SRM SPOTLIGHT

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

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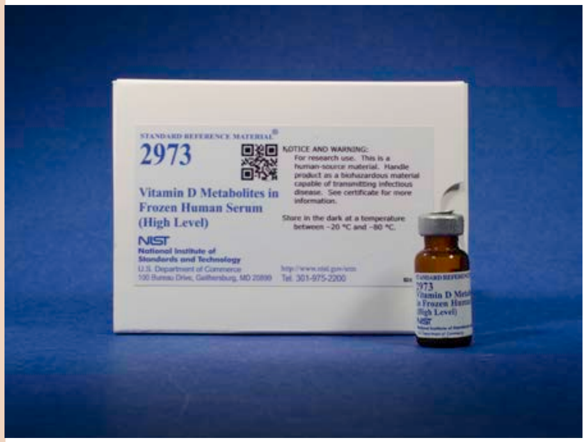
Inside:

Editor:
Regina R. Montgomery

NIST SRM 2973 Vitamin D Metabolites in Frozen Human Serum (High Level)



Testing for vitamin D has increased dramatically in the past several years, with clinical laboratories performing hundreds of thousands of vitamin D tests per year. The most widely used indicator of vitamin D levels is the measurement of a metabolite known as 25-hydroxyvitamin D [25(OH)D] in either serum or plasma. A number of studies have reported inconsistencies between the results of different techniques used to measure 25(OH)D, indicating that an accurate assessment of vitamin D status has remained problematic.

For the majority of the U.S. population, serum concentrations of 25(OH)D typically range from 40 nmol/L to 75 nmol/L. About 10 % of the population have 25(OH)D concentrations from 75 nmol/L to 120 nmol/L. SRM 2973 was prepared specifically to provide a serum material with a 25(OH)D concentration near 100 nmol/L, which will complement the lower levels available in SRM 972a Vitamin D Metabolites in Frozen Human Serum and SRM 1950 Metabolites in Frozen Human Plasma with values assigned for 25(OH)D.

NIST, in collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS), has developed SRM 2973, Vitamin D Metabolites in Frozen Human Serum (High Level), for use in evaluating the accuracy of procedures for the determination of 25(OH)D in human serum. SRM 2973 was prepared from unmodified human serum. A unit of SRM 2973 consists of one vial of frozen serum. In addition to a certified value for 25(OH)D$_3$, there are reference values for 25(OH)D$_2$ and 3-epi-25(OH)D$_3$. SRM 2973 also has a reference value for another metabolite 24R,25-dihydroxyvitamin D$_3$, which is an important vitamin D metabolite used as a catabolism marker and an indicator of kidney disease. SRM 2973 will provide a mechanism for in-vitro diagnostic (IVD) manufacturers and clinical laboratories to identify and address variations in 25(OH)D and 24R,25-dihydroxyvitamin D$_3$ measurements.

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=2973

NIST SRM 1934 Fluorescent Dyes for Quantitative Flow Cytometry (Visible Spectral Range)

Flow cytometry is a widely used technique for the analysis of single cells and micro particles. It is an essential tool for immunological research, drug and device development, clinical trials, disease diagnosis, and therapy monitoring. However, the measurements made on different instrument platforms are often inconsistent resulting in unreliable diagnostic decisions and impeding advances in biomedical research. In response, NIST and International Society for Advancement of Cytometry (ISAC) have developed a two-step methodology to enable comparable and quantitative measurements. The first step calibrates the fluorescence signal using micro particles with assigned values of equivalent number of reference fluorophores (ERF).

SRM 1934 is used in the second step to assign fluorescence intensity values to microspheres used to calibrate the fluorescence intensity scale of flow cytometers. This reference scale for fluorescence intensity is based upon ERF units. SRM 1934 consists of four ampoules, each containing a different fluorophore (e.g., fluorescent dye) solution or suspension. The SRM solutions, fluorescein in phosphate buffer solution (part A), Nile Red (NR) in acetonitrile solution (part B), Coumarin 30 (C30) in acetonitrile solution (part C), are certified for concentration of fluorophore with a certified purity. The fourth ampoule contains a Reference Material (RM), allophycocyanin (APC) in phosphate buffer with 60% saturated ammonium sulfate (part D), supplied with information values for purity, concentration, and extinction coefficient. The ERF scale is established for a particular set of experimental conditions, i.e., excitation wavelength and emission wavelength range, by comparing the fluorescence intensity of known concentrations of SRM/RM fluorophores and the fluorescence intensity of the microsphere suspension. Specific information for these fluorophores are provided in their corresponding sections of the certificate. Though SRM 1934 is developed for quantitative flow cytometry measurements, it can also be used to establish measurement traceability in other fluorescence based instrumentation such as spectrofluorometers, fluorescence microscopes, and quantative polymerase chain reaction (qPCR).

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=1934

NIST SRM 3530 Iodized Table Salt (Iodide)

Iodine (I) is an essential element that is required for proper thyroid function. Iodine deficiency is indicated by goiter, which is an enlargement of the thyroid gland, and is the most common cause of preventable mental retardation in the world. Salt iodization programs, which have been implemented in over 70 countries, have dramatically reduced the prevalence of iodine deficiency world-wide. Iodine is added to salt either as the iodide or iodate species. In the United States and other countries in the Americas, iodide is used, but iodide is not very stable and can easily be lost. Recent studies have suggested that the amounts of iodine present in table salt in the retail market vary widely, and many products are not consistent with the FDA recommended amount of 0.45 mg/kg.

NIST, in collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS) and Centro Nacional De Metrología, Mexico (CENAM), has developed Standard Reference Material (SRM) 3530 Iodized Table Salt (Iodide). SRM 3530 is intended primarily for validation of methods for the determination of iodide in table salt or similar materials and can additionally be used for quality assurance when assigning values to in-house reference materials. A unit of SRM 3530 consists of one bottle containing approximately 200 g of iodized table salt sealed inside an aluminized pouch containing an oxygen absorber-pack. Material for SRM 3530 was originally manufactured for retail food sale and was obtained from a commercial supplier. The table salt contains high-purity sodium chloride, sodium silicoaluminate, calcium sulfate, dextrose, and potassium iodide. Samples were assayed for iodine as iodide at NIST using a combination of analytical approaches, including a gravimetric titrimetric procedure based on the Winkler titration for iodide and schemes using inductively coupled plasma mass spectrometry (ICP-MS). These schemes included quadrupole ICP-MS, sector ICP-MS and ion chromatography in combination with quadrupole ICP-MS. Additional assays were performed at CENAM using linear voltammetry.

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=3530

NIST RM 8321 Peptide Mixture for Proteomics

A typical analytical strategy for the identification of proteins in complex mixtures is the enzymatic digestion of the proteins into constituent peptides. The resulting highly complex mixture of peptides is subjected to amino acid sequence analysis using techniques such as liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS). The sequence data of the peptides is then used to identify the proteins in the original sample using search algorithms and protein sequence databases. Due to the complexity of the multi-step sample and data analysis pipelines in these measurements, it can be difficult to identify analytical problems and optimize measurement procedures and outcomes. There is need in proteomic analysis for resources to address measurement quality assurance.

To support measurement quality in the analysis of the complex peptide mixtures that are common to protein identification strategies, NIST has prepared Reference Material (RM) 8321 Peptide Mixture for Proteomics. RM 8321 is a frozen aqueous solution containing approximately 440 synthetic peptides estimated to be in the concentration range of 0.1 pmol/µL to 10 pmol/µL. The synthetic peptides in RM 8321 have the same amino acid sequences as tryptic peptides from 50 high-abundance human plasma proteins that have been frequently observed through published identifications by the proteomics community. They are prototypic peptides as they have been observed frequently and with high confidence through mass spectrometry-based proteomic investigations of human plasma samples. The synthetic peptides in RM 8321 were each individually evaluated to confirm that they yield high-quality tandem mass spectrometry (MS/MS) spectra, have sufficient purity, and chromatographic retention characteristics to produce a peptide mixture with a complex elution profile in a typical reversed-phase chromatographic analysis. The presence of the 440 synthetic peptides in RM 8321 was confirmed by NIST through multiple analyses using two types of LC-MS/MS measurements and comparison to mass spectral libraries.

RM 8321 was developed to be used as a "trueness" control sample for the identification of peptides in complex mixtures. RM 8321 can be used to help assess the confidence of peptide identification within a laboratory or comparability between laboratories or among different measurement approaches. RM 8321 can also be used in the development and validation of new investigative approaches for identifying peptides in complex peptide mixtures.

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=8321

Renewal SRMs/RMs

NIST SRM 131h Refined Cast Iron

For years, manufacturers of metals from cast iron to high alloy steels have worked with composition specifications that require control of numerous elements at trace levels. Among the most important elements are carbon and sulfur. Carbon is used as an alloying element, but often, it must be kept out of alloys when other elements are more important. Sulfur is often undesirable, because it weakens alloys, especially under high-temperature operating conditions. SRM 131h is the ninth version of cast iron or cast steel designed to provide homogeneous distributions of C and S at levels < 10 mg/kg of each element. The certificate of analysis provides a certified value for S at 7.4 mg/kg and a reference value for C at 7.8 mg/kg. Sulfur analyses were performed by X-ray fluorescence spectrometry at NIST and by ASTM International combustion methods at collaborating laboratories. Carbon analyses were provided by collaborators using ASTM International combustion methods.

SRM 131h is different from the past three versions of SRM 131 by being cast iron that started out with percent levels of C and S, rather than high silicon steel. The manufacturer refined it carefully to remove these two elements. That is why the material has been dubbed "refined cast iron" to denote its history. SRM 131h was prepared from a single billet of iron by chipping on a lathe, grinding the chips for better uniformity, and bottling at 100 g per bottle. The material is designed to be used to validate chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials and, if necessary, to calibrate carbon/sulfur analyzers. The function of calibration of analyzers is the least preferred use for NIST SRMs. It is better to use NIST certified materials to establish traceability of values for a wider range of reference material compositions provided by commercial reference materials producers and by manufacturing laboratories producing in-house reference materials. For more information on development of in-house reference materials, consult ASTM E2972 – 15 Standard Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials.

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=131h

NIST SRM 2100a and 2100b Fracture Toughness of Ceramics

The property "fracture toughness" quantifies how brittle a ceramic or glass material is. It is more fundamental than "strength." The latter depends upon the material's intrinsic fracture resistance as measured by the property fracture toughness, and the size and distribution of flaws that may exist in a component. The latter are highly variable. Although ceramics and glasses have been used as structural materials for millennia, they were primarily used under compression loadings. Preexisting flaws usually do not propagate and hence ceramics are very strong in compression mode of loading. It was only in the latter half of the 20th century that engineers and designers began to use ceramics with tensile stress loadings. Refined, highly-engineered ceramics were created that were able to sustain tensile stress loadings. It was imperative to quantify a ceramic's intrinsic brittleness, i.e., fracture toughness. A proliferation of test methods with conflicting outcomes led to massive confusion in the 1970s – 1980s, which was only resolved when NIST released SRM 2100 in 1999. This SRM was the very first certified reference material in the world for the property fracture toughness for any class of material: ceramics, glass, metal, polymer, or composite. The SRM is a set of 5 unprecracked hot-pressed silicon nitride specimens. The material was a commercially produced material, versions of which are used worldwide in engine parts and ball bearings. SRM 2100 was certified for the property, independent of the test method. Three very different test methods were used to develop the SRM and the outcomes were independent of the method.

SRM 2100a and 2100b Fracture Toughness of Ceramics (continued)

SRM 2100 paved the way for rapid test method standardization for measurement of a material's intrinsic brittleness. The SRM complements ASTM International, ISO, and CEN international standards developed in the late 1990s and early 2000s. The existence of the SRM has facilitated international trade and harmonized test method procedures around the world.

All test pieces of the original SRM 2100 were from a single plate of silicon nitride and were depleted in 2015. SRMs 2100a and SRM 2100b are now available and were prepared from two plates of the same type of silicon nitride as used in the original SRM. There are subtle differences in the microstructures between the plates, however, and hence the certified fracture toughness values differ. This is not uncommon with ceramics, and the ability of the tests methods to detect and quantify variations in brittleness is important.

These SRMs can be used by industrial, government, and academic institutions to check their testing procedures including the precision and accuracy of their test procedures.

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https://www-s.nist.gov/srmors/view_detail.cfm?srm=2100a
https://www-s.nist.gov/srmors/view_detail.cfm?srm=2100b

Renewal SRMs/RMs

SRM 931h Liquid Absorbance Standard for Ultraviolet and Visible Spectrophotometry

SRM 1685b Nitric Oxide in Nitrogen (Nominal Amount-of-Substance Fraction 250 µmol/mol), Lot #43-M-XX

SRM 1686b Nitric Oxide in Nitrogen (Nominal Amount-of-Substance Fraction 500 µmol/mol), Lot #42-N-XX

SRM 3102a Antimony (Sb) Standard Solution, Lot #140911

SRM 3104a Barium (Ba) Standard Solution, Lot #140909

SRM 3147a Samarium (Sm) Standard Solution, Lot #140115

SRM 3166a Ytterbium (Yb) Standard Solution, Lot #140114

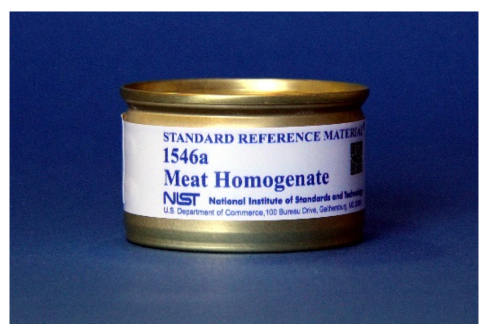
Certificate Revisions

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. Certificates are the official source for values and expiration dates. Users of NIST Standard Reference Materials should ensure that they have the current certificates. You can print or view a copy of the current certificate at our website at http:/www.nist.gov/srm or contact the Office of Reference Materials at **phone** 301-975-2200, **fax** 301-926-4751, or **email** srminfo@nist.gov

NIST SRM 1546a Meat Homogenate

SRM 1546a Meat Homogenate is one of several food-matrix SRMs currently available to support the requirements of the Nutrition Labeling and Education Act of 1990 (NLEA) whereby nutrition information must be provided on labels of processed foods sold in the U.S. The food industry uses this SRM, as well as other food-matrix SRMs, for quality assurance in their analyses in support of their labeling requirements. This SRM is also intended for use in method development and validation and for use in assigning values to in-house control materials. SRM 1546a is a mixture of pork and chicken products blended by a commercial process and is located in sector 4 of the AOAC food triangle (shown below), in which foods are positioned based on their fat, protein, and carbohydrate content, and can be used to help select an appropriate quality control material. Certified values are assigned for cholesterol and sixteen fatty acids, nine elements, and six vitamins. Reference values are assigned for seven elements, eight vitamins, choline and carnitine, proximates, calories, eleven fatty acids, and nineteen amino acids. This material was originally released for sale in 2014. A unit of SRM 1546a consists of four cans, each containing approximately 85 g of material.

The certificate for SRM 1546a was updated recently with a certified value for 25-hydroxyvitamin D_3 and reference values for vitamin D_3 and carnitine. Recent research has indicated that 25-hydroxyvitamin D may be 5 times more biologically relevant than unmetabolized vitamin D, therefore having a significant impact on vitamin D health status. As a result, the US Department of Agriculture (USDA) and National Institutes of Health (NIH) Office of Dietary Supplements (ODS) have joined efforts to document the quantity of 25-hydroxyvitamin D in common foods, and coauthored an interlaboratory study with NIST to compare analytical methods for measuring vitamin D metabolites. Values for 25-hydroxyvitamin D_3 and vitamin D_3 were obtained as a part of this interlaboratory study[1], in combination with data collected by NIST. The addition of these values for vitamin D and metabolites will increase the value of SRM 1546a to the food industry.



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https://www-s.nist.gov/srmors/view_detail.cfm?srm=1546a

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[1] Roseland, J.M., Patterson, K.Y., Andrews, K.W., Phillips, K.M., Phillips, M.M., Pehrsson, P.R., Dufresne, G.L., Jakobsen J., Gusev, P.A., Savarala, S., Nguyen, Q.V., Makowski, A.J., Scheuerell, C.R., Larouche, G.P., Wise, S.A., Harnly, J.M., Williams, J.R., Betz, J.M., Taylor, C.L.; *Interlaboratory Trial for Measurement of Vitamin D and 25-Hydroxyvitamin D [25(OH)D] in Foods and a Dietary Supplement Using Liquid Chromatography–Mass Spectrometry;* J. Agric. Food Chem., Vol. *64* Issue 16, pp 3167–3175 (2016).

Revisions (continued)

SRM 32e Carbon Low Alloy Steel (SAE 3140) (chip form)
Editorial changes

SRM 911c Cholesterol
Editorial changes

SRM 1595 Tripalmitin
Editorial changes

SRM 1624d Sulfur in Diesel Fuel Oil (Nominal Mass Fraction 0.4 %)
New expiration date: 31 January 2022
Editorial changes

SRM 1886b White Portland Cement
Editorial changes

SRM 1945 Whale Blubber
New expiration date: 31 July 2026
Editorial changes

SRM 1950 Metabolites in Frozen Human Serum
New expiration date: 30 September 2023
Editorial changes

SRM 2266 Hopanes and Steranes in 2,2,4-Trimethylpentane
New expiration date: 31 March 2026
Editorial changes

SRM 2570 Lead Paint Film for Building Surfaces (Blank) (Color: White)
New expiration date: 01 July 2026
Editorial changes

SRM 2571 Lead Paint Film for Building Surfaces (Nominal Pb 3.5 mg/cm^2) (Color: Yellow)
New expiration date: 01 July 2026
Editorial changes

SRM 2572 Lead Paint Film for Building Surfaces (Nominal Pb 1.6 mg/cm^2) (Color: Orange)
New expiration date: 01 July 2026
Editorial changes

SRM 2573 Lead Paint Film for Building Surfaces (Nominal Pb 1.0 mg/cm^2) (Color: Red)
New expiration date: 01 July 2026
Editorial changes

SRM 2574 Lead Paint Film for Building Surfaces (Nominal Pb 0.7 mg/cm^2) (Color: Gold)
New expiration date: 01 July 2026
Editorial changes

Revisions (continued)

SRM 2575 Lead Paint Film for Building Surfaces (Nominal Pb 0.3 mg/cm^2) (Color: Green)
New expiration date: 01 July 2026
Editorial changes

SRM 2576 Lead Paint Film for Building Surfaces (Nominal Pb 5.6 mg/cm^2) (Color: Blue)
New expiration date: 01 July 2026
Editorial changes

SRM 2579a Lead Paint Films for Building Surfaces (SRM 2570 through SRM 2575)
New expiration date: 01 July 2026
Editorial changes

SRM 2702 Inorganics in Marine Sediment
Editorial changes

SRM 2770 Sulfur in Diesel Fuel Oil (Nominal Mass Fraction 40 mg/kg), Lot #050716
New expiration date: 31 January 2022
Editorial changes

SRM 2921 Human Cardiac Troponin Complex
Editorial changes

SRM 3103a Arsenic (As) Standard Solution
New expiration date: 31 December 2018

SRM 3185 Nitrate Anion (NO_3^-) Standard Solution
New expiration date: 30 December 2017
Editorial changes

SRM 3275 Omega-3 and Omega-6 Fatty Acids in Fish Oil
Editorial changes

SRM 3280 Multivitamin/Multielement Tablets
New expiration date: 31 October 2019
Editorial changes

SRM 3282 Low-Calorie Cranberry Juice Cocktail
New expiration date: 30 November 2025
Editorial changes

SRM 3290 Dry Cat Food
Editorial changes

RM 8441a Wheat Hardness
New expiration date: 03 January 2022
Editorial changes

UPCOMING NIST 2016 SRM EXHIBIT SCHEDULE

Clinical Lab Expo AACC
Booth #3914
July 31- August 4, 2016
Pennsylvania Convention Center, Philadelphia, PA

American Chemical Society Fall Meeting ACS
Booth #1119
August 21-25, 2016
Pennsylvania Convention Center, Philadelphia, PA

International Annual Meeting AOAC
Booth #304
September 18-21, 2016
Sheraton Dallas Hotel, Dallas, TX

Materials Science & Technology Conference and Exhibition MS&T 16
Booth #419
October 24-27, 2016
Salt Palace Convention Center, Salt Lake, UT

Material Research Society Fall Meeting MRS
November 27-December 2, 2016
Hynes Convention Center
Boston, MA
Sheraton Dallas Hotel, Dallas, TX

ORDER NIST SRMs ONLINE

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

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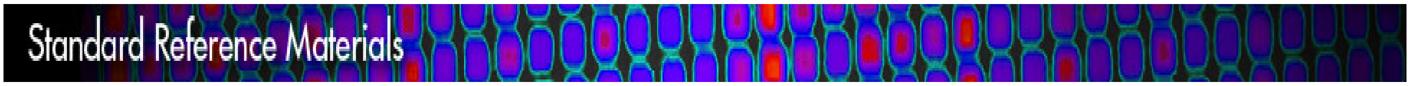
NIST Measurement Services Websites of Interest



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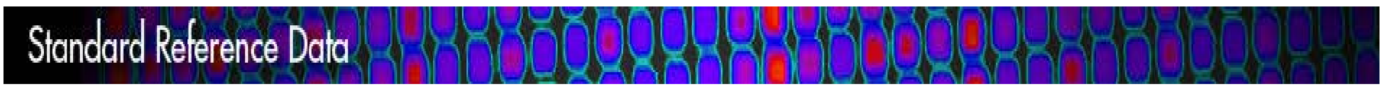
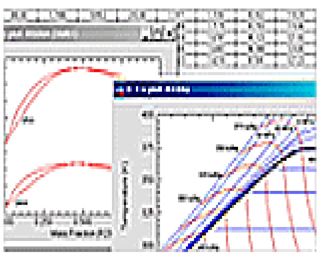
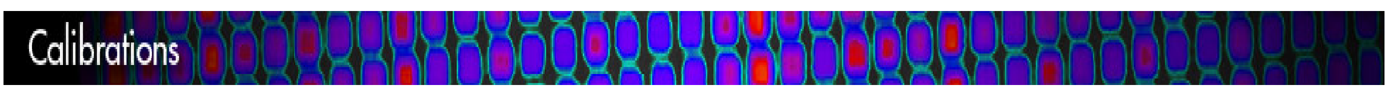
Historical Archived Certificates/Reports of Investigation
https://www-s.nist.gov/srmors/certArchive.cfm



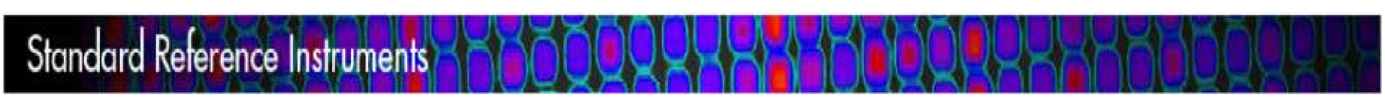


NIST Scientific and Technical Databases
http://www.nist.gov/srd

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



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



Standard Reference Instruments

http://www.nist.gov/srm/standard-reference-instruments.cfm

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