NIST Ceramics Division Quality Manual

for

Standard Reference Materials

QM-II-852

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INTRODUCTION

QM-II-852 (the present document) is the quality manual (QM) of the Ceramics Division (CD) for the production of Standard Reference Materials (SRMs). This document conforms to the requirements of the National Institute of Standards and Technology (NIST) Quality Manual (NIST-QM-I) and expands the policy and objectives of NIST-QM-I to provide a framework within which CD staff members realize these quality objectives. CD is committed to the use of good laboratory practices and quality management. Management and staff recognize that many CD activities within the scope of this quality system (QS) are non-routine. Therefore, CD policy as set forth in this document encompasses a number of means by which objectives are achieved.

1.1 Commitment to Quality

The Ceramics Division serves as the nation's reference laboratory for data and metrology related to advanced ceramic materials and nanotechnology. To that end, the Ceramics Division works with industry, standards bodies, universities, and government laboratories and conducts programs pertinent to measurement issues for ceramic materials and nanotechnology. CD programs contribute to fundamental and applied research, the development of standard test methodologies, the preparation of standard reference materials, and the evaluation and dissemination of standard reference data. CD programs are designed to enhance productivity and competitiveness of U.S. industry, assure equity in trade, and provide quality assurance for data and measurements used for assessing and improving public health, safety, environment, homeland security, and commerce. Measurement methods are developed and critically evaluated to provide U.S. industry and the nation with tools for achieving traceability and international comparability of property measurements. These tools include:

- Standard Reference Materials (SRMs®)
- Standard Reference Databases (SRDs)
- NIST Recommended Practice Guides
- Standard Test Methods via ASTM International (ASTM, formerly American Society for Testing and Materials) and the International Organization for Standardization (ISO)
- Interlaboratory comparisons of measurement methods and standards with other National Metrology Institutes via the Versailles Project on Advanced Materials and Standards (VAMAS) and the International Energy Agency (IEA)
- Critically evaluated data dissemination via the NIST Ceramics WebBook
- Peer-reviewed technical publications

CD is committed to providing its customers and the nation with measurement services that are of documented quality. The quality policy and its related technical requirements that have been adopted by CD are covered in this document. Their purpose is to ensure that all CD staff know the levels of performance needed to assure the quality of its products and services. All CD staff are expected to familiarize themselves with the requirements of the quality policy and apply the guidelines. As stated in NIST-QM-I Section 4.1.1, NIST requires its staff to have high standards

of ethical conduct, be impartial, have objectivity, and protect the confidential or proprietary information of its customers.

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Debra L. Kaiser, Ph.D.
Chief, Ceramics Division

1.2 Scope

CD maintains a quality system for the production of standard reference materials. Other services may be included at the discretion of the Groups. Such services are identified in QM-III-852.xx documents. QM-II-852 conforms to the requirements of NIST-QM-I and describes CD quality policies, procedures, and objectives.

1.3 Outline of CD Quality Manual

The CD Quality Manual (QM-II-852, this document) is a second-level document as described in NIST-QM-I section 1.3. It contains policies and procedures established and maintained by the CD to meet its quality goals. The CD quality system also includes the third-level documents (QM-III-852.xx) that are the quality manuals for Standard Reference Materials developed by the CD. The QM-III-852.xx documents contain quality-specific policies and procedures established and maintained for each SRM of the CD.

2. References

2.1 Normative Reference

NIST Quality Manual (NIST-QM-I)

2.2 Informative References

ILAC-G12:2000 – Guidelines for the Requirements for the Competence of Reference Material Producers - www-i.nist.gov/ts/tsintranet/quality/ilac-g12.pdf

International Vocabulary of Basic and General Terms in Metrology, 2nd ed., BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML, International Organization for Standardization (ISO), 1993 (VIM) (stored in the NCSCI Standards library, NIST North, Room 164)

ISO/IEC 17025 – "General requirements for the competence of testing and calibration laboratories" – www-i.nist.gov/ts/tsintranet/quality/iso17025.pdf

ISO Guide 31:2000 – "Reference materials -- Contents of certificates and labels" (stored in the NCSCI Standards library, NIST North, Room 164)

ISO Guide 32:1997 – "Calibration in analytical chemistry and use of certified reference materials" (stored in the NCSCI Standards library, NIST North, Room 164)

ISO Guide 33:2000 – "Uses of certified reference materials" (stored in the NCSCI Standards library, NIST North, Room 164)

ISO Guide 34:2000 – "General requirements for the competence of reference material producers" –www-i.nist.gov/ts/tsintranet/quality/iso_34_2000.pdf

ISO Guide 35:1989 – "Certification of reference materials -- General and statistical principles" (stored in the NCSCI Standards library, NIST North, Room 164)

Mutual Recognition Arrangement- Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes, Comité international des poids et mesures (CIPM) Paris, 14 October 1999, www.bipm.org/utils/en/pdf/mra.pdf

NIST Administrative Manual – complete copy stored in the CD Office;
Sections available at www-i.nist.gov/admin/mo/adman/contents.htm

NIST R&D Relationships Guide – www-i.nist.gov/div222/InventorHandbook/

NIST Special Publication 260-126 – "The NIST Traceable Reference Material Program for Gas Standards" – http://patapsco.nist.gov/srmcatalog/sp_publications/documents/sp260-126.pdf

NIST Special Publication 260-136 – "Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements" – http://patapsco.nist.gov/srmcatalog/sp_publications/documents/sp260-136.pdf

NIST Special Publication 260-140 – "Technical Specifications for Certification of Spectrophotometric NTRMs" - http://patapsco.nist.gov/srmcatalog/sp_publications/documents/sp260-140.pdf

NIST Special Publication 811 – "Guide for the Use of the International System of Units (SI)" – http://physics.nist.gov/Pubs/SP811/contents.html

NIST Technical Note 1297 – "Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results" – http://physics.nist.gov/Document/tn1297.pdf

Position Classification Manual of the NIST Alternative Personnel Management System – www-i.nist.gov/admin/pers/hrdemo.htm

3. Definitions

calibration: The set of operations that establish, under specific conditions, the relationship between values for quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (ISO VIM: 1993, 6.11)

CD calibration service: A service performed by CD on instruments and devices that are metrologically suitable as reference or transfer standards. Services directly link a customer's instrument or transfer standards to national and international measurement standards using well-characterized, stable and predictable measurement processes.

CD Official Report: A document produced by CD to record and report the measurement procedures used in providing standard reference materials or other analytical services. It typically includes a summary of instrumentation and procedures, analytical results, statistical analysis of data, and references. CD Official Reports include Reports of Analysis, Reports of Certification, and Reports of Investigation. See section 5.11.1 for descriptions**.**

certificate: See NIST-QM-I for definition. Numerous certificates are used by CD. See section 5.11.2 for descriptions**.**

Certified Reference Material (CRM): Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. (ISO VIM: 1993, 6.14)

measurement: The set of operations having the object of determining a value of a quantity. (ISO VIM: 1993, 2.1)

NIST Certified Value: An assigned value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. (NIST SP 260-136: 2000)

NIST Information Value: A value that will be of interest and use to the SRM/RM user, but insufficient information is available to assess the uncertainty associated with the value. Typically, the information value has no reported uncertainty listed on the certificate. (NIST SP 260-136: 2000)

NIST Reference Value: A best estimate of the true value provided by NIST where all known or suspected sources of bias have not been fully investigated by NIST. (NIST SP 260-136: 2000)

NIST Traceable Reference Material^{CM} (NTRM^{CM}): A commercially-produced reference material with a well-defined traceability linkage to existing NIST standards for measurements. This traceability linkage is established via criteria and protocols defined by NIST to meet the needs of the metrological community to be served. Commercial NTRM producers are allowed to affix the NTRM certification mark to materials produced according to these criteria and protocols. (NIST SP 260-126: 1996)

quality system (QS): The organizational structure, responsibilities, procedures, and processes for implementing quality throughout the laboratory.

Reference Material (RM): Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO VIM: 1993, 6.13, 7)

reference standard: A standard, generally having the highest metrological quality available at a given location or in a given organization, from which measurements made there are derived. (ISO VIM: 1993, 6.6)

repeatability: Closeness of the agreement between the results of successive measurements of the same measurand and carried out under the same conditions of measurement. (ISO VIM: 1993, 3.6)

reproducibility: Closeness of the agreement between the results of measurements of the same measurand carried out under changed conditions of measurement. (ISO VIM: 1993, 3.7)

SRM Statement of Work: a document created to describe the services and materials to be provided or procured by the CD (in collaboration with Measurement Services Division (MSD) and Statistical Engineering Division (SED), as appropriate) for development, production, certification, and delivery of SRMs and RMs. Guidelines for SRM Statements of Work are included in Appendix A of NIST Administrative Manual Subchapter 5.19.

Standard Reference Material® (SRM®): A CRM issued by NIST that also meets additional NIST certification criteria. (NIST SP 260-136: 2000)

statement of work: a document created to describe in detail: 1) a service procured from an external contractor, collaborator, or vendor, or 2) a CD measurement service procured by or provided to a customer. The NIST Acquisitions and Logistics Division provides guidelines for writing statements of work for services procured externally (see http://www-i.nist.gov/admin/aad_new/acquisition/aad-sow2.shtml).

technical procedure (TP): A compilation of evaluated steps leading to the completion of a specific task.

Technical Project Leader (TPL): CD member responsible for overseeing the technical aspects of a project.

traceability: The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, all having stated uncertainties. (ISO VIM: 1993, 6.10)

uncertainty of a certified value: An estimate attached to a certified value of a quantity which characterizes the range of values within which the "true value" is asserted to lie within a stated level of confidence. (ISO Guide 30: 1992 3.4 (8)

uncertainty of a measurement: Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand. (ISO VIM: 1993 3.9)

4. Management Requirements

4.1 The Ceramics Division

4.1.1 Description

The CD mission is to:

- Work with industry, standards bodies, universities, and government laboratories in providing the leadership for the Nation's measurements and standards infrastructure for ceramic materials and nanotechnology
- Conduct programs pertinent to measurement issues for ceramic materials and nanotechnology through fundamental and applied research
- Develop standard test methods
- Prepare standard reference materials
- Evaluate and disseminate standard reference databases

CD technical experts provide:

- Research in materials metrology
- Tools for achieving national traceability and international comparability of materials characterization and property measurements
- Evaluated data, databases, and evaluation methodologies.

Activities related to materials metrology within the CD are carried out in the following areas:

- Electronic and Optoelectronic Materials
- Characterization Methods

- Nanotribology
- Data and Standards Technology
- Nanomechanical Properties

4.1.2 Physical Locations

CD measurement services are conducted in NIST facilities in the Materials Science and Engineering Laboratory (Building 223), the Advanced Measurement Laboratory (Buildings 217 and 219), and the Center for Neutron Research (Building 235) in Gaithersburg, Maryland, and in the National Synchrotron Light Source at Brookhaven National Laboratory, Upton, New York.

4.1.3 Organizational Structure of the CD

CD is part of the Materials Science and Engineering Laboratory (MSEL) of the National Institute of Standards and Technology (NIST) which is an agency within the Technology Administration of the Department of Commerce.

4.1.3.1 Organizational Chart

The current NIST organizational charts are found at the following website: http://orgchart.nist.gov. The management chain for CD is given in the chart for the Materials Science and Engineering Laboratory http://www.msel.nist.gov/mselorg.html.

4.1.3.2 Responsibilities, Authorities, and Delegations

For every CD employee, detailed descriptions of employee responsibilities are documented in official Position Descriptions, and in individual yearly Performance Plans.

As delineated in NIST-QM-I section 4.1.3.2, the CD Chief (CDC) and Group Leaders (GLs) are responsible for the technical and scientific work involved in the development, maintenance, and provision of the CD technical research program. The CD Chief or designee has resource allocation authority and the responsibility for maintaining the infrastructure necessary to meet these needs.

Group Leaders have the responsibility for the research conducted within their groups, with the concordance and oversight of the CD Chief. Group Leaders or designees generate SRM Statements of Work. Group Leaders or designees are responsible for providing the required documentation for other technical outputs covered by the scope of this QS. The CD Chief, in consultation with the Group Leaders, designates Technical Project Leaders (TPLs). Group Leaders assign research tasks to qualified personnel, and ensure that personnel have appropriate training. Group Leaders or designees oversee research tasks and review resulting data and reports.

The Technical Project Leader is responsible (in collaboration with other staff as appropriate) for designing the project, preparing the statement of work, monitoring project progress, interacting with support divisions, and completing certificates, associated documentation, publications, and the annual technical activities report for the project. Technical Project Leaders and others requiring CD measurement services relay their measurement needs to the Group Leaders, who then assign tasks as described.

The individual employee and Group Leader share the responsibility for ensuring that the training and guidance received by the employee are sufficient to perform specific required tasks. Individual employees are required to document their capabilities (as described in Section 5.2.1). These records are updated annually and maintained by the NIST Human Resources Management Division.

General responsibilities for SRM development and production are described in the NIST Administrative Manual Subchapter 5.19. Any specific responsibilities, authorities, or delegations unique to the provision of a particular measurement service are documented in the QM-III-852.xx series, or included on a case-by-case basis in statements of work.

4.2 CD Quality System

4.2.1 CD Quality Policy

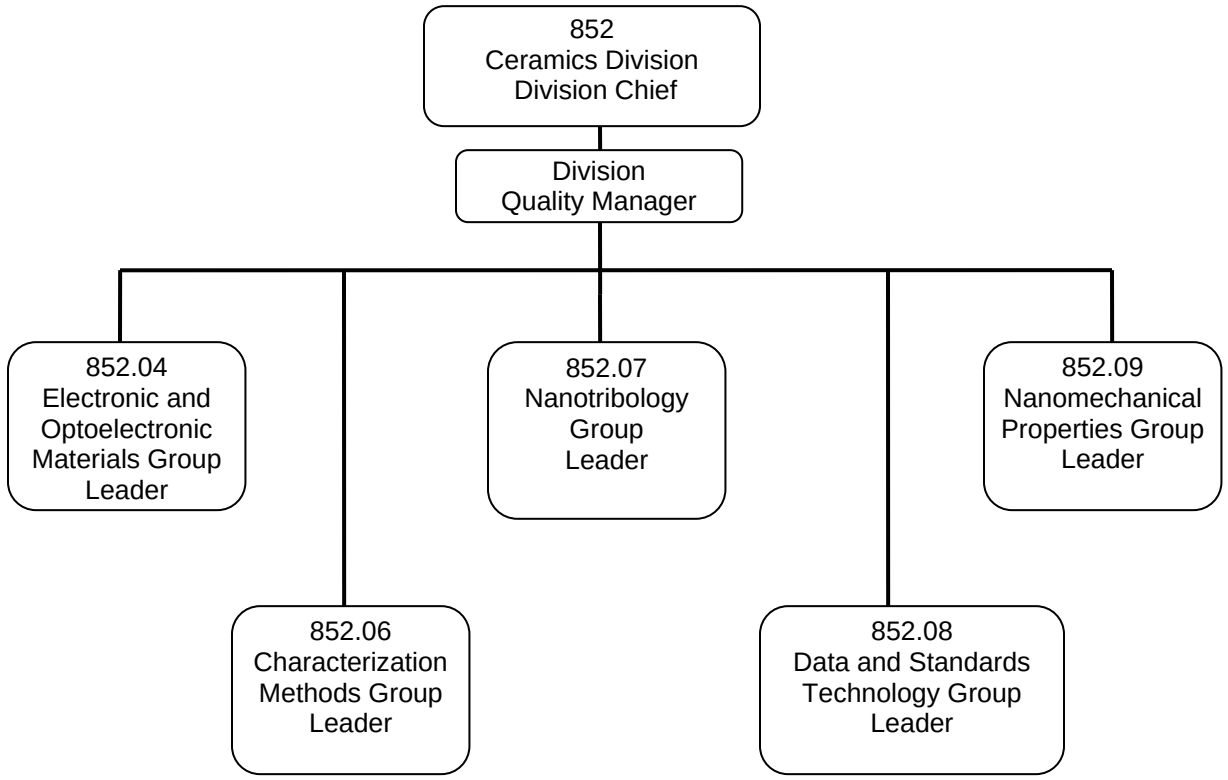
CD is committed to following the NIST Quality System outlined in QM-I, section 4.2.1.

4.2.2 CD Quality Objectives

The CD quality objectives are those put forth in QM-I, Section 4.2.2.

4.2.3 Organizational Structure of the CD QS

4.2.3.1 Organizational Chart of Quality Managers



The organization of quality officers in the CD.

4.2.3.2 Responsibilities, Authorities, and Delegations

The CD Chief is responsible for implementing, at the division level, the NIST Quality System described in QM-I. The CD Chief is responsible for the quality of all measurements and certifications conducted by CD staff, for assuring completion of assessments and reviews in a timely manner, and for implementing actions resulting from the findings of these assessments and reviews. The CD Chief appoints a Division Quality Manager (DQM). Each Group Leader is responsible for implementing the CD Quality System at the group level. The Group Leader or designee acts as a Group Quality Manager (GQM) in the implementation of QM-III-852.xx documents at the Group level. Additional responsibilities of Quality Managers may be included in the QM-III-852.xx documents.

The Division Quality Manager is responsible for:

- Obtaining CD management approval of QM-II-852
- Issue and control of QM-II-852
- Organizing, scheduling, and maintaining records of Division quality system assessments and reviews

- Assuring timely completion of any required revisions of QM-II-852 and obtaining approval thereof
- Assuring that QM-II-852 is kept consistent with successive editions of NIST-QM-I and applicable international standards
- Notifying CD staff of the issue of new versions of NIST-QM-I and QM-II-852
- Controlling records for nonconformity and corrective actions (see section 4.5)
- Maintaining and reviewing the CD F&C log
- Reporting F&Cs, corrective actions, and suggestions for preventive actions to CD management.

4.3 Control of Documents, Records, and Data

4.3.1 Quality Documents

Quality documents (including QM-II-852 reports from internal assessments, reviews by management, and records of non-conformities and corrective and preventive actions) are managed following the specifications in NIST-QM-I and the NIST Administrative Manual Subchapter 2.06 on Records Management. CD staff comply with Subchapter 11.02 (www-i.nist.gov/admin/mo/adman/1102.HTM) governing computer and information security.

4.3.2 Quality Document Approval and Issue

The Division Quality Manager is responsible for obtaining managerial approval of QM-II-852 prior to implementation of revised versions. The official version of QM-II-852 is maintained by the Division Quality Manager on the CD web site. This official version of QM-II-852 is a read/print-only document. This copy is updated (replaced) with each new and approved version of QM-II-852, and previous versions and dates of applicability are retained. Printed copies are clearly marked as uncontrolled documents.

4.3.3 Quality Document Changes

Changes to QM-II-852 may result from assessments, staff recommendations, and changes in QM-I, or other reasons. Recommended changes are submitted to the Division Quality Manager. The Division Quality Manager begins and oversees the review process. Proposed revisions to the document are reviewed by Group Leaders and the Division Quality Manager to determine whether the suggested changes are appropriate. The Division Quality Manager prepares a document containing recommended revisions and makes it available to CD staff for comments. Staff comments are addressed by the Group Leaders or designees, and the Division Quality Manager. If changes are recommended, the Division Quality Manager submits the resulting new document for approval by the CD Chief.

The Division Quality Manager notifies CD personnel when a revised version of QM-II-852 is official and available on the CD web site.

4.3.4 Technical Documents, Records, and Data

Due to the diverse nature of CD activities, there may be multiple procedures, contained in QM-III-852.xx documents, to control (identify, collect, index, access, file, store, maintain, and discard) documents, records, and data. It is the responsibility of individual staff members to maintain appropriate technical records in support of research covered by the scope of this Quality System. Records sufficient to reproduce assigned values from analysts' determinations are maintained and are readily retrievable. Technical records include:

- Statements of work;
- Notes, notebooks, and/or documents that describe material handling, sample selection, sample preparation, and the analysis;
- Data used to produce final measurement results.

Observations and data are recorded at the time they are made and are identifiable to the specific task. When mistakes occur in written records, particularly in laboratory notebooks, each mistake is crossed out and the correct information entered alongside. Alterations to records are initialed by the person making the correction. Records are legible and stored to prevent damage and minimize deterioration. Electronic files are backed-up as described in QM-III-852.xx.

Records are maintained as described in NIST Administrative Manual Subchapter 2.06 (http://www-i.nist.gov/admin/mo/adman/206.htm) or for a minimum of ten years if not otherwise specified. CD staff comply with Subchapter 11.02 (www-i.nist.gov/admin/mo/adman/1102.HTM) governing computer and information security.

4.4 Administrative Requirements

4.4.1 Review and Approval of Requests for Measurement Services

4.4.1.1 Reference Material Activities

On an annual basis, CD develops a list of potential reference material (WCF and SD) projects in response to customer needs. Each proposed project is described in the form of an SRM Statement of Work. CD management reviews and prioritizes the SRM Statements of Work. CD follows the procedures regarding reference materials activities set forth in the NIST Administrative Manual Subchapter 5.19 for all funded SRM projects.

4.4.1.2 Other CD Measurement Services

Agreements for other CD measurement services covered by the scope of this QS must be approved by the CD Chief or a staff member to whom authority for a specific measurement

service has been delegated. The steps involved in fulfilling these agreements are addressed in the relevant QM-III-852.xx documents or included on a case-by-case basis in statements of work.

4.4.2 Procuring Products and Services from External Sources

CD follows the policies set down in NIST-QM-I section 4.4.2 for the procurement of products and services from sources external to NIST. Supplies, reagents, and consumables are tested at point of use as necessary to confirm that they are acceptable. Services are evaluated on a case-by-case basis, as necessary. Records of these tests or evaluations are maintained at the group level.

4.4.3 Interaction with NIST Supporting Divisions

NIST-QM-I Section 4.3.3 and the NIST Administrative Manual govern interactions with supporting services. It is the responsibility of the CD management team to communicate concisely and clearly the actions desired/required of NIST supporting services to allow CD quality goals to be achieved.

4.4.4 Subcontracting of Measurement Services

CD follows NIST policy on subcontracting of measurement services as delineated in QM-I, Section 4.4.4

4.4.5 Reference Materials Production Planning and Control

4.4.5.1 General guidelines, procedures, and nomenclature for planning and control of RM production are included in NIST-QM-I Section 4.4.5, NIST Administrative Manual Subchapter 5.19, and NIST Special Publication 260-136. Prioritization of reference material production is the responsibility of CD management. Priorities are set for work in progress (both renewal and new SRMs), renewals, and new SRMs. Priorities are set considering customer needs for SRMs and the availability of resources such as technical capabilities, staffing, and funds.

4.4.5.2 Project planning and control for other measurement services is described in QM-III-852.xx level documents. Priorities are set considering customer needs and the availability of resources.

4.4.5.3 The TPL is responsible for including elements of production control in the design of the project and for monitoring that aspect of production.

4.4.5.3.1 For each material to be produced, the TPL revises the "Experimental Design/Certification Plan" section of the SRM Statement of Work, as needed, to address the final plan for production and value assignment steps, as applicable:

- Assessment of material homogeneity and stability;
- Experimental design and selection of the measurement methods;
- Appropriate storage and handling of materials
- Material preparation for testing and analysis;
- Selection and use of appropriate control materials.

SRM Statements of Work are maintained by the TPL and the Group Leader. When the SRM Statement of Work must be modified, the revised version replaces the original. Deviations from the SRM Statement of Work that occur during production are described in the Report of Analysis (see 5.11.1).

4.4.5.3.2 The TPL makes sure all staff participants in the measurement process have access to the current SRM Statement of Work. Samples are provided to the analysts according to the design developed in collaboration with SED. Analysts should ensure that they have all of the technical design details before starting the analysis.

When collaborating laboratories are participating in the value assignment process, the TPL provides any required protocol to the collaborators including instructions for submitting required documentation to CD.

4.4.5.3.3 As analyses are completed, Reports of Analysis are sent to the CD Office with copies to the TPL and CD Group leaders as appropriate. The TPL monitors the data as they are reported to detect any apparent discrepancies or deficiencies. Apparent problems are discussed with the analysts. Appropriate actions are documented, where necessary.

The TPL is responsible for assembling the data into a package for the statistician. The format of the data, certification goals, and intended approach should be discussed with the statistician. When the statistical analysis is completed, the TPL works with the statistician and other analysts to complete the value assignment process.

Deviations from this general procedure are permissible and are addressed in QM-III-852.xx.

4.4.5.3.4 The TPL coordinates the drafting and review of the certificate. The draft is sent to MSD along with the appropriate documentation, which may include CD Official Reports, results from SED, results from collaborating laboratories, and a Project Completion Memorandum (Appendix B). MSD formats the draft and returns it to the TPL, who coordinates final review and approval by CD and SED.

4.5 Corrective and Preventive Actions

4.5.1 Non-Conformity and Corrective Actions

If there is evidence derived from any source, including customer F&Cs, that any activity within CD supporting the provision of a measurement service is not or has not been in conformity with the requirements of NIST-QM-I or QM-II-852, the CD management team:

- institutes a timely investigation of root causes
- assesses the significance of the non-conformity to all completed and in-progress services, and, if warranted, notifies customers and/or ceases work, with resumption upon authorization by the Group Leader
- develops and executes corrective and/or preventive actions, if warranted, particularly in cases where the possibility of recurrence exists.
- monitors implementation and determines outcomes of such actions
- initiates an internal assessment as soon as possible, if required, per section 4.6.1.3
- maintains records of the non-conformities, action plans, implementation and outcomes thereof
- notifies customers of outcomes, as appropriate.

4.5.2 Customer Feedback and Concerns

CD follows the NIST policy, described in NIST-QM-I Section 4.5.2, for receiving, recording, and responding to customer feedback and concerns (F&Cs). F&Cs must be received in writing. Staff members report any F&C to their direct supervisor. Individual staff members (or their direct supervisor, as appropriate) report the F&C to the Division Quality Manager, who records the F&C and any actions taken in the CD F&C log. The division F&C log is maintained and monitored by the Division Quality Manager. The following are maintained in the CD F&C log:

- the nature of the F&C, date received, the name of person registering the F&C, CD recipient, and the initial response to the complainant;
- the final resolution of the F&C to include applicable elements of Section 4.5.1,
- a brief summary of all follow-up and the (required) final communication with the complainant.

Collecting and recording this information can be facilitated by use, as appropriate, of the NIST F&C Response Form found in QM-I, Appendix B.

New F&Cs are discussed at the next scheduled CD management meeting for the purpose of identifying preventive actions. See Sections 4.2.3.2 and 4.5.3.

4.5.3 Preventive Actions

As noted in ISO Guide 34 section 4.10.1 and ISO 17025 section 4.11 – Preventive action is a proactive process to identify improvement opportunities rather than a reaction to the identification of problems or concerns.

It is the responsibility of all CD staff to identify potential sources of non-conformity, both technical and related to the QS, and opportunities for improvement within their areas of responsibility. Suggestions are given to the Group Leader or Division Quality Manager who determine whether the suggestions should be addressed at the group or division level. If preventive action is necessary, action plans are developed, implemented, and monitored. Suggestions for preventive actions in technical activities are documented, reviewed by the Group Leader, implemented, and summarized for the Division Quality Manager, where appropriate. Suggestions for modifications to quality system documents are handled according to QM-II-852 Section 4.3.3.

4.6 Assessments and Management Reviews

4.6.1 Assessments

4.6.1.1 NIST-Level Assessments

CD participates in the NIST-level assessments described in NIST-QM-I Section 4.6.1.

4.6.1.2 CD-Level Assessments

CD-level assessments are coordinated by the Division Quality Manager and conducted by a team that includes one member from the work being assessed, one member from another technical area within CD, and the Division Quality Manager. The assessment includes a review of the relevant quality documents and any associated records. The findings of the assessment team are provided in writing to the appropriate Group Leader(s). Written responses to these findings are compiled by the Group Leader(s) and submitted to the Division Quality Manager for review, with assistance from the assessment team if necessary. Records pertaining to the assessment are maintained by the Division Quality Manager.

CD-level assessments are initiated

- within one year of the start date of a new CD research activity that is added to the scope of QM-II-852
- as necessary, at a frequency to be determined by the Division Management Team, based on results of management reviews, or in response to a report of non-conformity and resulting corrective actions

4.6.2 Reviews by CD Management

Reviews are the responsibility of the CD Management Team and shall be conducted annually.

Typically, management reviews will address the following during the course of regularly scheduled meetings:

- the suitability of policies and procedures
- the results of internal assessments

- corrective and preventive actions
- client feedback
- concerns
- other relevant factors, such as quality control activities, resources, and staff training

4.6.2.1 Quality Documents

QM-II-852 is reviewed annually by the Division Quality Manager for consistency with NIST-QM-I and relevant international standards. Reviews of Group quality documents are described in the QM-III-852.xx series.

4.6.2.2 Technical Competence

Group Leaders review the technical quality of the work produced by their staff on an on-going basis by reviewing results of analyses, technical publications, and all reports generated. Individual staff members maintain records documenting competence as described in Section 5.2, which are also reviewed by Group Leaders. CD members participate in both national and international quality assurance programs and laboratory comparison exercises, as appropriate. The Division Management Team or individual Group Leaders review the results of these exercises.

4.7 Service to the Client

CD follows the policies and procedures established by NIST in NIST-QM-I Section 4.7.

For the purposes of QM-II-852, the client (customer) may be internal or external, a single entity or a user community. For reference materials, the client is viewed as the potential end-user of the RM. Their individual identities are typically unknown until the RM is purchased. MSD maintains a database of purchasers of RMs. Exceptions to this policy are documented in QM-III-852.xx.

5. Technical Requirements

5.1 Introduction

The technical requirements in QM-II-852 are only those that apply across the CD. All other technical requirements are found in, or referenced in, the QM-III-852.xx series.

5.2 Personnel

CD follows the policies and procedures established by NIST in QM-I, Section 5.2.

5.2.1 Competence

Individual employees document their qualifications and capabilities in records known collectively as the Employee Qualifications and Capabilities (EQC) documents. The EQC records are maintained by the NIST Human Resources Management Division (for records such as the SF-171 (or equivalent) and all official personnel actions) and the CD (for records such as annual accomplishments and annual performance appraisals). The EQC records contain the following information:

- Education
- Training
- Relevant work experience
- Professional organization memberships
- Committee memberships and activities
- SRM and SRD Projects (detailing level of involvement)
- Professional Honors and Awards
- Publications
- Talks

These records document the employee's capabilities to contribute to CD research projects.

5.2.2 Education and Training Goals

Training needs are identified by the Group Leader and employee in the performance plan, which is reviewed and modified as needed to meet the competence requirements of projects to which the employee is assigned.

5.2.3 Job Descriptions

CD follows the policies and procedures established by NIST in NIST-QM-I Section 5.2.3.

5.2.4 Collaborators

CD follows the policies and procedures established by NIST in NIST-QM-I Section 5.2.4.

5.3 Accommodations and Environmental Conditions

5.3.1 CD determines the general environmental conditions, and, working in collaboration with the Facilities Division, Plant Division, and Engineering, Maintenance, Safety, & Support Division, is responsible for assuring that environmental conditions do not adversely affect the quality of measurement services. Critical environmental conditions (i.e., those that adversely affect measurement results) and the procedures for preventing them from compromising the quality of results are detailed in the QM-III-852.xx series and associated records. Work does not

proceed until environmental conditions are met and may continue only as along as conditions continue to be satisfactory.

5.3.2 As described in the NIST Administrative Manual Subchapter 5.19, CD works together with MSD to determine appropriate environmental conditions for material preparation, packaging, and storage. The personnel responsible for the area in which activities are carried out record environmental conditions, when relevant.

5.3.3 Materials and activities are segregated to the extent needed to prevent cross-contamination. Measuring equipment is installed and operated in ways that prevent interferences between electronics. This includes segregation, as needed, from activities that are outside the scope of this manual. Chemicals and other laboratory consumables are stored under the conditions necessary to maintain their stability or integrity and to prevent cross-contamination with other materials.

5.3.4 Access to laboratories and storage areas is usually limited to members of the group to which they are assigned. Others may not work in an area or use the equipment without the knowledge of the person responsible for the room or equipment.

5.3.5 Measures shall be taken to ensure good housekeeping in the laboratory. Where necessary, special procedures shall be prepared and documented in the QM-III-852.xx series.

5.3.6 CD addresses the environmental conditions of collaborators through experimental design and descriptions of necessary conditions in a statement of work.

5.4 Measurement Procedures and Procedure Validation

In the majority of cases, CD uses methods developed and validated in CD laboratories for the provision of measurement services. If a standard method of test is used, it is treated by CD as a laboratory-developed method and validated according to Section 5.5.2.

5.4.1 Measurement Procedures

CD measurements and procedures that lead to NIST Standard Reference Materials must be documented. These documented procedures (DPs) may take various forms, including (but not limited to) internal reports (NIST IRs), NIST Special Publications (NIST SPs), and peer reviewed articles. Wherever possible, the technical descriptions must be complete, current, easily accessible, and either peer-reviewed or internally reviewed publications. CD Group Leaders determine the activities within their groups for which DPs are required.

Each Technical Project Leader for the development of an SRM maintains a copy of each DP appropriate to the project. DPs are referenced appropriately in all SRM certificates issued by the project.

Examples of items that may be the topics of or sections of DPs are listed below.

- Title and Approval Page
- Introduction
- Instrumentation and instrument set-up
- Required environmental conditions
- Calibrants used, calibration logs, and calibration intervals (include a reference to the calibration DP if not described in this DP)
- Sample preparation
- Sample delivery to the instrument
- Analysis of the sample
- Sequencing of samples, calibrants, blanks, and controls
- Method validation
- Data collection, reduction, and storage
- Assignment of final value and uncertainty assessment (including Statistical Engineering Division involvement)
- Homogeneity assessment
- Sample storage, treatment, and disposal before and after analysis
- Maintenance of equipment
- Traceability
- Physical and electronic security
- Special safety precautions
- References

5.4.2 Method Validation

5.4.2.1 CD critically evaluates all methods used for official measurements. Critical evaluation includes all relevant components of method validation described in ISO 17025, a complete analysis of all significant sources of uncertainty, and additional procedures and processes specific to a given method deemed necessary by a CD expert. Method evaluation practices include (but are not limited to) the following tasks:

- Document the specificity and selectivity of the measurement process
- Estimate the limit of detection (LOD) or the limit of quantification (LOQ), or both, as needed to ensure that the measurement process has adequate sensitivity
- Assess the working range and/or linear range – Show that the measurements are within the scope of the method and that the calibration model is appropriate
- Assess the recovery of the method as an indicator of possible bias
- Demonstrate the repeatability and reproducibility of the method
- Test for sources of bias.

A measurement control, such as a previously certified RM, is used throughout the procedure to monitor the measurement process.

5.4.2.2 It is not necessary to demonstrate and document every aspect of method validation at every use of the method. The expert may use documented evidence from prior applications of the method to demonstrate validity. For example, the working range of the method may have been demonstrated in a published paper or the Report of Analysis for a previous SRM.

5.4.2.3 Methods that have not been published but have been used successfully for past projects and adequately documented in a CD Official Report are considered in-house methods, with internal review according to CD policy and procedures. Newly developed methods and methods never before used by CD staff must be validated with all relevant data reported in a CD Official Report.

5.4.2.4 Method validation must include documentation of the handling and preparation steps that were applied for the purpose of obtaining test portions or specimens from the units of candidate material specifically chosen for analysis by the method. Again, prior experience may be used, unless the method has not been previously applied to the material. The influence of handling and preparation must be considered where such procedures may affect the measurement result. Selection of units from the lot of material for distribution to analysts is not covered in this section.

5.4.3 Estimation of Uncertainty

All certified and reference values reported in certificates (see section 5.10.2) for reference materials and values reported as other official measurements (see section 1.2) are accompanied by quantitative statements of uncertainty. To ensure that the uncertainty statements are consistent across the organization and with international practice, NIST policy requires that official measurements be accompanied by statements of uncertainty as discussed in QM-I, Section 5.4.3 and NIST-QM-I Appendix C. Measurement results without uncertainties shall be explicitly labeled in the certificate as information values. An assessment of uncertainties affecting each measurement method is conducted, and uncertainties from all the methods employed are then combined under the guidance of SED using approved methods. Details of the measurement uncertainty assessment are contained in the QM-III-852.xx level documents.

5.4.4 Evaluation and Control of Data

Procedures for checking calculations, verifying data transfer, storing data, and evaluating associated data processing software are the responsibility of the Groups and are included in the QM-III-852.xx documents. These procedures address the following, as appropriate:

- Evaluation of computer software used to capture, reduce, and process data
- Evaluation of software or spreadsheets developed in-house
- Protection of stored data.

5.5 Equipment

It is CD policy that laboratories are supplied with equipment and instrumentation necessary to fulfill the requirements of the measurement services it provides.

CD instrumentation must be in good working order and demonstrated to perform properly. Evidence of this testing shall be kept in the appropriate notebook.

Group processes for identification, check out, calibration, status, demonstration of performance, use, maintenance, identification, tracking of instrumentation, with incumbent record keeping are included in the QM-III-852.xx documents. Test equipment (e.g. balances, thermometers, transducers, etc.) or artifacts that have an influence on the certified value must have a documented calibration schedule, and be appropriately maintained.

Equipment or instrumentation on loan from an outside source or returned to CD from loan is tested to ensure that it functions properly. Evidence of this testing will be kept in the appropriate notebook.

When there is evidence that an instrument is not performing properly it is labeled and taken out of service until any defects can be assessed and repaired. The effects of the malfunction on previous tests are investigated by the appropriate staff and the results reported to the Group Leader, if necessary (see Section 4.5.1).

5.6 Traceability

CD follows the NIST policy on measurement traceability as delineated in QM-I, Section 5.6 and NIST Administrative Manual Subchapter 5.16 (http://www-i.nist.gov/admin/mo/adman/516.HTM). As the nation's reference laboratory for ceramic property measurements, CD establishes traceability of its work to amount of substance or stated references, as appropriate, through an unbroken chain of comparisons all having stated uncertainties. Procedures for accomplishing this goal may be unique to each CD measurement service and are documented in QM-III-852.xx. Specific evidence of traceability is documented or referenced in CD Official Reports and records.

5.7 Sampling, Preparation, Homogeneity, and Stability for Reference Materials

Plans for material acquisition, processing, and packaging are provided in SRM Statements of Work or, for NTRMs, in NIST SP260-126 or NIST SP260-140. Sampling plans for homogeneity testing, results, value assignments, and stability testing are provided in SRM Statements of Work and/or CD Official Reports and associated records.

5.7.1 TPLs, with the assistance of MSD and other CD staff as appropriate, obtain or produce a batch of material for use as an SRM. Information pertaining to material acquisition, processing, and packaging is provided in project records.

5.7.2 The material is tested to determine if the degree of homogeneity is sufficient for the purposes of that SRM. When representative samples of a batch of reference material are required, a sampling plan is developed in cooperation with SED. Preparation of test portions for measurements is specific to each reference material and the test method used. The processes for assessing homogeneity and stability are documented in the QM-III-852.xx series.

5.8 Handling of Test and Calibration Items

It is CD policy that SRMs, candidate SRMs, and customer samples must be carefully identified, separated, stored, and shipped under appropriate environmental conditions that assure the integrity of the material with respect to its certified value and uncertainty. CD works in concert with MSD as appropriate to develop and implement procedures for preparation and storage of reference materials. Specific procedures for identifying, preparing, packaging, handling, storing, and shipping of these materials are documented in QM-III-852.xx series, SRM Statements of Work, notebooks, or CD Official Reports, as appropriate.

5.9 Quality Assurance Practices

The CD staff uses quality assurance practices to ensure the validity of measurement results and their uncertainties. Such practices can include:

- Results of national and international comparisons, including round-robin studies
- Repeat measurements/calibrations compared over time
- Comparison of results obtained using multiple reference standards
- Use of check standards and control charts
- Use of redundant experimental designs
- Comparison of results obtained using two or more differing measurement approaches
- Correlation of results for different characteristics of an item.

The QM-III-852.xx series details the quality assurance practices for the measurement services provided by each Group.

5.10 Reporting Results

Construction and transmission of CD reports conform to NIST Administrative Manual, Subchapter 5.19, Standard Reference Materials, subsection 5.19.06.j, Documentation review.

5.10.1 CD Official Reports

CD Official Reports are the written records of analytical or measurement services which CD provides to its customers. CD Official Reports may take the form of Reports of Analysis, Reports of Investigation, or Reports of Certification. The locations of all technical records associated with that measurement service are included as references.

All Reports of Analysis or Reports of Certification follow the NIST Administrative Manual, Subchapter 5.19 Standard Reference Materials, for formatting, routing, and transmittal of reports. Only a complete, signed version is official. In the case of transmission of portions of a report or results by telephone, facsimile, or other electronic means, the transmission is clearly designated as unofficial.

All CD Official Reports follow NIST Special Publication 811 – "Guide for the Use of the International System of Units (SI)" (www.physics.nist.gov/Pubs/SP811/contents.html) when reporting results.

CD Official Reports and the references listed in them include complete descriptions of the analytical processes, results, and conclusions. CD Official Reports contain the following sections (additional sections may be included by the authors as appropriate):

- Title
- Client: If appropriate, include contact information
- Background: Information about the background and purpose of the analysis
- Experimental: Information on the samples (including controls or blanks, if used), equipment, procedures, and measurement method(s)
- Results and Conclusions: Presentation and discussion of the results including a description of the methods used to determine results and uncertainties and, where relevant, a statement to the effect that the results relate to a specific, tested item (e.g., a spectrophotometric filter set with a unique serial number or a recertified gas cylinder).
- References: References to literature, laboratory notebooks, charts, computer records, etc., in which raw and processed data, experimental details, and statistical evaluation are recorded or documented
- Attestation: Signatures and titles of those who have responsibility for the content of the report or authority for its review and release
- Tables, Figures, and Appendices.

NOTE: When using pre-existing electronic documents as templates for new versions of the documents, special care must be exerted in reviewing data entries to ensure that all previous values and terms are updated correctly.

When a CD Official Report is amended after issue, it is reissued as a new document clearly identified as a revision with reference to the original. The revision is uniquely identified following procedures outlined QM-III-852.xx. Such amendments meet all requirements of CD Official Reports. Copies of the revised report are provided to all recipients of the original version.

5.10.2 Certificates

Certificates accompany a reference material, reference data, or calibration, and contain the certified information in a standard format. There are a number of documents that may serve as certificates in different situations. They are described below and, where appropriate, in greater detail in the QM-III-852.xx.

5.10.2.1 Certificate of Analysis

This document accompanies SRMs in which assigned values and their associated uncertainties are provided.

5.10.2.2 Certificate of Traceability (NTRM)

This document accompanies NTRMs in which assigned values and their associated uncertainties are provided.

5.10.2.3 Report of Analysis

This document accompanies RMs in which assigned values and their associated uncertainties are provided.

5.10.2.4 Report of Investigation

This document is equivalent to a Report of Analysis.

5.10.3 Signatory Authority

5.10.3.1 CD Official Reports

CD Official Reports (including those that serve as certificates) are reviewed and approved by the Division Chief or other designee.

5.10.3.2 Certificates

Certificates for SRMs are reviewed and approved by the TPL, analysts, Group Leader, and CD Chief. Review and approval by MSD and SED personnel may be required also. The procedure and documentation comply with NIST Administration Manual Subchapter 5.19 and may be prepared in cooperation with Measurement Services Division and Statistical Engineering Division.

Appendices

Appendix A: Acronyms

ASTM	ASTM International; formerly, American Society for Testing and Materials
CD	Ceramics Division
CRM	Certified Reference Material
DP	Documented Procedure
DQM	Division Quality Manager
EQC	Employee Qualifications and Capabilities
F&C	Feedback and Concerns
GL	Group Leader
GQM	Group Quality Manager
IEA	International Energy Agency
ISO	International Organization for Standardization
LOD	Limit of Detection
LQD	Limit of Quantification
MSD	Measurement Services Division
NIST	National Institute of Standards and Technology
NIST SP	NIST Special Publication
NIST IR	NIST Interim Report/Internal Report
NTRM	NIST Traceable Reference Material
QM	Quality Manual
QS	Quality System
ROA	Report of Analysis
RM	Reference Material
SED	Statistical Engineering Division
SRM	Standard Reference Material
TP	Technical Procedure
TPL	Technical Project Leader
VAMAS	Versailles Project on Advanced Materials and Standards

Appendix B: Project Completion Memorandum

At the completion of an SRM production project, CD must provide the following documentation to Measurement Services Division:

- Draft Certificate
- Report of Analysis
- Project Completion Memorandum

The following is a template for the Project Completion Memorandum as prescribed in the Guide to NIST SRM Development and Production, version 09/29/2004. This memo should be written at about the time the draft certificate of analysis is prepared

MEMORANDUM TO: *Group Leader or Designee*
Documentation and Quality Services (230.04)
Measurement Services Division (230)

THROUGH: *CD Group Leader*
Name of Group (852.##)
Ceramics Division (852)

FROM: *Technical Project Leader*

SUBJECT: Project Completion Memorandum for SRM XXXX *Name*

This memo provides the necessary information to price the SRM units, post the SRM to the website, and properly code the SRM in the SRM sales and inventory system.

SRM Number and Name: XXXX (*Name*)

Division Responsible for the SRM: 852

Technical Contact: (Name of appropriate CD expert)

SRM Category: Number – Name (see SRM catalog)

Number of Units Delivered: (if known, or amount of stock)
Date Units Delivered: (mm/dd/yyyy)

Estimated Annual Sales: (number of units per year; give brief justification)

Number Units to Stock: (total for 5 years to recover WCF)

WCF Costs: (cost center(s), labor costs, and total costs to be recovered in the SRM price)

Cost center	*CC Amount*	*Labor*
<<cc #1>>	<<$cc1 amnt>>	<<$amnt1 used for labor>>
<<cc #2>>	<<$cc2 amnt>>	<<$amnt2 used for labor>>
Total:	<<$cc total>>	<<$total labor>>

Laboratory Base Unit Cost: (Total CC Amount divided by the Number of Units to Stock)

Amount and Type of Other Funds: (give sponsor, cost, and cost center number)

Document is uncontrolled in printed form.

Key Words: (to facilitate searches on the SRM website)

Related SRMs: (for assignment to the proper SRM website table(s))