SRM SPOTLIGHT

September 2012

Editor: Regina R. Montgomery

New and Renewal NIST SRMs/RMs

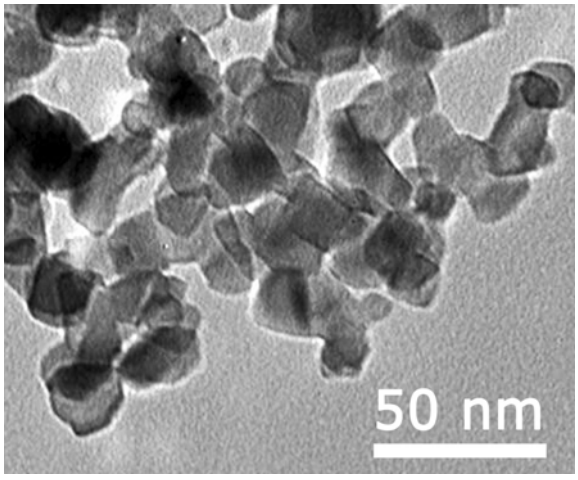
NIST SRM 1898 Titanium Dioxide Nanomaterial

This Standard Reference Material is intended primarily for use as a benchmark and investigative tool for evaluation of the potential environmental, health and safety risks that may be associated with manufactured nanomaterials during their product life cycle. The SRM consists of a commercial titanium dioxide (TiO_2) powder, an industrially relevant nanomaterial, which has been widely studied and reported in the environmental, health and safety (EHS) literature. The certificate of analysis contains a wide range of physical and chemical characterization data as well as dispersion protocols for its use in toxicity and fate studies.

This SRM is also intended for use in the calibration and performance testing of gas sorption instruments used for determining the Brunauer-Emmet-Teller (BET) specific surface area of powders and porous solids. Along with SRMs 2206 and 2207, these SRMs will provide a suite of reference materials covering a wide range of specific surface area values for instrument calibration and performance testing.

Finally, the SRM may be of ancillary use as a benchmark for photoreactivity studies, as the source material has been utilized for more than two decades by the photocatalysis and photochemistry communities as a de facto standard or benchmark against which other materials are compared; however, photoreactivity data are not included on the certificate of analysis.

A unit of SRM 1898 consists of an amber glass bottle containing nominally 15 g of mixed-phase (anatase and rutile) nanocrystalline TiO_2 in the form of a dry agglomerated powder certified for BET specific surface area determined by nitrogen gas sorption at liquid nitrogen temperature (77.3 K) using the static volumetric gas sorption technique.



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NIST SRMs 2206 and 2207 Controlled Pore Glass – BET Specific Surface Area

NIST has released two new Standard Reference Materials for the calibration and performance testing of instruments used for the determination of the Brunauer-Emmett-Teller (BET) specific surface area (SSA) by the static volumetric gas sorption technique. The new SRMs consist of granular controlled pore glass (CPG), a high purity, thermally stable porous silicate with a well-defined pore structure and pore size. SRMs 2206 (nominally 11 m^2/g) and 2207 (nominally 178 m^2/g) replace SRMs 1899 and 1897, respectively. These new SRMs provide a more chemically and thermally stable reference material for performance verification of gas sorption analyzers.

A unit of SRM 2206 consists of one bottle containing approximately 5.1 g of high-purity granulated controlled-pore glass with a nominal pore diameter of 300 nm. A unit of SRM 2207 consists of one bottle containing approximately 5.1 g of high-purity granulated controlled-pore glass with a nominal pore diameter of 18 nm. Both SRMs are certified for BET SSA determined by nitrogen gas sorption at liquid nitrogen temperature (77.3 K) using commercial instrumentation.

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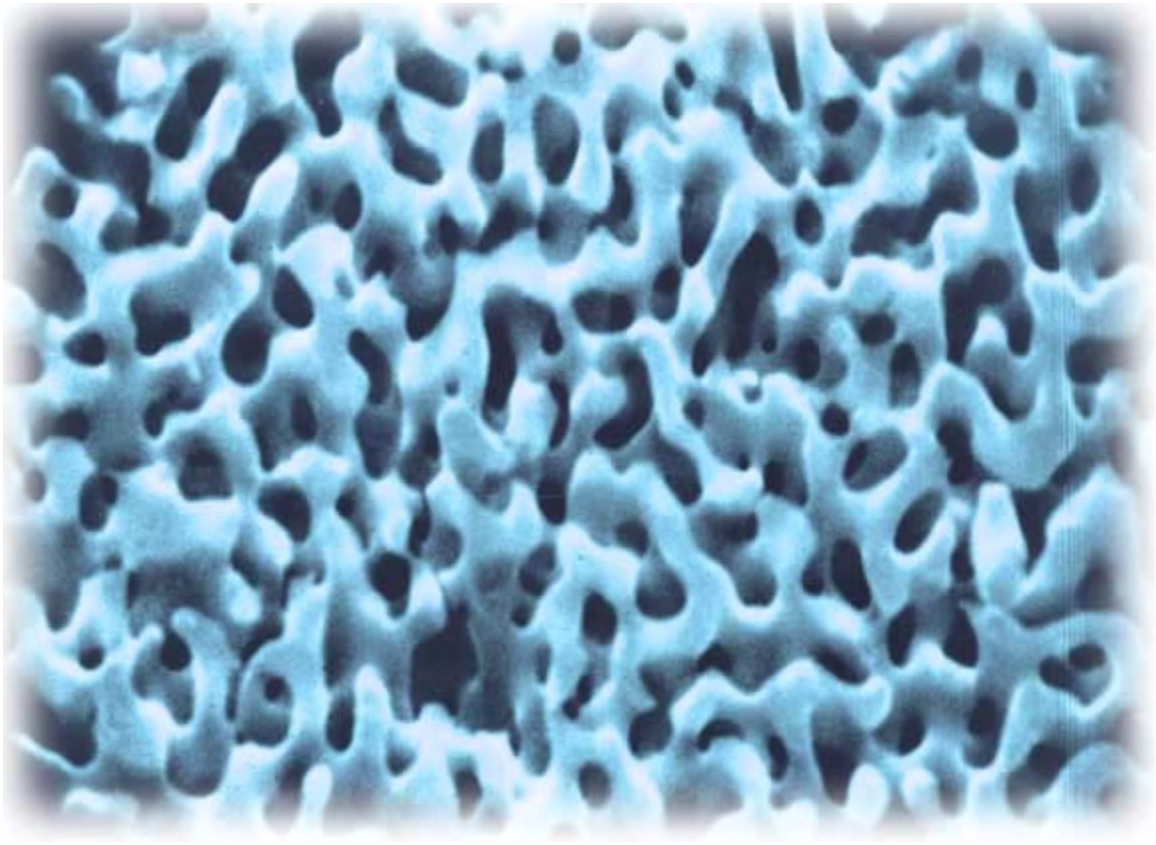

Scanning electron image showing characteristic bicontinuous porous structure of controlled pore glass used in SRMs 2206 and 2207.

NIST Standard Reference Material 2461 Standard Cartridge Case

Standard Reference Material 2461, developed in partnership with the Bureau of Alcohol, Tobacco, Firearms, and Explosives (ATF), will help crime laboratories to verify that automated optical equipment for cartridge case image acquisition and correlation is operating properly. It will also help them to establish ballistics measurement traceability and achieve accreditation from standards organizations. SRM 2461 is a physical standard that provides markings of a fired cartridge case. The figure shows both the overall shape and the detailed surface structure. Each unit of SRM 2461 consists of a circular electroformed nickel plate, replicated from the head of a fired master cartridge case, which contains the surface topography signatures of a breech face impression, a firing pin impression, and an ejector mark. The electroformed plate is cemented to a brass cylinder holder so that the assembly resembles a real fired cartridge case. The master cartridge case was fired at the National Laboratory Center of the ATF. The SRM cartridge cases produced from that master by electroforming have virtually the same surface topography signatures.

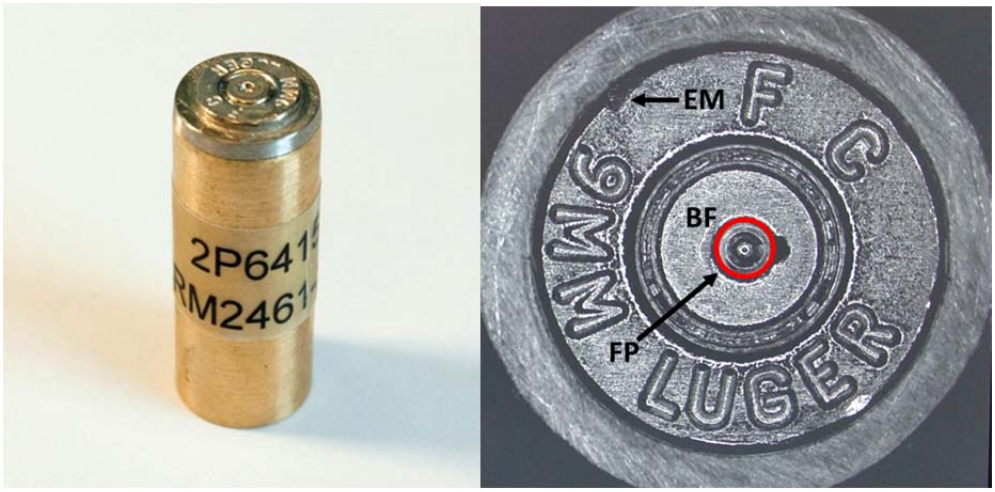

Figure 1. Left – a NIST SRM 2461 Cartridge Case mounted on a brass cylinder holder; right – optical micrograph showing the three certified areas of the SRM, breech face impression (BF), firing pin impression (FP), and ejector mark (EM).

SRM 2461 complements the SRM 2460 Standard Bullet developed previously. Each unit of the SRM may be used to test automated optical microscopes used in crime labs and to test systems that directly perform topographic imaging of cartridge cases.

Two properties of the surface topography are used to characterize the similarity of the cartridge case surface topography images: the areal cross correlation function maximum $ACCF_{max}$ and the signature difference D_s. The certified values for these parameters are obtained from statistical correlations between the surface topography of breech face, firing pin and ejector mark regions of the SRM 2461 cartridge cases and those of reference standards, acquired from three reference SRM cartridge cases. When two correlated cartridge case signatures are exactly the same (point by point), D_s must be equal to 0 and $ACCF_{max}$ must be equal to 100 %.

All units of the SRM as well as the reference images were measured with a confocal microscope at NIST. The topography images were bandpass filtered to minimize form and waviness and to emphasize the fine roughness features of the measured topographies. The topography images were then correlated with those of the three reference standards. For all three regions of the SRM cartridge cases being distributed, the lower limit for $ACCF_{max}$ and upper limit for D_s, each with a 95 % confidence level are reported in the SRM certificate.

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NIST SRM 2492: First SRM for Rheological Properties of Paste Mixtures

Standard Reference Material 2492 Bingham Paste Mixture was designed to help the industry develop new tests to measure the flow or rheological properties of concrete.

In the construction industry, concrete is the most used material in the world (3.1 billion metric tons in 2012). Concrete is conventionally a mixture of aggregates (10 mm - 30 mm), sand (1 mm - 3 mm), cement (1 μm – 100 μm) and water that behaves as a fluid until it hardens to become a load-bearing solid. During the design and construction process, the flow properties need to be predicted during mix development and measured at the construction site to ensure the proper placement and quality of the finished product. The typical instrument used to measure rheological properties of a fluid is called a rotational rheometer, in which the tested fluid is sheared between two surfaces, one of which is rotating [1]. Commercial laboratory rheometers are mainly designed for homogeneous liquids containing little or no solid particles, such as polymers, mayonnaise or creams. The rheometer manufacturers recommend using a standard oil of known viscosity to verify that the instrument is operating correctly. In the case of fresh concrete, the distance between the shearing surfaces needs to be sufficiently large to accommodate aggregates of at least 20 mm in diameter. This increase in the gap size between the rotating surfaces leads to generally unknown shear patterns and test results that cannot be expressed in fundamental units. Therefore, calibration of such large and non-standard rheometers is nearly impossible using the traditional method involving oils. A standard reference material is the logical solution for the calibration of concrete rheometers. Moreover, given the volumes being tested, the material must be relatively inexpensive, safe and incorporate aggregates.

NIST adopted a three-phase approach consisting of first developing a paste that can be measured with a conventional rheometer (SRM 2492). In future SRMs in this series, a mortar will be produced by adding fine beads to the paste (SRM 2492 with fine beads) and, finally, a concrete will be made by adding coarse aggregates (mortar with coarser beads). The rheological parameters of mortar and concrete will be determined from the paste by a combination of numerical simulations and experimental measurements. The simulation will be used to calculate the viscosity of the suspensions (mortar or concrete) from the medium's viscosity (cement paste) for various aggregate volume fractions and size distributions. SRM 2492 is the first step in obtaining a reference material for a concrete rheometer. The last two SRMs will be developed in future years.

[1] V. A. Hackley, C.F. Ferraris, "The Use of Nomenclature in Dispersion Science and Technology," NIST Recommended Practice Guide, SP 960-3, (2001) http://www.nist.gov/public_affairs/practiceguides/SP960-3.pdf (2001).

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NIST SRM 2907 Trace Terrorist Explosives Simulants

NIST has released SRM 2907 Trace Terrorist Explosives Simulants to promote the most sensitive and accurate detection of two chemicals, PETN (pentaerythritol tetranitrate) and TATP (triacetone triperoxide), often used as the explosive charge in terrorist attempts, such as the Lockerbie disaster, the shoe and underwear bombers, and the London subway bombing.
As with past SRMs of trace explosives, instrument developers, academic researchers and government labs will likely use SRM 2907 to test, refine and validate their new detector designs. SRM 2907 is the third SRM supporting the detection of trace explosives. The Department of Homeland Security Science and Technology Directorate funded the production of the work presented in this material under HSHQDC-10-00297 with NIST.

The two materials that comprise this SRM are designed to simulate the size and behavior of residues that remain after handling explosives. In airport security, typically these residues are collected with hand-held swipe wands and heated to vaporize the explosives for detection. The Semtex 1A (PETN) material in the SRM was prepared by completely dissolving the plastic explosive in solvent. The TATP material required custom synthesis of this improvised explosive. Each of the two materials was rendered inert by coating from solution onto particulate substrates using a rotary evaporative process.

Certification of the PETN and TATP content of the simulants used measurements by liquid chromatography with ultraviolet absorbance or mass spectrometric detection (LCUV and LCMS, respectively). Analytical challenges included development of a new ionization enhancing additive (for PETN) and a custom synthesized stable-isotope labeled $^{13}C_3$-TATP for the LCMS measurements. The development of the materials and analytical methods for value assignments is described in the journal Analytical Chemistry [Anal. Chem. 83:9054–9059, (2011)].

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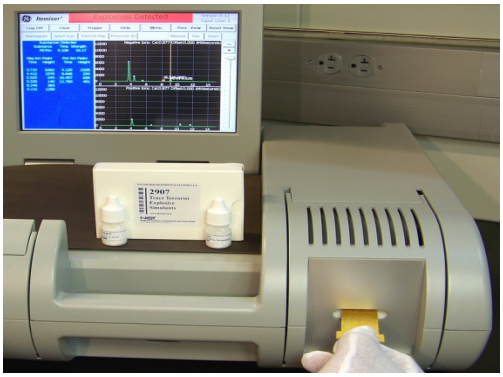

Renewals

Steady-state Thermal Transmission Properties: NIST SRM 1450d – Fibrous Glass Board

Standard Reference Material 1450d is intended for use in the measurement of the thermal conductivity or thermal resistance of insulation materials that are tested in plate-type instruments such as the guarded-hot-plate apparatus or the heat-flow-meter apparatus. The guarded-hot-plate method has been used for over 100 years in the determination of design heat transmission data for building materials and was standardized by ASTM International in 1945, designated Test Method C 177. Thermal insulation SRMs are used in standard test methods for the purposes of checking guarded-hot-plate apparatus, calibrating heat-flow-meter apparatus, and, when necessary, checking or calibrating hot-box apparatus. These SRMs also assist insulation manufacturers in the United States in complying with federal requirements for labeling and advertising of home insulation (also known as the U.S. Federal Trade Commission "R-value Rule"). Standard Reference Material 1450d is the fifth renewal of the 1450 Series, which was introduced in 1958 by NIST (previously the National Bureau of Standards) as part of a formal measurement program to assist the U.S. thermal insulation industry. Prior versions of SRM 1450 were issued in 1978, 1979, 1982, and 1997.

A unit of SRM 1450d consists of a square panel of fibrous glass and phenolic binder molded into a semi-rigid board. The nominal dimensions of a unit are 611 mm by 611 mm by 26 mm and the bulk density of the material lot ranges from 114 $kg \cdot m^{-3}$ to 124 $kg \cdot m^{-3}$. The thermal conductivity value assignments for 1450d were developed using the NIST 1016 mm Guarded-Hot-Plate Facility [1]. The measurements were conducted in accordance with a randomized full factorial design at three levels of density covering the material lot range (114 kg/m^3 to 124 kg/m^3) and at five temperature levels from 280 K to 340 K. The resultant thermal conductivity data were fit to several models, but the final certification equation was found to be simply a linear model in temperature. The expanded uncertainties (coverage factor of k equal to 2) for the certified values of thermal conductivity were determined to be ± 1 % over the temperature range of 280 K to 340 K. The development and production of SRM 1450d, Fibrous Glass Board have been documented in NIST Special Publication 260-173.

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[1] NIST Engineering Laboratory; Line Heat-Source Guarded Hot Plate, available at http://www.nist.gov/el/facilities_instruments/line_heat_source_guarded_hot_plate_fac.cfm (accessed Aug 2012).

Renewals (continued)

SRM 1635a Trace Elements in Coal (Subbituminous)

SRM 1666b Propane in Air (Nominal Amount-of-Substance Fraction – 10 µmol/mol) Lot # 84-K-XX

SRM 1668b Propane in Air (Nominal Amount-of-Substance Fraction – 100 µmol/mol) Lot # 82-L-XX

SRM 2035a Near-Infrared Wavelength/Wavenumber Transmission Standard

SRM 2092 Low-Energy Charpy V-Notch Impact Specimen NIST-Verification (ASTM E23) Lot # LL-129

SRM 2096 High-Energy Charpy V-Notch Impact Specimen NIST-Verification (ASTM E23) Lot # HH-129

SRM 3107 Boron (B) Standard Solution

SRM 3124a Indium (In) Standard Solution

SRM 3129a Lithium (Li) Standard Solution

SRM 3155 Tantalum (Ta) Standard Solution

SRM 3165 Vanadium (V) Standard Solution

SRM 3180 Iodide Anion (I^-) Standard Solution

Revisions

Certificate Revisions: Are You Using These Materials?

This is a list of our most recent certificate revisions. NIST updates certificates for a variety of reasons, such as to extend the expiration date or to include additional information gained from stability testing. Users of NIST Standard Reference Materials should ensure that they have the current certificates. If you do not have the current certificate for your material, you can print or view a copy at our website at **http**://**www.nist.gov/srm** or contact the Measurement Services Division at:

Phone: 301-975-2200 **Fax:** 301-926-4751 **Email**: srminfo@nist.gov

SRM 141d Acetanilide
New expiration date : 30 September 2017

SRM 610 Trace Elements in Glass
Technical changes
Editorial changes

SRM 611 Trace Elements in Glass
Technical changes
Editorial changes

SRM 612 Trace Elements in Glass
Technical changes
Editorial changes

SRM 613 Trace Elements in Glass
Technical changes
Editorial changes

SRM 614 Trace Elements in Glass
Technical changes
Editorial changes

SRM 615 Trace Elements in Glass
Technical changes
Editorial changes

SRM 616 Trace Elements in Glass
Technical changes
Editorial changes

SRM 617 Trace Elements in Glass
Technical changes
Editorial changes

Revisions (continued)

SRM 676a Alumina Internal Standard for Quantitative Analysis by X-ray Powder Diffraction
Technical changes
Editorial changes

SRM 864 Nickel Alloy UNS N06600
Values updated
Editorial changes

SRM 968e Fat-Soluble Vitamins, Carotenoids, And Cholesterol in Human Serum
Editorial changes

SRM 1244 Nickel Alloy UNS N06600
Values updated
Editorial changes

SRM 1575a Trace Elements in Pine Needles (*Pinus Taeda*)
New expiration date : 01 August 2022
Editorial changes

SRM 1580 Organics in Shale Oil
New expiration date : 31 March 2027
Values updated
Editorial changes

SRM 1582 Petroleum Crude Oil
New expiration date : 30 September 2020
Values updated
Editorial changes

SRM 1584 Priority Pollutant Phenols in Methanol
New expiration date : 31 March 2027
Values updated
Editorial changes

SRM 1623c Sulfur in Residual Fuel Oil
New expiration date : 13 June 2015
Editorial changes

Revisions (continued)

SRM 1635a Trace Elements in Coal (Subbituminous)
Editorial changes

SRM 1662a Sulfur Dioxide in Nitrogen (Nominal Amount of-Substance Fraction 1000 µmol/mol
Lot # 93-H-XX
New expiration date: 23 September 2019
Editorial changes

SRM 1664a Sulfur Dioxide in Nitrogen, (Nominal Amount of-Substance Fraction 2500 µmol/mol
Lot # 91-E-XX
New expiration date : 07 May 2018
Editorial changes

SRM 1849a Infant/Adult Nutritional Formula
Values added
Editorial changes

SRM 1897 Specific Surface Area Standard
New expiration date : 01 November 2012
Editorial changes

SRM 1932 Fluorescein Solution
New expiration date : 05 June 2017
Editorial changes

SRM 1941b Organics in Marine Sediment
Reference values added
Editorial changes

SRM 2234 Gallium for Thermal Analysis
New expiration date : 01 August 2015
Editorial changes

SRM 2235 Bismuth for Thermal Analysis
New expiration date : 01 August 2015
Editorial changes

SRM 2385 Slurried Spinach
New expiration date : 30 September 2017
Editorial changes

SRM 2396 Oxidative DNA Damage Mass Spectrometry Standards
New expiration date : 01 March 2021
Editorial changes

Revisions (continued)

SRM 2569 Lead Paint Film for Children's Products
Editorial changes

SRM 2627a Nitric Oxide in Nitrogen (Nominal Amount-of-Substance Fraction - 5 µmol/mol)
Lot # 48-H-XX
Editorial changes

SRM 2642a Carbon Monoxide in Nitrogen (Nominal Amount-of-Substance Fraction 8 % mol/mol)
Lot # 51-D-XX
New expiration date : 15 July 2019
Editorial changes

SRM 2646a Propane in Nitrogen (Nominal Amount-of-Substance Fraction 1000 µmol/mol)
Lot # 103-C-XX
Editorial changes

SRM 2683c Bituminous Coal (Sulfur, Mercury, and Chlorine)
Editorial changes

SRM 2702 Inorganics in Marine Sediment
New expiration date : 01 October 2022
Editorial changes

SRM 2740a Carbon Monoxide in Nitrogen (Nominal Amount-of-Substance Fraction 10 % mol/mol)
Lot # 59-B-XX
New expiration date : 15 July 2019
Editorial changes

SRM 2779 Gulf of Mexico Crude Oil
Editorial changes

SRM 3075 Aroclor 1016 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3076 Aroclor 1232 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3077 Aroclor 1242 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

Revisions (continued)

SRM 3078 Aroclor 1248 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3079 Aroclor 1254 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3080 Aroclor 1260 in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3081 Aroclor 1016 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3082 Aroclor 1232 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3083 Aroclor 1242 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3084 Aroclor 1248 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3085 Aroclor 1254 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3086 Aroclor 1260 in Methanol
New expiration date : 31 July 2030
Editorial changes

SRM 3090 Aroclors in Transformer Oil
New expiration date : 31 July 2030
Editorial changes

SRM 3091 Aroclors in Methanol
New expiration date : 31 July 2030
Editorial changes

Revisions (continued)

SRM 3280 Multivitamin/Multielement Tablets
Editorial changes

RM 8456 Ultra High Molecular Weight Polyethylene
Editorial changes

RM 8457 Ultra High Molecular Weight Polyethylene
Editorial changes

ORDER NIST SRMs ONLINE

You can now order NIST SRMs through our new online ordering system, which is continually updated. **PLEASE NOTE:** Purchase orders and credit cards may be used when ordering an SRM online. This system is efficient, user-friendly, and secure. Our improved search function finds keywords on SRM detail pages as well as words in titles. Also note that we are placing many historical archive certificates online for your convenience.

https://srmors.nist.gov

Please Register Your Certificate Online!

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http://tsapps.nist.gov/msdsurvey/register/default.aspx?ID=2

NIST SRM Remaining 2012 Exhibit Schedule

AOAC International
September 30 – October 3, 2012
Planet Hollywood
Las Vegas, NV

Materials Science & Technology Conference
October 7 – 11, 2012
David L. Lawrence Conference Center
Pittsburgh, PA

Material Research Society Fall Meeting (MRS)
November 26 – 29, 2012
Hynes Convention Center
Boston, MA

IMPORTANT MESSAGE about accessing the SRM website at http://www.nist.gov/srm

PLEASE NOTE: New security settings to protect your private information have been mandated by the U.S. government. The following are instructions to upgrade your browser settings so you can view SRM documents, perform searches and order online.

For Mozilla Firefox

1) You must have version 3.0.5 or later
2) Enable SSL 3.0
3) Enable TLS 1.0

To enable SSL 3.0 and TLS 1.0

1) Go to Tools > Options
2) Click on the Advanced icon
3) Click the Encryption tab
4) Under Protocols, make sure both boxes are checked

For Internet Explorer

1) You must have version 6.0 or later
2) Enable SSL 3.0
3) Enable TLS 1.0

To enable SSL 3.0 and TLS 1.0

1) Go to Tools > Internet Options
2) Click on the Advanced tab
3) Scroll down to Security
4) Make sure that both SSL 3.0 and TLS 1.0 are checked

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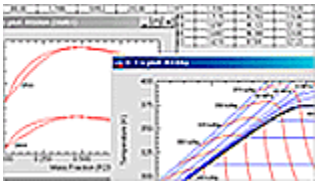
NIST Measurement Services Websites of Interest

Standard Reference Materials

Standard Reference Materials
www.nist.gov/srm

Historical Archived Certificates/Reports of Investigation
https://www-s.nist.gov/srmors/certArchive.cfm

Standard Reference Data



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