Fall 2002

Editor:
Regina R.Montgomery

SRM SPOTLIGHT

In this Issue…

New SRMs

Crude Oil SRMs for Sulfur, Mercury, and Water Analysis

NIST announces the release of two Standard Reference Materials (SRMs) - 2721 Crude Oil (Light-Sour) and 2722 Crude Oil (Heavy-Sweet) which are certified for sulfur, mercury, and water. These two SRMs are commercial crude oils from two different Texas fields. They will be useful for evaluating current and new analytical techniques and calibrating instruments for total sulfur, mercury, and water in crude oil and materials of a similar matrix.

Each crude oil used was passed through a 10 µm filter and blended before being ampouled. A unit of each crude oil consists of five amber ampoules, each containing approximately 10 mL of crude oil.

With recent increased global interest in sulfur and mercury contaminants in fuels, these SRMs are expected to be valuable additions to petroleum laboratories worldwide.

Technical Contact: Dr. William Kelly
E-mail: william.kelly@nist.gov

SRM 1897 Specific Surface Area

This SRM has been developed for calibration of instruments used to measure specific area (SSA) in the range of 0.1 m^2/g to 1000 m^2/g The SSA of this material was measured using a nitrogen gas static volumetric Branauer-Emmett-Teller (BET) surface area analysis instrument. A unit of SRM 1897 consists of one vial containing approximately 7 grams of silica-alumina compound, sieved to pass a 106 µm (No. 140) sieve. This SRM replaces RM 8572 Silica-Alumina (SSA).

Technical Contact: Dennis Minor
E-Mail: dennis.minor@nist.gov

PHONE 301-975-6776 **FAX 301-948-3730** **E-MAIL SRMINFO@NIST.GOV**

SRM News

Check *American Laboratory* for the latest NIST SRM News….

Beginning in November, 2002, The National Institute of Standards and Technology (NIST) is publishing articles highlighting various SRMs in *American Laboratory* bi-monthly. The November issue features articles on Microscopy Optics and Micro Liquid Chromatography. You can contact *American Laboratory* by phone at 203-926-9300 or you can also fax them at 203-926-9310 to receive this magazine free of charge.

If you would like to receive your newsletter via e-mail, please send your e-mail address to:

Spotlight@nist.gov

NIST SRM Exhibit Schedule

Association of Analytical Communities (AOAC) -Los Angeles, CA - Biltmore Hotel, September 22-26, 2002

Metrosul III - Curitiba Parana, Brazil – September 30 - October 3, 2002

Bio-Expo Korea - October 2 - October 6, 2002

ASM Materials – Columbus, OH, Columbus Convention Center, October 7 –October 10, 2002

Eastern Analytical Symposium (EAS)-Somerset, NJ– Somerset Convention Center, November 18-21, 2002

Magnetism and Magnetic Materials Conference – Tampa, FL - Tampa Marriott Waterside Hotel November 11 - 13, 2002

PHONE 301-975-6776 FAX 301-948-3730 E-MAIL SRMINFO@NIST.GOV

New SRMs continued...

Mercury Porosimeter Standard

SRM 1918 Mercury Porosimeter Intrusion Standard is a new SRM for use in calibrating mercury Porosimeter intrusion analytical instruments. Mercury is the most commonly used penetrating liquid in commercial instruments. The surface tension and contact angle for mercury with many materials are well known. Modern mercury porosimeters allow for several methods of intrusion and data manipulation software allows for numerous views of the pore filling process. Each unit of SRM 1918 consists of one vial containing approximately 12 g of an extruded silica-alumina compound.

Certified Values for SRM 1918

Mean Pore Diameter
8.847 ± 0.363 nm
Median Pore Diameter
8.503 ± 0.218 nm
Total Intruded Volume
0.547 ± 0.018 mm^3/g

Technical Contact: Dennis Minor
E-mail: dennis.minor@nist.gov

SRM 2853 Magnetic Moment Standard -
Yttrium Iron Garnet Sphere

This SRM is intended for calibrating magnetometers (such as vibrating sample magnetometers) used in the measurement of the magnetic properties of materials. As a consequence of its small size and weight, this SRM can be used in the relatively recently developed very- high sensitivity "alternating gradient magnetometers" (AGM), thereby providing for the first time a means to put measurements from those devices on an absolute basis. The low magnetization value of the YIG sphere SRM also enables it to be measured in the more commonly available vibrating sample magnometer (VSM) and superconducting quantum interference device (SQUID) magnetometers for independently calibrating the most sensitive scales in those instruments. The ferromagnetic character of the SRM along with its low saturation field make it unnecessary for the user to have calibrated the magnetic field of the magnetometer prior to calibrating its magnetization scale.

SRM 2853 consists of a yttrium iron garnet (YIG) sphere with a nominal diameter of 1 mm and a nominal mass 2.8 mg. The lot was produced by grinding pure (99.5 %), single-crystal YIG into spheres.

Technical Contact: Robert Shull
E-Mail: robert.shull@nist.gov

New SRMs continued…

L-Band Wavelength Calibration Standards

SRM 2514 Wavelength Calibration Reference (carbon monoxide $^{12}C^{16}O$) and SRM 2515 Wavelength Calibration Reference (carbon monoxide $^{13}C^{16}O$ are two wavelength calibration transfer standards for the new L-band of wavelength division multiplexed (WDM) optical fiber communication systems. These standards are based on the absorption spectrum of carbon monoxide. The Optoelectronics Division has characterized the pressure-induced shifts of these absorption lines and certifies the center wavelengths of 41 lines with uncertainties ranging from 0.4 to 0.7 pm. Because they are based on the quantized vibrational and rotational motion of molecules, these SRMs provide wavelength references that are very stable under changing environmental conditions.

SRM 2514 can be used to calibrate the wavelength scale of measurement equipment in the 1560 to 1595 nm region. Each unit is a single-mode optical-fiber-coupled absorption cell containing carbon monoxide ($^{12}C^{16}O$) gas at a pressure of 133 kPa (1000 Torr). The absorption path length is 80 cm, and the absorption lines are about 50 pm wide.

SRM 2515 is nearly identical to SRM 2514 except that it contains the $^{13}C^{16}O$ isotopic species of carbon monoxide. This isotopic species has numerous absorption lines at longer wavelengths, ranging from 1595 to 1630 nm .

SRM 2414 and SRM 2515 are each packaged in a small instrument box (approximately 32 cm long x 12.5 cm wide x 9 cm high) with two FC/PC fiber connectors for the input and output of a user-supplied light source.

Technical Contact: Sarah Gilbert
E-mail: sgilbert@boulder.nist.gov

SRM 2017 Multi-Angle White Reflectance Standard

The NIST Physics Laboratory has developed a new SRM to aid the many industries dependent on special-effect or gonioapparent paints to improve the appearance of their products. Examples of special-effect paints include the pearlescent and metal-flake coating found on automobiles, which change color or brightness with viewing angle. The continued appeal of these novel paints to consumers ensures that they will attract an increasingly larger faction of the color pigment market, estimated to be about $3.5 billion dollars in 2005. The value that these coatings add to products is even greater, as yearly approximately $700 billion dollars worth of goods are shipped for which overall appearance is critical to their sale. The successful use of these special-effect paints, particularly in automobile manufacturing, requires repeated measurements of the optical reflectance properties of the coatings as a function of angle to ensure that the paint application is performed correctly, reproducible between manufacturing plants, constant over time, and can be replicated during a repair.

SRM 2017 is an opal-glass white reflectance standard developed in response to industries' demands for multi-angle reflectance standards to calibrate the spectrometer instruments used to measure the optical properties of special-effect paints.

The reflectance of the opal-glass sample has been accurately measured for an illumination angle of 45° and viewing angles of 15°, 25°, 45°, 75°, and 110° for 10 nm wavelength increments between 360 nm and 780 nm in the visible spectrum.

This standard will help ensure that the measured properties of the special-effect paints are independent of instrument manufacturer or locale. The standard was produced using the Optical Technology Division's Spectral Tri-function Automated Reference Reflectometer or STARR.

Technical Contact: Maria Nadal
E-mail: maria.nadal@nist.gov

New SRMs continued…

Relative Intensity Correction Standard for Raman Spectroscopy

SRM 2241 Relative Intensity Correction Standard for Raman Spectroscopy is a certified spectroscopic standard for the correction of the relative intensity of Raman spectra obtained with instruments employing 785 nm laser excitation. SRM 2241 consists of an optical glass that emits a broadband luminescence spectrum when excited with 785 nm laser radiation. The relative spectral intensity of the glass luminescence has been determined through the use of a white-light, uniform-source, integrating sphere that has been calibrated for its irradiance at NIST.

The shape of the luminescence spectrum of this glass is described by a polynomial expression that relates the relative spectral intensity to the wavenumber (cm^{-1}) expressed as the Raman shift from the excitation wavelength of 785 nm. This polynomial, together with a measurement of the luminescence spectrum of the standard, can be used to determine the spectral intensity response correction that is unique to each Raman system. The resulting instrument-intensity response correction may then be used to obtain Raman spectra that are instrument independent.

This SRM is intended for use in measurements over the range of 20 °C to 25 °C and with Raman systems that employ laser excitation at 785 nm. It may also be used for Raman excitation with lasers that range from 784 nm to 786 nm in excitation wavelength.

Technical Contact: Steven Choquette
E-mail: steven.choquette@nist.gov

RENEWAL SRMs

SRM 1575 Pine Needles

NIST announces a new release of SRM 1575a Pine Needles**,** which is intended primarily for use in the evaluation of inorganic analytical techniques used to determine element content of botanical and agricultural materials. The needles were collected in North Carolina from freshly felled loblolly pine (*Pinus taeda*) trees of about the same age and origin. One unit of SRM 1575a consists of approximately 50 g of dried, jet-milled, and sterilized pine needles. Certified and reference concentrations are provided for 23 elements. This material replaces the original SRM 1575 Pine Needles, which has not been available since 1998.

RENEWAL SRMs continued…

SRM 1941b Organics in Marine Sediment

This SRM is widely used by marine pollution monitoring programs for assessing the condition of the marine environment. Most contaminants in aquatic systems, including polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, and organo-tin compounds, are associated with particulate matter and tend to accumulate in sediments. Resuspension of sediment by dredging, storms, and currents often result in desorption of the contaminants into the water column.

This is the third issue of SRM 1941b since 1989. The sediment used for SRM 1941b was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) harbor, very near the sites where SRM 1941 and SRM 1941a were collected. The sediment contains natural levels of contaminants in this area (not spiked). SRM 1941b is intended for use in evaluating analytical methods for the determination of PAHs, PCB congeners, and chlorinated pesticides in sediment. The material is certified for the concentrations of 24 PAHs, 29 PCB congeners, and 7 chlorinated pesticides. There are also reference values for 43 additional PAHs; 13 additional PCB congeners; 2 additional pesticides; mono-, di-, and tributyltin; total tin; and total organic carbon. SRM 1941b has certified and reference values for approximately 50 more organic constituents than in the previous issue of SRM 1941 and is the first SRM with values assigned for the organotins. In addition, there are information values for carbon, hydrogen, and nitrogen content. SRM 1941b complements SRM 1944 (New York/New Jersey Waterway Sediment) with concentrations of the PAHs and PCB congeners approximately an order of magnitude lower in SRM 1941b as compared to SRM 1944. The concentrations in these two materials span the range found in most urban waterways around the world.

Other Renewals Now Available

SRM 3190, 3198, & 3199 - Aqueous Electrolytic Conductivity Standard
SRM 1473b - Polyethylene Resin
SRM 1003c - Glass Spheres (Particle Size)

Revisions - - - - - - Certificate Revisions- Are You Using These Materials?

Below is a list of our most recent certificate revisions. To gain maximum benefit from a NIST SRM, the certificate in possession must be current. NIST updates certificates for a variety of reasons, such as the extension of the certificate date or to include additional information gained from stability testing. If you do not have the most recent certificate for your material, download a copy from the website at: www.nist.gov/srm, or contact SRM at: telephone (301) 975-6776; fax (301) 926-4751; or email srminfo@nist.gov.

SRM 869a Expiration Date Has Been Extended

The expiration date for SRM 869a Column Selectivity Test Mixture for Liquid Chromatography has been extended. This material is now certified until 30 September 2007 (extended from 30 September 2002).

SRM 1951a Lipids in Frozen Human Serum Expiration Date Has Been Extended

The expiration date for SRM 1951a Lipids in Frozen Human Serum has been extended. This material is now certified until 31 October 2005 (extended from 31 October 2002).

REVISIONS continued...

SRM 3164 Uranium Standard Solution

New expiration date: This material is valid until 01 January 2004.

SRM 2627a Nitric Oxide in Nitrogen

New expiration date: This material is valid until 07 January 2004.

SRM 2719 Calcined Petroleum Coke

A note regarding volatile matter was added.

SRM 2383 Baby Food Composite

Reference values for individual fatty acids, total dietary fiber, and vitamin K were added, and expiration date was extended to 31 August 2007.

SRM 1935 Potassium Dichromate Soln/UV Absorbance

New expiration date: This material is valid until 31 August 2005.

SRM 699 Alumina

Si value updated.

SRM 1581 Polychlorinated Biphenyls in Oils

This SRM has been discontinued and will be replaced by new SRMs 3075 to 3080, and SRM 3090. They will be available sometime in May 2003.

NIST and IAEA Collaborations Highlighted

NIST and the International Atomic Energy Agency (IAEA) have a long history of collaboration in the development and certification of reference materials. Over the years NIST and IAEA have shared information, materials, personnel and data resulting in a wide variety of important certified reference materials for isotopic, organic, and inorganic analysis.

Isotopic Analysis

For over 30 years, NIST has worked closely with the Isotope Hydrology Section of the IAEA and the U.S. Geological survey to ensure that a wide range of artifact standards and intercomparison materials are available to the scientific community for the precise measurement of isotopic variations in light elements, such as hydrogen, carbon, nitrogen, oxygen, silicon, and sulfur. These standards and reference materials are critical for performing accurate isotopic analysis of geological, biological, and environmental substances.

Natural and man-made materials show small but significant variations in the isotopic make-up of their constituent atoms and molecules because of the physical and chemical processes that fractionate or change the relative isotopic abundances. These small shifts in the isotopic ratios, typically a few parts per thousand or less, carry important information on parameters such as temperature, mass fluxes, reaction kinetics, bacterial processes, diffusion and filtration processes that gave rise to the materials. Such measurements have impacted diverse fields ranging from medical diagnosis to paleoclimate reconstruction and global atmospheric change.

NIST and the IAEA have overseen the development and distribution of more than 50 standards and materials. Recent activities have gone beyond the distribution of intercomparison standards to the development of samples of gaseous CO_2 that support reconciliation of fundamental algorithms and different data reduction methods important in global climate research. NIST has also participated in several consultative meetings held at the IAEA that have sought to meet the needs of the stable isotope community as applications and instrumentation have evolved.

Inorganic Analysis

In the area of inorganic analysis, NIST and IAEA have recently collaborated in the area of air particulate matter (APM). NIST SRM 2783 Air Particulate on Filter Media has been developed based on collaborative research initiated in the 1990s by the IAEA and extended at NIST. IAEA provided urban dust, collected by the IAEA Laboratories in Seiberdorf. An IAEA collaborative research program demonstrated that the APM can be homogeneously deposited on a filter. NIST coordinated the certification of SRM 2783, working with eight laboratories from around the world, including the US-EPA in Triangle Park NC, resulting in an 2.5 μm nominal size APM on a filter media. This SRM has 27 certified and reference values for evaluating and calibrating analytical methods used for common and toxic elements contained in APM.

Other collaborations in inorganic analysis include SRM 1570a Trace Elements in Spinach Leaves, IAEA 085 and 086 Human Hair, and SRM 4355 Environmental Radioactivity-Peruvian Soil.

Organic Analysis

Organic contaminant measurements have also been studied jointly by NIST and IAEA. Since 1986, NIST has participated in eight IAEA interlaboratory comparison exercises, providing measurements to assist in the determination of the certified values for organo-chlorine contaminants and petroleum hydrocarbons in marine samples such as sediment, fish tissue, mussel tissue, and plant material. NIST, the IAEA Marine Environmental Laboratory in Monaco, and the National Research Council of Canada collaborated to prepare and value assign organic contaminants in two NIST mussel tissue SRMs: SRM 2977 Mussel Tissue (Organic Contaminants and Trace Elements) and SRM 2978 Mussel Tissue (Organic Contaminants Raritan Bay, New Jersey). The IAEA Monaco Laboratory has also assisted in the certification of the first NIST SRMs with values for methylmercury, SRM 1974a Organics in Mussel Tissue (*Mytilus edulis*) and SRM 2974 Organics in Freeze-Dried Mussel Tissue.

As two of the leaders in measurement science, the natural partnership between the IAEA and NIST is resulting in increased analytical capability for laboratories throughout the world. The future promises new opportunities for collaborations and advancements to chemical measurement science.

PHONE 301-975-6776 FAX 301-948-3730 E-MAIL SRMINFO@NIST.GOV

Group News

John R. Rumble, Jr. has been selected to be the new Chief of the Measurement Services Division at the National Institute of Standards & Technology (NIST). John is currently the Leader of the Standard Reference Data Group, the Acting Leader of the Calibration Group and the Acting Leader of the Standard Reference Materials Group.

John will be bringing many capabilities to his new position. He received a Ph.D. (1976) in chemical physics from Indiana University. Prior to joining NIST in 1980, he was at the Joint Institute for Laboratory Astrophysics in Boulder, Colorado, and the International Atomic Energy Agency in Vienna, Austria. He has also worked as a chemist in industry. In 1993-1994, he was a Department of Commerce Fellow working in the Office of Science and Technology Policy in the Executive Office of the President. John has published extensively in atomic and molecular physics and scientific informatics, including several books. He has been active in developing scientific database standards, including an international standard for industrial data exchange. He is a Fellow of the American Society for Testing and Materials, a Fellow of ASM International, a member of the Russian Federation Academy of Metrology, a Fellow of the International Union of Pure and Applied Chemistry, and recipient of the U.S. Department of Commerce Silver Medal. In 1998, John was elected President of the Committee on Data (CODATA) of the International Council of Scientific Unions.

John is excited about the challenges of leading the SRM group into the 21st century. He feels he has an excellent staff that will assist him to accomplish several ambitious goals including: development of a national measurement strategy and moving even more supporting measurement documentation to the web to help customers.

More Group News……

Jennifer Colbert, Project Manager for NIST Standard Reference Materials Group (SRMG) retired on June 28, 2002, after 29 years of dedicated NIST service. She began her career as a chemist in the Methods Development Laboratory at Miles Laboratories in Elkhart, Indiana. Her NIST tenure began in 1973 as a research chemist. She conducted experimental oxygen bomb calorimetric and microcalorimetric measurements on pure chemical systems, living systems, biographical compounds, and fuels to determine their heats of combustion. She also performed measurements on a series of coals that were certified for enthalpy of combustion and was instrumental in the development of a synthetic refuse derived fuel Standard Reference Material. She was awarded the Department of Commerce Bronze Metal in 1986.

In 1989, Jennifer became a Project Coordinator for SRMG. Jennifer managed the SRM documentation processes and also coordinated the complex certification process for standards in such diverse technical categories as clinical, biotechnology, food and agriculture, primary chemicals, spectrometric solutions, gas mixtures, ion activity, thermodynamic properties, polymeric properties, and optical properties. Jennifer jointly received the R&D 100 award in 1993 for the development of SRM 2390 DNA Profiling Standard. Among her high-impact accomplishments includes the management of the certification process of standard reference materials for health care including definitive methods for 12 health status markers including calcium for blood clotting, chloride for kidney function, cholesterol for heart disease and glucose for diabetes. Jennifer's dedication and perseverance in management of the certification process has resulted in many SRMs being released and impacting the health, agriculture, transportation, fuel, and transportation industries.

Jennifer served on the NIST Washington Editorial Review Board (WERB) for a number of years and organized the Infant Formula Workshop held at NIST. She served on the planning committee for the Workshop on Measurement Traceability for Clinical Laboratory Testing and In-Vitro Diagnostic Test Systems. She also was on the steering committee and organized the Workshop on Biomedical Devices and Materials. Jennifer's technical affiliations include the American Society for Testing and Materials (ASTM), American Association for Clinical Chemistry (AACC), AOAC International, and the National Committee for Clinical Laboratory Standards, (NCCLS).

The SRM Group wishes Jennifer Colbert happiness and much success in the future.

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