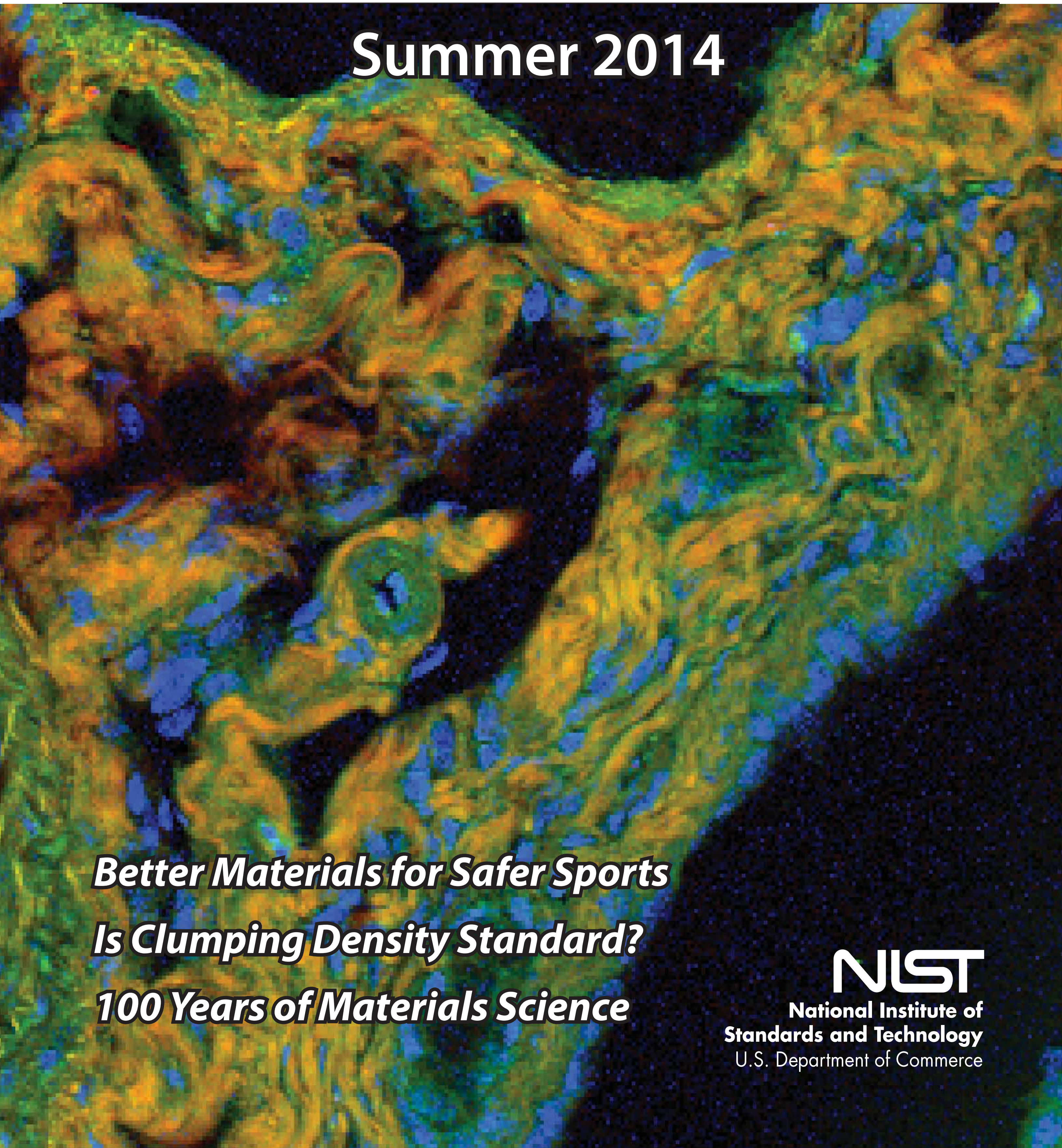
Material Matters

The Quarterly Magazine of NIST's Material Measurement Laboratory



Summer 2014

Better Materials for Safer Sports

Is Clumping Density Standard?

100 Years of Materials Science

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

About NIST's Material Measurement Laboratory

The Material Measurement Laboratory (MML) is one of two metrology laboratories within the National Institute of Standards and Technology (NIST). The laboratory supports the NIST mission by serving as the national reference laboratory for measurements in the chemical, biological and material sciences. Our activities range from fundamental and applied research on the composition, structure and properties of industrial, biological and environmental materials and processes, to the development and dissemination of tools including reference measurement procedures, certified reference materials, critically evaluated data, and best practice guides that help assure measurement quality. Our research and measurement services support areas of national importance, such as:

- Advanced materials, from nanomaterials to structural steels to complex fluids
- Energy, from characterization and performance of fossil and alternative fuels to next-generation renewable sources of energy
- The environment, from the measurement of automotive exhaust emissions and other pollutants to assessment of climate change and the health and safety aspects of man-made nanomaterials
- Food safety and nutrition, from contaminant monitoring to ensuring the accuracy of nutrition labels
- Health care, from clinical diagnostics to tissue engineering and more efficient manufacturing of biologic drugs
- Infrastructure, from assessing the country's aging bridges and pipelines to the quality of our drinking water
- Manufacturing, from lightweight alloys for fuel-efficient automobiles to biomanufacturing, advanced electronics, and data for chemical manufacturing
- Safety, security and forensics, from gunshot and explosive residue detection, to ensuring the performance of body armor materials, to DNA-based human identity testing

The Material Measurement Laboratory also coordinates the NIST-wide Standard Reference Materials® (SRM) and Standard Reference Data programs, which include production, documentation, inventory, marketing, distribution and customer service.

The Material Measurement Laboratory is home to more than 900 staff members and visiting scientists at six locations:

- NIST main campus in Gaithersburg, MD
- NIST Boulder Laboratories in Boulder, CO
- Hollings Marine Laboratory in Charleston, SC , where NIST staff work side-by-side with scientists from NOAA, the South Carolina Department of Natural Resources, the College of Charleston, and the Medical University of South Carolina to provide the science, biotechnology and standards needed to understand links between environmental conditions and the health of marine organisms and humans
- Institute for Bioscience and Biotechnology Research (formerly CARB) in Rockville, MD, where scientists from NIST, the University of Maryland College Park, and the University of Maryland School of Medicine conduct research on measurement science and standards issues associated with advanced therapeutics
- Brookhaven National Laboratory in Upton, NY where, in partnership with the Department of Energy, the laboratory has a user facility that enables researchers from industry, academia and other government agencies to apply synchrotron-based x-ray spectroscopy techniques to the development of products like oil additives and next-generation electronics
- The Advances in Biological and Medical Measurement Science (ABMS) Program at Stanford University in Palo Alto, CA, where NIST staff are working elbow-to-elbow with Stanford faculty groups and commercial affiliates to develop standards and tools that enable translation of innovations in quantitative biology and engineered biology to clinical and commercial practice

Cover image - A false-color, broadband, coherent anti-Stokes Raman scattering (BCARS) image of mouse liver tissue picks out cell nuclei in blue, collagen in orange and proteins in green. The image shows an area about 200 micrometers across. Credit: Camp/NIST

A Message from the MML Director

One of NIST's most important roles is serving as "Industry's National Laboratory." As NIST's national reference laboratory for measurements in the chemical, biological, and material sciences, MML works across a wide range of fields to help American industry grow. As the 2014 Fiscal Year comes to a close, I would like to highlight a few ways MML has done so over the past year.

Laurie Locascio, Ph.D.
Director, Material Measurement Laboratory
NIST

Our programs in bio- and nano-manufacturing, along with our role in the White House-led Materials Genome Initiative have all made significant contributions to manufacturing across a wide range of sectors. As Co-Chair of the National Science and Technology Council Subcommittee on the Materials Genome Initiative, I was particularly pleased that we were able to announce a $25 million multi-stakeholder NIST Center of Excellence, focusing on the development of industrially ready advanced materials in emerging fields such as organic photovoltaic materials, advanced ceramics, and novel polymer and metal alloys for structural applications. MML's biomanufacturing program supports the U.S. biopharmaceutical industry delivery of high quality and low cost protein drugs around the world. This year, MML and their collaborators from industry, academia, and regulatory agencies have exhaustively characterized a recombinant IgG1κ monoclonal antibody reference material that is critical for advancing bioanalytical system suitability and method validation as well as developing and evaluating novel technology. And MML researchers supported the growing nanomanufacturing sector with tests that explored the safety of modern plastics incorporating multiwalled carbon nanotubes - a special form of carbon with a unique potential for use in a wide range of applications.

MML's goal of advancing measurement science, standards, and technology in the biological, chemical, and materials sciences helps to provide critical support for American manufacturing. As you will read in this issue of *Material Matters*, MML works to solve manufacturing related problems across a wide range of disciplines, including medicine, electronics, and forensics. This concern for finding scientific solutions to real world problems, grounded in a fundamental commitment to supporting American trade and industry, is big part of what drives us to keep doing what we do.

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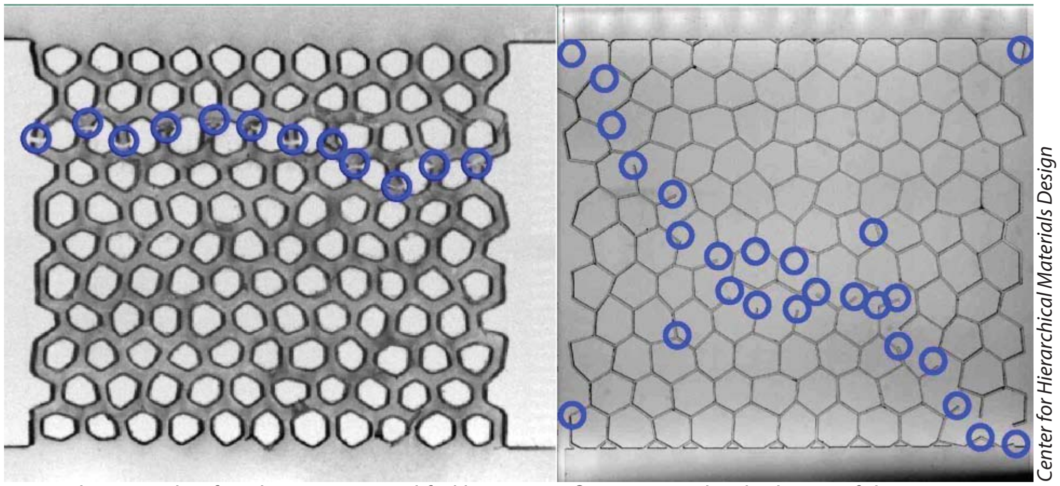


A simple example of making a material fail better : By fine-tuning the thickness of the connecting spokes in a sheet of acrylic, we can change how it transmits force when fractured. With thick spokes (left), fractures propagate in a straight line and concentrate the impact. Thin spokes (right) divert the fracture across the sheet, diffusing the impact.

Better Materials for Safer Sports: Time to Use Our Heads

Note: A version of this story previously appeared on the U.S. Department of Commerce blog (www.commerce.gov/blog) on May 30, 2014.

Earlier this year, at the White House Healthy Kids and Safe Sports Concussion Summit, President Obama highlighted both the need for greater national awareness of the risks our young athletes face from traumatic brain injuries and the need for increased research on how to combat these potentially life-altering injuries.

In 2009, for example, the Centers for Disease Control and Prevention, emergency departments in the United States treated more than 250,000 sports- and recreation-related traumatic brain injuries, including concussions, among children and adolescents—a figure that's risen by 60 percent in the past decade.

At NIST, we recognize that the use of advanced materials in protective equipment, such as helmets, can play a critical role in this effort. For that reason, NIST is investing $1 million per year for 5 years on tools to accelerate the development of advanced materials that can provide better protection against concussions for the athlete.

Sports equipment often leads the way in adopting new advances in materials—think of carbon nanotubes in high-end tennis rackets and golf clubs. But modern materials science offers the possibility of specifically designing new materials, from the ground up, that are tailored to the special needs of helmets and other protective equipment.

As an example, "shear-thickening suspensions"—specially designed particles suspended in a liquid polymer—can be a high-tech shock absorber that instantly adapts to offer greater resistance to stronger shocks. You've encountered a sheer-thickening suspension if you've ever tried to stir cornstarch in water quickly.

Other possibilities include micro- or nanostructured materials that either absorb shocks by crumpling in specific ways, rather like some automobile components are designed to protect passengers in a crash, or that selectively deform to channel the energy of shocks away from highly sensitive areas, like the skull. Self-healing polymers and shape-memory metal alloys can both provide reinforcement and extend the longevity of the equipment.

All of these depend on concepts from the President's Materials Genome Initiative (MGI): replacing trial-and-error experimentation with physical theory, advanced computer models, vast materials properties databases and complex computations to design new materials with specific properties. NIST plans to work closely with the recently created Center for Hierarchical Materials Design, a NIST Center of Excellence that was established specifically to pursue tools for creating custom materials.

Our principal focus will be to provide the research community with tools to advance this work: improved methods for determining material response to stress, sophisticated models and standards to support materials testing. An important part of our effort will be directed at better measurement tools—such as impact sensors that can be embedded in sports equipment—to help physicians and medical researchers better understand health effects and gear performance.

As a civilian technology effort, NIST's program will be focused on sports helmets; however, preventing traumatic brain injuries also is of great interest to the U.S. military. So we anticipate working closely with defense R&D agencies to explore how our results may be adapted to better protect our service men and women.

Sports provide tremendous positive, healthy experiences for so many of the nation's youth and adults. It's time to make sure our protective materials and equipment are just as smart as our plays.

- Laurie Locascio, Director
NIST Material Measurement Laboratory

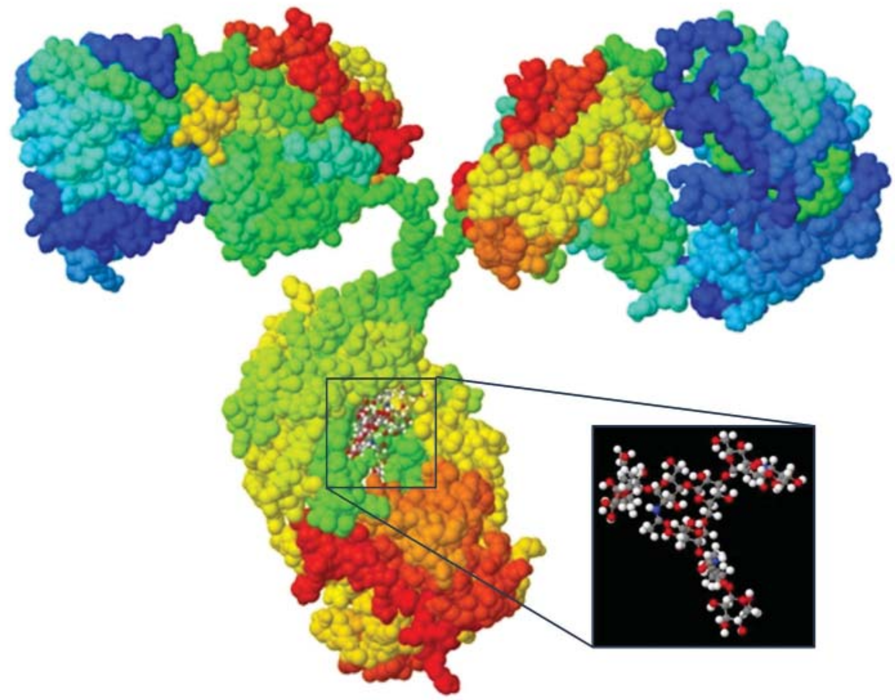
New NIST Web Tool Makes Working with Glycan Sugars a Lot Sweeter

Note: A version of this story previously appeared in NIST's TechBeat *on July 11, 2014.*

When researchers at NIST need a special tool to do their work more effectively, they often prove that necessity is truly the mother of invention. Such was the case recently for M. Lorna De Leoz and Stephen Stein, NIST MML chemists working in the growing specialization of glycomics. Glycomics is the study of the abundant, often-branched sugar chains called glycans that are attached to proteins and lipids and influence cellular processes, including immunity, protein folding and, sometimes, changes associated with cancer.

Like their fellow scientists in the glycomics field, De Leoz and Stein rely heavily on mass spectral (MS) analyses that yield "chemical fingerprints" used to characterize the mass, composition and organization of individual glycan molecules. The human body produces thousands of different glycans and, unfortunately, MS analysis is slow and laborious, involving lots of number crunching by hand. Out of their frustration with this low-tech system for calculating high-tech MS data, the NIST duo came up with a tool that automates most of the process.

Their new Glyco MS Calculator automatically determines the mass of individual glycan components and breaks them down element-by-element. Designed in a spreadsheet format, the user inputs the number of residues (the individual units that make up a polymer; in this case, the monosaccharide sugars in the polysaccharide chain) in the glycan and the program calculates the masses and elemental composition within the molecule. It also provides mass and composition for glycans that are chemically modified. Finally, the calculator generates the mass of molecules formed as byproducts of mass spectrometry so that they can be considered when interpreting the MS data.



RCSB Protein Data Bank

Immunoglobulin G (IgG) antibody molecule with glycan attached. Inset shows glycan structure.

The NIST Glyco MS Calculator may be accessed and used free-of-charge at www.nist.gov/customcf/glyco-mass-calc.

- Michael E. Newman, NIST

New White Paper Examines High Throughput Materials Science

The Materials Genome Initiative (MGI), announced by President Obama in 2011, aims to support U.S. institutions to discover, develop, and deploy advanced materials twice as fast, at reduced cost. While much progress has been made in computational design of materials, discovery, deployment, and commercialization of new materials would benefit greatly from the increased support of experimental data. High-throughput experimental techniques, where hundreds of samples are synthesized, characterized, and catalogued for their functional properties, provide an important tool toward these aims. In May of 2014 a workshop was held in San Francisco, CA, and it was attended by more than 90 experts in the field of high-throughput experimentation. On June 25, the Materials Research Society released a white paper resulting from this workshop. The workshop was co-organized by Applied Materials Inc., and the University of South Carolina with support from the National Science Foundation (NSF), the National Institute of Standards and Technology (NIST), and the Office of Science and Technology Policy (OSTP). The white paper can be accessed from this website: http://www.mrs.org/mgi-workshop-2014/

Snowballs to Soot:
The Clumping Density of Many Things Seems to Be a Standard

Note: A version of this story previously appeared in NIST's TechBeat *on June 10, 2014.*

Particles of soot floating through the air and comets hurtling through space have at least one thing in common: 0.36. That, reports a NIST research group, is the measure of how dense they will get under normal conditions, and it's a value that seems to be constant for similar aggregates across an impressively wide size range from nanometers to tens of meters.

NIST hopes the results will help in the development of future measurement standards to aid climate researchers and others who need to measure and understand the behavior of aerosols like carbon soot in the atmosphere.

Soot comes mostly from combustion and is considered the second biggest driver of global warming, according to NIST chemist Christopher Zangmeister. It is made up of small round particles of carbon about 10 or 20 nanometers across. The particles stick together randomly in short chains and clumps of a half dozen or more spheres. These, in turn, clump loosely together to form larger, loose aggregates of 10 or more which over a few hours will compact into a somewhat tighter ball which is atmospheric soot.

The interesting question for chemists studying carbon aerosols is how tight? How dense? Among other things, the answer relates to the balance of climate effects from soot: heating from light absorption versus cooling from light reflection.

The maximum packing density of objects is a classic problem in mathematics, which has been fully solved for only the simplest cases. The assumed density in models of atmospheric soot is 0.74, which is the maximum packing density of perfect spheres, such as billiard balls, in a given space. But when Zangmeister's team made measurements of the packing density of actual soot particles, the figure they got was 0.36. "We figured, man, we've got to be wrong, we're off by a factor of two," Zangmeister recalls, but "a bunch more measurements" convinced them that 0.36 was correct. Why?

Enter the summer help. Two students, one in college and one in high school, who were working with Zangmeister's group last summer were set to the task of modeling the packing question with little 6 mm plastic spheres sold for pellet guns. They glued thousands of random combinations of spheres together in clumps of from 1 to 12 spheres, and then filled every available size of graduated cylinders and hollow spheres with their assemblies, over and over, and over.

Baum/NIST

High school student Jessica Young checking the packing density of random aggregates of plastic spheres in a cylinder. Young's work as a summer intern at NIST contributed to a paper arguing that rigid aggregates like those she's testing tend to clump together at roughly the same density regardless of scale, from microscopic soot to large comets.

Their charted results, as a function of clump size, form a curve that levels off at … 0.36.

It gets better. Inspired by a book on the solar system he was reading with his son, Zangmeister checked NASA's literature. Comets are formed very much the same way as soot particles, except out of dust and ice, and they're a lot bigger. NASA's measurements on a collection of 20 comets estimate that packing density at between 0.2 and 0.4. So 0.36 may be an all-purpose value.

NIST's interest in the nature of soot particles is driven by a desire to imitate them, according to Zangmeister. "It's amazing how much uncertainty there is in optical measurements of particles in the atmosphere. The reason for this uncertainty is rooted in something really important to NIST: there are no real methods for calibrations. You can calibrate any CO2 measurement using one of our Standard Reference Materials for CO2 in air, but there's no such thing as a bottle of standard aerosol or a standard aerosol generator. That's really at the heart of what we're trying to do: make a black material that simulates carbon that you can put into an aerosol and know it will come out the same way every time. It's a real materials chemistry project."

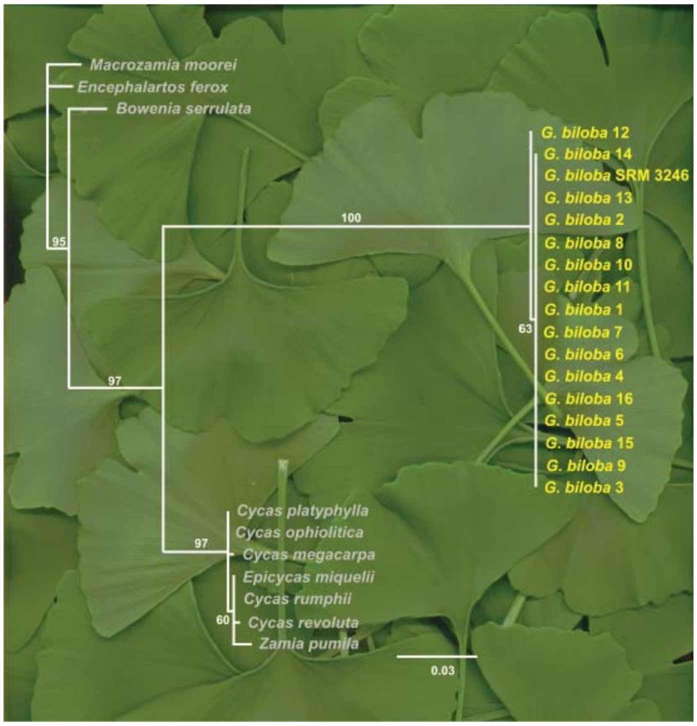
The agency is working with the National Research Council of Canada and Environment Canada on the project.

- Michael Baum, NIST

NIST MML-Developed Naming Protocol for Molecules Helps Promote Global Trade and Innovation

The chemical structure of complex molecules is difficult to convey and often the source of confusion, especially in computer databases and on the internet. NIST MML scientists have developed an international chemical identifier, or InChI, that uniquely and unambiguously defines the chemical structure of complex molecules using a string of numbers and letters. This new naming protocol has been readily adopted worldwide by chemical researchers, software developers, scientific publishers, industry, and federal agencies to exchange chemical structure information over the internet and link chemical data between diverse databases, scientific publications, patent literature, and popular news sources. The use of InChI promotes global trade and innovation by facilitating the electronic exchange of chemical information and the linking of diverse chemical data compilations. Prominent customers using InChI include 20 chemical structure software vendors; ChemSpider, a database containing more than 26 million unique molecules; NIH's PubChem, a database containing 10s of millions of chemical compounds; the journals of the Nature publishing group; and Wikipedia which lists the InChI and InChIKey for all chemical compounds it describes.

What's Really in My Herbal Supplement?



The NIST MML Chemical Sciences Division has recently added species DNA identification as a certified property to a newly released dietary supplement Standard Reference Material 3246 Ginkgo biloba (Leaves). Current Good Manufacturing Practices (cGMP), enforced by the United States Food and Drug Administration (FDA), require identity testing of dietary ingredient components to prevent accidental or intentional (economically motivated) adulteration of dietary supplement products. To directly address this issue, the DNA of the Ginkgo biloba material has been sequenced via Sanger sequencing by AuthenTechnologies, in a collaborative effect with the Chemical Sciences Division. This addition of DNA identity data to dietary supplement SRMs will have a direct impact on protecting public health as well as promoting the nation's ever-evolving dietary supplements manufacturing industry.

Each test material is compared to an "inclusivity" data set comprised of pressed plant sample (namely a herbarium voucher specimen) of the same species (or presumed species) and an "exclusivity" data set of a specimen from the same genus, but different species. The complete data set (inclusivity versus exclusivity) allows for the statistical determination of certainty of identity. This validation step is essential, as closely related species may have similarities in sections of the DNA sequence, potentially resulting in false positive identifications. Sanger sequencing on two independent chloroplast gene regions (psbA-trnH intergenic and trnL intron) was employed for the authentication of plant materials. Chloroplast DNA sequences from authenticated Ginkgo biloba samples were used to establish inclusivity; chloroplast DNA sequences from close relatives were used to establish exclusivity.

While the collection of DNA information was first envisioned to authenticate these dietary supplement materials, this data can also reveal the identification (and in relative amounts) of unwanted biological contaminants, such as mold and fungi. In addition to establishing the authenticity of NIST Standard Reference Materials, AuthenTechnologies is creating and maintaining a publically available database of DNA sequences obtained from herbarium species. Authenticity data are currently being generated for other dietary supplement SRMs and RMs, including: Actaea racemosa (Black Cohosh), Camellia sinensis (Green Tea), Hypericum perforatum (Saint John's Wort), Serenoa repens (Saw Palmetto), Trifolium pratense (Red Clover), Pueraria montana (Kudzu), Panax ginseng (Asian Ginseng), Eleutherococcus senticosus (Siberian Ginseng), Zingiber (Ginger), and Curcuma longa (Turmeric).

The erccdashboard: A New Software Package for Method Validation of Gene Expression Experiments

NIST MML has developed a new software tool enabling complete turnkey assessment of the technical performance of gene expression experiments. Evaluation of biological hypotheses with gene expression experiments is critically important for a multitude of application areas such as cancer biology, drug discovery, tissue engineering, stem cell biology, and biomanufacturing. Irreconcilable and irreproducible gene expression measurement results spurred industry leaders to approach NIST to host the External RNA Control Consortium (ERCC) to build metrology products that would enable confidence in gene expression measurement results.

The new erccdashboard method validation tool produces performance measures with ERCC RNA spike-in controls derived from NIST SRM 2374. The performance metrics are presented in an analytical "dashboard" for coherent evaluation of an experiment. These performance measures include dynamic range, biases and variability in ratio measurements, diagnostic performance (discrimination of true positive and true negative controls), and a new metric: the limit of detection of ratios.

The erccdashboard software package is written in the "R" statistical computing language, to support automated, consistent, reproducible method validation of gene expression experiments. R packages are open source, freely accessible, and can be rapidly installed from online code repositories such as Bioconductor and GitHub. The erccdashboard GitHub source code includes guidance on package installation and a user manual describing analysis of example data provided in the package.

Standard analysis of the ERCC controls with the erccdashboard package allows scientists to confidently assess technical performance of any gene expression experiment on any measurement platform as well as to establish the reproducibility of gene expression experiments across space and time.

NIST at the Institute for Bioscience & Biotechnology Research

Objective

The mission of IBBR is to: (1) leverage collective research strengths of the partnering institutions in bioscience, medical and measurement sciences and engineering; (2) develop integrated, cross-disciplinary teams to advance scientific discovery, technology development, measurement science and standards, and education; (3) support the biotechnology economy of Maryland and the Nation through translational research and measurements and technology transfer.

University of Maryland
College Park

College of Computer, Mathematical and Natural Sciences

A. James Clark School of Engineering

College of Agriculture and Natural Resources

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University of Maryland Baltimore

School of Medicine

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National Institute of Standards and Technology

Materials Measurement Laboratory (MML)

NIST Center for Neutron Research (NCNR)

Sectors and Partnership

The Institute for Bioscience and Biotechnology Research (IBBR), a collaboration among the University of Maryland College Park (UMCP), the University of Maryland Baltimore (UMB) and the National Institutes of Standards and Technology (NIST), focuses on fundamental measurement problems in structural biology, biophysics, bio-engineering and computational biology to support innovation in biotechnology. NIST research themes that benefit from strong interactions across IBBR include:

- Biomanufacturing
- Healthcare & Diagnostics
- Complex Drug Discovery & Development

Capabilities

IBBR operates a suite of facilities that support state of the art measurement technologies for both basic and translational research.

- NIST/UMD Biomolecular Labeling Laboratory (BL^2)
- W.M. Keck/NIST/UMD Laboratories for Structural Biology
    - Biomolecular NMR Facility
    - Macromolecular X-ray Crystallography Facility
- NIST/UMD Mass Spectroscopy Laboratory
- MTECH Biopharmaceutical Advancement Facility (BAF)

Projects

Precision Protein Structure Determination:

Macromolecular X-ray Crystallography and Biomolecular Nuclear Magnetic Resonance

We provide accurate measurements of the diffracted X-rays from crystals and from the nuclear magnetic resonance (NMR) spectra of biological macromolecules which allow their structures to be determined at high resolution. Knowledge of the atomic structure of biologically important macromolecules and their complexes facilitates understanding of function and how that function can be altered for biotechnology applications or targeted in drug development.

Figure 1. American Recovery and Reinvestment Act funded 900 MHz NMR

Biomolecular Labeling Lab (BL2)

Stable-Isotope Labeling to support Physical and Analytical Methods Applied to Biomolecules

We provide users (IBBR, NIST, and external researchers from academia and industry) the infrastructure, equipment, and expertise to produce stable isotope-labeled biomolecules, to advance bioanalytical and biophysical measurements (e.g. mass spectroscopy, NMR and neutrons).

Figure 2. Biomolecular Labeling Lab

Biomanufacturing:

Higher-order Structure Assessment of Protein Biologics

We develop validated measurement methods and standards for analysis of the higher-order structure of protein biologics. These tools allow comparison of the structural "sameness" of biologics (process monitoring) and between innovators and biosimilars, with an overarching goal of correlating higher-order protein structure and dynamics with drug function, efficacy and safety.

Neutron analysis for Biological Research:

Measurements for Integral Membrane Protein Structure and Function

Together with our collaborators in JILA and NCNR, we develop new measurement methods based upon cold neutron scattering, ultra-stable atomic force microscopy (US-AFM), and ultra-high field nuclear magnetic resonance (UHF-NMR) for membrane proteins that have been functionally reconstituted in "native-like" model membranes.

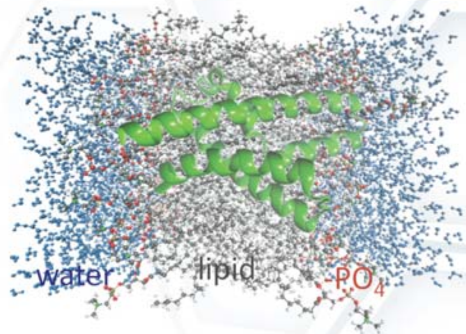

Figure 4. Neutron Diffraction used to characterize membrane proteins in model membrane bilayers.

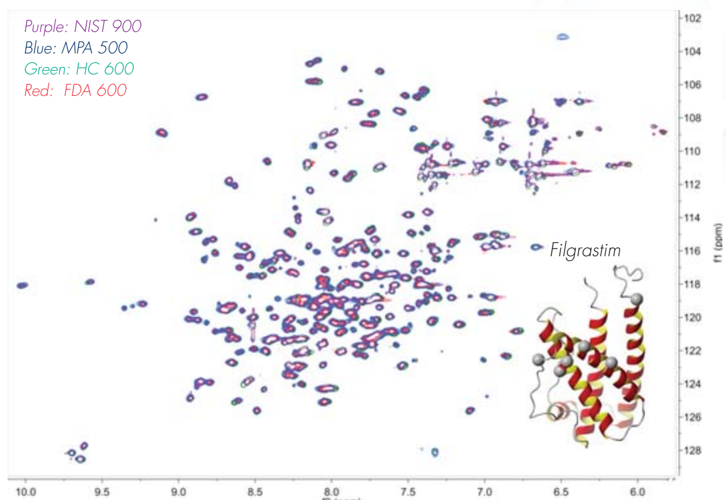

Figure 3. Overlay of the NMR spectral fingerprints of Filgrastim from the labs involved in an interlaboratory study.

Learn More

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http://www.nist.gov/mml/bmd/

55h_bio-4/13

Impacts

Precision measurement methods for the structure and dynamics of biomolecules yield reference data for improved modeling that aids drug development, supports development and regulatory approval of complex biological drugs and accelerates innovation in bioengineering and biotechnology.

Biomolecular measurements broadly support new NIST programs in the areas of Bioscience and Health e.g., protein measurements, data and standards for biologics and diagnostics.

The scientific outputs and BL² facility established through our partnership with the NCNR, a unique national user facility, excite the NCNR user community, expand its user base, and broaden the range of applications in the area of biological samples and material.

NIST MML Analytic Technique Offers Arson Investigators Faster, More Accurate Results


Burrus/NIST

Shown here are the tools for testing arson samples using NIST's new PLOT capillary method. Clockwise from top are a test chimney with burned sample of Douglas Fir (used in test burns), paint can equipped with a septum cap, test sample of Douglas Fir, scraper, collection tray, fire debris samples, then at the bottom a PLOT capillary and two containers used with the capillary.

Note: A version of this story previously appeared in NIST's TechBeat *on June 17, 2014.*

A NIST MML research group has demonstrated a new method for detecting ignitable liquids that could change the way arson fires are investigated. The new process for analyzing debris for traces of fire accelerants is faster and more accurate than conventional methods and produces less waste.

An arson investigation typically requires collecting one or two liters of ashes and debris from various locations within a fire scene in metal cans similar to those used for paint, and sending the material to a lab. The testing methods typically include gas and liquid chromatography or various versions of spectroscopy, with gas chromatography being the most widely used in fire debris analysis, according to the lead NIST researcher, Tom Bruno.

When the fire debris is received at the testing facility, samples are taken for testing. Sometimes this will involve suspending a strip with activated charcoal in the air or "headspace" directly above the sample in the paint can for a period of time that can vary, depending on the judgment of the analyst, for 2-3 hours or up to 16 hours.

Other testing methods include "dynamic purge and trap" of the headspace. And still another sampling method involves a newer solid-phase microextraction method (SPME) that does not destroy the sample. This later method, however, has a high displacement rate of heavier over lighter ignitable liquid components, is difficult to automate, makes preserving and archiving samples difficult and has not shown a consistent ability to obtain repeatable and quantitative results. Also, the SPME sampling method requires expensive equipment, and the SPME fibers are easily damaged. Still other methods are less sensitive and produce large amounts of chemical waste.

The vapor collection method developed by Bruno's group involves the dynamic adsorption of headspace vapors on short porous layer open tubular (PLOT) columns maintained at low temperature (as low as -40 C). The benefits of this method are many. The collection sensitivity is high; below 1 part per billion (ppb). The low temperature is achieved using a vortex tube connected to compressed air; it has no moving parts, and is attractive for use in environments with explosive or flammable materials.

After vapor collection, the PLOT capillaries can be heated (up to 160 C, again with the vortex tube), releasing the vapor. The capillaries used are robust and cheap, and this process is especially effective with relatively nonvolatile substances because of its wide operating temperature range. It also is not limited to water-borne samples, as most commercial sampling instruments are. And best of all, this PLOT-cryo method can be used to simultaneously test for up to eight different ignitable liquids from a single sample. This allows investigators to take multiple samples from each of several locations in a fire scene (such as a grid approach) in a short amount of time. This method also enables high repeatability and quality assurance of the testing process and is available in a portable unit that can perform the sampling in remote locations.

"This sampling method is faster, more efficient, recovers more analytics and produces much less waste than traditional methods," Bruno said. "And the sampling device and its components are much cheaper than traditional equipment." While the present study involved samples measured in the laboratory, Bruno has further developed the method to be field portable. A patent is pending for a device that will offer these same vapor collections, even at fire scenes. The self-contained portable unit is carried in a standard briefcase and may be available to arson investigators in as little as two years.

- James Burress, NIST

NIST Celebrates 100 Years of Materials Science

On May 20, 2014, NIST's Materials Science and Engineering Division (MSED) celebrated its rich history of materials science research, including 100 years of metallurgy in the Metallurgy Division and 100 years of polymer science, including 50 years in the Polymers Division. That research has prevented bridge collapses; ended a rash of thousands of early-20th-century train wrecks, developed the science of precipitation hardening; preserved the original copies of the Declaration of Independence and the Constitution of the United States; created science-based dental fillings and adhesives; quantified the molecular structure of today's polymers; generated the model necessary to understand the complex performance of synthetic rubber and the processes underlying food processing, the mixing of paints and the fabrication of plastic objects; elucidated the structure and behavior of polymer blends (alloys); discovered reasons for the World Trade Center collapse; uncovered quasicrystals; prepared the first thin film high temperature superconductors; pioneered nanocomposite magnetic refrigerants; and much more.

The one-day event was composed of a series of six presentations by previous and current NIST employees, outlining just some of the significant breakthroughs and milestones that occurred in the past 100 years in these disciplines at the National Bureau of Standards (NBS) and its successor NIST. These and many other milestones were documented in two 6 m long timelines, one for each discipline, taped across the windows where the symposium was held. The symposium was opened by Dr. Willie May, the NIST Associate Director for Laboratory Programs at the time. Eric Lin, Chief of the Materials Science and Engineering Division, followed by announcing the creation of a Centennial Collection in the NIST Digital Library (http://cdm16009.contentdm.oclc.org/cdm/landingpage/collection/p16009coll44) and setting the tone for the upcoming 6 talks. These technical talks were given by Professor Isaac Sanchez (UT-Austin), Dr. Richard Fields (NIST, retired), Dr. Frank Gayle (NIST), Professor Gregory McKenna (Texas Tech), Dr. Robert Shull (NIST Fellow), and Dr. Freddy Khoury (NIST, retired).

Following the symposium, a series of tours of laboratories in MSED were conducted, along with a poster display of division projects in NIST's Hall of Flags. A highlight of the evening was a presentation by Dr. Patrick Gallagher, the NIST Director at the time, to Dr. John Cahn (NIST, retired), National Medal of Science winner and Kyoto Prize recipient, of an original micrograph taken by NIST scientist Dr. Leonid Bendersky and signed by the assembled participants. This was followed by a short talk by John in which he spoke about how beneficial it was to him to have worked in the collaborative environment of NBS/NIST amongst so many other wonderful scientists.


Gale Holmes, NIST

Presentation of Micrograph to John Cahn by Patrick Gallagher at the evening reception.

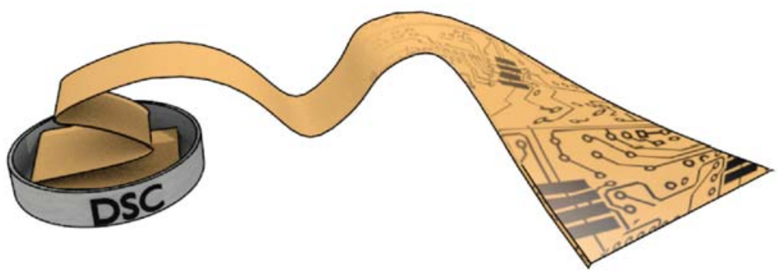
The Centennial was sponsored by the American Society of Metals International (ASM), The Minerals, Metals and Materials Society (TMS), the American Chemical Society's (ACS) Polymer Chemistry Division (POLY), and the ACS Polymeric Materials: Science and Engineering Division (PMSE).

New NIST-Developed Standard Improves Explosive Trace Detection

Note: A version of this story previously appeared in the May/June 2014 issue of ASTM's Standardization News.

Trace explosive detector systems are used at airports, embassies, federal buildings, military checkpoints and many other places to protect the public. Since these security networks involve multiple detection systems from multiple manufacturers, focus on various threat compounds, and at times cross international borders, these same manufacturers and agencies need to know how detection systems compare. A statistically robust and consistent method for comparing different trace detector performance metrics was needed that would be applicable across the variety of technologies used for trace explosives detection, applicable to any particular analyte and any detector in the field, and work in the presence of background material challenges. In response to this need, NIST scientists serving as members of ASTM International Committee E54 on Homeland Security Applications have developed a new ASTM International standard to quantitatively test the ability of a trace detector to reliably sense and identify very small amounts of explosives. The standard will be used by manufacturers, vendors, testers and users of explosive trace detectors to determine limits of detection, which are typically defined as the smallest signals that may be reliably discerned above variations in background and attributed to particular analytes with a certain level of confidence. Reliable values for limits of detection give security planners the ability to make informed decisions about the most appropriate systems to procure, whether a system is performing within certain requirements and whether a system is capable of detecting an amount of a particular compound expected from a sample protocol used on test residues.

NIST MML Researchers Develop a Framework for Quantifying Crystallinity in Semiconducting Polymers for Flexible Electronics



Researchers in NIST MML's Materials Science and Engineering Division, in collaboration with researchers from the University of Washington Seattle, have developed methods to quantify the degree of crystallinity in polymeric semiconductors such as those used for flexible electronics applications. This research attempts to address one of the most critical challenges in organic flexible electronics: characterizing the link between molecular order and ultimate device performance. While many metrics for order exist in these systems, quantitative metrics for the degree of crystallinity were lacking. This work demonstrates a framework for quantifying crystallinity in these systems.

Poly(3-hexylthiophene) (P3HT) is the most widely studied semiconducting polymer. Unfortunately, over the 20+ years it has been studied, truly quantitative measures of its crystalline fraction have been lacking. Typically, X-ray diffraction has been used to determine the degree of crystallinity in semicrystalline polymers; however, because of P3HT's weak reflections, the inability to create 0 % and 100 % crystalline fraction reference samples, and other problems associated with its nanoscale paracrystalline-like structure, it has been extremely difficult in the case of P3HT. Calorimetric measurements have been used in the past to report the degree of crystallinity in P3HT using an estimate for the enthalpy of fusion per crystalline repeat unit, but studies in recent years have strongly suggested that this estimate for the enthalpy of fusion value might be in error by up to 300 %.

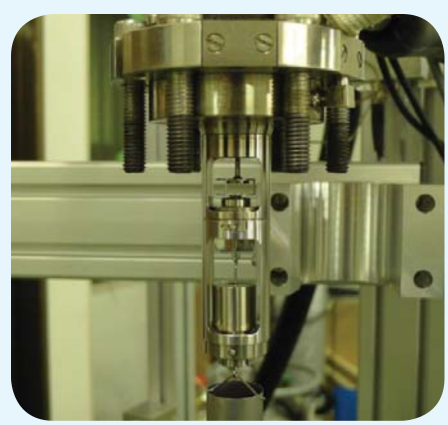
In work recently described in Macromolecules (dx.doi.org/10.1021/ma500136d), a combination of solid-state 13C nuclear magnetic resonance (NMR) spectroscopy and differential scanning calorimetry (DSC) measurements on a series of well-characterized P3HT fractions of varying molar mass were combined to yield a precisely determined value for the enthalpy of fusion for P3HT and further demonstrated that because of finite crystal size effects, a value for the equilibrium melting temperature, which is also reported in the Macromolecules article, is necessary for calculating crystallinity via DSC.

For further information on this topic, a PDF of the article can be downloaded from the journal website at dx.doi.org/10.1021/ma500136d.

NIST Partners with DOE's ARPA-E on New Gas Sorption Facility

Sorbent materials are candidates for many industrial sustainable development applications, including hydrogen and methane storage, gas separation, catalysis, methane conversion, and natural gas purification. While great advances continue to be made, the sorbent development community currently lacks the proper resources to accelerate materials innovation due the significant effect of slight variations in sample activation and measurement protocols, which makes reproducibility of results very difficult. NIST has recently partnered with the U.S. Department of Energy's Advanced Research Projects Agency – Energy (ARPA-E) on a new Facility for Adsorbent Characterization and Testing ("FACT") – a state-of-the-art facility built on the NIST campus in Gaithersburg, MD, with $5M support from ARPA-E. The goal of the facility is to serve government, industry and university research communities as an independent facility for accurate and reproducible characterization of gas sorption properties of materials. FACT is equipped to support programs developing adsorbents and serves the sorbent materials research community by providing impartial testing and characterization of material sorption properties, establishing testing procedures, and disseminating sorbent material property data and measurement "best practices."

More information on FACT is available at http://www.nist.gov/mml/fact/



NIST Diffusion Workshop and CHiMaD/NIST Calphad Data Workshop

Dr. Suzana Fries, ICAMS

The twelfth annual NIST Diffusion Workshop was held April 28-29, 2014 and was attended by thirty-one researchers and software developers from industry, government laboratories, and universities. The NIST Diffusion Workshop series began in 2003 with the goal of improving communication and collaboration between theorists and experimentalists in the field of multicomponent multiphase diffusion. Recently, the workshop series has focused the needs for multicomponent diffusion for materials design applications, the measurement and calculation of diffusion data, and the application of diffusion data within a materials data process.

The first day of the workshop emphasized some of the challenges in measuring and representing different types of diffusion data. The opening presentations highlighted the complexities associated with trying to measure grain boundary diffusion associated with recrystallization, including whether the diffusivity reported is along the grain boundary or across the grain boundary. The importance of reporting composition units associated with measured diffusion coefficients was also illustrated and discussed. The workshop also highlighted the value of first principles computational diffusion simulations. First principle simulations provided new insights into the diffusivity of oxygen interstitials and substitutional solutes in Ti alloys. The power and efficiency of high throughput first principles calculations were used to develop a database of impurity diffusion related properties, including vacancy migration energies. The database then allowed qualitative relationships to be determined between the diffusion behavior and other material properties, such as lattice constants.

The second day of the Diffusion Workshop was held in conjunction with the opening of the NIST/CHiMaD Calphad Data Workshop. This joint session discussed the requirements for building a diffusion genome and the development of materials informatics and data curation tools. Thirty-two researchers and software developers from industry, government laboratories, and universities attended the NIST/CHiMaD Calphad Data Workshop that was held on April 29-30th. The participants focused on what data and meta-data are needed to develop a Calphad proto database to improve the efficiency of Calphad assessments. The group discussed a variety topics related to the structure and content of these data repositories, including phase names, data uncertainty, federation of data systems, and data sharing initiatives. The participants agreed to follow-up actions to encourage data curation and sharing.

Institute for Systems Biology Enlists NIST Support on Landmark Study

In the 100K Project, the Institute for Systems Biology (ISB) will recruit 100,000 individuals into a unique longitudinal study that will enable the discovery of disease transitions for the most common human diseases. For some diseases, including type II diabetes and selected osteoporosis, early disease can in fact be reversed, and in other cases the Institute hopes to learn how this might be done in the course of the study. The Hundred Person Wellness Project (Pioneer 100) was initiated to begin this transformative process. ISB recently met with NIST MML scientists to discuss a role for NIST in developing the standards and technology necessary for this landmark study. Topics of discussion included challenges of developing standards that could serve as references for many biomarkers, as well as challenges involving maintaining, handling, and integrating all the data associated with the studies. NIST and ISB will be working together in this metrology test bed to provide improved confidence in the measurements of this landmark study designed to improve the global health.

Institute for Systems Biology
Revolutionizing Science. Enhancing Life.

Senator Whitehouse Visits Hollings Marine Laboratory

On April 22, 2014, Senator Sheldon Whitehouse (RI), member of the Senate Environment and Public Works Committee and Chairman of the Subcommittee on Clean Air and Nuclear Safety, visited the Hollings Marine Lab in Charleston, SC to learn about research related to climate change. NIST MML has several programs related to marine health and the monitoring of climate change including banking of bird, coral, sea turtle, and marine mammal specimens, and health assessment of marine animals. These specimens enable scientists to monitor changes in the marine environment over time and to better understand the effects of climate change on marine health.

Recent NIST MML Awardees

Schiel Honored with 2014 Nebraska Alumni Early Achiever Award

John Schiel, from NIST MML's Biomolecular Measurement Division was awarded the 2014 Nebraska Alumni Early Achiever Award. Established in 2011, the Early Achiever Award gives each UNL college the opportunity to acknowledge one of their outstanding young graduates and receive recognition at the Alumni Awards Banquet.

NIST Researchers Honored with Prestigious Mike Lynch Award

Researchers from the NIST Mass Spectrometry Data Center were awarded the Chemical Structure Association Trust Mike Lynch Award at the 10th International Conference on Chemical Structures in Noordwijkerhout, The Netherlands. The NIST researchers honored were Stephen E. Stein, Dmitrii V. Tchekhovskoi, and NIST associate Stephen R. Heller. The group is recognized for their outstanding work on the development and dissemination of the InChI (IUPAC International Chemical Identifier) standard.

Scanlan Wins Prize at the 7th International Nanotoxicology Congress

Leona Scanlan, NRC Postdoctoral Fellow in the NIST MML Biomolecular Measurement Division, working with Miral Dizdar won the Best Poster Presentation Award in the Ecotoxicology / Systems Biology (Omics) / Exposure Assessment and Life-cycle Analysis Session at the 7th International Congress on Nanotoxicology, on April 26 in Antalya, Turkey.

Vreeland named to the 2014-2015 Embassy Science Fellows Program

Wyatt Vreeland, of NIST MML's Biomolecular Measurement Division, was recently named to the 2014-2015 Embassy Science Fellows Program at the U.S. Embassy Prague, Czech Republic. Vreeland will be fostering ties and communication between the biotechnology industries in the Czech Republic and the U.S.

DelRio Receives A.J. Durelli Award from Society of Experimental Mechanics

Frank DelRio of NIST MML's Applied Chemicals and Materials Division was recently named the 2015 recipient of the A.J. Durelli award from the Society of Experimental Mechanics (SEM). This award recognizes young professionals for their early-career research contributions.

Beers Selected as 2014 ACS Fellow

Kathryn Beers, NIST MML Group Leader for Polymers and Complex Fluids was recently selected as a 2014 Fellow by the American Chemical Society. Beers was recognized for conducting research in measurements and methods for advanced polymeric materials, including high throughput and microreactor methods, and polymer brush and (co)polymerization metrology.

Selected Recent Publications

MML researchers publish over 400 journal articles each year. Here are a few recent examples:

A. R. Laesecke, J. L. Burger, "Viscosity Measurements of DNA Solutions with and without Condensing Agents" Biorheology, Vol. 51, pp. 15-28, (07-Jul-2014) (PubID: 914036)

B. D. Ravel, W. Klysubun, G.L. Carr, C. Hauzenberger, "The red color in 19th century Thai glass studied by X-ray and optical spectroscopy" Spectrochimica Acta, Part B, (02-Jul-2014) (PubID: 914254)

R. C. Tung, J. P. Killgore, D. C. Hurley, "Liquid Contact Resonance Atomic Force Microscopy Via Experimental Reconstruction of the Hydrodynamic Function" Journal of Applied Physics, Vol. 115, 8 pp., (11-Jun-2014) (PubID: 915745)

E. Michailidou, M. J. Assael, M. L. Huber, I. M. Abdulagatov, R. A. Perkins, "Reference Correlation of the Viscosity of n-Heptane from the Triple Point to 600 K and up to 248 MPa" Journal of Physical and Chemical Reference Data, Vol. 43, No. 2, pp. 023103-1-023103-13, (05-Jun-2014) (PubID: 915635)

C. R. Snyder, R. C. Nieuwendaal, D. M. DeLongchamp, C. K. Luscombe, P. Sista, S. D. Boyd, "Quantifying Crystallinity in High Molar Mass Poly(3-hexylthiophene)" Macromolecules, Vol. 47, No. 12, pp. 3942-3950, (03-Jun-2014) (PubID: 915163)

J. L. Staymates, S. Orandi, M. E. Staymates, J. G. Gillen, "Method for combined biometric and chemical analysis of human fingerprints" Forensic Science International, Vol. 17, No. 2, pp. 69-72, (02-Jun-2014) (PubID: 913415)

G. Stan, R. S. Gates, "Intermittent contact resonance atomic force microscopy" Nanotechnology, (23-May-2014) (PubID: 914896)

T. P. Forbes, E. R. Sisco, "Chemical Imaging of Artificial Fingerprints by Desorption Electro-Flow Focusing Ionization Mass Spectrometry" Analytical Chemistry, Vol. 139, No. 12, pp. 2982-2985, (19-May-2014) (PubID: 915130)

M. J. Bachman, J. M. Keller, K. West, B. Jensen, "Persistent organic pollutant concentrations in blubber of 16 species of cetaceans stranded in the Pacific Islands from 1997 through 2011" Science of the Total Environment, Vol. 488-489, pp. 115-123, (11-May-2014) (PubID: 914466)

A. Galkin, L. Kulakova, K. Lim, C. Chen, W. Zhang, I. V. Turko, O. Herzberg, "Structural Basis for Inactivation of Giardia lamblia Carbamate Kinase by Disulfiram" The Journal of Biological Chemistry, Vol. 289, No. 15, pp. 10502-10509, (11-Apr-2014) (PubID: 915235)

P. Y. Hsieh, J. A. Widegren, T. J. Fortin, T. J. Bruno, "Chemical and thermophysical characterization of an algae-based hydrotreated renewable diesel fuel" Energy and Fuels, (08-Apr-2014) (PubID: 914759)

C. G. Simon Jr, S. Parekh, C. K. Tison, G. Kumar, T. M. Farooque, C. H. Camp, "Measuring Stem Cell Dimensionality in Tissue Scaffolds" Proceedings of the National Academy of Sciences of the United States of America, Vol. 35, pp. 2558-2567, (01-Apr-2014) (PubID: 913148)

Full text versions of many papers and a full list of MML publications can be accessed through the NIST Publications Database at www.nist.gov/publication-portal.cfm

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