SRM SPOTLIGHT

June 2005

Editor: Regina R. Montgomery

Inside:

New NIST RMs/SRMs

NIST SRM 1955 Homocysteine and Folate in Human Serum

Homocysteine is an amino acid found at low concentrations in blood. It is not normally found in the sequence of proteins but it is an intermediate in the conversion of methionine to cysteine, the two common sulfur containing amino acids. In recent years considerable attention has focused on the levels of homocysteine in blood. There have been numerous studies that have reported a strong correlation between elevated homocysteine and risk of developing cardiovascular diseases such as heart disease and stroke. Because of these studies, assays for homocysteine are finding increased use in clinical laboratories for diagnosis. Folic acid is an essential nutrient found in leafy vegetables and is a common supplement found in bread. Studies have found that folates, the metabolites of folic acid, can reduce homocysteine levels in blood, and perhaps reduce the risk of diseases associated with elevated homocysteine levels. In addition dietary supplements of folic acid for pregnant women are known to reduce the risk of certain types of birth defects. Measurement of serum folic acid levels is an important part of the evaluation of nutritional status.

NIST, in collaboration with the Centers for Disease Control and Prevention (CDC) has developed a new human serum-based SRM for measurements of homocysteine and folate. A unit of the SRM consists of three vials of frozen human serum with certified levels of homocysteine and 5-methyltetrahydrofolic acid (5MT), the most common form of folate found in human blood, that cover the range normally found in the U.S. population. The concentrations of homocysteine were determined at NIST using multiple methods based on isotope dilution combined with gas chromatography/mass spectrometry (GC/MS), liquid chromatography/mass spectrometry (LC/MS), or liquid chromatography/tandem mass spectrometry (LC/MS/MS). The concentrations of 5MT were determined at NIST and CDC using methods based upon isotope dilution LC/MS/MS. These methods were also used to determine the low levels of folic acid present in the SRM. In addition, CDC provided results for total folate, 5-formaltetrahydrofolic acid, and vitamin B_{12}.

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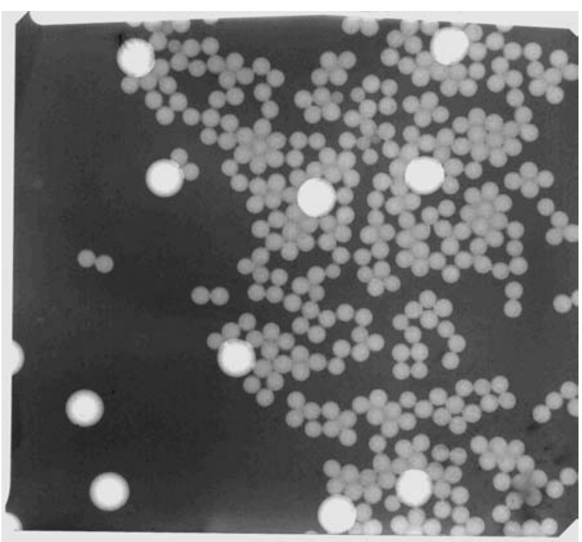
NIST SRM 1963a and 1964 Particle Diameter Standards

These SRMs consist of nominal 100 nm diameter polystyrene spheres (SRM 1963a) and of nominal 60 nm diameter polystyrene spheres (SRM 1964) in 5 mL of deionized filtered water. The spheres have a mass fraction of 0.5 % and are supplied in a dispensing vial. The SRMs are certified for the modal diameter, the peak in the number size distribution. The estimated expanded relative uncertainty (95 % confidence level) in the model size is about 1.0 %. In both cases the size distributions are narrowly distributed. Information is provided for the number mean diameter, volume mean diameter, light scattering mean diameter, and dynamic light scattering Z average mean diameter. The size distributions are tabulated and the standard deviations of the distribution provided. The size distribution was measured using differential mobility analysis. Dynamic light scattering was used to verify that there was no appreciable amount of agglomeration of the primary spheres. SRM 1963a replaces SRM 1963 because of the extensive agglomeration in many of the vials of SRM 1963.

The major application of these SRMs is in the production of deposition standards for surface scanning inspection systems used by the semiconductor industry and in the calibration of differential mobility analyzers used in making these depositions. The 60 nm SRM is near the minimum detectable size for surface scanning inspection systems. Other applications for these standards include the magnification calibration of transmission electron microscopes, the validation of the performance of dynamic light scattering instruments, and primary standards for basic aerosol and colloidal research. A transmission electron micrograph of the 100 nm spheres along with a few 205 nm spheres is below.



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NIST SRM 2037 Solvent Red 24 Diesel Fuel Dye

In the United States diesel fuel is subject to a variety of taxes depending on how it is consumed. If employed in vehicles operating on the nation's highways, it is subject to the full fuel-tax levy. However, diesel fuel that is either used in off-road vehicles, such as bulldozers and farm equipment, or that is burned as heating oil is exempt from the "road tax" portion of the assessment. To differentiate diesel fuel destined for on-road use from that to be used in off-road applications, the Code of Federal Regulations (CFR) prescribes that the latter be dyed with a red dye mixture--Solvent Red 164. Once this dye is mixed with the fuel at the distribution terminals, tax-exempt fuel must be handled separately and kept apart from "on-road" fuel. Getting caught with red diesel fuel in the tank of your Mercedes can be a very costly proposition.

To monitor the concentration of the red dye in tax-exempt fuel at the distribution terminals, fuel companies measure the absorbance of the dyed fuel using visible spectrophotometry. Actually, second-derivative optical spectrometry is employed to overcome problems caused by the variable color of diesel fuel, which depending on its source, can range from water clear to nearly black. SRM 2037 is a highly purified version of a red dye known by a variety of names such as Solvent Red 24, Sudan IV, or Scarlet Red that is made by diazo-coupling of two molecules of o-toluidine with one ß-naphthol (see Figure 1). Solutions of SRM 2037 can be used to calibrate the spectrophotometers used in the terminals to determine the amount of red dye added to the diesel fuel. SRM 2037 is used as the standard because it is a pure material whose amount can be accurately determined from its mass. The actual dye, Solvent Red 164, is a complex mixture made from less pure starting materials, which is much more easily dissolved in diesel fuel than Solvent Red 24.

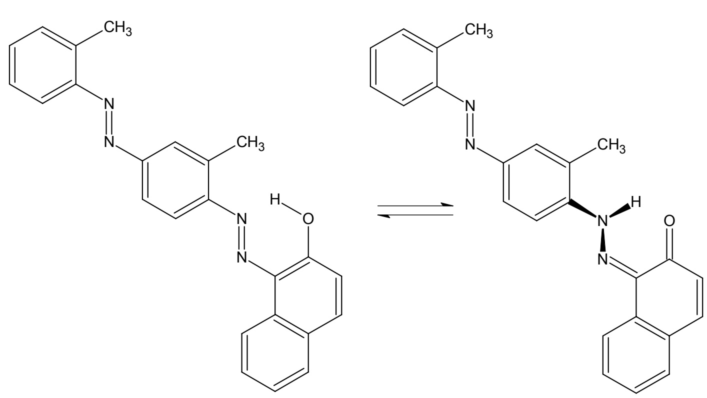

Figure 1. Tautomeric Structures of Solvent Red 24 Dye

NIST SRM 2037 Solvent Red 24 Diesel Fuel Dye (continued)

An ASTM procedure (D6258-98) and the current CFR section [26 CFR Part 48, Section 4081, Subsection 48-4082-1(b)] specify a very similar red dye (Solvent Red 26) as the standard for diesel fuel dye. Solvent Red 26 is structurally the same as Solvent Red 24 except it contains one additional methyl group on the middle ring. Solvent Red 26 was not used for SRM 2037 because it could not be obtained in sufficient quantities with high purity. The visible absorbance spectra of Solvent Red 24 and Solvent Red 26 are virtually identical, but converting the pertinent second derivative absorbance value $\Delta D2$ between Solvent Red 24 (SRM 2037) and Solvent Red 26 requires a conversion factor, C:

$$\Delta D2_{SR26} = C\Delta\, D2_{SR24} \text{ where } C = 0.54 \text{ +/- } 0.04.$$

C is calculated with the concentration of both solutions in milligrams per liter (or grams per liter), which are the concentration units used in standard methods for diesel fuel, such as ASTM D-6258-98. The solvent is kerosene:p-xylene (97:3 by volume). The expanded uncertainty for C above is given at the 95 % confidence level (k=2)[1]. C can also be used to convert the "Second-Derivative-Based Absorption Coefficient" for Solvent Red 24 given in liters per gram-centimeter in the certificate to the corresponding value for Solvent Red 26.

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1 ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at **http://physics.nist.gov/Pubs/**

NIST SRMs 2452- 2454 Hydrogen in Titanium Alloy

Since hydrogen is one of the chief contributors to brittleness in metals, its control in manufacturing processes is crucial. Rapid measurement methods used in industry are calibrated with working standard materials. NIST is producing a new series of titanium alloy SRMs certified for hydrogen concentrations. The process exploits the reversibility of the reaction
$Ti + H_2 = TiH_2$, in which the equilibrium lies far to the right (with hydrogen tightly bound as hydride) at room temperature and far to the left (with hydrogen as gas) at high temperature. Reaction is rapid at 500°C. These SRMs are produced by degassing a widely used titanium alloy (containing 6 % aluminum and 4 % vanadium) at 700 °C in a high vacuum, then adding a measured quantity of hydrogen gas to the system. The hydrogen content of the degassed metal and the final products are further characterized by prompt-gamma activation analysis (PGAA) at the NIST Center for Neutron Research. SRMs 2452, 2453, and 2454, with nominal mass fractions of 50, 100, and 200 mg/kg hydrogen, have been prepared by this method and are now available for purchase. This series will be available to check the linearity of instrument calibration, and provide check samples at levels significantly above and below the critical level of about 100 mg/kg. Selection of the concentrations was made in consultation with the ASTM E-01 task group.

The aerospace industry and, to an increasing extent the automotive and consumer goods industries, employ titanium alloys for their excellent combination of high strength, light weight, and good high-temperature properties. These SRMs will help ensure that fabricated titanium components in fact have these desired properties.

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NIST SRM 2585 Organics in House Dust

House dust is a repository of pesticides, flame retardants, and other chemicals used indoors and tracked in from outdoors. Pesticides become associated with house dust primarily through interior use of pest control formulations, intrusion of vapors from foundation and crawl space treatments, and track-in of lawn and garden chemicals. Polycyclic aromatic hydrocarbons (PAHs) are derived from indoor sources such as combustion, cooking, and smoking, as well as track-in of contaminated yard soil or residues from garage floors. Once indoors where they are protected from environmental degradation, pollutants associated with dust persist for long periods, particularly if the dust is embedded in carpets.

NIST has produced two house dust SRMs for lead and other trace elements (SRM 2583 Trace Elements in Indoor Dust, Nominal 90 mg/kg Lead and SRM 2584 Trace Elements in Indoor Dust, Nominal 1 % Lead). SRM 2585 Organics in House Dust is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and brominated diphenyl ether (BDE) congeners in house dust and similar matrices. The table below summarizes the number of compounds characterized in SRM 2585.

	Certified Values	Reference Values	Information Values
PAHs	34	29	
PCB Congeners	30	13	
Pesticides	4	9	
BDE Congeners	15	3	9

The concentrations (all on a dry mass fraction basis) of the individual PAHs range from approximately 20 ng/g to 4500 ng/g; those for the PCB congeners range from approximately 2 ng/g to 40 ng/g; those for the pesticides range from approximately 4 ng/g to 300 ng/g; and those for the BDE congeners range from approximately 4 ng/g to 2500 ng/g. This is one of the first matrix reference materials available with values assigned for the BDEs.

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NIST SRM 2770 Sulfur in Diesel Fuel Oil
NIST SRM 1616b Sulfur in Kerosene

New Sulfur in Fossil Fuel SRMs Support Introduction of Ultra Low Sulfur Diesel (ULSD)

NIST has developed new low level sulfur fossil fuel SRMs in support of the U.S. Environmental Protection Agency's 15 mg/kg cap on sulfur in transportation diesel fuel, which takes effect beginning June 1, 2006. EPAs highway heavy-duty and 2010 Tier 4 nonroad diesel regulations when fully implemented will provide roughly $150 billion annually in health and welfare benefits to the American public.

In preparation for the introduction of ULSD, the U.S. EPA is undertaking a test program this summer to determine the reproducibility among more than 100 laboratories of sulfur test methods and the readiness of industry to measure Ultra Low Sulfur Diesel (ULSD) fuel. Due to the low levels involved and the need for laboratories to measure within tight tolerances, NIST certified natural matrix reference material are being used as calibration and accuracy check standards to help insure the success of the test program. The certified sulfur content for NIST fossil fuel SRMs is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS), one of NIST's most accurate methods.

Recently Developed Low Sulfur Fossil Fuel SRMs

SRM	Description	Unit Size	S mg/kg
2298	Sulfur in Gasoline (High-Octane)	5 x 20 mL	4.7
2299	Sulfur in Gasoline (Reformulated)	5 x 20 mL	13.6
1616b	Sulfur in Kerosene	100 mL	8.41
1617a	Sulfur in Kerosene	100 mL	1730.7
1624d	Sulfur in Diesel Fuel Oil	100 mL	3882
2723a	Sulfur in Diesel Fuel Oil	10 x 10 mL	11.0
2724b	Sulfur in Diesel Fuel Oil	10 x 10 mL	426.5
2770	Sulfur in Diesel Fuel Oil	10 x 10 mL	41.57
In Prep	Sulfur in Diesel Fuel Blend Stock	100 mL	< 0.1

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Renewals

NIST announces the availability of SRM 114q Portland Cement Fineness Standard. This SRM, which has been provided by NIST since 1934, is used to calibrate cement fineness testing equipment according to ASTM Standard Methods. The details of the development of SRM 114q, which was prepared at NIST with the collaboration of numerous laboratories, will be detailed in a publication available later this year at NIST [Ferraris, C.,F. Avilés A.I., Guthrie W., Haupt, R., "Certification of SRM 114q; Phase I", SP260-161, 2005].

The surface area of SRM 114q is certified for Blaine air permeability (ASTM C 115-96a (2003)) and the Wagner turbidimeter (ASTM C 204-00) methods. The 45 µm residue (ASTM C 430-96 (2003)) is also certified. In the next phase of this project, the particle size distribution of this material based on laser diffraction methods will be provided.

The SRM 114q can be purchased for $ 155.00 per set of 20 vials (each vial contains 5 g).

Other SRM Renewals…

SRM 2806a Medium Test Dust (MTD) in Hydraulic Fluid

SRM 1491a Methyl-Substituted Polycyclic Aromatic Hydrocarbons in Toluene

SRM 1d Limestone, Argillaceous

SRM 929a Magnesium Gluconate

SRM 1624d Sulfur in Diesel Fuel Oil

SRM 1616b Sulfur in Kerosene- New low level (see page 6)

SRM 2519a High Resolution Wavelength Calibration Reference for 1530 nm – 1565 nm Hydrogen Cyanide $H^{13}C^{14}N$

Revisions

Certificate Revisions—Are you Using These Materials?

This is a list of our most recent certificate revisions. Users of NIST SRMs should ensure that they have the most recent certificates. NIST updates certificates for a variety of reasons, such as to extend the expiration date or to include additional information gained from stability testing. If you do not have the most recent certificate for your material, you can print or view a copy from the website at: http://www.nist.gov/srm, or contact SRM at:
Phone: (301) 975-6776
Fax: (301) 926-4751
Email: srminfo@nist.gov.

SRM 3127a Lanthanum Standard Solution
Lot # 890402
New Expiration Date:
01 April 2006

SRM 3132 Manganese Standard Solution
Lot # 890903
New Expiration Date:
01 April 2006

SRM 3156 Tellurium Standard Solution
Lot # 892901
New Expiration Date:
01 April 2006

SRM 3167a Yttrium Standard Solution
Lot # 790412
New Expiration Date:
01 January 2006

RM 8640 Microspheres With Immobilized Fluorescein Isothiocyanate
New Expiration Date:
31 January 2006

SRM 3113 Cobalt Standard Solution
Lot # 000630
New Expiration Date:
11 May 2009

SRM 3129a Lithium Standard Solution
Lot # 000505
New Expiration Date:
13 September 2008

SRM 3140 Platinum Standard Solution
Lot # 000615
New Expiration Date:
27 July 2009

SRM 3186 Phosphate Anion Standard Solution
Lot # 000330
New Expiration Date:
30 March 2009

SRM 3185 Nitrate Anion Solution
Lot # 991508
New Expiration Date:
30 March 2009

SRM 3181 Sulfate Anion Standard Solution
Lot # 000630
New Expiration Date:
30 March 2009

Revisions continued…

RM 8535 VSMOW Vienna Standard Mean Ocean Water
New Expiration Date: Indefinitely

RM 8536 GISP Greenland Ice Sheet Precipitation (Water)
New Expiration Date: Indefinitely

RM 8537 SLAP Standard Light Antarctic Precipitation (Water)
New Expiration Date: Indefinitely

RM 8562 CO_2 Heavy Paleomarine Origin (Carbon Dioxide)
Changes in Reference Values and Uncertainties
New Expiration Date: 31 December 2006

RM 8564 CO_2 Biogenic, Modern Biomass Origin (Carbon Dioxide)
Changes in Reference Values and Uncertainties
New Expiration Date: 31 December 2006

SRM 3199 Aqueous Electrolytic Conductivity Lot # 043001
Change in the Certified Value and Uncertainty
New Expiration Date: 30 January 2006

SRM 1482a Polyethylene
Editorial Changes
New Expiration Date: 22 November 2012

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In addition, we are in the midst of a project to add numerous certificate references for each SRM online. Please also note we are also adding numerous historical archive certificates online for your convenience.

https://srmors.nist.gov

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Email: srminfo@nist.gov. These CDs are helpful to SRM users who do not have access to our online catalog on the Internet.

NIST SRM 2005 Exhibit Schedule

BIO2005 PPCA (BIO2005)
June 19 - 22, 2005
Philadelphia, PA

Institute Food Technologists (IFT) 2005 Food Expo
July 17 - 19, 2005
New Orleans, LA

American Association of Clinical Chemists (AACC)
July 26 –28, 2005
Orlando, FL

NCSL Symposium (NCSL)
August 7 - 10, 2005
Washington, DC

American Chemical Society (ACS)
August 29 - 31, 2005
Washington, DC

Association of Analytical Communities (AOAC)
September 11-15, 2005
Orlando, FL

Materials Science & Technology (MS&T 05)
September 25 - 28, 2005
Pittsburgh, PA

CHEM Show (CHEM)
November 1 - 3, 2005
New York, NY

Eastern Analytical Symposium (EAS)
November 14 - 17, 2005
Somerset, NJ