

December 2008

Editor: Regina R. Montgomery

New NIST RMs/SRMs

NIST 2277 Organic Acids in Methanol: Methylene Chloride NIST 2278 Deuterated Organic Acids in Methanol: Methylene Chloride

Organic Acid Calibration Solutions Developed in Collaboration with U.S. Environmental Protection Agency (EPA)

A working group of investigators is characterizing and quantifying the organic compounds in particulate matter (PM) as part of the U.S. EPA's PM 2.5 research program and related studies. The group has included organic acids as one of the priority groups of compounds for preparation of new solution SRMs. To address this need, NIST prepared two solution SRMs: SRM 2277 and SRM 2278. SRM 2277 Organic Acids in Methanol: Methylene Chloride is a solution of 24 organic acids. SRM 2278 Deuterated Organic Acids in Methanol: Methylene Chloride is a solution of seven deuterated organic acids. The deuterated acids in SRM 2278 include benzoic acid, decanoic acid, hexanoic acid, myristic acid, palmitic acid, phthalic acid, and succinic acid. These solutions will be useful for validating the chromatographic separation step (retention times and detector response), spiking samples, studying analyte recoveries, and determining response factors for the specific analytes.

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The identification of any commercial product or trade name does not imply endorsement or recommendation by the National Institute of Standards and Technology.

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NIST Standard Reference Material 1546 Meat Homogenate



Standard Reference Material (SRM) 1546 Meat Homogenate was released in 1999 with certified values assigned for fatty acids, cholesterol, and three elements. Reference values were assigned for other elements, several vitamins, and proximates. In 2008, the Certificate of Analysis was updated to include certified values for calcium, magnesium, phosphorus, potassium, sodium, and zinc. Reference values for amino acids are now provided for this material.

This SRM is intended primarily for validation of methods for determining fatty acids, cholesterol, proximates, calories, vitamins, and elements in canned meat products and similar materials. This SRM can also be used for quality assurance when assigning values to in-house reference materials. The meat homogenate is a mixture of pork and chicken products blended together in a commercial process.

A unit of SRM 1546 consists of four cans, each containing approximately 85 g of material. This material was originally prepared at the request of the U.S. Department of Agriculture's (USDA's) Food Safety Inspection Service. Amino acid values have been added at the request of USDA's Nutrient Data Laboratory to underpin the quality of the amino acid values in their National Nutrient Database for Standard Reference.

Updated Certificate of Analysis for SRM 1939a Polychlorinated Biphenyls (Congeners) in River Sediment A

SRM 1939a Polychlorinated Biphenyls (Congeners) in River Sediment A is an air-dried river sediment collected from a site where transformer oils containing polychlorinated biphenyl (PCB) mixtures had been spilled. Each unit of SRM 1939a consists of one bottle containing approximately 50 g of sediment.

SRM 1939a was originally released in 1998 with certified concentration values provided for 20 PCB congeners and reference concentration values for 4 additional PCB congeners. The Certificate of Analysis for SRM 1939a has been updated recently to include certified concentration values for 34 PCB congeners and reference concentration values for 30 others plus a reference concentration value for total PCBs. This is the first sediment SRM that has a concentration value for total PCBs included. Several methods for quantifying total PCBs were used, and the uncertainty in the reference concentration value reflects the differences seen among the methods. Many



environmental monitoring programs use total PCB concentration for assessing clean-up efforts so this additional information on SRM 1939a will make it more useful as a control material for those analyses.

Updated Certificate of Analysis for SRM 2977 Mussel Tissue (Organic Contaminants and Trace Elements)

SRM 2977 Mussel Tissue (Organic Contaminants and Trace Elements) was first released in 2000 with certified concentration values for 14 polycyclic aromatic hydrocarbons (PAHs), 25 polychlorinated biphenyl (PCB) congeners, 7 chlorinated pesticides, 6 trace elements, and methylmercury.

This SRM consists of one bottle containing approximately 10 g of freeze-dried mussel tissue. Since the original release of SRM 2977, interest in monitoring brominated flame retardants, particularly polybrominated diphenyl ethers (PBDEs), has increased. PBDE concentrations are present at relatively low concentration levels in mussel tissue, i.e., <1 ng/g dry mass to 40 ng/g dry mass in SRM 2977. The Certificate of Analysis for SRM 2977 was recently updated to include certified concentration values for five PBDE congeners and reference concentration values for five others. This represents the first mussel tissue SRM with concentration values included for the PBDE congeners.



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Gravimetric Approach to the Standard Addition Method in Instrumental Analysis. 1.

W. Robert Kelly, Bruce S. MacDonald, and William F. Guthrie

A mathematical formulation for a gravimetric approach to the univariate standard addition method (SAM) is presented that has general applicability for both liquids and solids. Using gravimetry rather than volumetry reduces the preparation time, increases design flexibility, and makes increased accuracy possible. SAM has most often been used with analytes in aqueous solutions that are aspirated into flames or plasmas and determined by absorption, emission, or mass spectrometric techniques. The formulation presented here shows that the method can also be applied to complex matrixes, such as distillate and residual fuel oils, using techniques such as X-ray



fluorescence (XRF) or combustion combined with atomic fluorescence or absorption. These techniques, which can be subject to matrix-induced interferences, could realize the same benefits that have been demonstrated for dilute aqueous solutions

Determination of Sulfur in Biodiesel and Petroleum Diesel by X-ray Fluorescence (XRF) Using the Gravimetric Standard Addition Method–II Lydia R. Barker, W. Robert Kelly, and William F. Guthrie

ULTRA-LOW SULFUR HIGHWAY DIESEL FUEL (15 ppm Sulfur Maximum) Required for use in all model year 2007 and later highway diesel vehicles and engines. Recommended for use in all diesel vehicles and engines. Sulfur in petroleum diesel is typically detected by wavelength dispersive X-ray fluorescence (XRF) spectrometry by comparing the response of the unknown to a linear calibration curve composed of a series of matrix-

identical standards. Because biodiesel contains about 11% oxygen by mass and diesel is oxygen-free, the determination of sulfur in biodiesel using petroleum diesel



calibrants is predicted to be biased $\approx -16\%$ due to oxygen absorptive attenuation of the X-ray signal. A gravimetric standard addition method (SAM) was hypothesized to overcome this bias because it should be matrix-independent. Samples of both petroleum diesel (SRM 2723a and European Reference Material EF674a) and biodiesel (candidate SRM 2773, NREL 52537, and NREL 52533) were analyzed, comparing the traditional calibration curve method to the gravimetric SAM approach. As expected, no significant difference was found between the two methods when measuring sulfur in petroleum diesel. Sulfur determinations in biodiesel with petroleum diesel calibrants were lower by $\approx 19\%$ relative to the gravimetric SAM at the 3, 7, and 12 µg/g levels. It is concluded that XRF using gravimetric SAM approach is insensitive to differences in the C/H ratio.

Renewals

- SRM 17f Sucrose Optical Rotation
- SRM 1598a Inorganic Constituents in Animal Serum
- SRM 1935a Potassium Dichromate Solution/UV Absorbance Standard
- SRM 2036 Near Infrared Transmission Wavelength
- SRM 2092 Low-Energy Charpy
- SRM 2096 High-Energy Charpy
- SRM 2098 Super-High Charpy
- SRM 2627a Nitride Oxide in Nitrogen Lot #48-H-XX
- SRM 2637a Carbon Monoxide in Nitrogen Lot #56-F-XX
- SRM 2910a Calcium Hydroxyapatite
- SRM 3169 Zirconium Standard Solution Lot #071226
- SRM 1877 Beryllium Oxide Powder



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

SRM 46h Portland Cement Fineness Standard Changed ASTM Standard from 3.4 to 4.3

SRM 114q Portland Cement Fineness Standard Editorial Changes

SRM 965a Glucose in Frozen Human Serum New Expiration Date: 31 December 2009

SRM 1082 Cigarette Ignition Strength Standard Editorial Changes

SRM 1261a LA Steel (AISI 4340) Editorial Changes

Revisions (continued)

SRM 1544 Fatty Acids and Cholesterol in a Frozen Diet Composite New Expiration Date: 30 April 2015

SRM 1546 Meat Homogenate

New Expiration Date: 30 April 2014 ; Editorial Changes

SRM 1570a Trace Elements in Spinach Leaves New Expiration Date: 31 August 2013 ; Editorial Changes

SRM 1619b Sulfur in Residual Fuel Oil (0,7 %) Editorial Changes

SRM 1641d Mercury in Water New Expiration Date: 01 October 2014; Editorial Changes

SRM 1677c Carbon Monoxide in Nitrogen 10 umol/mol New Expiration Date: 29 February 2016

SRM 1736 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1737 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1738 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1739 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1740 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1741 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1742 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 1877 Beryllium Oxide Powder Editorial Changes

SRM 1939a PCBs in River Sediment A Editorial Changes

Revisions (continued)

SRM 1980 Positive Electrophoretic Mobility

New Expiration Date: 01 September 2012; Editorial Changes

SRM 2035 Near Infrared Transmission Wavelength

New Expiration Date: 31 December 2018

SRM 2065 Ultraviolet-Visible-Near-Infrared Transmission Wavelength/Vacuum Wavenumber Standard

New Expiration Date: 31 December 2018

SRM 2139 Zinc-Aluminum Alloy Removal of Expiration Date; Editorial Changes

SRM 2241 Relative Intensity Correction Standard for Raman Spectroscopy : 785 nm Excitation New Expiration Date: 31 July 2013

SRM 2242 Relative Intensity Correction Standard for Raman Spectroscopy : 532 nm Excitation New Expiration Date: 01 January 2014

SRM 2391b PCR-based DNA Profiling Standard New Expiration Date: 31 December 2013

SRM 2395 Human Y-Chromosome DNA Profiling Standard New Expiration Date: 31 December 2013

SRM 2586 Trace Elements in Soil Containing Lead from Paint New Expiration Date : 30 September 2013 ; Editorial Changes

SRM 2587 Trace Elements in Soil Containing Lead from Paint New Expiration Date : 30 September 2013 ; Editorial Changes

SRM 2720 Sulfur in Di-*n***-Butyl Sulfide** Editorial Changes

SRM 2890 Water Saturated 1-Octanol New Expiration Date: 30 June 2015; Editorial Changes

SRM 2977 Mussel Tissue (Organic Contaminants and Trace Elements) New Expiration Date: 31 December 2017; Editorial Changes

Revisions (continued)

SRM 3122 Hafnium Standard Solution Lot #000406 New Expiration Date : 13 September 2009

SRM 4401H Iodine-131 Solution Editorial Changes

SRM 4401L Iodine-131 Solution Editorial Changes

SRM 4361C Hydrogen-3 Radioactivity Standard Editorial Changes ; Expiration Date Extended

SRM 4412H Molybdenum Solution Editorial Revisions

SRM 4412L Molybdenum Solution Editorial Revisions

SRM 4926E Hydrogen-3 Radioactivity Standard Expiration Date Revised

SRM 4927F Hydrogen-3 Radioactivity Standard Editorial Changes ; Expiration Date Extended

SRM 5001 Two-Dimensional Grid Photomask Standard Added an Expiration Date : 01 November 2017

RM 8456 Ultra High Molecular Weight Polyethylene

New Expiration Date : 01 January 2012 ; Editorial Changes

RM 8457 Ultra High Molecular Weight Polyethylene

New Expiration Date : 01 January 2012 ; Editorial Changes

ORDER NIST SRMS ONLINE

You can now order NIST SRMs through our new online ordering system, which is constantly being 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 picks up keywords on the detail page along with the words in the title of each SRM.

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

https://srmors.nist.gov

Please Register Your Certificate Online!

Users of NIST SRMs should ensure that they have the most recent certificates.

http://www.nist.gov/srd/srmregform.htm

COMING SOON: SRM 2009 CATALOG/CD/Price List

NIST SRM 2009 Exhibit Schedule



American Academy for Forensic Science (AAFS) February 16-21, 2009 Colorado Convention Center Denver, CO

Pittsburgh Convention PITTCON *March 8 -13, 2009* McCormick Convention Center Chicago, IL

American Chemistry Society ACS March 22-26, 2009 Salt Palace Convention Center Salt Lake City, UT

Materials Research Society Spring Meeting MRS

April 13-17, 2009 Moscone West San Francisco, CA **NOBCChE** *April 13-18, 2009* Renaissance Grand Hotel St. Louis, MO

IFT – FOOD EXPO June 6-10, 2009 Anaheim Convention Center Anaheim, CA

AACC Clinical Lab Expo July 19-23, 2009 McCormick Convention Center Chicago, IL

NCSL Symposium July 26-30, 2009 San Antonio Convention Center San Antonio, TX

American Chemistry Society ACS

August 16-20, 2009 Washington DC Convention Center Washington, DC

Association of Official

Chemists AOAC September 13-16, 2009 Philadelphia Marriott Downtown Philadelphia, PA MS&T Show October 25-29, 2009 David L. Lawrence Convention Center Pittsburgh, PA

Chem Show *November 17-19, 2009* Jacob Javits Convention Ctr New York City, NY

Materials Research Society Fall Meeting MRS November 30 – December 4, 2009 Hynes Convention Center Boston, MA



Other NIST Measurement Services Websites of Interest



Standard Reference Materials – Historical Archived Certificates/Reports of Investigation http://ts.nist.gov/MeasurementServices/ReferenceMaterials/archived_certificates.cfm



NIST Scientific and Technical Databases http://www.nist.gov/srd

NIST Data Gateway http://www.srdata.nist.gov/gateway



Calibrations Services http://www.nist.gov/calibrations