



Designation: F 1569 – 94 (Reapproved 1999)

Standard Guide for Generation of Consensus Reference Materials for Semiconductor Technology¹

This standard is issued under the fixed designation F 1569; 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 procedures for producing a single set of consensus reference materials (ConRefs) in the absence of suitable certified reference materials from an established source.

1.2 This guide covers the steps to be taken to generate a set of ConRefs for a specific property or family of related properties required in semiconductor technology.

1.3 The procedure for generating the set of ConRefs is based on interlaboratory testing in accordance with Practice E 691. It is assumed for the purposes of this guide that the test method evaluated by the interlaboratory study (ILS) is appropriate for determining the property values of the ConRef. This guide does not cover the selection of one of several possible test methods nor does it cover the case for which other reference materials must be used in the measurement of the properties of the ConRef.

1.4 This guide also describes procedures that may be used to generate consensus property values that may form the basis for the generation of multiple sets of CRMs or reference materials (RMs).

2. Referenced Documents

2.1 ASTM Standards:

E 456 Terminology Relating to Quality and Statistics²

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method²

2.2 ISO Standards:

Guide 30:1981 Terms and Definitions Used in Connection with Reference Materials³

ISO 8402 Quality—Vocabulary³

ISO 10012-1 Quality Assurance Requirements for Measuring Equipment—Part 1: Management of Measuring Equipment³

3. Terminology

3.1 *Definitions* (see Fig. 1 for a schematic presentation of the hierarchy of reference materials):

3.1.1 *certified reference material (CRM)*—a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body (ISO Guide 30:1981).

3.1.2 *consensus reference material (ConRef)*—a reference material one or more of whose property values have been established by a documented ILS that is based on a technically valid test method.

3.1.3 *reference material (RM)*—a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, (for) the assessment of a measurement method, or for assigning values to materials (ISO Guide 30:1981; ISO 10012-1).

3.1.3.1 *Discussion*—A reference material made by a laboratory for its own use is referred to as a working reference material (WRM). This term is gradually replacing the term, secondary reference material, which has been used widely in the past.

3.1.4 *Standard Reference Materials (SRM)*⁴—a certified reference material issued by the U.S. National Institute of Standards and Technology.

3.1.5 *traceability*—the ability to trace the history, application, or location of an item or activity, or similar items or activities, by means of recorded identification (ISO 8402).

3.1.5.1 *Discussion*—ISO 8402 states that in a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, or basic physical constants or properties. In this guide, as in ISO 10012-1, the term “measuring equipment” is extended to include both measuring instruments and measurement standards (including reference wafers).

3.2 Terminology related to the conduct and analysis of the ILS may be found in Terminology E 456 and Practice E 691.

4. Summary of Guide (See Fig. 2 for a flow chart of the steps covered.)

4.1 The test method to be used is selected or developed, and

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¹ This guide is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Dec. 15, 1994. Published February 1995.

² Annual Book of ASTM Standards, Vol 14.02.

³ ISO Central Secretariat, C. P. 56, CH-1211 Genève 20, Switzerland; available in the U.S. from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

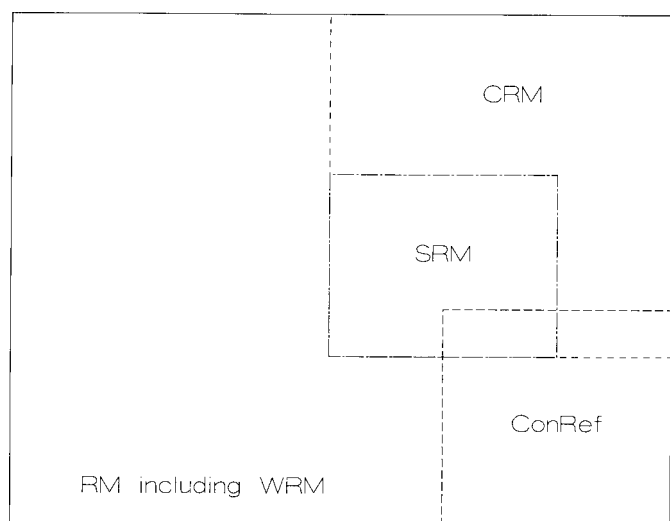


FIG. 1 Reference Material Hierarchy

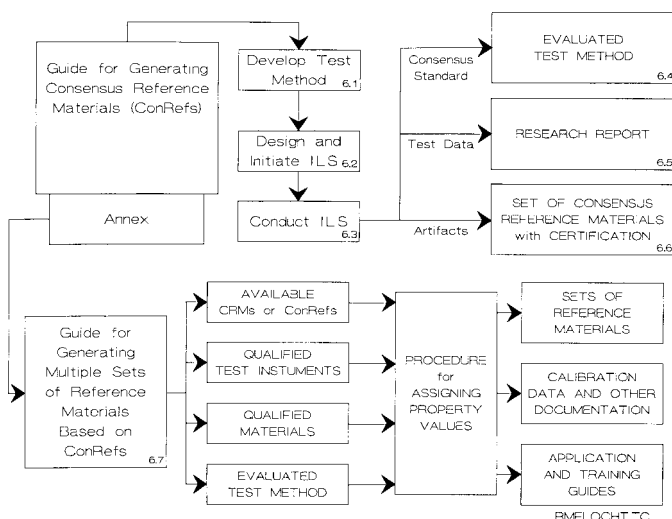


FIG. 2 Flow Chart for Generation of Consensus Reference Materials and Derived Sets of Reference Materials

acceptable levels of repeatability and reproducibility are defined.

4.2 An ILS test protocol for use with the selected consensus test method is developed in accordance with Practice E 691 and the proper materials and instrumentation are assembled.

4.3 The ILS is executed and analyzed.

4.4 The results are evaluated against the criteria selected in 4.1. If the results are acceptable, a precision statement is developed and balloted for inclusion in the test method. If the results are not acceptable, the test method validity and the usefulness of the materials tested are assessed.

4.5 The results are incorporated into an ASTM Research Report.

4.6 If the results are acceptable, the materials used in the test are now qualified as ConRefs. They have values of the desired property (or properties) that represent the consensus of the industry; the uncertainty in these values can be estimated from the repeatability and reproducibility obtained in the test, as documented in the research report. These materials are stored

in a location accessible for future use in preparing replicate sets of reference materials.

5. Significance and Use

5.1 The generated ConRefs can be used by a single laboratory for internal use or for interlaboratory comparison of related equipment and materials.

5.2 Most often, however, they are used for replicating multiple sets of reference materials. In such a case, the property values of these reference materials are traceable to the values of the property value of the ConRefs. Depending on the nature of the preparing organization, these multiple sets may be certified reference materials or working reference materials. For this purpose, a guide for generation of reference materials specific to the materials and property may be required; Annex A1 outlines the requirements for preparing a guide for generation of multiple sets of reference materials.

6. Procedure

6.1 *Select or establish the test method to be used.*

6.1.1 Determine feasibility of proposed test method.

6.1.2 Conduct screening experiments (ruggedness tests) of the proposed test method in a single laboratory.

6.1.3 Define acceptable levels of repeatability and reproducibility for the proposed test method.

6.1.4 Draft test method.

6.1.5 Obtain consensus within a representative task group or subcommittee.

6.2 *Design and initiate the ILS.*

6.2.1 Design the experiment in accordance with the principles of Practice E 691, taking into account whether the test is destructive or nondestructive.

6.2.2 Prepare an interlaboratory test protocol that specializes the test method to this design and also includes a provision for reporting deviations from the method if participants in the interlaboratory test are unable to follow the test method exactly.

6.2.3 Select, qualify, and prepare materials for the interlaboratory test, paying particular attention to issues of uniformity, stability, storage, transportation, and handling.

6.2.4 Qualify instrumentation in accordance with test method and applicable quality control practices.

6.2.5 Select and qualify participating laboratories.

6.3 *Execute the interlaboratory experiment.*

6.3.1 Collect the data in a round-robin or hub-and-spoke mode.

6.3.2 Analyze the data collected in accordance with Practice E 691 to establish the repeatability and reproducibility of the test.

6.3.3 Derive an appropriate "uncertainty" from the results of the analysis.

6.4 *Evaluate test results against the preselected criteria.*

6.4.1 If the result is acceptable, prepare and ballot "Precision" and "Bias" sections for the test method and proceed to 6.5.

6.4.2 If the result is not acceptable, assess the validity of the test method and the usefulness of the generated materials. Consider whether the test method may need to be revised or the ILS repeated with improved controls on the procedures used.