the prepared material during final characterization of the WRM. Depending on packaging requirements, equipment may be required for such things as sealing glass ampoules or packaging a WRM in a special atmosphere.

6.3.3 Use—The degree of attention given to some steps in producing a WRM may vary depending on its planned use. 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.

6.3.4 Characterization of Materials—Planning must provide for the characterization of materials used for a WRM (See

Appendix X1). Characterization may include the analysis of starting materials for impurities and major constituents. It should include a scheme for establishing the value to be assigned (reference value) to each constituent of interest. In planning for characterization, consideration must be given to the degree of reliability required for a reference value. This will involve planning for the statistical collection and analysis of characterization data (see 6.4).

6.3.5 Packaging—Packaging of the WRM should be planned. Decisions need to be made concerning the division of the WRM into portions, selecting containers, uniquely identifying containers, sealing containers, and using additional means to protect the integrity of the WRM. It may be necessary to package some WRMs soon after preparation to preserve integrity; in that case, packaging materials and equipment should be readied prior to material preparation. Inadequate packaging may lead to loss of the WRM's integrity through such consequences as contamination, evaporation, degradation and absorption.

6.4 Statistical Plan—A statistical plan for characterization should be developed during planning. Such a plan is necessary to allow an uncertainty to be determined for each reference value. The statistical plan establishes how characterization will be done. It includes sampling of the WRM, the frequency and number of measurements to be made of the WRM, any reference material to be measured with the WRM, and the order of measurements (see 9.3 and 9.4). The validation or calibration of the measurement method to be used for characterization may be addressed in the plan also (see 9.2.3). It is essential to have a qualified statistician involved in developing the plan, and the statistician should be brought into the planning process early (see Fig. 2). Developing a statistical plan is an iterative process that will go on throughout planning, and it must be integrated with the production plan (see 6.3).

7. WRM Preparation

7.1 The 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 the materials involved. Various aspects of preparation are discussed in this section.

7.2 Starting Materials—The starting materials for the preparation of WRMs may be the WRM forms desired 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_3O_8) to prepare a WRM or matrix material (see **Appendix X2**).

7.3 WRM Form—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.

7.4 *Procedure*—A preparation procedure should be written using a scheme for preparing the WRM developed during the planning stage (see 6.3). The procedure should include the necessary steps for making the required chemical and physical manipulations, and it should include requirements for recording data generated during preparation. If it is planned that the reference value will be calculated based on process or make-up parameters (weights, volumes, etc.), write the procedure accordingly to minimize the possibilities of losing any material during processing (see 9.1). Procedures to illustrate the preparation of two WRM solutions are given in Appendix X3.

8. Packaging and Storage

8.1 *Packaging*—Once preparation is complete, the WRM is packaged for use. A frequent practice is to divide the WRM material into essentially equal portions or units, each of which represents enough material for a one-time use. If a WRM is sufficiently stable, it could be divided into larger portions for multiple use. There is a risk here, however, because each time a container is opened there is a potential for loss of WRM integrity. The key to packaging is to contain the WRM portions in such a manner as to preserve their integrity for the life of the WRM (see 6). A technique sometimes used for solutions is to evaporate each weighed portion to near-dryness in its packaging container, giving a weighed amount of the element of interest for a one-time use. Various aspects of packaging are discussed in this section. A procedure to illustrate packaging a WRM solution is given in Appendix X3.

8.1.1 *Container*—Containers often used for WRMs are screw-cap glass vials or bottles and glass ampoules. It is

important that the container material is compatible chemically with the WRM matrix and that the material will not contribute to the contamination of the WRM. To avoid contamination, containers are often specially cleaned before packaging. When radioactive material such as plutonium is involved, the primary container is often packaged in a secondary or outer container to protect against radioactive contamination.

8.1.2 *Addition to Container*—The manner of adding WRM material to containers depends on the nature of the material, the type of container, and whether the weight of each WRM portion is required. It is exceedingly important that the WRM material be delivered into each container without any part of the material adhering to the neck or top of the container (or outside of the container), particularly when solution is added to glass ampoules that will be heat sealed. Special apparatus is sometimes used for delivery to glass ampoules (see Fig. 4 as an example). When a WRM is to be apportioned by weight, WRM material is usually added to tared containers, which are reweighed after addition. When radioactive material is involved, special care is required to keep the outsides of the containers free of contamination. Each container should be surveyed after addition, and those contaminated should be discarded.

8.1.3 *Cover Gas*—With some materials, stability is enhanced by packaging the WRM in an inert gas or dry air. A common way to do this is to package in a glove box containing the atmosphere desired. The materials most often packaged in an inert and dry atmosphere or simply in dry air are the oxides, particularly powders. This is done to ensure stability and integrity, even when an oxide is basically stable. When a special atmosphere is used, care must be taken to ensure that containers will not lose the atmosphere over the shelf life of the WRM.

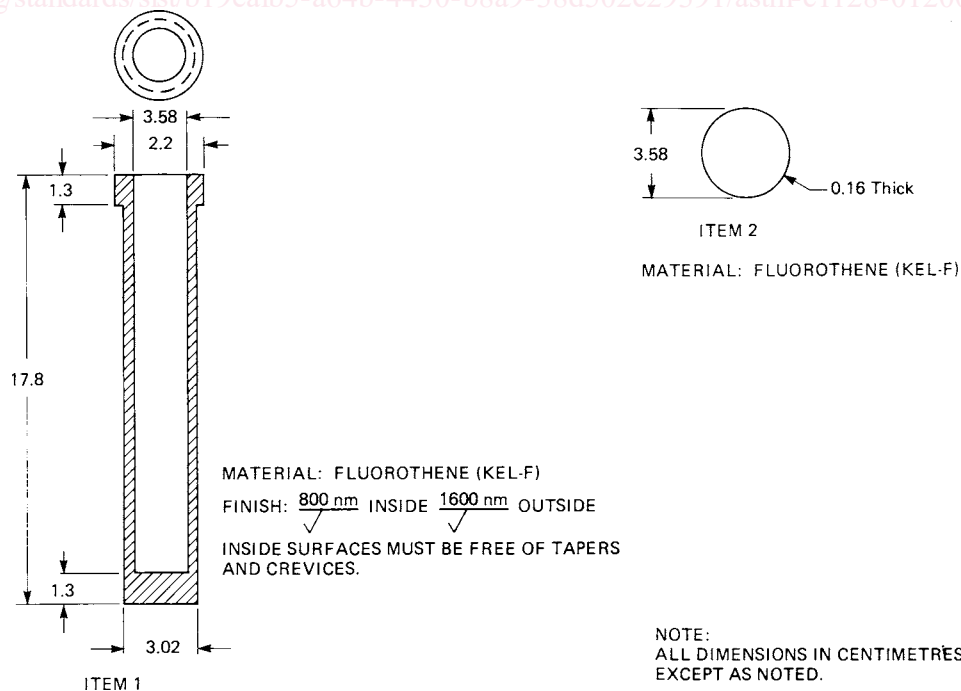


FIG. 4 Fluorothene Knockout Cylinder

8.1.4 *Sealing Containers*—If a special atmosphere is used as discussed in 8.1.3, the method of sealing the containers is important. For screw cap containers, sealing the caps with a sealant over the cap is one way. Using glass ampoules that are heat sealed is another approach (a procedure for sealing glass ampoules is given in Appendix X3). Glass ampoules are commonly used for solutions to avoid loss of integrity through evaporation. When simply closing a vial or bottle with a screw cap is satisfactory, a cap liner that provides a reasonably air-tight seal should be used.

8.1.5 *Labeling*—Each WRM container should be labeled for identification. Individual identification of each container or unit is not usually required unless each unit is uniquely identifiable by a characteristic that affects the use of the WRM, such as the net weight of the WRM in the container. As a minimum, information on a label must provide traceability to the WRM. It would be desirable to have the date of preparation and any limitations on shelf life indicated on the label. It is essential that labels be firmly attached to the containers and that their markings be nonsmearing and nonfading. Bar-code labeling may be desirable since more information can be added in a smaller space.

8.2 *Storage*—Although a major purpose of packaging is to preserve the integrity of WRMs, attention should also be given to how and where the packaged WRMs are stored. Exposure over time to large fluctuations in temperature, or to above-ambient temperatures, could adversely affect the container seals and the WRM materials themselves. Exposure to conditions that would damage or destroy labels, or even damage containers, should be avoided.

9. Characterization

9.1 Characterization, as discussed in this section, applies to the analysis or measurement of a prepared WRM to establish a reference value for the WRM. Characterization normally begins after the prepared WRM has been packaged. The required number of WRM units is selected, and the specified number of measurements (analyses) is made using the designated measurement method or methods. If a WRM is to be used for calibration purposes because a CRM is not available, the decision might be made to use two methods if two comparable and applicable methods are available. In some instances, the reference value is based on a make-up value in which the starting material is weighed and processed quantitatively through the preparation procedure with a final weight or volume determination. Even then, the make-up value is often confirmed or verified by measurement. The selection and use of the measurement method is briefly discussed below. An outline to illustrate a chemical characterization of a WRM is given in Appendix X1.

9.2 *Measurement Method:*

9.2.1 *Type*—Often the measurement method selected is the method used for the analysis of the samples for which the WRM is prepared. If another method is used, it should be equal to or better than the sample method in terms of precision and bias.

9.2.2 *Conditions of Measurement*—A decision, which is based on the intended use of the WRM, must be made

regarding how much care will be taken when measuring the WRM. If the WRM is to be used as a control standard, the measurements might be made under the routine conditions used for sample analysis. The alternative is to make the measurements under more rigidly controlled conditions. For example, the method might be qualified first using the criteria given in Guide C1068, and then only highly qualified analysts might be permitted to make the measurements.

9.2.3 *Validation of Method*—Before the measurement method is used for characterization, the method must be validated in the sense that it is calibrated by using a selected calibration standard and by following a prescribed calibration procedure. The ideal standard would be a CRM that has the same matrix as the WRM. A second choice could be a CRM with a different matrix but still certified for the element of interest. If possible, the calibration standard should have a higher standing in the metrological hierarchy of standards than the WRM will have. A calibration procedure should be prepared and integrated with the statistical plan (see 6.4).

9.3 *Sampling*—Sampling is addressed in the statistical plan. After preparation and packaging, a random sample of the required number of WRM units will be selected for characterization. Consideration should be given to minimizing the number of units taken for characterization to maximize the number available for the planned use of the WRM.

9.4 *Measurement Scheme*—The measurement scheme is addressed in the statistical plan. There are various factors that could be considered when devising a measurement scheme. In addition to the possibility of using more than one measurement method, more than one analyst might be used. Instead of two different methods, there might be duplicate setups for one method. The degree of replication of each step in the analysis and the time period for the analysis would be considerations. These and other factors will affect the measurement scheme and the amount of work required. A balance should be decided upon between the cost of characterization and the degree of reliability desired.

10. Statistical Analysis

10.1 A statistical analysis of the characterization data is made to derive the reference value and to determine an appropriate uncertainty for that value. The statistical analysis is based on the statistical plan, and it should be done by a statistician if possible. The meaning of the uncertainty value assigned to the reference value should be defined (see Guide C1215 and ISO Guide to the Expression of Uncertainty in Measurement).

11. Documentation

11.1 Records generated during the preparation and chemical or isotopic characterization of a WRM provide the documentary evidence and support for the technical interpretation, judgments, and decisions regarding the quality of the WRM. Records provide the historical evidence needed for future reviews and evaluations should the credibility of the WRM ever be questioned. They provide linkage (traceability) between the WRM's assigned value(s) and a nationally recognized measurement base as represented by CRMs or other