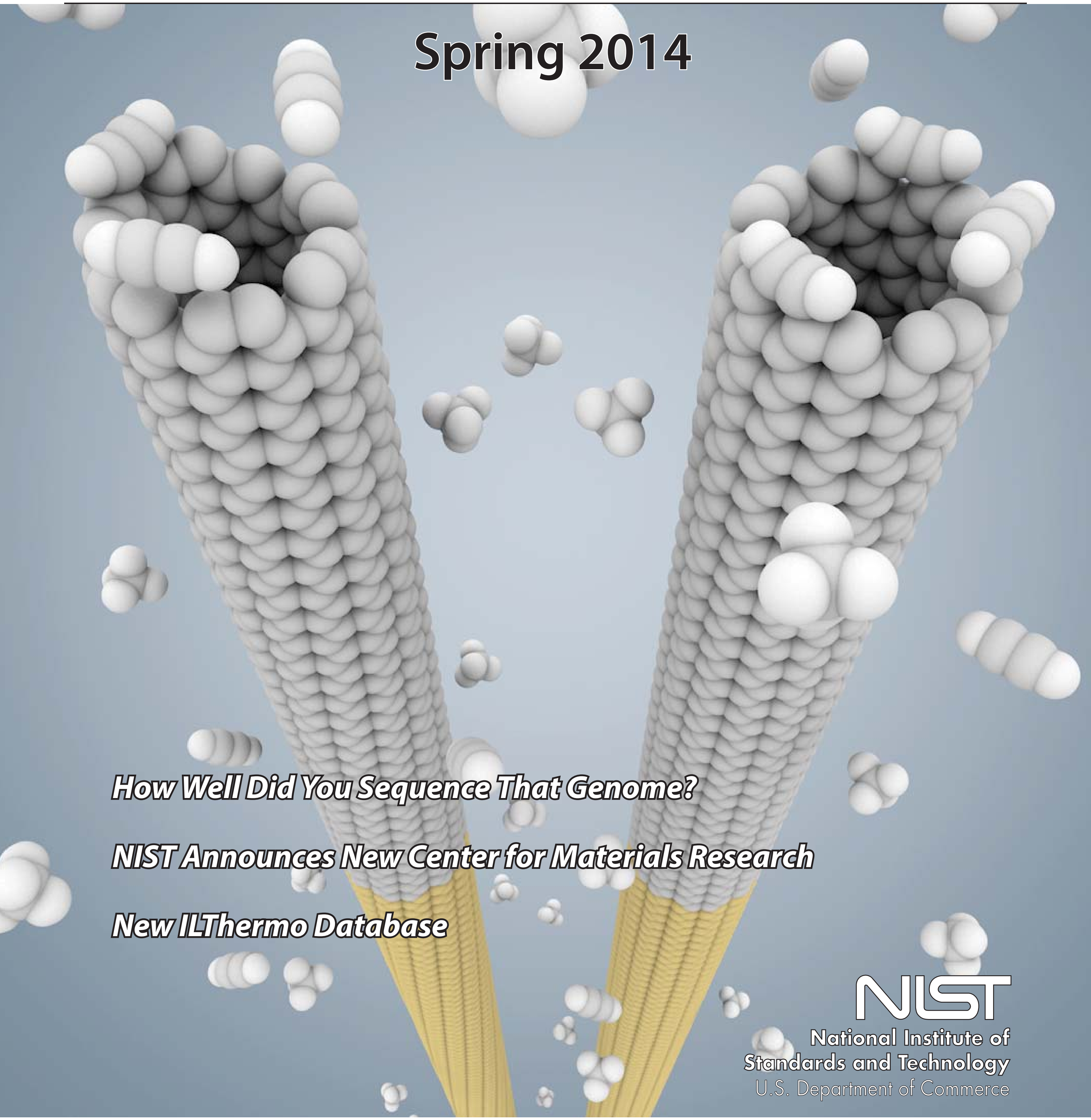
Material Matters

The Quarterly Magazine of NIST's Material Measurement Laboratory

Spring 2014



How Well Did You Sequence That Genome?

NIST Announces New Center for Materials Research

New ILThermo Database

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 - Computer Model of Carbon Nanotubes - courtesy of the University of Southern California

A Message from the MML Director

Materials matter. From the water we drink to the cars we drive, we truly live in a material world. Understanding materials and their potential depends upon our knowledge of their basic properties. Scientific measurement is the only route to this knowledge, and is fundamental to a huge number of endeavors, from global trade to modern medicine. Our ability to diagnose and cure diseases, protect the environment, and improve manufacturing productivity and economic competitiveness depends on accurate methods and reliable standards to support measurement of a broad range of materials from structural and functional materials to biological materials and chemical substances.

The Material Measurement Laboratory (MML) of the National Institute of Standards and Technology (NIST) supports these goals by advancing measurement science, standards, and technology in the biological, chemical and materials sciences. NIST's role as the U.S. National Metrology Institute means that we work towards global harmonization and traceability to the International System of Units, and support measurement quality for confidence in measurements performed in the U.S. NIST also plays a role as "Industry's National Laboratory," and in that capacity we work closely with our industry partners to help maintain U.S. leadership across a broad range of sectors. And as a part of the U.S. Department of Commerce, we develop standards to support the U.S. in international trade and business.

I am pleased to introduce the first issue of MML's quarterly magazine, *Material Matters*. *Material Matters* offers readers a chance to learn about some of the cutting-edge measurement science and services that MML is producing. Look for a new issue every quarter to find out about the exciting and innovative work going on in MML.

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

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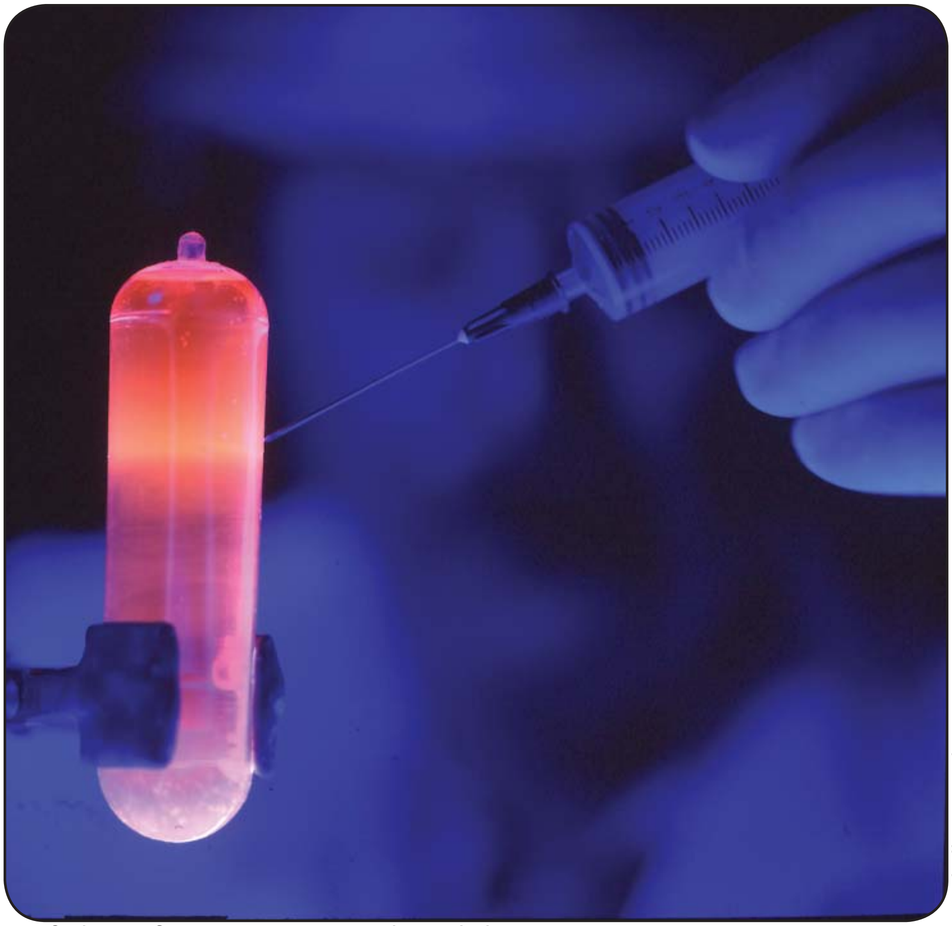


Mitchell/National Cancer Institute

Purified DNA, fluorescing orange under UV light.

'How Well Did You Sequence that Genome?' NIST MML, Consortium Partners Have Answer

Note: A version of this story previously appeared in NIST's TechBeat *on February 25, 2014*

In December 2013, the U.S. Food and Drug Administration approved the first high-throughput DNA sequencer (also known commonly as a "gene sequencer"), an instrument that allows laboratories to quickly and efficiently sequence a person's DNA for genetic testing, medical diagnoses and perhaps one day, customized drug therapies. Helping get the new device approved was another first: the initial use of a reference set of standard genotypes, or "coded blueprints" of a person's genetic traits. The standard genotypes were created by NIST and collaborators within the NIST-hosted Genome in a Bottle consortium.

"Two years ago, NIST hosted Genome in a Bottle—a group that includes stakeholders from industry, academia and the federal government—to develop reference materials that could measure the performance of equipment, reagents and mathematical algorithms used for clinical human genome sequencing," says NIST biomedical engineer Justin Zook. "Our goal is to provide well-characterized, whole genome standards that will tell a laboratory how well its sequencing process is working, sort of a 'meter stick of the genome.'"

Modern DNA sequencers take a genetic sample in the form of long strings of DNA and randomly chop the DNA into small pieces that can be individually analyzed to determine their sequence of letters from the genetic alphabet. Then, bioinformaticians apply complex mathematical algorithms to identify from which part of the genome the pieces originated. These pieces can then be compared to a defined "reference sequence" to identify where mutations have occurred in specific genes.

There are several different DNA sequencing technologies and computer algorithms to do this very complex analysis, and it's known that for any given sample, they will produce similar, but not identical results. Built-in biases as well as what are essentially "blind spots" for certain possible sequences contribute to uncertainties or errors in the sequence analysis. "These biases can lead to hundreds of thousands of differences between sequencing technologies and algorithms for the same human genome," Zook says.

In a recent paper in Nature Biotechnology,* Zook and his colleagues describe the methods used to make the Genome in a Bottle consortium's pilot set of genotype reference materials. The source DNA, known as NA12878, was taken from a single person. The reference set is essentially the first complete human genome to have been extensively sequenced and re-sequenced by multiple techniques, with the results weighted and analyzed to eliminate as much variation and error as possible.

The University of Maryland Announces New Director of the Institute for Bioscience and Biotechnology Research

The University of Maryland recently announced the appointment of Thomas R. Fuerst, Ph.D., as the new director of the Institute for Bioscience and Biotechnology Research (IBBR). IBBR is a joint research enterprise created to enhance collaboration among the University of Maryland, College Park (UMD), the University of Maryland, Baltimore (UMB), and NIST in the fields of medicine, biosciences, technology, quantitative sciences and engineering.

Under Dr. Fuerst's direction, IBBR will continue its work as an initiative of The University of Maryland: MPowering the State, a formal collaboration among University System of Maryland (USM) institutions designed to focus their collective expertise on critical statewide issues of public health, law, biomedical informatics, and bioengineering.

In his new role, Dr. Fuerst will lead IBBR in leveraging advances in innovative science and technology to facilitate the transition from basic scientific discoveries to the development of new medical products and diagnostics that address unmet medical and public health needs. Dr. Fuerst will also steer IBBR's work in the development of new analytical tools, standards and approaches for the characterization of safe and effective biological products and innovative manufacturing platforms. He will lead the institute in networking with leading academic research groups to create interdisciplinary programs, and work closely with industry partners to facilitate commercial application and economic growth.

Dr. Fuerst has a long-standing and distinguished track record as a senior biotechnology executive and former government official with more than two decades of experience in the research, development, and manufacturing of biological products, and strategic planning. He has served as a leader at the nexus of government, industry, and academia and brings extensive scientific expertise and knowledge of state-of-the-art technologies and practices used in the discovery and development of vaccines and biotherapeutic products.

"We minimized bias in our reference materials toward any specific DNA sequencing method by comparing and integrating data from 14 sequencing experiments generated by five different sequencing platforms," Zook says.

The findings in the Nature Biotechnology paper are publicly available from the Genome in a Bottle website, www.genomeinabottle.org. In addition, the Genome Comparison and Analytic Testing (GCAT) website enables real-time benchmarking of any DNA sequencing method using the paper's results. The research was conducted by a team of scientists at NIST; Harvard University; the Virginia Bioinformatics Institute at Virginia Tech University; and an Austin, Texas, genetic company, Arpeggi Inc. (now part of Gene by Gene Ltd.).

After characterizing the NA12878 pilot, samples of the DNA will be issued as a NIST Reference Material. The Genome in a Bottle consortium also plans to develop well-characterized whole genome reference materials from two genetically diverse groups: Asians and Ashkenazi Jews. Both reference sets will include sequenced genes from father-mother-child "trios" to utilize genetic links between family members.

- Michael E. Newman

For more information on the Genome in a Bottle consortium, go to www.genomeinabottle.org.

*J.M. Zook, B. Chapman, J. Wang, D. Mittelman, O. Hofmann, W. Hide and M. Salit. Integrating human sequence data sets provides a resource of benchmark SNP and indel genotype calls. Nature Biotechnology Published online Feb. 16, 2014. doi:10.1038/nbt.2835

NIST MML Cell Membrane Model Studied as Future Diagnostic Tool

Note: A version of this story previously appeared in NIST's TechBeat *on January 29, 2014*

Researchers at NIST and in Lithuania have used a NIST-developed laboratory model of a simplified cell membrane to accurately detect and measure a protein associated with a serious gynecological disease, bacterial vaginosis (BV), at extraordinarily low concentrations. The work illustrates how the artificial membrane could be used to improve disease diagnosis.

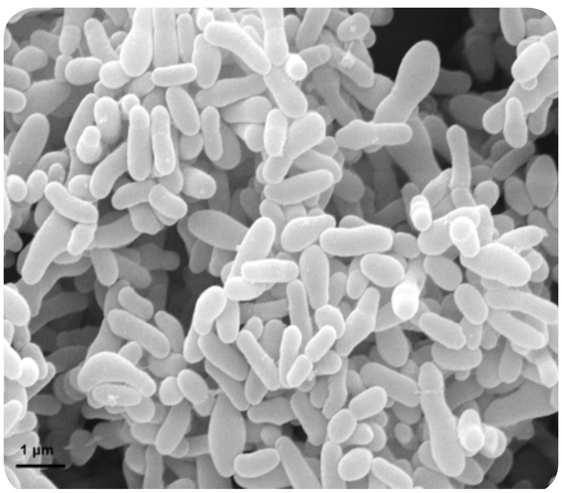
Caused by the bacteria Gardnerella vaginalis, BV is a very common health problem in women and has been linked to infertility, adverse pregnancy outcomes, post-surgery infections and increased risk for acquiring sexually transmitted diseases. Current diagnosis relies on time-consuming, labor-intensive and somewhat inconsistent laboratory cultures or immunological assays.

In a recent paper in the journal PLoS One,* researchers at NIST and Vilnius University (Vilnius, Lithuania) reported that they were able to reveal the presence of G. vaginalis by rapidly detecting and quantifying vaginolysin (VLY), a protein toxin produced exclusively by the bacteria, using the NIST model of cell membranes known as a tethered bilayer lipid membrane (tBLM).

The NIST tBLM is a two-layer sheet of simple lipid molecules analogous to the more complex structures that form the outer shell of animal cells. The membrane is anchored to a substrate with molecular "tethers" that allow it to be surrounded, top and bottom, by typical cellular fluids. Researchers can use the model to study how various factors, such as proteins, affect the integrity of the lipid membrane.

In nature, the protein VLY binds to cholesterol-containing membranes and forms pores in the structure, causing the cell to burst open and die. The researchers prepared a molecular fishing line by baiting their laboratory membrane with cholesterol in concentrations ranging from 0 percent (serving as the control) to 40 percent. VLY proteins hooked by the cholesterol obligingly created pores in the test membranes, which in turn altered the electrochemical behavior of the membranes in a way that could be detected in real time by a sensitive technique called electrochemical impedance spectroscopy (EIS).

The researchers found that they could detect the presence of VLY down to 28 nanograms (billionths of a gram) per milliliter, a four-fold improvement over antibody detection methods now in use. The speed of detection also is faster, with the tBLM-EIS system yielding results in hours rather than days. Additionally, different G. vaginalis strains produce different amounts of VLY, so in many cases, the corresponding EIS readings can help define the specific type of bacteria present in an infection.



K.K. Jefferson/Virginia Commonwealth University

Scanning electron micrograph of Gardnerella vaginalis bacteria.

Now that they have proven the viability of the tBLM-EIS detection system, the researchers plan to begin tests on clinical samples early this year.

- Michael E. Newman

*R. Budvytyte, M. Pleckaityte, A. Zvirbliene, D.J. Vanderah and G. Valincius. Reconstitution of cholesterol-dependent vaginolysin into tethered phospholipid bilayers: implications for bioanalysis. PLos One (December 2013), DOI:10.1371/journal.pone.0082536

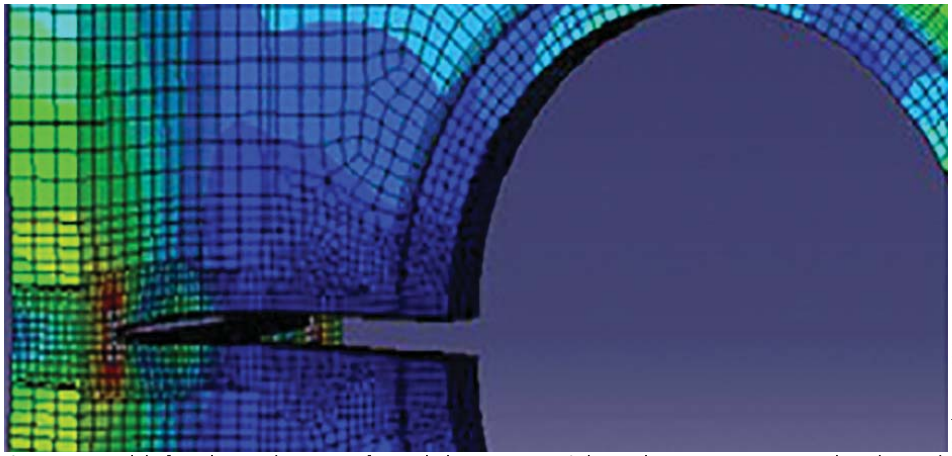
NIST leads US Effort on New ISO Technical Committee on Biotechnology

The NIST Materials Measurement Laboratory (MML) and Standards Coordination Office (SCO) have recently agreed to host the US Technical Advisory Group (TAG) for ISO/TC 276 Biotechnology. Sheng Lin-Gibson (MML) is serving as Chair and Clare Allocca (SCO) is serving as TAG Administrator.

The US TAG has been established with strong participation from industry and other US government agencies. The initial meeting in December 2013 was used to develop the US position with respect to Technical Committee (TC) scope definition and directions, in anticipation of the TC meeting later in December. Sheng Lin-Gibson led the US Delegation to the inaugural ISO/TC 276 meeting, during which a consensus scope was developed, covering terms and definition; biobanks and bioresources; analytical methods; bioprocessing; data processing (including annotation, analysis, validation, comparability and integration); and metrology.

Four task groups were also formed with conveners to identify standardization needs, gaps and priorities in the areas of terms and definition (Germany); biobanks and bioresources (France); analytical methods (USA); and bioprocessing (Japan). These international task groups, all with strong US participation, will make recommendations that will guide the overall TC business plan to be completed during the May 2014 TC meeting. US TAG mirror task groups have also been formed to ensure our understanding of needs, gaps and priorities of importance to US industry. These areas will also be the focus of the April 2014 TAG meeting, during which the US consensus position on all four of these areas will be determined in anticipation of the May 2014 TC meeting.

NIST Announces New Center for Materials Research to Advance Manufacturing and Innovation



Computer model of crack growth in a part from a helicopter rotor. Colors indicate stresses surrounding the crack (red is high and blue is low). Such computations will be a key part of CHiMaD's research.

Note: A version of this story previously appeared in NIST's TechBeat *on Dec. 3, 2013*

NIST recently announced that it has selected a consortium led by Northwestern University to establish a new NIST-sponsored center of excellence for advanced materials research. The new Center for Hierarchical Materials Design (CHiMaD) will be funded in part by a $25 million award from NIST over five years.

Other members of the CHiMaD consortium include the University of Chicago, the Northwestern-Argonne Institute of Science and Engineering (a partnership between Northwestern and the Department of Energy's Argonne National Laboratory) and the Computation Institute (a partnership between the University of Chicago and Argonne.) The consortium also plans to work closely with QuesTek Innovations, a small business spin-off of Northwestern; ASM International, a well-known professional society of materials scientists; and Fayetteville State University.

"I'm particularly excited to announce this new alliance between NIST and two prominent research universities to drive innovation in the development of advanced materials," said Patrick Gallagher, Under Secretary of Commerce for Standards and Technology and NIST Director. "This new Center for Hierarchical Materials Design is a natural fit for NIST, which has a long tradition of serving as a nexus with academia and industry to advance research and innovation for the nation's benefit."

"The launch of this new center represents a major milestone in support of the President's Materials Genome Initiative and our national goal of doubling the pace of discovery and development of novel materials," said Cyrus Wadia, assistant director for Clean Energy and Materials R&D at the White House Office of Science and Technology Policy. "By integrating the complementary strengths of computation, instrumentation, and creative modeling, this center promises to help keep America at the forefront of the materials revolution and a leader in the economically important domain of advanced manufacturing."

The new center will focus on developing the next generation of computational tools, databases and experimental techniques to enable "Materials by Design*," one of the primary goals of the administration's Materials Genome Initiative (MGI). "Materials by design" employs physical theory, advanced computer models, vast materials properties databases and complex computations to accelerate the design of a new material with specific properties for a particular application—perhaps an extremely tough, lightweight composite for auto bodies or a biocompatible cell scaffold for medicine. It stands in contrast to the traditional trial-and-error method of materials discovery (think of Thomas Edison and his dogged quest for the best lightbulb filament.)

Materials-by-design techniques have the potential to revolutionize the development of new advanced materials, which in turn have created whole industries. It's estimated that the average time from laboratory discovery of a new material to its first commercial use can take up to 20 years. The MGI aims to halve that.

The new center's work is expected to encompass both "hard" (inorganic) and "soft" (organic) advanced materials in fields as diverse as self-assembled biomaterials, smart materials for self-assembled circuit designs, organic photovoltaic materials, advanced ceramics and metal alloys.

CHiMaD will focus these techniques on a particularly difficult challenge, the discovery of novel "hierarchical materials." Hierarchical materials exploit distinct structural details at various scales from the atomic on up to achieve special, enhanced properties. An example in nature of a hierarchical material is bone, a composite of mineral and protein at the molecular level assembled into microscopic fibrils that in turn are assembled into hollow fibers and on up to the highly complex material that is "bone."

The award to the Northwestern consortium for the Center for Hierarchical Materials Design is for $5 million per year for 5 years, subject to available funds. NIST may, at its discretion, extend the award for an additional 5 years after a performance review. The Northwestern-led consortium is contributing another approximately $4.65 million to the center.

- Michael Baum

*"Materials by Design" is a registered trademark of QuesTek Innovations LLC.

nSoft Consortium Holds First Virtual Member Meeting

On February 11, 2014, nSoft (see next page) held its first "virtual meeting" of its members and collaborators. The meeting, open only to those participating in the consortium, featured five 30 minute presentations on research being pursued by the consortium, including protein characterization, rheology of complex fluids, flow through membranes, macromolecular topology, and semicrystalline polymers. The meeting was hosted at the NIST Center for Neutron Research (NCNR) with 33 participants (including 20 industrial members) from NIST and consortium members. The meeting highlighted many of the most recent accomplishments of the consortium, including a new flow cell to study complex fluids under extreme elongational stresses and strains, and a new vapor cell capable of studying rapid changes in membrane structure during operation. The virtual meetings are intended to be held after each NCNR fuel cycle, approximately 7 weeks in duration.

nSoft Consortium

Objective

The overall goal of the nSoft Consortium is to develop and transfer new neutron-based measurement science to soft materials manufacturers. “Soft materials” are the basis of plastics and composites and are critical to the production of consumer goods, automobiles and other everyday items, as well as to materials for increased energy efficiency, protein-based therapeutics, and water filtration and desalination. The consortium is led by the Materials Science and Engineering Division and the Center for Neutron Research, both at NIST. Expertise is transferred to consortium members through co-participation in experiments, training programs and annual meetings. Members then adopt the technology into their analytical research programs through reliable and predictable access modes at the NIST Center for Neutron Research.

nSoft

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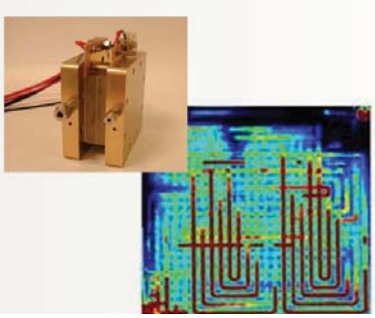

Impact and Customers

- The nSoft research program is designed to transfer to manufacturers of soft materials expertise in neutron technology that will provide a competitive advantage in future product development. Expertise is transferred through co-development of new measurements, sample environments and data analysis software designed to attack specific areas of interest to members.
- Proprietary access to the neutron source enables the incorporation of these advanced tools in members’ manufacturing research programs. The nSoft Consortium also provides a clear route to access NIST’s leading expertise in soft materials, biomanufacturing and neutron technology.
- Members are from many industrial sectors including plastics and light-weight composites, protein-based therapeutics, consumer products, sustainable materials, foods, and energy.

Approach

The nSoft Consortium enables member companies to participate with NIST in the development of advanced measurements of materials and manufacturing processes, and to develop their own expertise in state-of-the-art measurement technologies to include in their analytical research programs.

The nSoft Consortium will operate a 10-meter small angle neutron scattering (SANS) instrument to develop measures of morphology and molecular topology. nSoft will also develop methods based on neutron imaging , neutron spectroscopy (dynamics and chemistry) and neutron reflectivity (depth profiling).

Key areas of expertise include polymer melt and solution structure under elongational strain and extensional flow, high strain rate behavior of composites, and structure and flow of concentrated protein solutions.

The consortium is led by the Materials Science and Engineering Division at NIST, the NIST Center for Neutron Research, and the University of Delaware. Up to 40 for-profit institutions can join by paying an annual membership fee and signing a collaborative research agreement (see nSoft website). Not-for-profit institutions can join by providing materials and resources.

Accomplishments

Formal operations at nSoft began with a kick-off meeting on August 14, 2012. The initial membership consists of 10-15 companies with businesses spanning petroleum-based energy products, basic chemicals, consumer products and pharmaceuticals. The technical objectives of the consortium are divided into three tasks:

Material morphology and topology

Molecular topology, specifically molecular shape in solution and solid form, is a critical parameter of manufacturing in soft materials. nSoft will develop neutron scattering and imaging methods to measure the spatial position and shape of long-chain branched polyolefins, used to control flow rate and mechanical properties of the commodity plastics industry. In collaboration with the NIST Biomanufacturing Program, these measurements will also elucidate the form of antibodies in solution while interacting with incipients and surfaces, where molecular shape indicates therapeutic function. New vapor adsorbtion cells will be used to advance the characterization of polymer matrix composites, ascertaining the effects of fillers on mechanical properties. In collaboration with a NIST neutron imaging facility, vapor adsorbtion studies will advance the characterization of moisture in encapsulants and porous media.

Materials characterization under flow and strain

The incorporation of a couette rheometer on a small angle neutron scattering instrument ("rheo-SANS") has demonstrated the capacity of neutrons to probe soft materials under dynamic conditions. nSoft will build on this successful effort at the NIST Center for Neutron Research to develop in situ measurements using a Sentemant extensional rheometer, large amplitude oscillatory shear (LAOS) technology, and hyperbolic inlets to measure materials under high shear rates (approaching 1 million s-1). These environments mimic conditions found in manufacturing of blown film, film coating, extrusion of melts and protein solution flow through a needle.

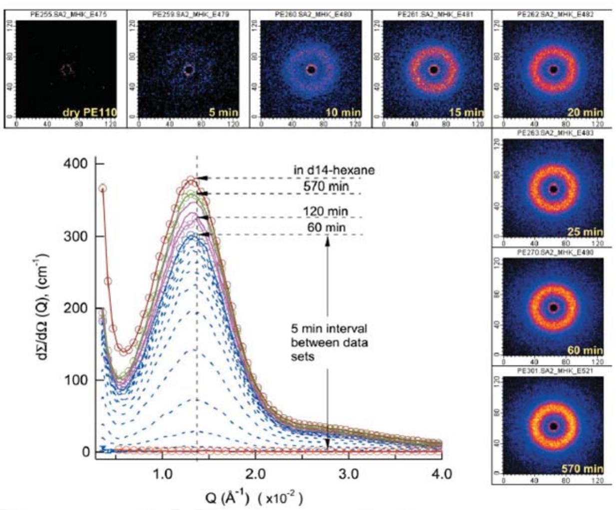

Figure 1. The in situ vapor cell for small angle neutron scattering allows labeling of two-phase materials; for example, to probe the amorphous region of a semicrystalline polyethylene, and contrast variation on as-produced materials. Shown here are SANS data demonstrating the increasing contrast of the long period of a linear polyethylene with increasing exposure time to d-cyclohexane vapor (from Kim and Glinka, 2009).

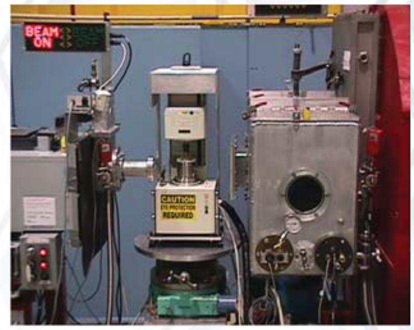

Figure 2. The couette rheometer sample environment for the 30 meter small angle neutron scattering instrument at the NIST Center for Neutron Research. The rheometer can handle a wide range of shear for fluids, providing structure and rheology simultaneously.

Shear and structure of soft material interfaces

Soft materials are defined by their interfaces, including surfactants at an oil/water interface, polymer adsorbtion at a filler interface in composites, and the interphase between amorphous and crystalline regions in polyolefins. The depth penetration and sensitivity to constrast variations at internal interfaces allows neutrons to probe soft material interfaces, even during the application of shear. Methods such as the overflowing cylinder, dynamic Langmuir trough, and time- resolved small angle neutron scattering will be developed to probe this key component of material design for manufacturing.

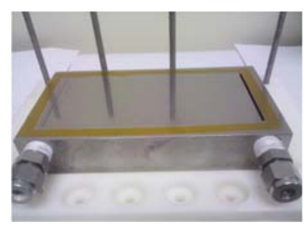

Figure 3. Shown is a flow cell being developed for interfacial studies of soft materials and fluids. The silicon enclosure is transparent to neutrons, allowing slot flow at an interface to be studied in real time.

Learn More

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Publications

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NIST MML Microanalysis Technique Makes the Most of Small Nanoparticle Samples

Note: A version of this story previously appeared in NIST's TechBeat on February 24, 2014

Kar/NIST

Small piezoelectric quartz crystals are the key to micro thermogavimetric analysis. Minute amounts of the test sample are deposited on the crystals.

Researchers from NIST MML and the Food and Drug Administration (FDA) have demonstrated that they can make sensitive chemical analyses of minute samples of nanoparticles by, essentially, roasting them on top of a quartz crystal. The NIST-developed technique, "microscale thermogravimetric analysis," holds promise for studying nanomaterials in biology and the environment, where sample sizes often are quite small and larger-scale analysis won't work.*

Chemical analysis of nanoparticles is a challenging task, and not just because they're small. They're also complicated. They can become coated with other materials in their environment, and the question becomes, what materials? Or they might have been engineered with a coating, perhaps to provide anchor points for drug molecules, and then the question can be, how complete is the coating? In nanoelectronics, the question may be, how pure is the sample and just what are the impurities?

Researchers have an alphabetic array of tools for this, including scanning, transmission or atomic force microscopy (SEM/TEM/AFM); dynamic light scattering (DLS); nuclear magnetic resonance (NMR); and sundry spectrometry techniques, but they all have a variety of limitations, including complex sample preparation or the difficulty of analyzing enough particles to get a statistically significant result.

On the other hand, one technique, thermogravimetric analysis (TGA) is quite straight-forward. The sample is heated and monitored for changes in mass as the temperature increases. Sudden changes in mass correlate with the energies needed to decompose, oxidize, dehydrate or otherwise chemically change components in the sample. If you have some idea of what you start with, TGA can tell you much more, but it requires pretty substantial sample sizes.

NIST's technique is essentially the same except that a small piezoelectric quartz crystal is substituted for the mass scale. A tiny amount of a nanomaterial sample deposited on the crystal dampens the crystal's resonant frequency, and as the sample grows lighter, the frequency shifts. NIST researchers originally applied it to measure the purity of carbon nanotube samples.**

In this latest paper, the research team tested the utility of microTGA on typical nanomaterial analysis problems, including assessing the purity of carbon nanotubes, determining the amount of surface-bound ligands (i.e., molecular anchors) on gold nanoparticles, and testing for the presence of PEG, a polymer commonly used in medicine on silicon oxide nanoparticles.

"Our results are a pretty close match to other techniques," reports NIST analytical chemist Elisabeth Mansfield, "but using far less of a sample."

In fact, the team reports, microTGA gets results using samples a thousand times smaller than conventional techniques. It can work with one microgram of sample and detect mass changes of less than a nanogram. "That's important because you often don't have much of a sample," Mansfield says," "If you're pulling nanoparticles out of a water sample from the environment to measure how much exists in a real world sample, you're going to have very little to work with."

"In nanomedicine, the surface chemistry is oftentimes critically important to the performance of the nanomaterial," notes FDA chemist Katherine Tyner. "When working with real life samples, we may only have a very small sample amount. MicroTGA allows us to obtain information that we otherwise would not be able to get with conventional techniques."

- Michael Baum

*E. Mansfield , K.M. Tyner, C.M. Poling and J.L. Blacklock. Determination of nanoparticle tsurface coatings and nanoparticle purity using microscale thermogravimetric analysis. Anal. Chem., 2014, 86 (3), pp 1478–1484 DOI: 10.1021/ac402888v.

**See the November 2010 NIST story "Quartz Crystal Microbalances Enable New Microscale Analytic Technique" at www.nist.gov/public_affairs/techbeat/tb20101124.cfm#qcm.

NIST MML Analysis Helps the U.S. Chemical Safety Board Pinpoint Root Cause of Pressure Vessel Failure

CSB

NDK Crystal damage after the incident. The force of the explosion blew out most of the wall panels.

In 2009, a violent rupture of a 50-foot pressure vessel used to produce synthetic crystals at the NDK Crystal facility in Belvidere, Illinois fatally injured a member of the public and caused significant property damage to the plant itself and the surrounding area. In response to the accident, scientists from NIST MML were approached by the U. S. Chemical and Hazardous Material Safety Board (CSB) to review data and assist in identifying the failure mechanism. The review found strong evidence of stress corrosion cracking (SCC) on and near the inner diameter of the vessel fragment, and a reduction in material toughness during service. This combination likely resulted in a flaw reaching critical size, causing the catastrophic failure. The results were included in CSB's recently published final reporton the incident investigation.

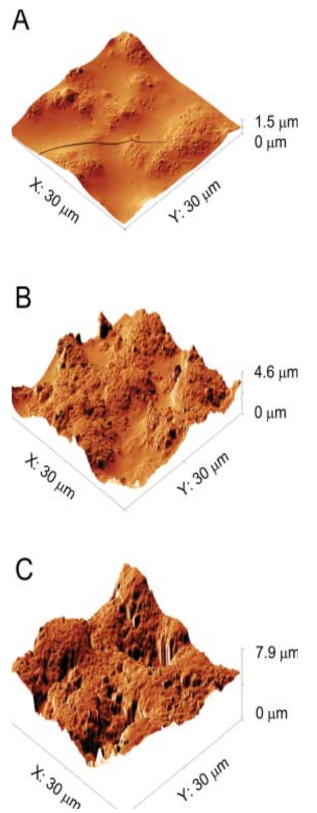
In order to reach its conclusions, NIST scientists reviewed over 1000 pages of documents, including chemical and mechanical property data, micrographs, material standards, code documents and results from ultrasonic examinations. The NIST review identified stress corrosion cracking as the most likely cause of the extensive cracking found in the vessel. The review also revealed possible reduction in fracture toughness caused by temper embrittlement in the failed vessel, based on comparisons of Charpy impact test data after fabrication and after failure. In addition, NIST provided a fracture mechanics analysis which concluded that though the vessel entered service in a safe condition, the SCC-caused cracking, along with the reduction in fracture toughness of the steel, resulted in the vessel reaching a critical stress intensity, and subsequent catastrophic failure.

CSB's mission is the investigation of industrial accidents and determination of their root cause, in order to recommend changes to safety procedures, codes and standards, resulting in safer plants, workers and communities. As part of CSB's network of experts, NIST provides expert, independent technical analysis based on its many years of research in structural materials reliability. CSB expects to continue engaging NIST in investigations where material failure is suspected of playing a role.

New NIST MML Tests Explore Safety of Nanotubes in Modern Plastics Over Time

Note: A version of this story previously appeared in NIST's TechBeat *on Dec. 17, 2013*

Who cares about old plastic? Researchers at NIST MML do, so that you won't have to years down the road, when today's plastic concoctions start to break down and disintegrate from weather exposure. Experiments* at NIST may help scientists devise better tests to make sure aging plastics won't turn into environmental or health hazards as time goes by.



NIST

Exposed to intense ultraviolet light and high temperatures, samples of epoxy containing multiwalled carbon nanotubes deteriorated. Exposure tended to destroy the epoxy, but on the surface remained a network of nanotubes, which NIST tests indicated were less damaged than the epoxy over time, ranging from approximately 6 months (A) to 18 months (B) to 4 years (C) in subtropical conditions.

Tests like this are more important now than ever, because plastics aren't what they used to be. Modern epoxies are frequently made stronger, lighter and more resilient with the addition of multiwalled carbon nanotubes (MWCNTs), a special form of carbon that under a microscope looks like rolls of chicken wire. MWCNTs already enhance plastics used in baseball bats, tennis rackets, bikes and airplanes, and though the tiny tubes appear to be long-lasting, no comprehensive set of tests exists to determine what happens to them over the long haul. So a NIST team took steps to change that.

"Some studies have been done about the effect of ultraviolet (UV) light, but not with a large number of analytical methods," says NIST's Elijah Petersen. "We wanted to begin developing a suite of tests for evaluating the performance of these nanocomposite materials, so that we can examine their potential risks, if any, during usage."

The team needed a way to simulate the degrading effect of years of high temperature, humidity and sunlight, but without waiting that long for results. They created samples of epoxy with 3.5 percent MWCNTs—a fairly typical mixture quantity—and put them into a NIST-developed device called SPHERE (Simulated Photodegradation via High Energy Radiant Exposure), which pours out powerful UV light into a chamber kept at 50 degrees Celsius and 75 percent humidity. Keeping the samples there for 100 days "was the equivalent of four years in the Florida sun," Petersen says.

After exposing the samples to this artificial Florida, the team ran a set of six different tests that analyzed changes ranging from mass to appearance to surface chemistry. One major discovery was that the UV light tended to destroy the epoxy, but on the surface remained a spaghetti-like network of nanotubes, which the group of tests indicated were less damaged by SPHERE exposure than the epoxy matrix.

So are these nancomposite materials stable forevermore? Petersen says the study did not answer every question, but that it should help researchers determine answers more effectively than was possible before by development and optimization of multiple analytical methods.

"We got a lot of new information from the test suite about how nanocomposites degrade," says Petersen, "and the most encouraging thing is that the results of the different tests generally back one another up. We hope the test suite allows for better analysis of the stability of these new plastics, which are growing more common in everyday life."

- Chad Boutin

* E.J. Petersen, T. Lam, J.M. Gorham, K.C Scott, C.J. Long, D. Stanley, R. Sharma, J.A. Liddle, B. Pellegrin and T. Nguyen. Impact of UV irradiation on the surface Chemistry and structure of multiwall carbon nanotube epoxy nanocomposites. Carbon, Dec. 16, 2013, doi: 10.1016/j.carbon.2013.12.016.

MML Helping to Make Cardiac Devices More Reliable

Implanted pace makers and defibrillators sense the electrical signals of the heart and deliver resynchronization therapy through bundles of wires (termed "leads" by the industry). Leads are meant to last 10 years, but fail at rates approaching 6% after five years, with two major recalls in the past seven years that have affected hundreds of thousands of patients. When leads fail, doctors and patients are faced with difficult decisions about extraction and/or replacement that can have major consequences to patient health. Most cardiac devices are designed and manufactured in the US, and the FDA has urged device manufacturers to develop new testing methods to ensure the reliability of the leads.

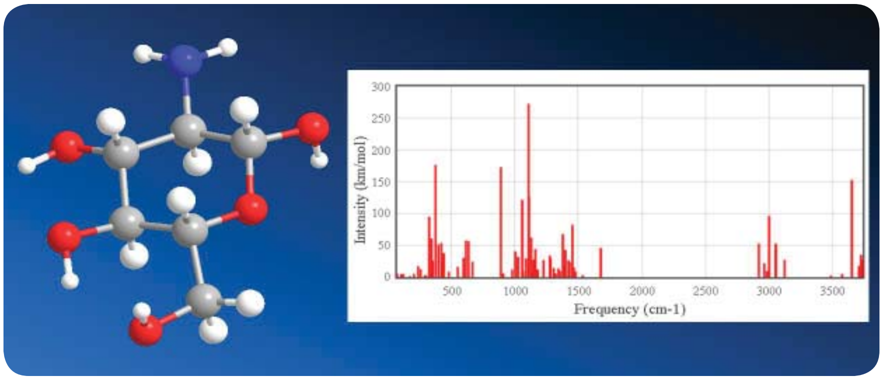
In response to this need, the five leading manufacturers of leads, physicians, the FDA, and NIST have come together on an AAMI PC-WG-01 committee to standardize improved lead testing methods. The challenge faced by the committee was to quickly standardize test methods that are able to distinguish leads that are reliable from those that might not be. The committee chose to address fatigue of the wires in the lead first as it is a prominent cause of failure.

Because each of the manufacturers had developed their own proprietary testing methods, MML engineers formed a CRADA consortium and reviewed the testing practices for all five of the principal cardiac device manufacturers. In April of 2013, they reported on the best practices of the industry and made recommendations on how to extend those practices to allow for an improved standard that would be able to distinguish leads that were acceptable from leads that were not. The report was immediately adopted by the AAMI committee as a basis for the new fatigue standard. Based on recommendations in the report, the AAMI committee is currently planning a large in vivo study of devices that were already implanted.

NIST MML Researchers Team Up with Summer Interns to Generate 50,000 Molecular Structures

A team of NIST researchers in the MML Chemical Sciences Division has worked with college and high school students over the past two summers to calculate high quality three-dimensional structures for important chemical species. Chemical intuition and insight is based largely on an accurate knowledge of molecular structure, therefore, a comprehensive catalog of molecular structures is an invaluable tool for the chemist or chemical engineer.

Each year a six member team is put together consisting of college students from the NIST Summer Undergraduate Research Fellows (SURF) program and high school students from the NIST Summer High School Intern Program (SHIP) to work on generating structures ranging from the commonplace to the exotic, from those found in every household to those found in interstellar dust clouds. Computational chemists in the NIST Chemical Informatics Research Group provided instruction and guidance to assist the students in their work. This included instruction in computational techniques, use of software and operating systems along with the underlying quantum theory associated with the computations. A new group of students will continue the project this summer.



Computed molecular structure and infrared vibrational intensities for glucosamine, a popular dietary supplement for people suffering from arthritis. Gray=carbon, white=hydrogen, red=oxygen, blue=nitrogen.

This work directly supports science, technology, engineering, and math (STEM) education in two ways. The first is the knowledge and experience the SURF and SHIP students gained in working on this project. As the students gained experience in the areas of molecular structure and chemical nomenclature throughout the summer program, they were often able to spot inconsistencies and errors in structures and identifiers without any aid from NIST staff. The second is that the structures are being made available to the public via the NIST Chemistry WebBook (http://webbook.nist.gov/chemistry/). By doing so the structures are used as visualization aids for educators. These structures represent the digital equivalent of "ball and stick" molecular models which have been traditionally used to teach students concepts in chemistry. In addition to being more accurate, the advantage of using computational models is that many more structures are available. In fact, over 50,000 structures have been generated so far. For students in more advanced areas of study, the structures and information derived from their computation (e.g., vibrational behavior) can be used as a starting point for additional molecular calculations.

The students also complete the project knowing that their efforts have a direct impact on both NIST's industrial customers as well as the broader scientific community. Professional researchers and engineers routinely use the structures and derived information to assist their work in a range of scientific fields. As an example, infrared absorption intensities derived from these computations can be used to help estimate properties associated with climate change for chemical species in the atmosphere.

Services Highlight: New Version of ILThermo Designed to Support Ionic Liquids Research

A new version of the Ionic Liquids Database ("ILThermo," NIST Standard Reference Database 147, see http://ilthermo.boulder.nist.gov/) has been released by the Thermodynamics Research Center (TRC) of the MML Applied Chemicals and Materials Division. ILThermo is a web-based, open-access ionic liquids database. It aims to provide users worldwide with up-to-date information on published results of experimental studies of ionic liquids, including numerical values of thermophysical and thermochemical properties, chemical structures, measurement methods, sample purity, critically evaluated uncertainty of property values, and many other significant measurement details. The database can be searched according to the ions constituting the ionic liquids, the ionic liquids themselves, their properties, and references. The new version of the database is equipped with a user-friendly interface and contains information on more than 1,000 ionic liquid systems, including pure ionic liquids as well as their binary and ternary mixtures with other chemicals, totaling near 300,000 experimental data points reported in more than 1,500 original sources.

ILThermo is produced from the NIST SOURCE Data Archival System, a comprehensive data storage facility supporting the NIST ThermoData Engine, developed to dynamically evaluate data for thermophysical and thermochemical properties. The original version of ILThermo, available since 2006, was developed by TRC in cooperation with other experts from the International Union of Pure and Applied Chemistry, Project 2003-020-2-100.

Ionic liquids have been the subject of enormous scientific interest over the past 10 years due to their extremely low vapor pressure, and therefore exhibit favorable solvent properties for new homogenous catalytic reactions and other chemical processes designed to be sustainable. Understanding the thermophysical and thermochemical properties of ionic liquids is a key part of developing their industrial potential.

U.S. Senator Mikulski Visits NIST Center for Automotive Lightweighting

On January 27 NIST hosted U.S. Senator Barbara Mikulski for a visit to the Center for Automotive Lightweighting (NCAL). The Center works to help automobile manufacturers meet ambitious goals like doubling automobile fuel economy by 2025, reducing the weight of automobiles by up to half a ton each while maintaining or improving safety and saving millions of dollars annually in redesign and re-tooling costs.

During her visit, the Senator was introduced to some of the advanced mechanical testing work done at NCAL, and she asked about the industrial drivers for using new materials in vehicles. She expressed her support for the important work done by NIST researchers to support U.S. industry.

U.S. Senator Barbara Mikulski and NIST Director Patrick Gallagher hear a presentation from NIST researcher Mark Iadicola as part of tour of the NIST Center for Automotive Lightweighting.

MML Researcher R. Joseph Kline Receives Presidential Early Career Award for Scientists and Engineers

MML researcher R. Joseph Kline was recently named among the 102 recipients of the Presidential Early Career Awards for Scientists and Engineers (PECASE), the highest honor bestowed by the United States Government on science and engineering professionals in the early stages of their independent research careers. Kline recently received his award at a Washington, D.C. ceremony.

Kline is recognized internationally for his scientific leadership in the characterization of molecular factors that are critical to the performance of organic electronics—an exciting class of materials that, if properly harnessed, promise a wealth of innovative technologies including flexible electronic devices and affordable solar cells that can be printed on a roll-to-roll manufacturing line. In particular, Kline has pioneered the use of grazing incidence X-ray diffraction (GIXD) as a now-widespread technique for quantifying the orientation and structure of organic electronic molecules in films, the knowledge and control of which is critical for the manufacture of high-quality organic electronic devices. The lack of methods to quantify these factors was a considerable road-block for organic electronics, but measurement advances like GIXD have fueled new interest in this technology. Kline has emerged at the forefront of this wave because his GIXD measurements reveal the molecular basis for charge carrier mobility transport in organic semiconductor materials. His stature in the field is reflected in over 60 peer-reviewed publications (including five invited review articles and two book chapters), over 5000 citations, an h-index of 26, 27 invited lectures in the U.S. and multiple countries, and several prestigious NIST and external awards.

Kline is widely known across NIST for his expertise in x-ray scattering and for generously contributing his talents to projects across the laboratories and divisions. He has co-organized three international symposia on organic electronics and a NIST workshop on "Advanced Data Analysis and Modeling Tools for Scattering Methods." He has led or co-led collaborations, CRADAs, or MTAs with Merck Chemical, Corning, IBM, Dow Chemical, KLA Tencor, SEMATECH, HGST, and Intel. He serves on the Advanced Photon Source Proposal Review Panel, the National Synchrotron Light Source Proposal Review Panel, the Center for Functional Nanomaterials Proposal Review Panel, and previously served on the Stanford Synchrotron Radiation Laboratory User Organizational Executive Committee, and participated in the planning of new beam lines for the National Synchrotron Light Source-II. Kline has mentored multiple undergraduates and graduate students, and NRC-NIST Postdoctoral Fellows.

Selected Recent Publications

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

K. W. Pratt, "*Measurement of pHT Values of Tris Buffers in Artificial Seawater at Varying Mole Ratios of Tris:Tris•HCl*" Marine Chemistry, Vol. 162, pp. 89-95, (20-May-2014) (PubID: 914253)

N. Bassim, K. C. Scott, L. A. Giannuzzi, "*Recent Advances in Focused Ion Beam Technology and Applications*" MRS Bulletin, Vol. 39, pp. 317-325, (11-Apr-2014) (PubID: 915390)

G. A. Cooksey, F. J. Atencia, "*Pneumatic valves in folded 2-D and 3-D fluidic devices made from plastic sheets and tapes*" Lab on A Chip, Vol. 14, pp. 1665-1668, (01-Apr-2014) (PubID: 915332)

S. Muramoto, T. P. Forbes, M. E. Staymates, J. G. Gillen, "*Characterization of Emerging Ambient Pressure Mass Spectrometric Techniques using Time-of-Flight Secondary Ion Mass Spectrometry*" Analytical Chemistry, (28-Mar-2014) (PubID: 915103)

K. D. Benkstein, P. H. Rogers, C. B. Montgomery, J. Jin, B. Raman, S. Semancik, "*Analytical Capabilities of Chemiresistive Microsensor Arrays in a Simulated Martian Atmosphere* " Sensors and Actuators B-Chemical, Vol. 197, pp. 280-291, (06-Mar-2014) (PubID: 913645)

J. B. Morrow, A. S. Downey, L. Delaney, "*Best Practices for Sample Collection and Transport in an Initial Response to Potential Biothreat Materials*" Technical Note (NIST TN) 1776, (05-Mar-2014) (PubID: 909556)

J. M. Zook, B. Chapman, W. Hide, M. L. Salit, "*Why human genome sequencing platforms differ: Integrating sequencing datasets to understand biases and form consensus variant calls*" Nature Biotechnology, Vol. 32, pp. 246-251, (16-Feb-2014) (PubID: 912658)

S.J. Oh, N. E. Berry, H.H. Choi, E. A. Gaulding, H. Lin, T. Paik, B. T. Diroll, S. Muramoto, M. B. Christopher, C. R. Kagan, "*Designing High-Performance PbS and PbSe Nanocrystal Electronic Devices through Stepwise, Post-Synthesis, Colloidal Atomic Layer Deposition*" Nano Letters, (06-Feb-2014) (PubID: 915267)

P. Y. Hsieh, T. J. Bruno, "*Measuring the sulfur content and corrosivity of North American petroleum with the advanced distillation curve method*" Energy & Fuels, Vol. 28, No. 3, pp. 1868-1883, (05-Feb-2014) (PubID: 914144)

G. Stan, S. Solares, B. Pittenger, N. Erina, C. Su, "*Nanoscale mechanics by tomographic contact resonance atomic force microscopy*" Nature Nanotechnology, (23-Jan-2014) (PubID: 914282)

S. Y. Rhieu, A. A. Urbas, D. W. Bearden, J. P. Marino, V. Reipa, K. A. Lippa, "*Probing the intracellular glutathione redox potential using in-cell NMR spectroscopy*" Nature Methods, (02-Jan-2014) (PubID: 914505)

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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