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Contents

Office of Measurement Services

SRM Quarterly

Standard Reference Materials® Program

National Institute of Standards and Technology
U.S. Department of Commerce, Technology Administration

Program News

Available Now! – Online Ordering of NIST Standard Reference Materials

The NIST SRM Website now provides easier access to the information and services that customers need most. Today U.S. customers can order NIST Standard Reference Materials using the Web for convenience and quick service. A single mouse click provides pages with a current price list, new and newly renewed SRMs and a general list of all SRMs in order by SRM/Catalog number. Orders can then be placed online with virtually no extra effort. The site is available at: http://www.nist.gov/srm

The NIST SRM Website still has all previous features such as NIST SRM/RM Certificates, material safety data sheets, NIST SRM technical contacts and ordering information. Users of the Website will find friendlier layouts for faster service.

The most important addition to the Website is online ordering, currently available to U.S. domestic customers only. Customers can register on the web site to use the new online shopping cart and place orders by credit card or by purchase order number, with billing to follow. Information, such as billing address, shipping address and end-user information is required to complete registration. While not mandatory, customers will also be asked to provide SIC codes and NAICS codes, which will help NIST SRM to better serve specific segments of industry. An active E-mail address is required and allows NIST SRM to send electronic confirmation and other pertinent information regarding an order.

All information obtained from any customer is electronically secure, completely confidential and will only be used to process or follow up on orders.

Visit: http://www.nist.gov/srm and place your next order online!

For further information, contact the NIST SRM Sales Office at:
E-mail: srminfo@nist.gov
Fax: 301-948-3770
Tel: 301-975-6776.

Getting To Know the SRMP Staff

In May 2001, John Rumble, Jr. became Acting Chief of the NIST Standard Reference Materials Program. Dr. Rumble joined NIST (then NBS) in 1980 and worked in the NIST Standard Reference Data Program for many years, becoming Chief in 1994. He is a chemist by training, having worked in an industrial analytical chemistry laboratory. In addition, he has worked on evaluating and disseminating property data for all types of engineering materials and chemicals. In early 2001, Rumble was also named Acting Chief of the NIST Calibrations Program.

The NIST SRM Program is also fortunate to have an outstanding set of project coordinators. Jennifer Colbert coordinates the SRM gases, spectrometric solutions, health/clinical, food/agriculture, pH and high purity SRMs. Bruce MacDonald is the coordinator for environmental SRMs, which includes inorganics, organics, fossil fuels, engine wear material, geological material, ceramic and glass, microanalysis, and EPA water proficiency testing program standards. Joy Thomas coordinates the physical properties SRMs, which includes standards for optical properties, optoelectronics, thermodynamic properties, polymers, and radioactivity. Carlos Beauchamp, currently on assignment from the NIST Material Science and Engineering Laboratory, has responsibility primarily for chemical property and engineering standards, but also coordinates some physical property standards.

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New and Renewal SRMs

SRM 2066
K-411 Glass Microspheres

Numerous requests from the microanalysis community for particle standards certified for selected elements prompted the development of Standard Reference Material (SRM) 2066. Users have expressed a need for a homogeneous particle-like standard in the 1 µm to 20 µm diameter range with known geometry. SRM 2066 is intended for use as a standard for the quantitative microanalysis of particles and for the development of particle matrix correction procedures. The elements present in these microspheres, common to many classes of particles, are silicon, calcium, magnesium, iron, and oxygen. The bulk material used to produce SRM 2066 is the same K-411 glass certified in SRM 470 Glasses for Mineral Analysis.

The certified compositions and uncertainties for silicon, calcium, magnesium, and iron, as well as the reference value for oxygen, are based on a comparative analysis of the SRM 2066 microspheres to the bulk K-411 glass. The certified values for SRM 2066 apply only to microspheres that are 2 µm or larger in diameter.

SRM 2066 consists of approximately 50 mg of glass microspheres ranging in size from 1 µm to 40 µm.

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SRMs 2885, 2886 & 2887 Polyethylene

Polymer standards certified for molar mass are widely used by industry to calibrate size exclusion chromatographs (SEC), the most widely used instrumentation for determining the molecular mass distribution of synthetic polymers. Although polyethylene is the dominant commercial polymer, few high temperature SEC calibration standards are available for use in its analysis. Because of the lack of suitable commercial standards and the rapid depletion of existing NIST Standard Reference Materials (SRMs) for polyethylene, SRMs 2885, 2886, and 2887 have been developed.

SRMs 2885, 2886, and 2887 are intended primarily for use in the calibration and performance evaluation of instruments used to determine the molar mass (previously expressed as molecular weight) and molar mass distribution, by size exclusion chromatography. The certified values for molar mass are listed below.

SRM 2885	6.28	±	0.56	x 10^3 g/mol
SRM 2886	87.0	±	6.0	x 10^3 g/mol
SRM 2887	196.4	±	13.7	x 10^3 g/mol

SRMs 2885, 2886, and 2887 are also certified for intrinsic viscosity in 1,2,4-trichlorobenzene at 130 °C. A single unit of each of these SRMs consists of approximately 0.3 g of polyethylene powder.

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SRM 200a
Potassium Dihydrogen Phosphate (KH_2PO_4)

SRM 200a is a highly purified and homogeneous lot of crystalline potassium dihydrogen phosphate (KH_2PO_4). It is intended primarily for use as a working standard in the calibration and standardization of procedures employed in the fertilizer industry, for the determination of potassium and phosphorus. This SRM serves as the primary benchmark for the evaluation of analytical procedures that are accepted for specifying the contents of potassium and phosphorus in marketed fertilizers.

The certified values for the mass fraction (%) of potassium and phosphorus in SRM 200a are:

Potassium:	28.735 %	±	0.012 %
Phosphorus:	22.7352 %	±	0.0032 %

The value for potassium is based on the results of ion-exchange separation followed by the gravimetric determination of potassium as potassium sulfate (K_2SO_4) from each of six randomly selected bottles. The value for phosphorus is based on duplicate coulometric acidimetric determinations of phosphate from each of ten randomly selected bottles.

A unit of SRM 200a consists of one bottle containing 90 g of crystalline potassium dihydrogen phosphate.

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SRM 154c
Titanium Dioxide

SRM 154c is intended primarily for use in the evaluation of techniques employed in the assay of titanium dioxide (TiO_2) in the paint and ceramic industries.

The certified value, expressed as the mass fraction of TiO_2 on a dry basis is:

TiO_2: 99.591 % ± 0.062 %

This value is based on the results of 12 independent coulometric reductimetric back-titrations performed on a stratified-random sampled set of 8 units of SRM 154c and referenced to coulometrically-standardized potassium dichromate ($K_2Cr_2O_7$). The dissolution and reduction procedures were adapted from American Society for Testing and Materials (ASTM) Standard Method D1394-76.

The certified value includes corrections for elements known to interfere in the reduction and coulometric titration used for the certification. However, it is the responsibility of the user to ascertain which elements interfere with the procedure being calibrated or evaluated and to make corresponding corrections for the given application for use of SRM 154c as a calibration or evaluation standard.

A unit of SRM 154c consists of a clear glass bottle containing 90 g of titanium dioxide as a fine (< 45 µm) powder.

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SRM 861
Nickel-based Superalloy

Modern aerospace nickel-based superalloys are used to fabricate single crystal turbine blades used in high performance jet engines. One of the many specifications for this type of alloy is that the sulfur concentration be less than 1 mg/kg (ppm). Single crystal alloys exhibit greatly improved cyclic oxidation resistance and scale adhesion when the bulk sulfur content is near or below 1 mg/kg. The accuracy of instrumentation used by the aerospace industry to determine sulfur in alloys is dependent on the availability of

accurate standards for calibration and quality control assessment. SRM 861 Nickel-based Superalloy is intended primarily for use in the evaluation of analytical techniques that determine sulfur and phosphorus in nickel-based alloys. A unit consists of a glass jar containing 50 g of metal chips that have passed through a 600 µm (30 mesh) screen.

The sulfur concentration was determined in SRM 861 by isotope dilution thermal ionization mass spectrometry to be 0.561 mg/kg ± 0.078 mg/kg. This is the lowest sulfur value ever certified on any metallic SRM.

A reference value for phosphorus, which typically must be below 50 mg/kg in aerospace alloys, was determined by a new radiochemical neutron activation procedure to be 12.71 mg/kg ± 0.71 mg/kg.

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Certification Revisions

SRM 1570a Trace Elements in Spinach Leaves
(Revised to Include Nutrition Composition)

SRM 1570a Trace Elements in Spinach Leaves was originally value-assigned for its elemental composition (major, minor, and trace elements), but no additional nutritional information was provided. The SRM 1570a was recently distributed by NIST to four collaborating laboratories with expertise in food analysis for the measurement of proximates (solids, fat, protein, etc.). As a result, values were recently assigned for solids, ash, protein, and total dietary fiber.

In addition to the validation of elemental composition, this SRM will now also be useful for validation of analytical methods for the measurement of nutrients in botanical, agricultural, and food materials of similar composition. This is based on AOAC's fat-protein-carbohydrate triangle, a nine-sectored triangle in which foods can be plotted based on the proportions of fat, protein, and carbohydrate that they contain. The SRM may also be used as a "primary control material" in the value-assignment of in-house control materials of similar composition.

The addition of the proximate information to this existing SRM as well as to other NIST food matrix RMs/SRMs was provided in response to industry and customer needs for assigned values for proximates in six of the nine sectors of the AOAC triangle.

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Notice of Revision:
SRM 2724b
Sulfur in Diesel Fuel Oil

The certificate of analysis for SRM 2724b, issued on 09 September 1999, has been revised for sulfur content based on recent stability testing measurements. Effective 28 March 2001, the new certified value for sulfur content is 0.04265 % ± 0.00057 % (n = 14). The previous certified value for sulfur was 0.04282 % ± 0.00015 % (n = 9).

The revised certificate for SRM 2724b is being mailed to each organization known to have purchased SRM 2724b. The revised certificate is also available from the SRMP website http://www.nist.gov/srm by selecting "Catalog" from the website homepage and then entering the SRM number "2724b" into the "Quick Search" search engine. Next click on the SRM's name and select "C" to view and subsequently print the certificate.

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Other NIST Measurements and Standards News

New NIST Policy on Traceability

In nearly all types of activities—be they related to manufacturing, finance, health, regulatory affairs or even sports—people and organizations are becoming sticklers for measurement accuracy. However, unlike beauty (said to be in the eye of the beholder), accuracy must be judged on the basis of a measurement pedigree—a documented line of descent from accepted standards.

NIST, the nation's measurement authority, has responded to this growing customer need for demonstrable accuracy. It has created an online resource devoted to matters pertaining to the topic of measurement traceability—whether the result of a specific measurement can be related to accepted international or national standards through an unbroken chain of comparisons.

"More and more of our customers are asking questions about traceability," explains NIST Acting Director Karen Brown. "These range from the most basic—What is it?— to the more complex—How can I judge the credibility of a supplier's claims that his measurements are linked to those developed and maintained by NIST and ultimately to the SI, the International System of Units?"

Factors driving the growth of traceability requirements include increasing world trade, growing reliance on laboratory accreditation as a means of assuring confidence in calibration and test reports, the continuing spread of quality standards and, in some technology areas, a proliferation of regulations.

At the new NIST web site, visitors can read the NIST policy on traceability. They also can review, among other resources, a glossary of terms, answers to an extensive set of frequently asked questions on traceability, examples of relevant NIST measurement programs, and a traceability checklist for users of calibration services.

To learn more, visit the NIST Traceability Web site at: http://www.nist.gov/traceability/

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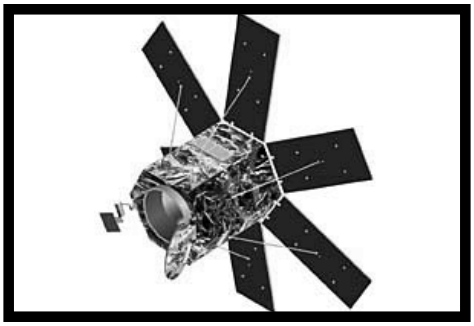
SRM 2035 in Space

The Air Force Research Laboratory (AFRL) Space Vehicles Directorate is partnering with Orbital Sciences Corporation in a unique program arrangement, called Warfighter-1 (WF-1). The program leverages commercial satellite technology and remote sensing capability with an Air Force hyperspectral imaging (HSI) sensor. This satellite system, Orbview-4, will provide remote sensing capability in a spectral-spatial data hypercube.



The spectral coverage will include the visible, near-infrared and short wave infrared regions (450 nm to 2500 nm) at a nominal 10 nm bandpass and characterize and validate the WF-1 sensor before the satellite launch in August of 2001.

As part of the effort to calibrate the wavelength axis of the HSI, the AFRL turned to NIST for help in providing a suitable calibration standard. SRM 2035 is a recently developed wavelength standard for transmission measurements in the near infrared (NIR) spectral region. This optical filter standard is a borate glass containing four rare earth oxides. It has seven absorption bands that are certified for use as wavelength features between 1.0 and 2.0 microns. Because SRM 2035 contains holmium and neodymium oxides, it also has a rich UV-visible spectrum with approximately 11 bands between 345 nm and 975 nm. Although SRM 2035 was originally developed with chemical applications in mind, the combination of its small size (25 mm diameter x 1.5 mm thick), light weight, broad spectral coverage and well characterized temperature dependence makes it ideally suited for use in extraterrestrial satellite applications. After several initial tests and discussions with Orbview-4 mission engineers, SRM 2035 was adopted for use as a key component in the On Board Health Unit (OBHU), a system which will periodically monitor and validate the system performance of the HSI unit.

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New NIST Recommended Practice Guides for Charpy, Particle Size, and Rockwell Hardness Standard Reference Materials

NIST has released three new practice guides for use with NIST SRMs. These new guides will help users of Charpy, Particle Size and Rockwell C Hardness SRMs get better test results from each type of standard. The Particle Size and Rockwell Hardness Practice Guides are both new this year.

The Charpy practice guide, entitled *Installing, Maintaining and Verifying Your Charpy Impact Machine*, (NIST Special Publication 960-4) is a revision of NIST Technical Note 15008, published last year. The new NIST Recommended Practice Guide is distributed with Charpy standard (SRM 2096) to provide technical assistance to engineers and technicians when installing, maintaining and verifying their Charpy impact machines.

Charpy machines are used to determine the temperature at which structural materials go through a ductile-to-brittle transition. Charpy impact testing is often specified as an acceptance test for materials used in critical structures such as bridges and pressure vessels. This publication is a compilation of manufacturers' mounting instructions and years of technical information gathered from verification of thousands of machines.

For a free copy of NIST Special Publication 960-4, *NIST Recommended Practice Guide: Installing, Maintaining and Verifying Your Charpy Impact Machine*, contact:

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The *NIST Recommended Practice Guide: Particle Size Characterization* (NIST Special Publication 960-1) will help industrial and academic laboratories measure particle size and size distribution of ceramic powders in a more reliable and reproducible way. Improper powder size measurements during processing can affect the mechanical, electrical and thermal properties of the final product, resulting in poor quality and high rejection rates. Designed for a general user, the guide includes aspects of particle characterization research conducted by NIST for well over a decade. This research also has resulted in the development of SRMs and improvements in measurement procedures.

The guide covers techniques commonly used in the ceramics manufacturing industry such as microscopy, sieving, gravitational sedimentation and laser light diffraction. For each technique, the book provides directions for sample preparation, instrument calibration, and set-up, details relevant national and international standards, and discusses capabilities, limitations and general principles. This guide is distributed with many of the particle sizing SRMs.

For a copy of NIST Special Publication 960-1, *NIST Recommended Practice Guide: Particle Size Characterization*, contact:

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The *NIST Recommended Practice Guide: Rockwell Hardness Measurement of Metallic Materials* (NIST Special Publication 960-5) is also available. Rockwell Hardness is a method-based test primarily used by metals and metal products producers to measure the hardness of metal parts such as those found in aircraft and automobiles. The new guide is aimed at promoting accuracy and consistency in test results in the laboratory and on the production floor. Offering good practice recommendations, the guide highlights the causes of variability in test results. To help machine operators avoid errors, the guide covers common problems such as using the correct Rockwell scale, surface preparation, speed of testing, machine verification and environmental factors.

As part of its Rockwell Hardness standardization program, NIST has developed Standard Reference Material test blocks for the Rockwell C scale, which is used for hard metals, primarily steel. SRMs 2810, 2811, and 2812 are used to calibrate commercial hardness machines. Copies of the new practice guide will be distributed with each of these standards.

To obtain a copy of NIST Special Publication 960-5, *NIST Recommended Practice Guide: Rockwell Hardness Measurement of Metallic Materials,* or for more information on NIST's Rockwell hardness research, contact:

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To order a single copy of these and future practice guides, contact *inquiries@nist.gov* or call 301-975-NIST (6478). For an electronic copy, visit: http://www.nist.gov/practiceguides to download practice guides as .pdf files.

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