SRM SPOTLIGHT

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NIST SRM 955c Lead in Caprine Blood

The SRM 955 series of lead (Pb) in blood standards has been heavily relied upon for quality assurance of clinical blood Pb measurements. It is estimated that over 300,000 U.S. children aged one to five years have blood Pb levels greater than the Centers for Disease Control and Prevention (CDC) recommended level of 10 µg/dL. SRM 955c, the fourth in a series of NIST standards certified for blood Pb concentration, was developed in conjunction with the Trace Elements/Lead Poisoning Laboratory, Wadsworth Center, New York State Department of Health and partially funded by CDC. A unit of SRM 955c consists of four vials of frozen caprine (goat) blood at four concentration levels: a base level and three progressively elevated levels that contain endogenous Pb and other toxic elements of interest. SRM 955c will be used by clinical laboratories and hospitals to evaluate the accuracy of Pb concentration measurements in blood and to validate working or secondary reference materials for Pb in blood analysis.

Lead is neurotoxic and particularly harmful to the developing nervous systems of fetuses and young children. In 1990 the U.S. Department of Health and Human Services established a national goal to eliminate blood Pb levels greater than 25 µg/dL by 2000, and set a new goal to eliminate blood Pb levels greater than 10 µg/dL in children under the age of six by 2010 [1].

In response to the continued need for a blood-based reference material with accurately certified Pb concentrations, NIST developed SRM 955c Lead in Caprine Blood. The composition of this material differs from previous issues of SRM 955. The first SRM 955 was based on heparinized porcine blood obtained from Pb-dosed hogs. SRMs 955a and SRM 955b were based on bovine blood from Pb-dosed cows. SRM 955c is based on caprine blood obtained from Pb-dosed goats. The hemopoietic system of the adult goat is much closer to that of humans, thus making it a better model for assessing proficiency for erythrocyte protoporphyrin, a biomarker of Pb exposure. Adult goats were dosed with lead acetate to produce blood pools containing Pb physiologically bound to red blood cells (erythrocytes). In addition, the blood pools were spiked with inorganic arsenic, cadmium, inorganic mercury, methylmercury, and ethylmercury. Certified values are provided for Pb and are based on analysis using a high-accuracy isotope dilution, inductively coupled plasma mass spectrography (ID ICP-MS) method [2].

NIST SRM 955c Lead in Caprine Blood (continued)

Table 1 lists the certified values and expanded uncertainties for Pb in the four levels of SRM 955c. Compared to previous issues of SRM 955, the Pb concentration for Level 1 is an order of magnitude lower and represents a true base level. Expanded uncertainties of less than 0.6 % relative were achieved for Levels 2, 3, and 4.

Table 1. Certified values (μg/dL) and expanded uncertainties for lead in SRM 955c.

Level 1	0.424	±	0.011
Level 2	13.950	±	0.080
Level 3	27.76	±	0.16
Level 4	45.53	±	0.27

Future Plans: Additional analyses will be performed to provide certified or reference values for inorganic arsenic, cadmium, inorganic mercury, methylmercury and ethylmercury.

References:

[1]Surveillance for Elevated Blood Lead Levels, 1997-2001, http://www.cdc.gov/mmwr/preview/mmwrhtml/ss5210a1.htm, accessed December 2006.
[2] Murphy, K.E.; Paulsen, P.J.; Fresenius J. Anal. Chem., Vol. 352, pp. 203-208 (1995).

SRM 955c is used to validate the accuracy of blood Pb measurements.

Impact: **Over two million blood Pb measurements are performed in the U.S. annually. SRM 955 is heavily relied upon for quality assurance of clinical blood Pb measurements, with over 200 units distributed per year. SRM 955c is the fourth issue of this material. Significant improvements over previous issues have been realized in the material composition, Pb levels, and expanded uncertainties of the certified Pb concentrations.**

Technical Contacts: K.E. Murphy and T.W. Vetter (Div. 839), P.J. Parsons, C. Geraghty, C.D. Palmer, and M.E. Lewis, Jr. (Wadsworth Center, New York State Department of Health)
Email: karen.murphy@nist.gov; thomas.vetter@nist.gov

NIST SRM 1994 Silicon Single Crystal Wafer for Crystalline Orientation

Crystallographic orientation is a critical parameter in the semiconductor industry, where the orientation with respect to the wafer surface is necessary for materials growth and processing. In other industries (such as the production of surface acoustic wave devices and quartz oscillators) crystal orientation is important as well. A primary calibration standard for on-site X-ray single crystal orientation systems and cross-calibration between multiple crystal orientation instruments would be of widespread importance to diverse manufacturing processes.

NIST has developed SRM 1994 Silicon Single Crystal Wafer for Crystalline Orientation as a crystal orientation standard for X-ray single crystal orientation systems. The SRM consists of a 100-mm diameter single crystal silicon wafer where the orientation of a major crystallographic plane with respect to the surface of the wafer has been established. The misorientation of the [001] silicon crystal planes with respect to the polished surface of each wafer has been measured about two orthogonal axes relative to the edge flat manufactured into each wafer. These measurements were performed using a high accuracy X-ray diffractometer that utilizes a polarization-encoded optical angle interferometer so that the angular measurements are traceable to the SI. The measured crystalline disorientations with respect to the two orthogonal axes have been certified to an accuracy of better than 5 arcseconds, or approximately 2.4×10^{-5} radians.

Technical Contact: Erick Benck
Email: erick.benck@nist.gov

NIST SRM 2842 Semiconductor Thin Film

Second New Standard Released for Compound Semiconductor Industry

Continuing a recently launched series of composition standards for the compound semiconductor, Standard Reference Material (SRM) 2842 is now available. This SRM is a semiconductor piece made of GaAs with a 3 µm thick layer of $Al_xGa_{1-x}As$ deposited on top. The Al mole fraction x of the layer has been certified by NIST to within an expanded uncertainty of 0.002. The piece is approximately one centimeter square and is mounted to a stainless steel disk for transport and handling. This is the second standard for the chemical composition of thin-film semiconductor alloys issued by NIST. This SRM series was requested by the compound semiconductor industry to help measure and control thin film composition as a basis for optimizing material and device properties. The SRM can be used to calibrate equipment for making or analyzing these materials. Expected customers include companies that grow or characterize thin films or use them to make devices, as well as government and university laboratories. SRM 2842 units have Al mole fraction near 0.30; NIST has previously released units of SRM 2841 with Al mole fraction near 0.20.

AlGaAs is used as a barrier material to increase conductivity in high-speed circuits for wireless communication; semiconductor lasers for optical disk drives, bar code scanning, xerography, and laser surgery; and light-emitting diodes for remote controls, traffic lights, and medical instruments.

NIST SRM 2842 Semiconductor Thin Film (continued)

The NIST standard is expected to increase the accuracy of chemical characterization of AlGaAs films by an order of magnitude over the current state of the art. Improved accuracy will reduce wasteful duplication of reference wafers, increase the free exchange of thin-film materials between vendors and their customers, and ultimately improve the accuracy of data on relationships between material and composition and properties.

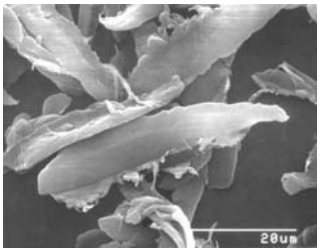
Additional information is also available in NIST Special Publication 260-163.

Media Contact: Laura Ost
Email: laura.ost@nist.gov
Technical Contact: Kris Bertness
Email: bertness@boulder.nist.gov

NIST SRM 2880 Controlled Size and Shape of Ultra-High Molecular Weight Polyethylene (UHMWPE) Wear Particles for Bioactivity Tests and Calibration (spherical shape)

The use of prosthetics, particularly artificial joints, is increasing steadily world-wide. Longer life span, more accessible health care, and improvements in materials technology all contribute to this trend. Ultra-high molecular weight polyethylene (UHMWPE) and cobalt-chrome alloys are the dominant materials used in making artificial joints, with a typical lifespan of about ten to fifteen years. The durability of an artificial joint in a human body is limited by a complex physiological-immuno response to UHMWPE wear particles developed in the joint. The wear particles are generated when the polymer and the metal surfaces rub together during walking, running, and exercising. The details of the mechanisms for the immuno-response are not well understood, especially the effects of the size and shape of the wear particles. To measure the effects of particle size and shape on response reactions–and thus optimize joint design and performance–an accurate UHMWPE particle reference material is required with sizes in the range appropriate to wear debris from artificial joints. Such a reference material would also allow validation of quantitative measurements of wear debris retrieved from revision surgeries.

NIST has developed SRM 2880 UHMWPE wear particles for bioactivity testing and joint retrieval analysis comparisons. An innovative textured-surface production method was used to generate the wear particles: Using a micro-texturing technique, cutting surfaces with specific sizes and shapes of micro-cutting edges were produced and then UHMWPE pins were rubbed against the cutting surfaces under controlled loads and speeds. In this manner, wear particles with well-controlled size and shape distributions were produced by adjusting the load or the number of passes. The SRM consists of five vials of wear particles dispersed in water: Three vials of round wear particles (R1, R2, R3), of nominal particle radii of 2.4 µm, 6.6 µm and 12.4 µm, and two elongated wear particles (E1, E2), with aspect ratios of 1.9 (9.4 µm length) and 4.0 (18.3 µm length).



Scanning electron micrograph of UHMWPE wear particles (E2) as part of SRM 2880 Ultra-High Molecular Weight Polyethylene (UHMWPE) wear particles for bioactivity testing and joint retrieval analysis comparisons.

Technical Contact: Robert Cook
Email:robert.cook@nist.gov

NIST SRMs 2940 and 2941 for Spectral Correction and Performance Validation of Fluorescence Instruments

Two ready-to-use, fluorescent glass SRMs have recently been released by NIST. They will enable the relative spectral correction and day-to-day performance validation of fluorescence instruments to be achieved with relative ease, even by non-expert users. Luminescence measurements have become the detection methods of choice for many clinical and biochemical assays, due to their extraordinary selectivity and sensitivity. As these analytical methods become more accurate and precise, they require better standards for both instrument calibration and method validation if they are to be included in quality systems or applied to analyses governed by regulation. Ideally, users would like to employ the same organic dye probes used for analyte detection as standards for fluorescence intensity and spectral correction. Unfortunately, organic dyes photodegrade quickly, do not have long shelf lives in solution, have environment-dependent fluorescence, and are expensive to produce at high purity.

After studying the characteristics of the different types of fluorescent materials, NIST researchers found metal-ion-doped glasses to be the best choice for use as fluorescence standards for spectral correction and intensity. These glasses are photostable, robust, relatively inexpensive, and can be made to suit most detection formats. SRM 2941 has green emission that peaks at 526 nm and an effective emission range from 485 nm to 605 nm. SRM 2940 has orange emission that peaks at 620 nm and an effective emission range from 520 nm to 780 nm. The certified values for relative steady-state emission spectrum power are supplied with each SRM, along with their estimated total uncertainties. Both SRMs are highly resistant to photodegradation and are, therefore, also recommended for use as day-to-day and instrument-to-instrument intensity standards for performance validation. These SRMs are in the form of a solid glass, standard sized cuvette (12.5 mm × 12.5 mm × 45 mm) with three polished long faces for 90° detection and one frosted long face for front-face or epi-fluorescence detection.

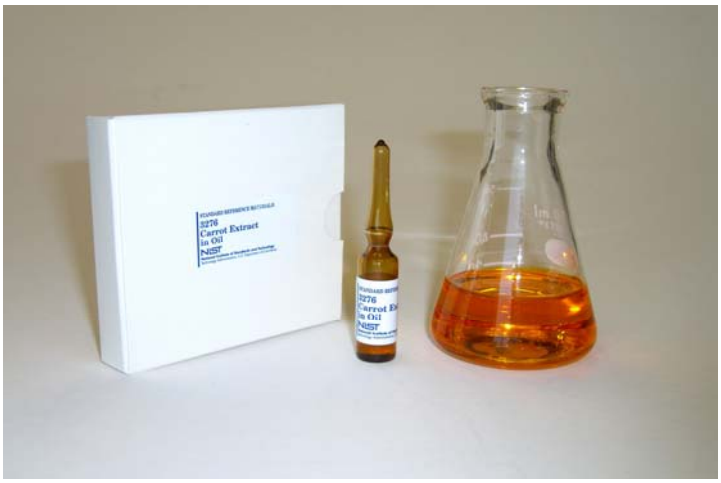
Relative Intensity Correction Standards for Fluorescence Spectroscopy

SRMs 2940 and 2941 can be used in combination with SRM 936a, quinine sulfate dihydrate, a pre-existing blue spectral correction standard. This combination allows the user to cover the visible region from 400 nm to 780 nm. The high photostability of SRMs 2940 and 2941 make them particularly useful as day-to-day intensity standards, even when spectral correction is not needed or when the excitation wavelength differs from that used for certification.

Technical Contact: Paul DeRose
Email:paul.derose@nist.gov

NIST SRM 3276 Carrot Extract in Oil

Beta-carotene is used in dietary supplements for its perceived cardiovascular benefits and cancer preventive effects and as a non-toxic source of vitamin A. The accurate measurement of beta-carotene is important to the food and nutrition community because nutrition labeling laws require that vitamin A content (including the contribution from carotenoids) be included on product labels. As part of an interagency agreement, NIST, the National Institutes of Health's Office of Dietary Supplements (NIH/ODS), and the Food and Drug Administration's Center for Drug Evaluation and Research (FDA/CDER) are developing Standard Reference Materials (SRMs) for dietary supplements. One of these materials is SRM 3276 Carrot Extract in Oil. This material is an infusion of carrots in vegetable oil, and is expected to complement NIST's other SRMs that have values assigned for carotenoids (i.e., SRM 968c Fat-Soluble Vitamins, Carotenoids and Cholesterol in Human Serum, SRM 2383 Baby Food Composite, and SRM 2385 Slurried Spinach). Values in SRM 3276 are assigned for alpha- and beta-carotene, delta- and gamma-tocopherol, and twelve fatty acids. This material is intended for use as a primary control material when assigning values to in-house (secondary) control materials and for validation of analytical methods. Other materials available in the dietary supplement series include cod liver oil and suites of materials containing ephedra and ginkgo. Future dietary supplement SRMs will include a multivitamin/multielement tablet, saw palmetto, bitter orange, green tea, cranberries, blueberries, bilberries, black cohosh, soy, kudzu, red clover, and St. John's wort.



SRM 3276 Carrot Extract in Oil

Technical Contact: Katherine E. Sharpless
Email: katherine.sharpless@nist.gov

NIST SRM 5000 Calibrated Overlay Wafer Standard

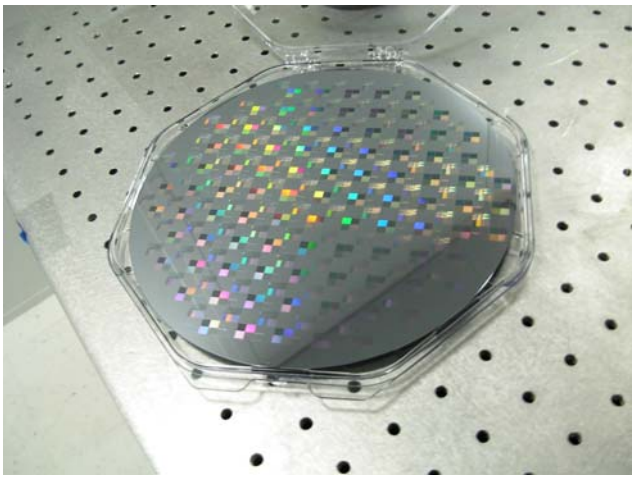
New SRM for establishing accuracy and traceability on optical overlay metrology tools.

Semiconductor devices are created by exposing successive patterns onto a photosensitive resist spun onto a wafer. The proper functioning of the devices depends on how well each successive layer is aligned to the previous layers. The measure of how well one exposure pattern aligns with the next is termed "overlay" (OL).

NIST is offering for the first time a 200 mm calibrated wafer for establishing accuracy and traceability on optical overlay metrology tools. The wafer has two types of overlay targets that are calibrated. It has both Frame-in-Frame (FF) and Bar-in-Bar (RR) overlay targets that have been calibrated.

SRM 5000 is a 200 mm double-etched silicon wafer containing various research and calibration targets. The wafer contains 93 dies, each die measuring 17.6 mm in X and 16.0 mm in Y, with a 17.6 mm pitch in the X-direction and a 16.0 mm pitch in the Y-direction. The center die contains five calibrated FF targets and five calibrated RR targets, with OL offsets ranging from +0.100 µm to -0.100 µm.

Technical Contact: Mike Stocker
Email: michael.stocker@nist.gov



SRM 5000 Calibrated Overlay Wafer Standard

NIST SRM 5001 Two-Dimensional Grid Photomask Standard

This SRM is intended for calibrating high accuracy two-dimensional photomask/reticle registration metrology tools. The standard is fabricated on a 15.2 cm × 15.2 cm × 0.64 cm (or 6 in × 6 in × 0.25 in) fused quartz plate. Although designed for semiconductor photomask metrology and inspection tools, the reticle has calibration applications for other two dimensional metrology instruments. Applications include machine tools and flat panel display fabrication where positional accuracy is higher than what conventional coordinate measurement machines are capable of. The standard consists of a grid of 27 × 27 unit cells having a nominal pitch of 5 mm. Each unit cell has a frame, a solid box, and a 13 × 13 micro array. The positions of the centers of the frames, every other row and column, are reported and traceable to the definition of the meter. The expanded uncertainty (k=2) is $7.0 \times 10^{-8} \times L + 8.7$ nm, where L is the distance between any two frame center positions.

Technical Contact: Richard Silver
Email: richard.silver@nist.gov

NIST RM 8327 Peptide Reference Material for Molecular Mass and Purity Measurements

Methods have been developed to extract small amounts of proteins from tissue samples at various stages of disease for subsequent identification and characterization as possible drug targets in the development of various therapeutic strategies. These methods involve the use of proteases to break down the proteins into smaller peptides, which are then separated by high performance liquid chromatography and subsequently analyzed by determining their masses by mass spectroscopy, such as matrix-assisted laser desorption ionization (MALDI) or electron solvent ionization. Although the peptide fragments differ in size, net charges, and physical properties, reference peptides have not been available to validate this method for protein fragmentation, separation, and identification. The need for a peptide RM has been long recognized by the Association of Biomolecular Resource Facilities (ABRF), a non-profit standards organization sponsored by 33 companies. The organization, with 800 members from industry, academia, and government, was created in part to provide a mechanism for the self-evaluation and improvement of procedural and operational accuracy, precision, and efficiency in resource facilities and research laboratories. In collaboration with NIST scientists, a Peptide Standards Project Committee (PSPC) was formed by ABRF to design and characterize three synthetic peptides to serve as a NIST peptide reference material, RM 8327.

RM 8327 is unique in that it consists of three peptides from 11, 14 and 26 amino acid residues in length, with net charges of -3, -1 and +3. The absence of methionine, cysteine and tryptophan in the peptides facilitates synthesis and improves stability. The peptides do incorporate protease cleavage sites, and contain tyrosine for concentration analysis by UV spectroscopy.

Technical Contact: Fred Schwarz
Email: frederick.schwarz@nist.gov

NIST RM 8573 and 8574 Carbon and Nitrogen Isotopes in L-glutamic acid

Nitrogen and carbon isotopic ratio variations are powerful tools for studying nutrient dynamics and relationships between trophic levels in terrestrial and aquatic ecosystems. The processes controlling nutrient flows between sources and sink are quite complex, especially when human interplay is possible and an understanding and quantification of possible impacts is central to implementing best practice and informed management of environments ranging from wetlands to agricultural settings.

Currently, the majority of organic C and N isotope ratio measurements are made by on-line combustion isotope ratio mass spectrometry (IRMS). Suitable isotopic reference materials are critical for calibrating measurements and laboratory working standards against the δ^{13}C scale relative to Vienna Pee Dee belemnite (VPDB) ($\delta^{13}C_{VPDB}$) and the δ^{15}N scale relative to N_2 in AIR ($\delta^{15}N_{AIR}$). They are also used to correct measurements for instrumental biases such as drift, mass dependent fractionation and delta scale contractions. Currently available reference materials suitable for on-line combustion, however, have C/N ratios and isotopic compositions that are not optimum for measurements of many biological materials.

The U.S. Geological Survey (USGS) (Reston, VA) undertook to create a suitable organic isotopic reference material and produced RM 8573 (USGS40) and RM 8574 (USGS41) from analytical reagent L-glutamic acid. L-glutamic acid was chosen because it has a molar C/N ratio of five (typical of many biological materials), is soluble in water, and is stable as a dry powder at room temperature. RM 8573 was derived from commercially available L-glutamic acid and contains light carbon ($\delta^{13}C_{VPDB}$ = -26.93 %) and light nitrogen ($\delta^{15}N_{AIR}$ = -4.52 %). RM 8574 was prepared by dissolving, mixing and then precipitating a precise blend of isotopically normal and ^{13}C and ^{15}N enriched L-glutamic acids. RM 8574 is approximately 5 % isotopically heavier in carbon ($\delta^{13}C_{VPDB}$ = +37.63 %) and nitrogen ($\delta^{15}N_{AIR}$= +47.53 %) than RM 8573. The range of carbon and nitrogen isotopic values bracketed by these two RMs captures the range and variations seen in natural biological systems.

The isotopic values for these materials were assigned by combining data from off-line dual-inlet IRMS and on-line combustion continuous flow IRMS measurements done at both Max Planck Institute (Jena, Germany) and USGS (Reston, VA).

These two new RMs will provide researchers in biological and ecology fields with a precise and timely tool for eliminating instrumental biases; thereby improving the quality and intercomparability of their carbon and nitrogen isotopic measurements.

Technical Contact : Robert Vocke
Email : robert.vocke@nist.gov

Renewals

SRM 918b Potassium Chloride (Clinical Standard)

SRM 919b Sodium Chloride (Clinical Standard)

SRM 967 Creatinine in Frozen Human Serum

SRM 1822a Refractive Index Standard

SRM 1866b Common Commercial Asbestos

SRM 1921b Infrared Transmission Wavelength Standard

SRM 2842 Semiconductor Thin Film: $A1_xGA_{1-x}AS$ Epitaxial Layers

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

SRM 114q Portland Cement Fineness Standard
Technical Changes

SRM 934 Clinical Laboratory Thermometer
Editorial Changes

SRM 1548a Typical Diet
Correction to barium value

SRM 1589a PCBs, Pesticides, PBDEs, and Dioxins/Furans in Human Serum
Editorial Changes

SRM 1687b Nitric Oxide in Nitrogen Lot #41-K-XX
New Expiration Date: 01 April 2013

SRM 1845 Cholesterol in Whole Egg Powder
Update of expiration date and editorial changes

SRM 1951b Lipids in Frozen Human Serum
Editorial Changes

SRM 1976 Instrument Sensitivity Standard for X-Ray Powder Diffraction
Editorial Changes

SRM 2630 Nitric Oxide in Nitrogen Lot #46-E-XX
New Expiration Date: 01 April 2013

SRM 3139a Phosphorus Standard Solution Lot #060717
Correction of nitric acid volume fraction

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

SRM 2007 MARKETING CATALOG/CD

If you would like a copy of our new January 2007 SRM Marketing Catalog, Price List or a CD, please call, fax, or email us at:

Ph: 301-975-6776/2200
Fax: 301-948-3730
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 2007 Exhibit Schedule

AACC-Clinical Lab Expo (AACC)
July 15-19, 2007
San Diego Convention Center
San Diego, CA

IFT-Food Expo
July 26-30, 2007
McCormick Place
Chicago, IL

NCSL Symposium
July 29-August 2, 2007
St. Paul River Centre
Minneapolis, MN

American Chemical Society (ACS)
August 20-22, 2007
Boston Convention Center
Boston, MA

AOAC Annual Meeting (AOAC)
September 16-20, 2007
Hyatt Regency Orange County
Anaheim, CA

MS&T-Materials Science & Technology
September 17-20, 2007
COBO Hall
Detroit, MI

Chem Show
October 30-November 1, 2007
Javits Convention Center
New York, NY