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Editor: Regina R. Montgomery

# **New NIST SRMs**

NIST SRM 2385: Certified Slurried Spinach



The National Institute of Standards and Technology (NIST) has certified the exact contents of Popeye's favorite food. Standard Reference Material (SRM) 2385 consists of small jars of slurried spinach, pure spinach that's been blanched, pureed and passed through filter screens. The concentrations of vitamins and other constituents have been measured and certified, so that the food industry can use the SRM to validate analytical methods and provide accurate nutritional information for its products. An analytical method is evaluated by using it to measure constituents in the SRM and then comparing the results to the NIST-certified values.

The NIST values confirm that spinach is rich in antioxidants both beta-carotene and lutein. Although the actual amounts look small (the antioxidants constitute 0.0019 percent and 0.0033 percent of the spinach by mass, respectively), spinach contains far more of the two combined than most other fruits or vegetables.

Antioxidants help fight formation of free radicals, the highly reactive molecules that can damage DNA and are implicated in the development of certain diseases. Beta-carotene converts to vitamin A in the body and is needed for healthy vision, skin and hair. Lutein is a pigment found in the retina and may help guard against eye diseases such as age-related macular degeneration. Among its other attributes, spinach also contains 1.55 percent dietary fiber by weight.

The new SRM was developed at the request of the food industry with the help of more than 10 food manufacturers.

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

## NIST SRM 2385: Certified Slurried Spinach

NIST now supplies 37 different food SRMs to the industry, one or more for each of the nine sectors of the Association of Analytical Communities' food triangle, which categorizes food based on its fat, carbohydrate or protein content. The food triangle helps to assure the availability of validated analytical methods for all types of foods.

The Nutrition Labeling and Education Act of 1990 requires that information for selected nutrients is provided on labels for processed foods. In response, NIST has been working to provide food-matrix SRMs with values assigned for the required nutrients. SRM 2385 Slurried Spinach is the most recent SRM in this series. SRM 2385 is intended for use as a primary control material for assigning values to in-house control materials and to validate methods for measuring nutrients such as protein, calcium, iron, calories, total dietary fiber, and carotenoids.

To study the robustness of analytical methods, AOAC International developed a ninesector triangle in which foods are positioned based on their fat, protein, and carbohydrate content. The idea was that one or two foods within each sector should be representative of other foods within that sector when validating an analytical method. Similarly, one or two food-matrix reference materials in each sector can be used as control materials for other foods within that sector. SRM 2385 occupies sector 7 of this triangle, along with SRM 1566a Oyster Tissue and SRM 1570a Trace Elements in Spinach.

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## NIST SRM 2285 Arson Test Mixture in Methylene Chloride

SRM 2285, Arson Test Mixture in Methylene Chloride, contains 15 compounds, including even chain aliphatic hydrocarbons from hexane to tetracosane, toluene, p-xylene, o- and m-ethyltoluene, and 1,2,4trimethylbenzene, and it is intended primarily for use in the calibration of chromatographic instrumentation used for the classification of an ignitable liquid residue. The classification of the ignitable liquid residues is primarily based on the carbon number of the various hydrocarbons, as well as the presence of various aromatic marker compounds. Gas chromatographic (GC) analysis of volatile mixtures relies upon accurate retention time data to provide qualitative identification of compounds. This is particularly important with hydrocarbon materials, whose mass spectra are often quite similar in structure and appearance. Therefore, a certified standard of homologous alkanes is critical for calibrating the retention time scale of chromatographic columns.

This SRM will benefit local, state, and federal law enforcement agencies; private sector laboratories serving as insurance fraud investigators; and forensic science services abroad. It is also anticipated that the petroleum industry will be served by this standard in their analysis of crude and refined petroleum products. In addition, SRM 2285 will be useful for the environmental analysis of hydrocarbons in various matrices.

A unit of SRM 2285 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution. The certified concentrations of the individual components range from 1.0 mg/g to 1.4 mg/g. The concentrations are also expressed as percent volume fraction on the Certificate of Analysis for information. In addition, two gas chromatograms appear on the Certificate of Analysis, one from analysis of SRM 2285 on a relatively nonpolar GC stationary phase and

one from analysis on a moderately polar stationary phase, showing the shift in the retention order between the aliphatic and aromatic components.

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### NIST SRM 2453: Hydrogen in Titanium Alloy

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 +  $H_2$  = Ti $H_2$ , 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 temperatures. The 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.



The first SRM (2453, with a nominal mass fraction of 100 mg/kg hydrogen) has been prepared by this method and is now available for purchase. Two additional materials, SRMs 2452 and 2454, nominally 50 mg/kg and 200 mg/kg hydrogen, respectively, are in the final stages of certification. 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, increasingly, the automotive and consumer goods industries, employ titanium alloys because of 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.

SRM 2453 consists of 5 g of chips, each approximately 15 mg in mass, contained in an amber glass bottle.

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## NIST SRM 2242: Relative Intensity Correction Standard for Raman Spectroscopy: 532 nm Excitation

This Standard Reference Material (SRM) is a certified spectroscopic standard for the correction of the relative intensity of Raman spectra obtained with instruments employing 532 nm laser excitation. SRM 2242 consists of an optical glass that emits a broadband luminescence spectrum when excited with 532 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<sup>-1</sup>) expressed as the Raman shift from the excitation wavelength of 532 nm. This polynomial, together with a measurement of the luminescence spectrum of the standard, can be used to determine the spectral intensityresponse correction that is unique to each Raman system. The resulting instrumentintensity-response correction may then be used to obtain Raman spectra that are instrument independent.

This SRM is the second in a series of SRM's (2241, 2242) that will provide relative intensity correction for Raman spectrometers employing lasers commonly used for Raman spectroscopy. 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 532 nm.

SRM 2242 is a manganesedoped (0.15 wt % MnO<sub>2</sub>) borate matrix glass. Each unit of this SRM consists of a glass slide that is approximately 10.7 mm in width x 30.4 mm in length x 2.0 mm in thickness, with one surface optically polished and the opposite surface ground to a frosted finish using a 400 grit polish. The frosted surface of the slide is characterized by a surface average roughness (root-mean-square) in the range of 1.30 µm to 1.49 µm, as determined by stylus profilometry. The slide is held in a 12.5 mm square cuvette-style optical mount. This mount is designed for the typical 12.5 mm sampling accessories widely used in chemical spectroscopy (i.e., absorbance, fluorescence, etc.). This mount can easily be placed on its side for use on a microscope stage.

The mount holds the glass slide, frosted side up, in place with a clip. The glass slide extends approximately 0.3 mm above the sides of the mount to allow its use with close focus objectives.

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## NIST SRM 1908 and 1909: Vickers Microhardness of Nickel

Standard Reference Materials (SRMs) 1908 and 1909 are intended primarily for use in calibrating Vickers-type microhardness testers and are certified for mean Vickers hardness values (HV) at loads of 2.943 N (0.300 kgf) and 9.81 N (1.000 kgf), respectively. Hardness values are reported in gigapascal (GPa) and kgf/mm<sup>2</sup>. These SRMs are individually measured and bear a serial number that is imprinted on the side of the epoxy mount.

The hardness of metals is a property that is important to the steel industry, the coatings industry, and the electroplating industry. All of these industries measure hardness to characterize their products for durability and uniformity, and for general quality control. Hardness is often measured using the "Vicker's test" where a sample is struck by the point of a diamond with a known amount of force. The Vicker's number is defined as the ratio of the surface area of the resulting indentation and the force used to create it.



SRM 1908 and SRM 1909 consist of a 1.35 cm square test block of electrodeposited bright nickel, approximately 750 µm thick, on an AISI 1010 steel substrate mounted in a thermosetting epoxy.

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## NIST SRM 2392-I Mitochondrial DNA Sequencing (Human HL-60 DNA)

This Standard Reference Material (SRM) is intended to provide quality control when performing the polymerase chain reaction (PCR) and sequencing of human mitochondrial DNA (mtDNA) for forensic identification, medical diagnosis, or mutation detection. It may also serve as a control when amplifying (PCR) and sequencing any DNA. This SRM can also be used for quality assurance when assigning values to in-house control materials. It is certified for the sequences of the entire human mtDNA (16 569 base pairs) from a promyelocytic cell line (HL-60) prepared from the peripheral blood leukocytes from an individual with acute promyelocytic leukemia.

SRM 2392-I Mitochondrial DNA Sequencing compliments and adds another DNA template to SRM 2392 for the amplification and sequencing of human mtDNA. The selection of the HL-60 cell culture line for this additional DNA template was based on a suggestion from the Federal Bureau of Investigation (FBI) that this DNA would be particularly useful to the forensic community.



A unit of SRM 2392-I consists of 65  $\mu$ L of extracted DNA from cell culture line HL-60 at a nominal concentration of 1.4 ng/ $\mu$ L, which is contained in a vial packaged in a protective plastic box.

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# **NIST SRM Exhibit Schedule**

Pittsburgh Conference (Pittcon) March 7-12, 2004 McCormick Place Chicago, IL

American Chemical Society (ACS) *March 29-31, 2004* Anaheim Convention Center Anaheim, CA

#### NOBBCChE

April 11-17, 2004 Marriott Hotel & Marina San Diego, CA

#### Analytica

May 11-14, 2004 Munich, Germany

### Institute of Food

**Technologist (IFT)** July 13-16, 2004 Las Vegas Convention Center Las Vegas, NV American Association of Clinical Chemists (AACC) July 25-29, 2004 Los Angeles Convention Center Los Angeles, CA

American Chemical Society (ACS) August 23-25, 2004 Philadelphia Convention Center Philadelphia, PA

Association of Official Analytical Chemists (AOAC) September 19-23, 2004 St. Louis Convention Center St. Louis, MO

Eastern Analytical Symposium (EAS) November 11-14, 2004 Somerset Convention Center Somerset, NJ



## Renewals

SRMs C1251a, C1252a and C1253a were prepared using high purity copper doped with trace elements and chill-cast to ensure homogeneity. This suite of three SRMs provides a total of 22 certified elements.

These Standard Reference Materials (SRMs) are intended primarily for use in evaluating chemical methods of analysis and in the calibration of instrumental methods for analysis of copper and its alloys. Each unit consists of a directionally solidified, chill-cast block, approximately 32 mm square and 19 mm thick.

RM 8040 was prepared to provide material of uniform highpurity for use as a working standard for oxidation-reduction reactions and is the successor to SRM 40h Sodium Oxalate. The material conforms to the American Chemical Society specification for reagent-grade material, but should not be considered as entirely free from impurities such as moisture, sodium hydrogen oxalate and sodium hydrogen carbonate. A unit of RM 8040 consists of one bottle containing 60 g of crystalline sodium oxalate.

**SRM 185h** Potassium Hydrogen Phthalate

SRM 191c pH Standards (191-I-c) Sodium Bicarbonate (191-II-c) Sodium Carbonate

SRM 1240c Aluminum Alloy 3004

**SRM 1659a** Methane in Air, 10 μmol/mol

SRM 1685b Nitric Oxide in Nitrogen, 250 µmol/mol

SRM 1686b Nitric Oxide in Nitrogen, 500 µmol/mol

SRM 1894a Vickers Microhardness of Copper

**SRM 1951b** Lipids in Frozen Human Serum

**SRM 3191** Aqueous Electrolytic Conductivity

**SRM 3193** Aqueous Electrolytic Conductivity

**SRM 3198** Aqueous Electrolytic Conductivity

SRM 1804c Toxic Volatile Organic Compounds in Nitrogen, 5.0 nmol/mol

SRM 2798a Vickers Microhardness of Nickel

SRM 2723a Sulfur in Diesel Fuel Oil In support of EPA regulations, this ultra low sulfur diesel has a sulfur certified content of 11.0 mg/kg ± 1.1 mg/kg sulfur; (SRM 2723 was 300 mg/kg but never issued).

**SRM 1694a** Sulfur Dioxide in Nitrogen, 100 µmol/mol

# 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 expiration 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 email: srminfo@nist.gov

SRM 1261a LA Steel (AISI 4340)

This SRM will not be replaced. The certificate was edited for clarification of the uncertainty

SRM 186g Potassium Dihydrogen Phosphate (186-I-g), Disodium Hydrogen Phosphate (186-II-g) Editorial Changes **SRM 706a Polystyrene** New Expiration Date: 26 August 2010

SRM 909b Human Serum Uncertainties for bilirubin updated

SRM 1508a Benzoylecgonine (Cocaine Metabolite) in Freeze-Dried Urine New Expiration Date: 31 July 2004

SRM 1546 Meat Homogenate Editorial Change; New Expiration Date: 01 January 2008

SRM 1646a Estuarine Sediment Editorial Changes

SRM 1647d Priority Pollutant Polycyclic Aromatic Hydrocarbons (in Acetonitrile) New Expiration Date: 31 December 2008

SRM 1664a Sulfur Dioxide in Nitrogen 2500 µmol/mol Information value for lead was updated SRM 1666b Propane in Air, 10 μmol/mol New Expiration Date: 01 December 2009

SRM 1668b Propane in Air, 100 μmol/mol New Expiration Date: 01 December 2009

SRM 1669b Propane in Air,500 µmol/molNew Expiration Date:01 December 2009

SRM 1675b Carbon Dioxide in Nitrogen New Expiration Date: 01 November 2009

SRM 1776 Naval Brass WK1 Removal and de-certification of Cu, Mn, Ni and P certified values

SRM 1777 Naval Brass WK2 Removal and de-certification of Cu, Mn and P certified values

SRM 1778 Naval Brass WK3 Removal and de-certification of Mn certified value

## Revisions continued...

**SRM 1779 Naval Brass WK4** Removal and de-certification of Bi, Cu, Fe, Mn, P, Sb, and Sn certified values.

SRM 1780 Naval Brass WK5 Removal and de-certification of Bi, Fe, Ni, P, and Si certified values.

**SRM 1846 Infant Formula** New Expiration Date: 30 September 2004

SRM 2035 Near Infrared Transmission Wavelength Standard New Expiration Date: 31 December 2008

SRM 2092 Low Energy Charpy V-Notch Editorial changes

SRM 2096 High Energy Charpy V-Notch Editorial changes

SRM 2098 Super High-Energy V-Notch Editorial changes

SRM 2517a High Resolution Wavelength Calibration Reference for 1510 nm -1540 nm Acetylene 12 C<sub>2</sub>H<sub>2</sub> Editorial and Contact Changes SRM 2519 Wavelength Calibration Reference for 1530 nm - 1560nm Hydrogen Cyanide H<sup>13</sup>C<sup>14</sup>N Editorial and Contact Changes

SRM 2541 Silicon Resistivity Standard Editorial Changes

SRM 2621a Carbon Dioxide in Nitrogen 1.5% mol/mol New Expiration Date: 17 July 2009

SRM 2619a Carbon Dioxide in Nitrogen 0.5% mol/mol New Expiration Date: 15 July 2009

SRM 2622a Carbon Dioxide in Nitrogen 2.0 % mol/mol New Expiration Date: 21 July 2009

SRM 2660a Total Oxides of Nitrogen New Expiration Date: 01 May 2009

SRM 2702 Inorganics in Marine Sediment Corrected Cu reference value, certified Hg value and uncertainty updated. SRM 2806 Medium Test Dust in Hydraulic Fluid New Expiration Date: 31 December 2004

SRM 3102a Antimony Standard Solution New Expiration Date: 01 June 2007

SRM 3104 Barium Standard Solution New Expiration Date: 15 July 2007

SRM 3105a Beryllium Standard Solutuion New Expiration Date: 15 December 2006

SRM 3107 Boron Standard Solution New Expiration Date: 15 October 2007

SRM 3108 Cadmium Standard Solution New Expiration Date: 01 February 2007

SRM 3110 Cerium Standard Solution New Expiration Date: 13 July 2008

## Revisions continued...

SRM 3114 Copper Standard Solution New Expiration Date: 01 March 2007

SRM 3119a Gallium Standard Solution New Expiration Date: 01 November 2006

SRM 3120a Germanium Standard Solution New Expiration Date: 14 July 2008

SRM 3121 Gold Standard Solution New Expiration Date: 27 July 2007

SRM 3123a Holmium Standard Solution New Expiration Date: 01 July 2006

SRM 3128 Lead Standard Solution New Expiration Date: 10 June 2007

SRM 3130a Lutetium Standard Solution New Expiration Date: 02 June 2007

SRM 3131a Magnesium Standard Solution New Expiration Date: 15 July 2007 SRM 3133 Mercury Standard Solution New Expiration Date: 27 July 2007

SRM 3134 Molybdenum Standard Solution New Expiration Date: 01 December 2006

SRM 3135a Neodymium Standard Solution New Expiration Date: 02 March 2008

SRM 3137 Niobium Standard Solution New Expiration Date: 14 July 2008

SRM 3138 Palladium Standard Solution New Expiration Date: 22 December 2007

SRM 3139a Phosphorus Standard Solution New Expiration Date: 14 October 2006

SRM 3141a Potassium Standard Solution New Expiration Date: 01 February 2007

SRM 3142a Praseodymium Standard Solution New Expiration Date: 04 April 2008 SRM 3145a Rubidium Standard Solution New Expiration Date: 19 June 2006

SRM 3147a Samarium Standard Solution New Expiration Date: 15 February 2008

SRM 3148a Scandium Standard Solution New Expiration Date: 13 July 2008

SRM 3152a Sodium Standard Solution New Expiration Date: 15 July 2007, Change in certified value and uncertainty

SRM 3153a Strontium Standard Solution New Expiration Date: 01 March 2008

SRM 3154 Sulfur Standard Solution New Expiration Date: 18 August 2006

SRM 3155 Tantalum Standard Solution New Expiration Date: 13 July 2008

SRM 3157a Terbium Standard Solution New Expiration Date: 01 September 2006

## Revisions continued...

SRM 3159 Thorium Standard Solution New Expiration Date: 30 November 2008

SRM 3160a Thulium Standard Solution New Expiration Date: 15 September 2006

SRM 3163 Tungsten Standard Solution New Expiration Date: 11 July 2008

SRM 3164 Uranium Standard Solution New Expiration Date: 01 March 2007

SRM 3166a Ytterbium Standard Solution New Expiration Date: 01 September 2006

SRM 3169 Zirconium Standard Solution New Expiration Date: 17 May 2008 SRM 3161a Tin Standard Solution New Expiration Date: 15 September 2007

SRM 3162a Titanium Standard Solution New Expiration Date: 01 March 2007

SRM 3165 Vanadium Standard Solution New Expiration Date: 02 September 2007

SRM 3182 Chloride Anion Standard Solution New Expiration Date: 30 March 2005

SRM 3183 Fluoride Anion Standard Solution New Expiration Date: 30 March 2005

SRM 3185 Nitrate Anion Standard Solution New Expiration Date: 30 March 2005

SRM 3230 lodine-129 lsotopic Standard (Low Level) Editorial Changes

SRM 3231 Iodine-129 Isotopic Standard (High Level) Editorial Changes SRM 8562 CO<sub>2</sub>-Heavy, Paleomarine Origin (Carbon Dioxide) Changes in reference values and uncertainties updated. New Expiration Date: 31 December 2006

SRM 8563 CO<sub>2</sub>-Light, Petrochemical Origin (Carbon Dioxide) Changes in reference values and uncertainties updated New Expiration Date: 31 December 2006

SRM 8564 CO<sub>2</sub>-Biogenic, Modern Biomass Origin (Carbon Dioxide) Changes in reference values and uncertainties updated New Expiration Date: 31 December 2006