

# News Briefs

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## General Developments

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### TRAINING PROGRAM FOR FORMER SOVIET EXPERTS

During 1995, NIST hosted a total of 82 technical and regulatory experts from Russia and the Commonwealth of Independent States (CIS) for briefings on U.S. standards development, conformity assessment, and quality management practices. NIST hosted four groups for separate 2 week orientation sessions that focused on key product sectors with high export potential: automotive, medical equipment, telecommunications, and aerospace equipment. After each of these orientation sessions, CIS experts visited various companies, private-sector standards developers, testing laboratories, and product certifiers over a 6 week period. The program objective is to provide technical training and contacts in support of commercial and technical cooperation between the United States and the CIS.

More than 40 companies hosted these groups for periods ranging from 1 day to an entire week. Participating companies report that these visits put in place key ingredients of the groundwork for increased business in the CIS region: face-to-face discussion, mutual understanding, and shared technical knowledge. Specific benefits were reported: new sales in the region, ranging from several thousand dollars to \$100 million; increased knowledge of CIS certification requirements for imported equipment; and facilitation of the product introduction process in key CIS markets. Nearly all companies would consider hosting again. Most got

more out of the program than they had anticipated, and the benefits from personal contact with key CIS decision-makers on standards-related issues will undoubtedly increase in value to them in the long run.

Three additional groups of CIS experts will visit NIST during 1996: experts in food processing and packaging, oil and gas equipment, and construction. OSS plans to continue follow-up activities with previous participants, both in the CIS and in the U.S. private sector. New sales realized by companies that have participated in this \$1 million training program have already repaid the initial investment more than 100-fold.

### STANDARDS SPECIALISTS ASSIGNED TO EMBASSIES

The Standards Training and Support Program has assigned standards representatives to two embassies to support U.S. industry by assisting in eliminating technical barriers to trade and promoting exports. They will provide technical advice and assistance related to product standards and conformity assessment through training and consultations in cooperation with in-country and U.S. government agencies, standards organizations, trade associations, and individual companies, collecting information, analyzing issues, and following up on initiatives and problems.

One NIST employee is assigned to the U.S. Embassy in Mexico City to address TBT (Technical Barriers to Trade) issues in Mexico and Central America, especially with regard to NAFTA. A former NIST employee and a new private-sector consultant is assigned to the U.S. Embassy in Buenos Aires, Argentina, and will cover issues there and in other South American countries, especially those in MERCOSUR (a South American trade group). Other U.S. standards experts are already in place in Saudi Arabia and at the U.S. Mission to the European Union in Brussels. NIST contractors provide additional standards assistance in India and Japan.

### **NEW VIDEOS ON '95 BALDRIGE AWARD WINNERS RELEASED**

Armstrong World Industries' Building Products Operations and Corning's Telecommunications Products Division are featured in two new videos on the 1995 Malcolm Baldrige National Quality Award winners. The recently released videos highlight the two companies' quality improvement strategies. A short version, "Quest for Excellence VIII," which runs 12 minutes, was premiered at the Quest for Excellence VIII Conference on Feb. 5, 1996. The longer version, 24 minutes, contains more in-depth information about the companies' quality efforts. VHS copies are available for purchase from ASQC at (800) 248-1946, Item Number T1033. For a single copy, the cost is \$20 plus \$4 shipping and handling.

### **ORDER-OF-MAGNITUDE REDUCTION REALIZED IN CONTRIBUTION OF ALIGNMENT ERRORS TO NIST WATT BALANCE UNCERTAINTY**

The NIST watt balance compares mechanical power to electrical power with high accuracy. An important future direction is to use the balance to monitor the drift in the artifact kilogram mass standard in terms of more stable electrical standards, the "electronic kilogram." Alignment errors constitute one of the significant factors contributing to the overall uncertainty of the balance. As a result of recent work by NIST scientists, critical alignment problems have been addressed with a resulting order-of-magnitude reduction in the contribution of this factor.

The balance operates by comparing the force on a mass in a gravitational field to the force on a current loop in a magnetic field, and then separately measuring the voltage induced around that loop when it moves at some velocity through the same magnetic field. By comparing the product of the voltage and the current to the product of the force and the velocity, the "electrical" watt is compared to the "mechanical" watt. If the direction of the velocity and the directions of the forces are not aligned, or if the electrical or gravitational forces produce torques, then the measurement of the watt will be in error. Also, even if the balance is perfectly aligned, a misalignment of the velocity measurement system could produce errors in the watt measurement. A systematic study has resulted in measurement procedures to monitor the uncertainty in the watt measurement from alignment errors, and this uncertainty has been reduced by more than an order of magnitude to 40 nW/W. Future improvements have been identified to further reduce that uncertainty. This accomplishment is a major step toward the goal of monitoring the kilogram,

which requires an overall balance uncertainty of less than 10 nW/W.

### **STANDARD DEVELOPED FOR RECHARGEABLE BATTERIES FOR PERSONAL TRANSCEIVERS**

NIST has developed a performance standard, issued as National Institute of Justice (NIJ) Standard 0211.01, Rechargeable Batteries for Personal/Portable Transceivers. Hand-held radios are a vital tool for law enforcement and other public safety officials; these devices depend critically on the performance of the rechargeable nickel-cadmium batteries that power them. The standard establishes both performance requirements and test methods for these batteries, which are classified into two types: those for which the manufacturer specifies only the nominal capacity, usually in milliampere-hours, and those for which the manufacturer specifies the nominal service life when used with a specific transceiver at a specified duty cycle. For either type, the standard specifies a test cycle for service life/capacity determinations based on the battery serving as a power source 10 % of the time in both the transmit mode and receive mode and 80 % of the time in the standby mode. The current drains for each mode are specified according to whether the battery capacity is less than or greater than 700 mAh. This work is part of the NIJ Law Enforcement and Corrections Standards and Testing Program.

### **TUTORIAL ON MICROWAVE MEASUREMENTS AIDS WIRELESS COMMUNICATIONS INDUSTRY**

As the wireless communications industry continues its explosive growth, system and component designers are recognizing the need for measurements to verify their models and simulations. In response to this demand, NIST scientists developed a two-day course entitled "Microwave Measurements for Wireless Applications," held just prior to the 46th Automated Radio Frequency Conference, Testing for Wireless Applications at Phoenix, AZ in November 1995. The automated radio frequency techniques group sponsored both events. The course attracted 89 participants from the microwave and wireless communications industries.

NIST scientists presented tutorial lectures on basic microwave measurements, which covered microwave circuit theory, transmission lines, scattering parameter measurements, impedance, and power measurements. Speakers from private industry covered digital modulation, spectrum analyzers, power amplifier characterization, automated test, and other areas of wireless testing. Favorable response from the participants has led to plans to give the course again.

### **UNIQUE 20-YEAR-OLD HIGH-ENERGY LASER CALORIMETERS RETURNED TO NIST FOR REFURBISHMENT**

In the mid-1970s, NIST scientists designed and built two 400 kg calorimeters for use in the Department of Defense high-power laser weapons program. As far as is known, these are the only two electrically calibrated standards ever developed that are capable of monitoring the greater-than 100 kW outputs of such lasers. They have been in continuous use by the Air Force primary standards laboratory to provide traceable measurements for various laser development activities scattered around the country. Along with associated electronics, they are transported to test ranges in a customized van 10 m long.

After nearly 20 years of regular use without failure, one of these calorimeters was recently returned to NIST for refurbishing and upgrading. This included cleaning all of the optical surfaces, replacing worn and broken electrical heaters, and modifying the water flow system to allow faster cooling and easier drainage and filling. A NIST scientist performed the work, with the assistance of two now-retired NIST scientists, who participated in the original design and development project. After preliminary testing by the Air Force, the calorimeter is now back in regular service and the second calorimeter is scheduled for the same treatment in the near future.

### **DIODE-LASER WAVELENGTH STANDARD FOR ABSOLUTE DISTANCE MEASUREMENT DELIVERED**

A major milestone has been achieved in a collaboration between two NIST laboratories that share responsibility in the realization of the meter as the international standard of length. A tunable diode laser, primarily developed as a wavelength reference standard by one laboratory, was delivered to the other laboratory that contributed to the design. The laser will now be characterized for use in absolute-distance interferometry, the shared goal of the collaboration.

NIST is responsible for providing U.S. industry with practical access to the meter as the international standard of length, now defined in terms of the length of path an electromagnetic wave travels in a specific interval of time. NIST also is responsible for providing access to the second as the international standard of time, in part via wavelength reference standards. By

comparison with commercial visible-light lasers, the NIST developed diode laser can sweep either 10 times farther than those with a comparable rate or 100 times faster than those with a comparable range. With its unique ability to continuously and rapidly sweep its central wavelength near 680 nm over a range of 2 nm at a rate of 10 Hz, the laser will allow development of an absolute-distance interferometry system, which can overcome the limitation of current displacement interferometry systems for an uninterrupted light beam. With the new interferometer system being developed, absolute distance between the reflectors of the interferometer can be measured directly rather than by changes in the position of one reflector relative to the other.

### **NIST'S APPROACH TO BE USED FOR UNIFYING THE INTERNATIONAL ROCKWELL HARDNESS SCALES**

The Ninth International Hardness Symposium was held on Nov. 23-24, 1995, in Düsseldorf, Germany, with 96 delegates from nine countries attending. A NIST scientist presented two papers. One of them, "A Metrology Approach to Unifying Rockwell C Hardness Scales" discussed NIST's approach for unifying the international Rockwell C hardness scales. By comparison, the European Community's performance-based HRC scale lacks traceability to fundamental metrology, has an unknown offset, and lacks reproducibility. Therefore, it cannot be used to unify the international HRC scales. The NIST approach, which includes accurate geometrical characterization of the hardness indenter, was supported widely, especially by delegates from other national metrology institutions.

Based on the metrology approach, a proposal entitled "Worldwide Unified Scales—Rockwell Hardness Test with Conical Indenters" was drafted by a staff member of a German testing laboratory. This proposal will be submitted to the BCR (Bureau Communautaire de Reference of EEC). Under the BCR proposal, an international round robin on Rockwell hardness tests will be held to establish worldwide unified Rockwell hardness scales (HRA, HRC, HRD, HRN15, HRN30, and HRN45). The round robin will use a common diamond indenter. Its geometry will be characterized by the NIST metrology approach. It is expected that the round-robin test program will take place over the next 3 years.

## **SURFACE WAVES**

Scientists at NIST are pursuing the determination of elastic constants of isotropic and anisotropic materials by methods that depend on the use of the leaky surface wave velocity. Leaky surface waves occur at the boundary between a solid and a fluid and generate wave motion only a short depth into the solid, while also radiating acoustic waves into the fluid. The leaky wave technique relies on liquid coupling. However, it is easier to relate the elastic constants to the vacuum surface wave velocity, so in the past the leaky wave velocity has been approximated in calculations by the vacuum surface wave velocity.

In support of material characterization work at NIST, a NIST scientist recently obtained a numerical solution for the velocity of surface waves on glass in water, a commonly used coupling fluid. The correction relative to surface waves on glass in a vacuum was about 1 %. Incorporating the corrected velocity values should lead to increased accuracy in determination of elastic constants.

## **NIST/IEEE CO-SPONSOR SMART SENSOR WORKSHOP**

NIST and the Institute of Electrical and Electronics Engineers co-sponsored a fifth workshop on Smart Transducer Interface Standards on Nov. 15-17, 1995, at NIST. More than 80 people attended. The objective of the workshop was to provide an open forum to discuss refining the proposed transducer interface standard for smart sensors and actuators. The proposed IEEE PI 451 specification is the result of a year-long industry/government effort involving large and small companies. The endeavor is not to develop another competing sensor bus or control network, but to define a standard transducer interface and common network object model by building on existing control networking hardware and software.

The standard transducer interface will ease the integration of a wide range of sensors and actuators to a network or system, while the common network object model will allow mapping sensor and actuator-related information onto the underlying network vendor's representation or protocol. The workshop started with presentations on the need and global economic impact of the standard, followed by an overview of the latest in sensor bus and control networking technologies. In the afternoon, specifications of the transducer digital

communication interface and transducer electronic data sheet (TEDS)—a key concept in the interface to support self-identification of sensor/actuator—were reviewed in detail.

The workshop subsequently addressed the concept of object modeling and object classes, and specifications for the transducer blocks were explained. Two demonstrations were given: one, a smart transducer to multi-network interoperability, and the other, a prototype application implementation of the standard. The workshop concluded with discussion on implementation concepts of the proposed standard for smart sensors and actuators, such as an integrated sensor, multivariable pressure transmitter, smart rotary actuator, and pressure-temperature sensor with a microprocessor.

## **NIST COORDINATES INTERNATIONAL LABORATORY TEST OF DNA PROFILING METHOD**

NIST is coordinating a major interlaboratory effort to validate a new test for human identification by DNA profiling. The test is based on the polymerase chain reaction (PCR) and allows the amplification and profiling of three different regions on a chromosome in a single run. NIST was selected as the coordinating site for the tests following discussions in June 1995 by the Technical Working Group on DNA Methods Analysis (TWGDAM) that reports to the National DNA Advisory Board.

The participating laboratories are being asked to provide genetic profiling of the human DNA from three different blood stains and extracted/purified DNA from two individuals. In addition, the DNA from two cell lines of SRM 2391, the NIST standard for PCR-based DNA profiling, is included for profiling. The genetic regions on the DNA being tested are from different chromosomes of the human genome and are termed CSF1PO, TPOX, and TH01 (commonly known as the CTT triplex).

Profiling will be performed by PCR amplification of the specific regions of the DNA samples followed by electrophoretic separation of the PCR products. Detection will be done by silver staining or by laser-induced fluorescence with a variety of instruments, including fluorimagers and automated gene sequencers.

Forty laboratories in 22 states and six foreign countries have asked to be included in the interlaboratory testing. Materials for the tests were donated by three commercial vendors of DNA reagents.

### **NOVEL MEASUREMENT METHOD FOR DETECTING PROTON PUMPING**

Bacteriorhodopsin (bR) is a large protein, which is isolated from the cell membranes of a bacterium. The purple color of the bacterium is due to retinal, a chromophore bound to the protein. bR has an intriguing ability to absorb light and to convert that light energy into intramolecular electronic and conformational changes that result in the transfer of a proton from one side of an insulating lipid membrane to the other. This capability has resulted in intense interest in bR as a potential component of electronic and optical devices, including night vision equipment and flat-panel displays.

While the optical properties of bR are relatively easy to study, measurement of the proton pumping ability often has been achieved by following the fluorescence of a pH sensitive molecule, a technique that is not entirely applicable to studying bR on a surface. Alternative electronic techniques detect only charge displacement without identifying the ions that carry the charge and thus are unable to ascertain whether the membrane protein is functioning properly.

Recently, NIST researchers have developed a method that allows for direct measurement of the proton-pumping activity of bR. The system is composed of a tin oxide electrode which responds to pH changes. The potential of the tin oxide layer changes as bR takes up or releases protons, and the amplitude of the resulting transient current associated with the reordering of the double layer is proportional to this change in proton concentration.

The detector is sufficiently sensitive that it permits monitoring of the effect of light on a monomolecular layer of proteins. It is the first measurement tool of its kind that allows direct measurement of the function of bR or similar proteins, because it responds only to up-take and/or release of protons.

### **CHEMICAL MECHANISM FOR FLUOROCARBONS IS PUBLISHED**

Recent work at NIST on the development of a detailed chemical kinetic mechanism for high-temperature fluorocarbon chemistry will appear as a special issue of *Progress in Energy and Combustion Science* in early 1996. It is also available as NIST Technical Note 1412, *Thermochemical and Chemical Kinetic Data for Fluorinated Hydrocarbons*. The work originated from a NIST effort to provide the underlying science for screening and predicting the performance of possible alternative chemical extinguishers to Halon 1301 (CF<sub>3</sub>Br), which is destructive to stratospheric ozone. A key aspect of the work was the heavy reliance on computational chem-

istry methods for obtaining thermochemical and chemical kinetic data where experimental data were unavailable. The chemical mechanism that was developed will find wide application in areas such as plasma processing, polymer formation, and atmospheric chemistry as well as fire suppression.

### **FEMTOCHEMISTRY AT SURFACES USING TUNNEL JUNCTIONS**

A promising new method for laboratory realization of control in hot electron femtochemistry at surfaces has been proposed by a NIST scientist. Both its conceptualization and theoretical basis were presented in an invited talk at "Femtochemistry: The Lausanne Conference" (Sept. 4-8, 1995), the major international forum for airing new developments in the emerging young field of femtochemistry.

Femtochemistry, as usually practiced, aims to control the intramolecular dynamics required for select chemical processes, by imaginative applications of ultrafast, temporally shaped and sequenced laser pulses. The new complementary method proposed at NIST involves a novel application of a solid-state metal-insulator-metal tunnel junction device to produce a monochromatic, tunable electron flux internally incident upon the outer metal-vacuum interface, where the controlled surface femtochemistry then occurs. Inelastic resonance scattering of the hot electron by a surface reaction complex can have a similar controllable influence on the molecular motion, hence chemical dynamics, as do the pulse sequences.

Model studies have demonstrated the feasibility of the proposition on elementary processes such as bond selective dissociation, desorption, and fragmentation of molecules adsorbed on metal surfaces. Mechanisms of this type point to a new, potentially fruitful symbiosis between the ultrafast domain of femtochemistry and the ultrasmall world of nanostructures.

### **BREAKTHROUGH IN DISCRIMINATING ANTHROPOGENIC FROM NATURAL SOURCES OF INDIVIDUAL CHEMICAL COMPOUNDS IN THE ATMOSPHERE**

Scientists at NIST collaborating with scientists from a private institution have demonstrated, for the first time, the ability to measure <sup>14</sup>C in individual, trace chemicals in atmospheric particles. The ability to measure tiny amounts of radiocarbon (<sup>14</sup>C) has revolutionized knowledge in such diverse fields as atmospheric chemistry, archaeology, and isotopic geophysics. In atmospheric chemistry, the fundamental significance of <sup>14</sup>C measurements is the ability to reliably and quantitatively distin-

guish fossil from biogenic sources of carbonaceous gases and aerosols, based on the fact that the fossil carbon sources are dead," with ages large compared to the  $^{14}\text{C}$  half-life of 5730 years, whereas the biogenic sources are, in effect, "living," with a  $^{14}\text{C}/^{12}\text{C}$  ratio of approximately  $1.2 \times 10^{-12}$ . The resulting scientific data permit, therefore, informed decisions about carbonaceous emissions sources that may affect human health, visibility, and even regional or global climate (through greenhouse effects and excessive absorption of solar radiation).

The NIST-private institute research builds on several years of pioneering work by NIST scientists in trace organic analysis and its application to urban particles, and atmospheric  $^{14}\text{C}$  metrology. The latter has been applied to the quantitative apportionment of urban and regional carbon in atmospheric methane, carbon monoxide, and aerosol particles, between fossil and biogenic sources such as motor vehicle emissions and residential woodburning. This past work has been instrumental in providing definitive answers to sources of atmospheric pollutants affecting such U.S. cities as Denver, CO, Los Angeles, CA, and Albuquerque, NM. The results have been critical in the development or validation of control strategies, as well as in establishing the limits of validity of other methods of apportionment that lack the definitive character of  $^{14}\text{C}$  technique.

The new work was presented as an invited talk at the "Symposium on Biogenic Hydrocarbons in the Atmosphere" at the December 1995 International Chemical Congress of Pacific Basin Societies. It was made possible by utilizing a special, well-characterized aerosol sample collected in Washington, DC: "urban dust" Standard Reference Material (SRM 1649). A representative portion of the SRM was processed at NIST for isolation of individual polycyclic aromatic hydrocarbon (PAH) compounds at the private institute for characterization by "radiocarbon dating" (accelerator mass spectrometry). This is the first time that such compounds have been "dated" in atmospheric aerosol.

The implications of this work are that: (1) we now have the capability of assigning fossil/biogenic sources to individual PAH compounds which are known or suspected atmospheric carcinogens or mutagens; and (2) the resulting compound-specific isotopic signatures can be used to rigorously define new "molecular markers" for the identification of suspected aerosol pollution sources. A case in point is the PAH compound, benzo[ghi]perylene (BghiP), which had been suggested previously as an urban fossil source combustion tracer on indirect, statistical grounds. For this particular sample, the results derived from the absolute,  $^{14}\text{C}$  tracer largely supported the above conclusion, based on the approximately 87 % fossil carbon content of the BghiP.

For other chemical fractions, such as the polar organic fraction, the NIST researchers found that biogenic sources were much more significant (only 62 % fossil), whereas the aliphatic portion was almost totally derived from fossil sources (98 % fossil).

#### NEW SYSTEM DEVELOPED FOR THE DIRECT TRANSFER OF FUNDAMENTAL WAVELENGTH STANDARDS

Wavelengths are the meter-sticks of nanotechnology, and scientists from NIST have developed a system to perform wavelength measurements of spectral lines to an accuracy unprecedented for broadband spectroscopy. The heart of the system is a UV/visible-Fourier transform spectrometer (FTS), one of four in the world of its type. A unique optical coupling scheme devised by NIST scientists allows the uncertainty associated with illumination effects (parts in  $10^7$ ) to be overcome and to achieve accuracy limited by the uncertainty of the measurement of the calibration standards (parts in  $10^9$ ).

The calibration factor, obtained by comparison to a single standard line, should be exactly the same for every standard line. However, preliminary measurements of argon lines revealed a trend in the calibration factor for different standards. The same trend was detected for well recognized  $^{198}\text{Hg}$  standard lines, and a series of experiments was performed, ruling out biases due to the operation of the  $^{198}\text{Hg}$  standard lamp or the operating conditions of the FTS. These accepted  $^{198}\text{Hg}$  standards in the UV and visible region were published in 1962 and were part of development work toward primary length standards. Four wavelengths from this publication were later designated as secondary standards of length by the Comité Consultatif pour la Définition du Mètre (CCDM), a consultative committee of the International Committee of Weights and Measures.

Recently an experiment by NIST scientists, using a frequency doubled laser, confirmed that the trend in the  $^{198}\text{Hg}$  wavelengths was not due to an FTS system artifact, but rather a bias in the reference wavelengths. Preliminary measurements referenced to an iodine transition, which is, in turn, referenced to a clock frequency, indicate a systematic bias of six parts in  $10^8$  for the reported wavelength standard "green" line of mercury (a discrepancy of 32 femtometers). A relative error of this size represents a difference of about 23 m between Earth and the moon. This standard line has been used over the years to calibrate spectra for assignment and calculation of atomic energy levels for several other elements in a number of laboratories. The team of NIST scientists will begin using this system to remeasure the mercury spectrum as well as other important reference spectra.

### **NEW METHOD TO FABRICATE GRATINGS FOR COUPLING LIGHT INTO PLANAR WAVEGUIDE SENSORS**

Scientists at NIST have submitted a patent application for a new method to fabricate gratings for coupling light into planar waveguide sensors. Planar optical waveguides are attractive tools for biological and chemical sensing. But, unlike optical fibers, the acceptance and use of planar waveguides has been limited due to the difficulty of coupling light into the waveguide.

Although either prisms or gratings can be used to couple light into planar waveguides, the NIST researchers have chosen to work with gratings and have developed a technique to fabricate them reproducibly directly onto the waveguides. To create such gratings easily and inexpensively, the researchers employ an embossing technique using a commercial grating as the master template. By chemically modifying the surface of the commercial grating with a suitable release agent, it is possible to replicate gratings in epoxy onto a variety of waveguide types. With care, this technique allows many replicates to be produced from a single master that have sufficient efficiency to be used as waveguide couplers. Furthermore, this technique is independent of waveguide type and is suitable for replicating grating profiles of different line spacing and groove shape. Because “submasters” or copies of gratings can be made readily and reused in conjunction with the new release agent, it is feasible to consider developing a custom grating for a particular application. This technology also should be applicable to other diffractive optical elements that are currently fabricated by holographic or lithographic means. Future work will concentrate on specialized gratings for white light applications.

### **PATENT ISSUED FOR FLOW INJECTION IMMUNOASSAY TECHNIQUE**

A patent entitled “Liposome Immunoanalysis by Flow Injection Assay” authored by two NIST scientists and a researcher was recently allowed. The patent describes a technology that utilizes liposomes, or synthetic cell-like structures, as detectable markers to measure the results of immunochemical reactions. Liposomes are attractive assay reagents since they have interior aqueous compartments for encapsulation of a large number of dye molecules providing substantial signal amplification for enhanced immunoassay sensitivity.

An important aspect of this patent is the unique combination of liposome immunoassays and flow injection analysis, which allows for rapid, sequential processing of samples for measurement of analytes in complex matrices. The assay is performed by mixing

the sample containing the analyte with analyte-coated liposomes. This mixture then flows through a column where antibodies are immobilized, and the analytes and liposomes in solution compete for binding to the antibody column. Liposomes that are not bound to the column may be measured as they flow out of the column and into a detector. The amount of bound liposomes is inversely related to the amount of analyte present in the sample. Therefore, the signal can be used to determine sample analyte concentration. Liposomes bound to the column may be also be used as a measure of analyte concentration by disrupting the bound liposomes with detergents and measuring the released dye at the detector. At NIST a device based on the patented technology has been used to detect both clinical analytes (therapeutic drugs, hormones, and antibodies) and environmental analytes (pesticides) with very high sensitivity.

### **CGPM ELIMINATES CLASS OF SUPPLEMENTARY UNITS IN THE SI**

The International System of Units (SI), the modern metric system, was established in 1960 by the General Conference on Weights and Measures (CGPM), an intergovernmental treaty organization created by the Meter Convention. (The Convention, 48 countries of which are now members, was initially signed in 1875 by 17 countries, including the United States.) The SI was established by the CGPM with three classes of units: base units such as the meter (m) for length, kilogram (kg) for mass, and second (s) for time; derived units such as the  $\text{m} \cdot \text{kg} \cdot \text{s}^{-2}$  for force (given the special name “newton” and symbol N); and the supplementary units radian (rad) for plane angle, and steradian (sr) for solid angle. However, the CGPM did not specify the nature of the supplementary units in relation to the base units and derived units. In 1980, the International Committee for Weights and Measures (CIPM), which is under the authority of the CGPM, interpreted the radian and steradian as so-called dimensionless derived units for which the CGPM allows the freedom of using or not using them in expressions for SI derived units. In 1995, the CIPM, at the request of its Consultative Committee for Units (CCU), asked the 20th CGPM to eliminate formally the class of supplementary units as a separate class in the SI. The 20th CGPM approved this request via Resolution 8 at its October 1995 meeting and thus the SI now consists of only two classes of units: base units and derived units, with the radian and steradian subsumed into the class of derived units of the SI. The option of using them or not using them in expressions for other SI derived units, as is convenient, remains unchanged.

**ISO TAG 4 PROPOSES CREATION OF FICOM**

Technical Advisory Group (TAG) 4, Metrology, is one of two TAGs currently providing advice to the Technical Management Board of the International Organization for Standardization (ISO). It consists of representatives from seven organizations: the International Bureau of Weights and Measures (BIPM); the International Electrotechnical Commission (IEC); the International Federation of Clinical Chemistry (IFCC); ISO; the International Union of Pure and Applied Chemistry (IUPAC); the International Union of Pure and Applied Physics (IUPAP); and the International Organization of Legal Metrology (OIML). The terms of reference of TAG 4 embrace two distinct types of concerns: (a) those directly related to the day-to-day work of a number of ISO technical bodies; and (b) those intended to promote coordination between broadly based institutions interested in harmonizing the basic concepts in metrology and their practical implementation in science and industry. To date, TAG 4 has focused on activities associated with (b). In particular, it has prepared and published the International Vocabulary on Basic and General Terms in Metrology (known as the VIM); and the Guide to the Expression of Uncertainty in Measurement (known as the Guide). Each has been widely distributed and each has gained world-wide acceptance and support.

Recognizing the difficulty of addressing concerns (a) and (b) in the same TAG, TAG 4 proposed at its November 1995 meeting that it be reconstituted with members from appropriate ISO technical committees in order to address concern (a); and that a new, completely independent body called FICOM—Forum for Inter-Organizational Cooperation in Metrology—be created to continue to address concern (b). The proposed terms of reference of FICOM are (i) to develop and maintain, at the international level, guidance documents addressing the general metrological needs of science and technology, and to consider arrangements for their dissemination; in particular, FICOM shall take responsibility for maintaining and up-dating the VIM and the Guide; (ii) to promote worldwide adoption and implementation of the results of its work; (iii) to provide advice, when requested, on questions related to the implementation of its guidance documents; and (iv) to assist in the coordination between the member organizations on subjects concerned with general metrology. The initial composition of FICOM would be BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, and OIML; however, membership would be open to other such international, broadly based organization at FICOM's discretion. ISO would provide the secretariat for FICOM, at least for the first several years of its existence. The proposal is to be sent to the seven founding organizations for their comment and approval, and it is hoped that the first meeting of FICOM can be held in November, 1996.

**SPONTANEOUS PATTERN FORMATION IN GROWTH OF IRON FILMS**

The growth and structure of semiconductor and magnetic thin films are closely linked to the performance of electronic, optoelectronic, and magnetic devices employing these materials. Current trends in industry are requiring film thickness variations or roughness controlled to the single atomic layer level. High-purity, well-ordered films can be realized by molecular beam epitaxy. However, the structure of such films varies widely depending on how effectively the roughness induced by the random nature of the impinging atom flux is counterbalanced by a smoothing process resulting from the mobility of the atoms diffusing on the surface.

Measurements of multilayer homoepitaxial growth, in this case iron on an iron (001) surface, have been carried out at NIST with the aim of better understanding the processes that control roughness in epitaxial growth. Rather surprisingly, the films do not exhibit scaling properties postulated by a number of theories, but rather there is a pattern formation imprinted in the film resembling a mosaic of tile patterns. The pattern is characterized by a well-defined separation, initially determined by the first atoms that were deposited which formed two-dimensional islands. Such behavior occurs at temperatures such that energy barriers at the island edges inhibit the downward transport of atoms to the next terrace relative to diffusion on a terrace. In this way, atoms tend to remain on the terrace on which they were deposited and a wedding-cake island structure develops. The very good agreement between these experiments and theoretical simulations incorporating step edge diffusion barriers indicates that the theoretical understanding of the fundamental processes of growth is developing to the point where it can be used to help grow films tailored to specific applications. These results were reported in the Dec. 4, 1995, issue of Physical Review Letters.

**LIQUID CRYSTAL DISPLAYS: BURIED INTERFACES CHARACTERIZED**

There are many methods of characterizing properties of solids, liquids, and gases, and for probing the interface between a solid and vacuum or air. However, probing buried interfaces, such as between a semiconductor and a metal, or between a liquid and a solid, is more difficult. A new optical approach, uniquely sensitive to interface properties and with high spatial, spectral, and temporal resolution, is the nonlinear spectroscopy method called sum frequency generation (SFG). In this method, two laser beams of very short pulses and different tunable colors reflect from an interface. Because of the broken symmetry at the interface, the two beams



combine to give a third beam at a frequency that is the sum of the two input frequencies (SFG is symmetry forbidden in bulk liquids, gases, and centrosymmetric solids such as silicon). The efficiency of the process depends on the molecular structure at the interface and the wavelengths and polarization of the lasers. An SFG spectrum thus provides information similar to absorption spectroscopy or ellipsometry but with special interface specificity.

In collaboration with scientists from a private company, a team of NIST scientists has done SFG studies to characterize the polymeric interfaces for liquid crystal displays (LCDs). In a LCD, the liquid crystal molecules that orient in response to the applied electric field must be pre-aligned. This "pretilt" of the molecules is imposed by contact with a thin alignment layer applied to the glass or transparent electrode between which the liquid crystal material is sandwiched. The standard method of inducing anisotropy in the alignment layer is to mechanically rub it in one direction. However, the chemistry and physics of the alignment process are not understood. Therefore, the NIST researchers used SFG to study alignment layers, finding that the SFG signal could be correlated to the quality of the LCDs assembled on the layer. Future work will include SFG studies in which one laser is in the infrared, resonant with the absorption of particular chemical bonds of the alignment layer, in order to understand the chemical basis for the observed alignment effects.

#### **A NEW DATABASE OF OPTICAL CONSTANTS FOR X RAYS**

A comprehensive new database for atomic scattering factors has been published (*Journal of Physical and Chemical Reference Data*, Volume 24, No. 1, pp. 71-643). This theoretical formulation retains robust empirical connections while offering functional approximations that have good analytic properties over their entire domain. The database provides scattering and absorption coefficients over the very broad range of photon energies from 30 eV to 300 keV. The improved accuracy offered by this database permits more effective modeling of the performance of multilayer optics for x rays and more efficient analysis of crystal structures. Such modeling and analysis can lead to improved performance of x-ray telescopes for astronomy and x-ray microscopes for biology. The work was done in collaboration with a University of Oxford Fellowship awardee working at NIST.

#### **NIST-DESIGNED CRYSTAL MONOCHROMATORS PROVIDE CALIBRATION RESOURCES FOR SPACE ASTRONOMY**

Three high-resolution double crystal monochromators have been designed, constructed, and delivered to NASA programs in x-ray astronomy. One such instrument is currently installed at the Marshall Space Flight Center in Huntsville, AL. It stands at one end of a nearly 1 km vacuum chamber and illuminates the entrance aperture of the AXAF (Advanced X-ray Astrophysics Facility) telescope soon to undergo preflight testing. This monochromator (as well as the other two described below) has a unique design within which a parallel (displaced) entrance beam illuminates the x-ray telescope. Five alternative crystal pairs mounted on pentagonal turrets can be manipulated under computer control to give x-ray standards from 0.3 keV to 12keV.

A second multichromator was delivered to NASA's Goddard Space Flight Center and is being used as a calibration source for the cryogenic bolometer spectrometer planned for deployment in the ASTRO-E mission scheduled for launch aboard a Japanese satellite in 1999. The main goals of this monochromator are to provide calibration markers at characteristic x-ray wavelengths and continuously tunable radiation through the critical absorption thresholds of the materials used in construction of the detectors and their associated window materials. By means of such calibrations, the spectrometer, once in orbit, can provide objective maps of the spectral distribution of radiation incident on earth.

The third instrument of this group was delivered to the program associated with the recently launched X-ray Timing Explorer.

#### **CIRMS HOLDS FOURTH ANNUAL MEETING AT NIST**

The Council on Ionizing Radiation Measurements and Standards (CIRMS) held its fourth annual meeting at NIST on Nov. 28-30, 1995. The organization represents thousands of users of ionizing radiation and radioactive sources engaged in industrial radiation processing and sterilization, medical radiation diagnostics and therapy, nuclear power, and worker radiation protection programs. CIRMS provides a forum for discussing ionizing radiation issues; identifying, defining, and prioritizing needed work; disseminating information on standards; and organizing workshops and meetings to advance ionizing radiation technology.

More than 100 participants attended the meeting, which highlighted advanced techniques in radiation dosimetry and radioactivity measurements for the different ionizing radiation communities. Representatives attended from 28 corporations, 10 federal agencies, eight national laboratories, 12 universities, and one state agency. Advanced techniques and future measurement needs were discussed in four areas: radiation effects, medical applications, public and environmental radiation protection, and occupational radiation protection. An additional session was added to this annual meeting on the implementation of ISO 9000 for those CIRMS members involved in instrument and product manufacturing, and those providing radiation measurement services. Participation this year was particularly high from the secondary calibration laboratories in the different fields and from small businesses involved in radiation detection instrument manufacturing, as CIRMS provides a forum for these groups to discuss directions for new programs and instrument needs.

#### **DOSIMETRY FOR INTRAVASCULAR RADIATION SOURCES**

An exciting development in coronary medicine is the use of beta-particle emitting sources for the prevention of restenosis (reclosing) of blood vessels after cardiac and peripheral artery interventional procedures such as angioplasty, atherectomy, and stent implantation. More than 400 000 of these procedures are performed each year in the United States, and restenosis occurs in about 30 % of the cases, requiring an additional treatment. Research has shown that a radiation dose of about 10 Gy, delivered to the wall of the blood vessel after such procedures have been performed, is effective in inhibiting restenosis.

Building on the expertise with dosimetric calibration of encapsulated radiation sources (so-called brachytherapy sources), NIST has taken an early and leading role in the calibration of absorbed dose from the catheter-based beta-particle sources to be used for this therapy. The calibration procedures are based on measurements with the NIST extrapolation chamber of the dose rate at a depth of 2 mm in water. These measurements are confirmed using radiochromic dye film, which also is used to characterize the cylindrical sources for uniformity and to provide the dose rate at arbitrary locations around the sources. A publication describing this work is in progress, and collaborations with other workers in this field are in the planning stages. The NIST calibration procedure for beta-particle brachytherapy sources will be adopted by the American Association of Physicists in Medicine in the recommendations

of Task Group 60 on Intravascular Brachytherapy, and by the International Commission on Radiation Units and Measurements in their upcoming report on beta-rays for therapeutic applications.

A recent 2 day conference, "Discoveries in Radiation for Restenosis" held in Atlanta, brought together cardiologists, radiation oncologists, medical physicists, industry representatives, and Federal regulators. A conclusion of this conference was that preliminary studies by several different groups using a variety of radiation sources and dose delivery techniques indicate that this form of therapy is very successful in reducing the incidence of restenosis after angioplasty. Routine use of radiation for the prevention of restenosis would result in savings in U.S. health care costs estimated at between \$0.8 and \$2 billion annually.

#### **VOLTAGE NOISE IN CHEMICAL CELLS**

A NIST scientist, working with students at the University of Colorado, recently reported measurements of voltage noise for a variety of chemical cells. Chemical cells often have been used in electronics for their low current drift, isolation of a particular circuit from other circuits, and low voltage noise. While it is clear that voltage noise on these cells is small, actual values for the voltage noise have not been reported, so there has been little evidence suggesting that one type of cell might be better than another.

This work was made possible by the development of a cross correlation measurement system capable of measuring voltage noise below  $-200$  dB (1 V/Hz). The method involves two parallel measurement channels. Because the measurement noises in these two channels are completely independent, a cross correlation of the outputs of the two channels effectively rejects the measurement noise while recovering the noise on the device under test.

Nickel-cadmium cells exhibited the lowest noise of all cells tested over the frequency range from 1 Hz to 60 kHz. An AA size nickel-cadmium cell showed a noise of  $-205$  dB (1 V/Hz) at 10 kHz. This noise level is consistent with the Johnson noise produced by the  $0.2 \Omega$  internal resistance of the cell. The study involved nickel-cadmium, alkaline, lithium, and mercury cells. A general conclusion of the study was that the dominant broadband noise process in cells is the Johnson noise arising from the internal resistance. This clearly indicates that larger capacity batteries with lower internal resistance should produce lower voltage noise. The voltage noise was found to be independent of bias current suggesting that shot noise is not significant. The results of this study should provide guidance in electronics applications demanding the lowest possible levels of voltage noise.

### RABI PEDESTAL SHIFTS AS A DIAGNOSTIC TOOL IN PRIMARY FREQUENCY STANDARDS

NIST scientists, in collaboration with a scientist from the Laboratoire Primaire du Temps et des Fréquences in Paris, have developed a new method for evaluating certain systematic frequency shifts in primary cesium frequency standards. It uses a digital servo system to measure the frequency offset between the Rabi pedestal and the Ramsey fringe for all seven of the components of the hyperfine transition observed in the standard. Measurement of the dependence of each of these shifts on microwave power enables one to separate three distinct causes: Rabi pulling, cavity pulling, and magnetic field inhomogeneity.

The method was used to evaluate these three shifts for NIST-7, NIST's new optically pumped cesium-beam frequency standard. The method indicates a shift due to magnetic-field inhomogeneity of  $2 \times 10^{-15}$  a shift due to cavity pulling of  $6 \times 10^{-15}$ , and a shift due to Rabi pulling of less than  $1 \times 10^{-16}$ . One advantage of the method is that it requires frequency measurements with an uncertainty of only  $1 \times 10^{-11}$ . This work is part of a continuing effort to find additional independent methods for measuring each of the systematic errors in NIST-7. The shifts measured in this manner are consistent with previous measurements using other methods, and add confidence to the NIST-7 uncertainty statement, which is now  $5 \times 10^{-15}$ .

### AN IMPROVED VARIANCE FOR CHARACTERIZING OSCILLATORS

A NIST scientist has developed an improved variance, closely related to the Allan variance, that provides an increased confidence level at long averaging time. This is important because the long-term data, requiring longer measurement time, are the most costly to obtain. As the method is adopted, manufacturers of oscillators can expect to either reduce measurement time or increase measurement accuracy.

The improvement follows from the simple observation that the procedure for the Allan variance, sometimes called the two-sample variance, measures only frequency variations with an odd symmetry at and near the longest averaging time, resulting in a bias. This work constructs a three-sample variance of even symmetry to improve the confidence interval. The process bears a similarity to the method of complex demodulation used in signal processing. The gain in measurement confidence depends on the noise type involved. For white phase-modulation noise, the improvement for the longest averaging time in a data set can be greater than an order of magnitude. The improvement is significant, but not quite as dramatic, for higher order noise processes.

### OBSERVATION OF A SCHRÖDINGER CAT STATE

In recent experiments NIST scientists generated a "Schrödinger cat-like" state of matter at the single atom level. In 1935 Schrödinger developed a thought experiment where a cat is placed in a quantum superposition of being dead and alive (correlated with a single radioactive atom which has and has not decayed, respectively). The state of the system is represented by an entangled quantum mechanical wavefunction involving a superposition of the two different states. This situation of course defies our sense of reality, where we only observe live or dead cats and we expect that only live and dead cats exist independent of our observation. This is a classic illustration of the conflict between the existence of quantum superpositions and our real world experience of observation and measurement. Although superposition states such as Schrödinger cat states appear to be absent from the macroscopic world, there is great interest in the creation of "Schrödinger cat-like" states in mesoscopic systems (systems having both microscopic and macroscopic features and hence bridge the gap between the quantum and classical worlds). These types of experiments may provide an interesting proving ground in the controversial theory of quantum measurement.

In the experiments, a single laser-cooled and trapped  ${}^9\text{Be}^+$  ion is prepared in a quantum superposition of two separate localized positions correlated with different internal states of the ion. This state is prepared by applying several pulses of laser radiation, which "entangle" the internal (electronic) and external (motional) states of the ion. The superposition is verified by detecting the quantum mechanical interference between the localized wavepackets. Of critical importance in the conduct of these experiments is the high level of control of the motion of the ion, from the initial laser cooling to the zero-point of energy to the excitation to higher-energy coherent states of motion in the harmonic potential.

### IMPROVED CO<sub>2</sub> LASER

A NIST scientist, using a new grating produced by an optical manufacturer, has developed a CO<sub>2</sub> laser that oscillates on about 250 lines with a maximum output power of 40 W. Most CO<sub>2</sub> lasers will oscillate on only about 80 lines. The laser uses a grating to couple power out of the laser in zero order. This new laser promises to be an excellent infrared frequency and wavelength standard as well as a source of radiation for pumping far-infrared lasers.

Three years ago, the scientist developed a ribbed laser tube that greatly increased the grating resolution of the laser cavity. This provided a substantial improvement in

laser performance, because the ribbed structure reduces or eliminates modes associated with radiation bouncing off the walls of the tube. The latest improvement is a direct result of the improved grating, which couples out 3 % of the power over the region from 9  $\mu\text{m}$  to 11.5  $\mu\text{m}$ . The new grating, with 150 lines per millimeter, was developed by an optical manufacturer in direct response to NIST requirements and was tested and proven at NIST. The laser has a mirror separation of 2.67 m and a diameter of 16.3 mm.

#### FUNDAMENTAL LIMITS ON THE FREQUENCY STABILITIES OF CRYSTAL OSCILLATORS

A NIST scientist and a researcher from the U.S. Army Research Laboratory recently published a review of the instabilities in precision bulk-acoustic-wave (BAW) quartz crystal oscillators. This is the most comprehensive review of this topic in the literature. Their examinations of the fundamental limits on achievable frequency stabilities and the degree to which these fundamental limits have been approached provide researchers with a road map for improving the performance of oscillators. Highlights of their study include thermodynamic limits to temperature stability, the limits imposed by noise generated in the electronic sustaining stage, the effects of static and dynamic temperature fluctuations, and the possible role of background ionizing radiation on long-term frequency drift. They conclude their study with a discussion of the ideal resonator and suggest a levitation method for suspending a resonator so as to eliminate (or minimize) the nonideal effects of resonator suspensions.

#### IMPROVEMENTS IN THE AT1 TIME SCALE

Three NIST scientists have implemented a number of improvements in the AT1 time scale allowing NIST to more accurately realize Coordinated Universal Time (UTC) in real time. Over a period of 280 days, UTC (NIST) has been held within 50 ns of the international UTC maintained by the BIPM. This provides a more accurate time scale for dissemination to high-end users in the United States and improves the quality of input data to the BIPM on the frequency of NIST's primary-frequency standard, NIST-7.

The key improvements involved the addition of new commercial hydrogen masers and cesium-beam standards to the scale and the development of new reset procedures in the AT1 algorithm. The fractional-frequency drift rate of the scale was reduced from  $1 \times 10^{-16}$  per day to  $3 \times 10^{-17}$  per day. The level of random

fluctuations of AT1 also was reduced by a factor of two to  $2 \times 10^{-15}$  using an averaging time of 100 days. These improvements continue a long history of world leadership in time scale operation. Further improvements are expected as two additional masers are added to the scale in 1996.

#### IR LASER STUDIES OF OZONE CHEMICAL CHAIN REACTION KINETICS

In the past decade there has been a steadily growing concern about the chemistry of the ozone layer, and in particular the influence of man-made sources of chemicals on the atmosphere. One of the dominant chemical reaction cycles responsible for the removal of ozone ( $\text{O}_3$ ) is the so-called  $\text{HO}_x$  chain cycle,  $\text{OH} + \text{O}_3 \rightarrow \text{HO}_2 + \text{O}_2$  and  $\text{HO}_2 + \text{O}_3 \rightarrow \text{OH} + 2\text{O}_2$ , which cycles OH into  $\text{HO}_2$  and back, thereby catalytically converting  $\text{O}_3$  into  $\text{O}_2$ . This has led to considerable concern with regard to proposed high-speed air traffic in the upper troposphere and lower stratosphere, which would release considerable amounts of water vapor (and  $\text{NO}_x$ ) into what would otherwise be a quite "dry" region of the atmosphere. Kinetic information on the  $\text{HO}_x$  chain reaction has, therefore, assumed particular importance in developing reliable atmospheric models. However, both steps in the chain, particularly the  $\text{HO}_2 + \text{O}_3$  step, are slow and require relatively high ozone concentrations to probe reliably. One serious technical problem is that the ultraviolet light used in the ultraviolet laser fluorescence method for monitoring the OH is strongly absorbed by the  $\text{O}_3$ , and furthermore can distort the kinetics by unwanted photolysis processes that lead to the generation of additional OH radicals.

NIST and JILA scientists recently developed alternative methods to investigate the  $\text{HO}_x$  chain cycle by monitoring the concentrations of OH radical directly by infrared laser absorption. By detecting OH in the near infrared, this method cleanly circumvents problems associated with ultraviolet laser fluorescence detection of OH radical. This IR method now permits operation at more than an order of magnitude higher ozone concentrations and has led to significantly improved rate constants for the first step in the chain reaction. The results indicate that the room temperature rate constant is considerably faster (20 % to 30 %) than previously recommended values. The method also permits the much slower step of the  $\text{HO}_x$  chain reaction to be observable directly in a real time measurement. This work will be important in reassessments of the various effects on atmospheric models.

## WORKSHOP ADDRESSES MOISTURE EFFECTS ON POLYMER PERFORMANCE

NIST recently organized a workshop, “Hygrothermal Effects on the Performance of Polymers and Polymeric Composites,” to identify moisture problems in polymer materials and processes that are important to industrial competitiveness, define scientific and technical needs, and provide a research agenda for developing theories, models, and better measurement techniques to address these problems. There were 24 participants from the private sector representing 17 companies, 12 representatives from the academic community and 13 NIST staff.

The workshop was divided into three sections: (1) current knowledge base, (2) moisture concerns in electronics packaging and composites, and (3) moisture issues in automotive applications. Participants identified needs in fundamental understanding and measurements of moisture effects on polymers and their end-use products. Representatives from the electronics industry were concerned about moisture absorption at the interface between the polymer and electronic components and its effects on reliability of electronic systems. Participants from the composites and automotive industries sought better measurement procedures to obtain data relevant to interfacial effects, including adhesive fracture and changes in the fracture response due to plasticization or stress cracking. Several participants identified changes in the glass-transition temperature with moisture uptake, which affect service temperature as a common issue. Finally, there was general agreement that some of the problems in estimating the impact of moisture, either in general or at interfaces, arise from contamination of materials and interfaces by substances that absorb moisture. Few studies of such effects have been made on materials relevant to these industries.

The workshop ended with the formation of working groups that cover two areas of research: (1) effects of moisture on the mechanical response of polymers with initial emphasis on viscoelasticity and fracture; and (2) influence of contaminants in the materials on the up-take of moisture and its deleterious effects at interfaces. The working groups have the goal of developing collaborative research efforts, involving investigators from industry, universities, and NIST.

## HIGH-SPEED LASER POLARIMETRY SYSTEM DEVELOPED

A high-speed laser polarimetry system for measurements of normal spectral emissivity of materials in pulse-heating experiments at high temperatures has been developed jointly by a NIST scientist and a scientist from a private company. This system, the first and the only one of its kind, consists of a novel high-speed laser

polarimeter integrated into the pulse-heating facility available at NIST for thermophysical measurements in the range 1300 K to 4000 K. The technique is based on rapid resistive self-heating of the specimen from room temperature to near its melting region in less than 1 s and on measuring, with millisecond-resolution, the radiance temperature of the specimen and the polarization states of the laser radiation reflected from the surface of the specimen.

Unlike the conventional methods for normal spectral emissivity measurements, the laser polarimetry technique does not require a blackbody configuration for the specimen. Performance of the system was assessed by measuring the normal spectral emissivity (at 633 nm) of molybdenum in the range 2000 K to 2800 K with the new laser polarimetry technique simultaneously with a conventional method involving spectral radiometry (pyrometry). Satisfactory agreement (within 1 %) between the results of the two different techniques suggests that the laser polarimetry can be a valid technique for measurements of normal spectral emissivity of metals and alloys at high temperatures. Normal spectral emissivity is not only an important property in its own right but plays a very significant and often essential role in the determination of the true temperature of a specimen from radiometric measurement of its surface radiance temperature. The polarimetry technique has the distinct advantage in measurements at high temperatures where it is either very difficult or impossible to have a blackbody configuration for the specimen to permit direct radiometric measurement of its true temperature.

## THERMAL ANISOTROPY IN POLYCRYSTALLINE $\text{Bi}_2\text{Te}_3$

Scientists are using a thermal wave technique to study thermal diffusivity anisotropy in functional ceramics. While nonisotropic thermal properties are crucial to many applications of functional ceramics, measuring these properties in polycrystalline materials as a function of crystallographic alignment is very difficult. To develop tools to make these types of measurements, a thermal wave technique based upon the mirage effect has been used to measure thermal diffusivity in  $\text{Bi}_2\text{Te}_3$ , a material with strongly anisotropic thermal and electric properties. Results show that this technique can measure even small variations in thermal diffusivity as a function of crystallographic alignment (texture) with high precision and reproducibility. For example, changes in thermal diffusivity from  $(0.013 \pm 0.0002) \text{ cm}^2/\text{s}$  to  $(0.016 \pm 0.00045) \text{ cm}^2/\text{s}$  have been observed as the material becomes more highly aligned (i.e., from being nearly random to being 50 % aligned, within 20 °).

Although the changes in diffusivity are small, they are easily distinguished by the thermal wave technique. Not only is the technique sensitive to thermal variations, but it is also nondestructive and can be used to analyze thin films, multilayers, composites, and monolithic materials. This information also can be used to develop models that predict the properties of anisotropic materials.

#### **IMPLEMENTING AGREEMENT BETWEEN NIST AND KOREAN INSTITUTE OF ENERGY RESEARCH**

In September 1995, an Implementing Agreement was signed between NIST and the Korean Institute of Energy Research of the Republic of Korea. The agreement is for a period of 5 years, and its purpose is to exchange scientific and technological knowledge and to encourage the joint research in the field of energy technology. The first 2 years of this agreement will involve conducting joint research and the possible exchange of personnel to develop methods for the automated real-time performance optimization, fault detection, and diagnosis of thermal systems, especially HVAC processes to improve energy efficiency, increase safety and reliability, and reduce operating cost.

#### **NEW PUBLICATION PRESENTS INFOSERVER CASE STUDY**

NISTIR 5757, *Sharing Information Via the Internet—An Infoserver Case Study*, describes NIST's experiences using software based on open standards to disseminate documents electronically on the Internet. The report discusses electronic mail, anonymous, FTP, Gopher, Z39.50, and the World Wide Web (WWW). It outlines installation and operation requirements for hardware and software, and gives guidance on implementation decisions.

#### **TECHNIQUE OF BASIS SET TESTING DESCRIBED**

NISTIR 5737, *A Method to Determine a Basis Set of Paths to Perform Program Testing*, examines a technique to determine a set of paths for basis testing and proves that the algorithm that is used fulfills the requirements of basis testing. A major problem in unit testing of programs is to ascertain which test cases to use. There must be sufficient tests to do thorough testing but not so many tests that the limited testing time is used up. The basis set testing technique takes the control flowgraph from each of the program functions and calculates a basis set of test paths, providing a good starting set of test cases. Also described is a prototype tool, Stest, built to implement the basis set algorithm.

#### **NIST COLLABORATES WITH THE AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI) ON ELECTRONIC ACCESS TO STANDARDS**

NISTIR 5708, *Electronic Access to Standards on the Information Highway*, describes an ongoing NIST testbed effort to provide technical assistance to the National Standard Systems Network (NSSN) directed by ANSI. The report discusses the use of the information highway to support electronic access to standards and standards information. It outlines how the NIST testbed effort contributes to the evolution of an architecture to support access to standards on the information highway by viewing the domain of standards as a virtual library. The document concludes with a description of future testbed investigations, including more complex documents, advanced document content and structure searching, automatic maintenance of hyperlinks and indexes, collaborative authoring tools, and security billing.

#### **TELECOMMUNICATIONS SECURITY GUIDELINES ISSUED**

NIST Special Publication 800-13, *Telecommunications Security Guidelines for Telecommunications Management Network*, gives guidance on enhancing the security of the Public Switched Network (PSN), which provides critical commercial telecommunications services and National Security and Emergency Preparedness (NSEP). The guideline focuses on two specific components of a Telecommunications Management Network (TMN), Network Elements (Nes) and Mediation Devices (Mds), with emphasis on the security features need to protect the operations, administration, maintenance, and provisioning of these components.

The document can assist telecommunications vendors in developing systems and service providers in implementing systems and service providers in implementing systems with appropriate security for integration into PSN. It also is useful to government agencies or commercial organizations in formulating a specific security policy.

#### **NIST OFFERS IMPROVED MAMMOGRAPHY X-RAY STANDARDS**

Women undergoing mammography exams at licensed U.S. facilities soon will have greater assurance of receiving proper x-ray exposure thanks to a new radiation standard and instrument calibration facility at NIST. The new facility will allow the operators and inspectors of more than 10 000 U.S. mammography centers to trace the accuracy of their x-ray exposure measurements to the primary x-ray standards at NIST. The calibration facility was established to help the U.S. Food and Drug

Administration implement the Mammography Quality Standards Act of 1992. This law requires the FDA to set up a certification and inspection program for U.S. mammography clinics. The instruments used by the FDA inspectors to measure the x-ray exposure from the clinical units will be calibrated using standards traceable to the new NIST reference x-ray beams. Anyone wanting to calibrate an x-ray exposure measuring instrument for mammography beams may send the instrument to NIST. Typical users of this service are expected to be secondary calibration laboratories, inspectors of mammography units, and instrument manufacturers. For information on the new facility, contact Michelle Johnson, C229 Radiation Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2014.

### STEP MAKING GREAT STRIDES

It took more than a decade to go from the ambitious, but abstract goal of creating a universal system for electronic exchanges of manufacturing product data to the official international and U.S. standard published in early 1995. In the year since, however, the initial release of the Standard for the Exchange of Product Model Data, or STEP, has progressed rapidly to useful reality and matter of policy for several major U.S. manufacturers. A major aircraft manufacturer, for example, announced in December 1995 that it will buy only products that comply with STEP and two other data exchange standards. Meanwhile work is proceeding on new elements of STEP. The standard is evolving to extend data exchange capabilities to all aspects of a product's life cycle—from material specifications to after-sale maintenance requirements—and to many industries beyond metalworking, the initial focus of STEP applications. The International Standards Organization subcommittee (ISO TC184/SC4) that is spearheading the development of STEP has elected its new chairman, who has extensive experience in the area of manufacturing information systems research and applications. He will be stationed at NIST, host to the subcommittee's secretariat. A NIST computer systems researcher who specializes in product-data exchange technology was recently appointed as the new executive secretary to ISO TC184/SC4.

### PARTNERS TO BETTER ASSESS "COATS OF MANY COLORS"

More than 30 years of technological advances and environmental requirements have transformed the nation's \$14 billion-a-year paint and coatings industry. Colors of virtually every shade add hundreds of billions of dollars of value to products from automobiles to bulk plastics.

Industry's ability to measure and predict appearance, however, has remained mired at pre-1960 levels. For instance, at least 10 different metrics exist for measuring "gloss." Old metrics also are considered inadequate for new coatings such as metallic and pearlescent finishes. A NIST team has set out to work with industry to change that situation. "In this era of global competition and ISO 9000 requirements, it is important that industry have the scientific tools necessary to accurately quantify the appearance and lifetime of their products," said a NIST scientist. NIST will host a workshop on May 20, 1996, at NIST's Gaithersburg, MD headquarters to discuss a framework for cooperative work in coatings appearance. Industry and government participants will consider a provisional technical plan, a research timetable, and the makeup of an advisory committee. Sponsors hope that the application of advances in optical technology (such as detectors, light scattering, and imaging), coupled with modern computational technology, should result in much more predictable and detailed understanding of appearance parameters. Interested parties should contact Jonathan Martin at B348 Building Research Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6707, e-mail: jonathan.martin@nist.gov.

### HIGH-FLYING EXPERIMENT SEEKS SAFER SPACEFLIGHT

A NIST-led experiment aboard the recent STS-75 space shuttle mission could help keep the future international space station and its crews safe from fire. NASA and NIST conducted the experiment, the Radiation Ignition and Transition to Spread Investigation (or RITSI), to look at how flames behave in microgravity. The researchers hope to use data from the experiment to learn how to keep sparks from growing into a life-threatening fire aboard a spacecraft. "The trick is to contain the momentary ignition, avoiding any possibility of flame spread," says a NIST materials research engineer serving on the RITSI team. RITSI data, he adds, will allow scientists to compare actual experimental results with computer simulations of fire behavior in near-weightless conditions. Detailed information on the RITSI experiment can be found on the NASA World Wide Web site via Internet at <http://zeta.lerc.nasa.gov/expr/ritsi.htm>. In a related announcement, NASA has chosen a NIST mechanical engineer to serve as a payload specialist aboard the Microgravity Science Lab space shuttle mission (STS-83) scheduled for March 1997. He will conduct experiments in fluid physics, combustion science, and materials science during the 7 d flight.

**NIST HELPS POWER GRIDS MAKE  
“LIGHTNING SAVES”**

A NIST technique has been incorporated into the latest version of an electrical standard used to test the ability of electric power grid high-voltage equipment to survive lightning strikes. Now a part of IEEE Standard 4-1995, “Standard Techniques for High-Voltage Testing,” the technique improves the measurement of specific high-voltage impulse waveforms that simulate lightning. Such test waveforms subject the equipment to up to 10 times its normal operating voltage. To certify that power grid equipment is lightning-proof, engineers must evaluate the strength of the waveform. They use a “high-voltage divider” device to scale the voltage down to levels that can be measured. However, the process can produce a distorted reading. The NIST technique improves the measurement process by enabling testers to mathematically estimate the distortion. The formulation offers an easy way to compare the input to the distorted output. Using the NIST technique, engineers no longer need a second divider to serve as a reference. The check can be done quickly at low voltage with a digital recorder to measure the response waveform and a personal computer to perform the calculations. Further, the test operator can see the distortion introduced by the divider by comparing the graphics of the input waveform and the calculated output. For more information, contact Gerald J. FitzPatrick, B344 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2737, e-mail: fitzpa@eel.nist.gov.

**SOFTWARE REDUCES TOUCH PROBE ERRORS**

A NIST computer model that compensates for a common source of error in coordinate measuring machines promises to greatly increase manufacturers’ ability to accurately check the shape and dimensions of products. The new SuperFit software corrects “probe-lobing” errors, a chronic and relatively large source of measurement uncertainty in 98 % of the some 30 000 CMMs in U.S. factories and laboratories. Such errors stem from small variations in the performance of touch trigger probes and depend on the angle that the probe approaches a part being measured. When programmed with data gathered during a simple test, the software anticipates and then compensates for probe-lobing errors, reducing them by as much as 90 %. Today, CMM users must pay a premium to eliminate the measurement uncertainty caused by probe-lobing errors. They can purchase CMMs with specialized probes that are as much as 10 times more expensive than the touch-trigger variety, placing them beyond the budgets of many CMM users. The NIST software could erase much of the trade-off between price and accuracy. Several U.S. CMM

manufacturers are now evaluating the software. Working with the NIST team, they are determining whether the model meets the specialized requirements of their particular products and whether refinements and adaptations are necessary. One CMM maker already has decided to refine and integrate the NIST error-compensation technology into future products. For more information, contact Steven Phillips, B113 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3565.

**PAPER SHOWS FIBER HEALTHY  
FOR COMMERCIAL SENSORS**

The Faraday effect—whereby the plane of polarization of a light beam is rotated by a magnetic field—has been used as a nonelectrical tool for measuring magnetic fields and electric currents for over 100 years. However, most of this measurement work has been in scientific laboratories. Because of recent advances in technology, current sensors utilizing optical fibers are being developed that meet the accuracy and durability needs of a large commercial sector: the electric power industry. The route to commercial success has been difficult; only within the last few years have significant numbers of optical current sensors been placed in service. A new paper from NIST discusses the fundamentals and problems of fiber current sensors, and should be helpful to those interested in applications of this technology. The paper describes the latest commercial developments and goes into detail on current research efforts to reduce sensor costs, increase accuracy, improve immunity to vibration, and create more rugged devices. Among topics discussed are improved techniques for removing fiber birefringence, low stress-optic coefficient fibers and compensation techniques. For a copy of paper no. 6-96, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, e-mail: sarabeth@micf.nist.gov.

**BOILING DATA OFFERS COOLER TOMORROW**

Recent NIST studies of the boiling process may help improve the efficiency of tomorrow’s large building air conditioners. In an air-conditioner chiller, water flows inside tubes and boils the refrigerant that surrounds them. The process chills the water that is used to cool large structures. Most of the data available for the design of chillers have been derived from tests in which refrigerants were boiled on heated electrical tubes. To determine how accurate such data are, a NIST mechanical engineer compared measurements of boiling induced by flowing water heat with those induced by electric resistance heat. He used four popular commercial heat



transfer surfaces in a pool of the alternative refrigerant-123. The result: surfaces heated with flowing water exhibited as much as a 32 % greater heat flux than those heated with an electric resistance heater. Based on this finding, the NIST engineer recommends that engineers not use an electrical test apparatus to acquire boiling data. “If one uses the data from such a unit, you could improperly design your commercial equipment,” he warns. “Boiling data taken on an electric-resistance heated surface should be cautiously—if at all—used to design chillers.” For more information contact Mark Kedzierski at B128 Technology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5282.

**NIST-LED ROUND ROBINS ESTABLISH  
INDUSTRY MEASUREMENT  
STATE-OF-THE-ART FOR GEOMETRICAL  
OPTICAL FIBER AND CONNECTOR  
PARAMETERS**

The increasing use of fiber communications systems, especially fiber local-area networks, in civilian applications demands improved measurements of fiber geometry and the devices used to connect fibers. To help ensure that U.S. suppliers have equal access to globalized markets, NIST has taken a leading role in a worldwide determination of the capabilities of the optical communications industry. A NIST scientist has completed the analyses of measurements from five round robins, which focused on test methods and calibration procedures for optical fiber and fiber ferrule connection geometries. Participants in the round robins, which attracted industry interest here and abroad, were drawn from the Telecommunications Industry Association and the International Telecommunications Union. The results are published in NIST Technical Note 1378.

The international fiber geometry round robin dealt with measurements of cladding (a fiber’s outer layer of glass) diameter, noncircularity, and concentricity error (with respect to the center of the core of the fiber). Results of previous round robins had demonstrated the need for accurate calibration of cladding diameter measurements to enable the industry to meet desired tolerance specifications. In this round robin, many participants’ test sets were calibrated by means of NIST Standard Reference Material (SRM) 2520 or similar calibration artifacts from other national standards laboratories in Europe and Asia. Agreement among those participants was significantly greater than among participants whose test sets were not calibrated to any national standards laboratory. U.S. fiber manufacturers now specify a tolerance of  $\pm 1 \mu\text{m}$  on nominally

125  $\mu\text{m}$  fibers, compared to  $\pm 2 \mu\text{m}$  before the round robin and SRM development.

Two round robins dealt with similar measurements of fiber acrylate coatings and of ceramic ferrules which position fibers within connectors. Another round robin dealt with diameter measurements of fiber-sized steel wires, called pin gages, which are used to determine the inside (bore) diameter of ferrules. In these three comparisons, large systematic offsets were observed among participants’ data for some measurement parameters, particularly for coating diameter, ferrule outside diameter, and pin gage diameter. The observed measurement spread for ferrule outside diameter indicated that most participants do not currently have the accuracy to verify measurements within the desired tolerance specification. For such parameters, accurate calibration will lead to improved interlaboratory agreement.

The remaining round robin involved measurements of ferrule inside diameter, which must be only slightly larger than fiber diameter, to obtain the snug fit that is needed for accurate fiber alignment in connections. The standard measurement method is a go/no-go procedure, performed by inserting pin gages into the ferrules. The round robin results showed this method to be effective in establishing upper and lower bounds for ferrule inside diameter; however, the bounded range, and hence, the precision of the measurements is limited by the incremental size differences between pin gages (typically 1  $\mu\text{m}$ ). A few participants used video methods, which offered improved resolution over the go/no-go method but did not lead to improved accuracy. Large uncertainties, with large random components, were observed for the video data.

**NEW SOFTWARE ADVANCES  
MEASUREMENTS FOR WIRELESS  
APPLICATIONS**

NIST’s High-Speed Microelectronics Metrology Project has released the measurement software package MultiCal, a major revision of a program popular with the microwave industry. MultiCal supports advanced NIST-developed data-processing algorithms for the measurement of microwave network parameters, particularly those on high-speed integrated circuits. Further, it allows industry laboratories to implement these methods on commonly available equipment. MultiCal was developed with the support of the NIST Industrial MMIC Consortium, which sponsors much of NIST’s work in monolithic microwave integrated circuits. Consortium members had exclusive use of the software in its first year of availability.

The name MultiCal reflects not only the use of the NIST-developed ‘‘multiline TRL’’ calibration, the most accurate yet developed for integrated circuits, but also the fact that the software supports a multitude of other calibration procedures and a variety of instruments. Important features new to this release include its compatibility with popular low-cost network analyzers and its ability to run on personal computers. New methods allowing compact standard sets and compensation for lossy dielectrics are also included.

One noteworthy application of the software occurred at the 46th Automatic RF Techniques Group Conference (Scottsdale, AZ, Nov. 30, 1995). In accordance with the conference’s theme of ‘‘Testing for Wireless Applications,’’ a paper from a private company focused on the limitations of a calibration technique that had recently been offered commercially for use with low-cost radio-frequency network analyzers popular in the wireless industry. The presentation immediately following, by NIST scientists, presented a practical and accurate solution to the problem: the implementation of the multiline TRL calibration using MultiCal.

#### **NIST CALIBRATES PROBES FOR INDUSTRY AND FOR NEAR-FIELD MICROWAVE ANTENNA TESTING**

NIST recently completed two calibrations, which required 4 months of measurement time, for a private company. The company is working with LORAL Space Systems to provide them with a near-field scanner and probes to be used for measuring communication antennas. The calibrations were for two COMSAT-built, dual-port linearly polarized probes in two different frequency bands in the 10 GHz to 15 GHz frequency range. The probes will serve as standards for Loral’s planar near-field measurement facility.

A 3 month probe calibration also was completed for the Naval Surface Warfare Center at Port Hueneme. The probe’s gain, axial ratio, tilt angle, and pattern were determined along with their respective uncertainties. The probe will be used as a transfer standard for their near-field facility. The Navy is in the process of acquiring a near-field scanner to allow the on-site testing of ship-borne radar antennas. These antennas, which were obtained from industry, range from 1.5 m prime focus dishes to 4.6 m sophisticated phased arrays. Both the Navy and industry want to accurately evaluate antenna performance on-site after installation and after deployment in the field. Diagnostics and repairs would be made on site, where possible.

#### **NIST HOSTS RADAR CROSS SECTION WORKING GROUP**

NIST recently hosted a meeting of a consortium of Radar Cross Section (RCS) range facilities, which are dedicated to ensure and to deliver high-quality RCS measurements to their customers. Twenty-five representatives concerned with reliably establishing RCS measurement uncertainties for both industrial and government ranges participated. NIST staff presented a summary of a recently published comprehensive report, A Review of Government Radar Cross Section Ranges, that details the research activities and accomplishments of the NIST RCS Working Group. The report provides: (1) identification of significant sources of uncertainty in RCS measurements, (2) procedures for estimating component uncertainties, and (3) methods for combining these into an overall uncertainty budget. The consortium now has a reasonable and uniform formalism for evaluating radar cross section measurements that can be used on both indoor and outdoor test ranges to produce compatible estimates of uncertainty.

Because uncertainty estimation also highlights sources of error that must be controlled to improve measurement quality, the report also includes a study of polarization errors, details of NIST’s participation on the IEEE RCS Standards subcommittee, and recommendations for future work. Further details can be found in the publications NISTIR 5019, 5022, 5033, and papers presented at meetings of the Antenna Measurement Techniques Association (AMTA). The ongoing research includes site specific uncertainty analyses, an in-depth study of polarization effects in RCS measurements, development of RCS standard artifacts, and analysis of near-field RCS measurements. In the long term, the overall research effort will lead to the development and implementation of an industry-wide certification process.

#### **NIST IDENTIFIES A MAJOR SOURCE OF BIAS IN THE INDUSTRIAL MEASUREMENT OF MOISTURE IN TRANSFORMER OILS**

Research conducted at NIST over the past 2 years has identified a major source of bias in the method most frequently used for determining trace levels of moisture in transformer oils. Electrical transformers use refined crude oils as insulation fluids. Moisture accumulates in the fluid over time and becomes critical at a mass fraction approaching 50  $\mu\text{g/g}$ .

Karl Fischer titration is the method of choice for determining low levels of moisture. The method is based

on a reaction of an organic iodide complex with water to release free iodide. The amount of water is determined by measuring either the amount of iodide that must be oxidized or the amount of iodine that must be added to reach a preset electrochemical potential. There are two common methods for this analysis. In the coulometric method, the iodide produced by the reaction with water is electrochemically oxidized in order to reach a preset null point. This approach provides a direct measure of moisture content and does not require external calibration if the null point is properly set. Theoretically, the number of moles of iodide oxidized is equal to the number of moles of water in the sample. Alternatively, the amount of water released can be determined by volumetric addition of iodine. The moisture content is determined through use of a calibration curve.

Most commercial testing laboratories use the coulometric Karl Fischer approach. This approach is simple, requires less calibration than the volumetric approach, and has the added appeal of being linked to a fundamental quantity, the faraday. NIST has shown, through both laboratory research and interlaboratory studies, that results from coulometric and volumetric Karl Fischer measurements of trace levels of moisture in petroleum-based oils (<100  $\mu\text{g/g}$ ) differ by as much as 50 %. The major source of this bias appears to be associated with the solvents or solvent blends used with the coulometric Karl Fischer instruments. Chosen for their electrochemical properties, these solvents do not completely dissolve the oil matrix to allow total release of the water.

Based on these studies, published recently in Analytical Chemistry, ASTM committee D-27 has asked manufacturers to reformulate their Karl Fischer solvent mixtures to allow complete dissolution of petroleum-based oils for subsequent titration of trace amounts of moisture. The ASTM method for moisture in oils also will be amended to incorporate the use of NIST moisture-in-oil standards for quality assurance.

#### **TWO NEW NIST PRECISION MEASUREMENT GRANTS AWARDED FOR FY 96**

Two new \$50,000 NIST Precision Measurement Grants have been awarded for fiscal year 1996. The recipients, Prof. Siu Au Lee of Colorado State University, and Prof. Jonathan Sapirstein of the University of Notre Dame, were selected from an initial group of 35 candidates. NIST sponsors these grants to promote fundamental research in measurement science in U.S. colleges and universities and to foster contacts between NIST scientists and researchers in the academic community actively engaged in such work.

The aim of Lee's project, "Measurement of the Magnetically-Induced QED Birefringence of the Vacuum," is to observe for the first time the birefringence of the vacuum as predicted by quantum electrodynamics (QED). The experiment combines advanced optical metrology with a long, high-field superconducting magnet. The same apparatus can be used to perform a laboratory search for axion-like particles, which are among the leading candidates for dark matter in the universe. The experiment will be the first step in developing a new class of precision QED tests, with future accuracy projected to complement or surpass the electron and muon magnetic moment anomaly tests. The axion search will set laboratory limits for the coupling strength of axions to photons that, until now, have been derived from astrophysical considerations.

The aim of Sapirstein's project, "Calculations of Higher Order QED Effects in Helium," is to improve significantly the theory of the energy levels of helium by extending the existing Bethe-Salpeter equation calculations, which only include corrections of order  $\alpha^2$  relative to the fine structure, to all corrections of order  $\alpha^3$  (alpha is the fine-structure constant). Because the existing calculation is extremely awkward, Sapirstein will reformulate it, incorporating recent progress made in other Bethe-Salpeter calculations. When completed, a highly accurate value of  $\alpha$  obtained by comparing experimental measurements of helium energy levels with theory will become available. This value can then be compared with values obtained from the quantum Hall effect (QHE) and the electron magnetic moment anomaly, thereby providing a critical test of the theory of the QHE and QED.

#### **NEW CALIBRATION OF $^{63}\text{Ni}$ PERFORMED, CONFIRMS PREVIOUS NIST CALIBRATIONS OVER PAST 27 YEARS**

Recent re-evaluations of the nuclear data of the low-energy  $\beta^-$ -emitter  $^{63}\text{Ni}$  have prompted a new calibration of  $^{63}\text{Ni}$ . The resulting standards will be disseminated by NIST as Standard Reference Material SRM 4226C. Nickel-63 is of interest to the nuclear reactor community because it is often found in reactor environments as the product of neutron-capture in nickel contained in the steel used in construction. Because of its low  $\beta^-$  end-point energy and relatively long half-life, it is also becoming increasingly important in radionuclidic metrology. These characteristics make it more sensitive to many of the experimental variables encountered in  $4\pi\beta$  liquid spectrometry (LS) including sample volume, aqueous content of the LS sample, and chemical quenching. All of these variables were exhaustively

studied by NIST scientists during this calibration and the information obtained will be of help in designing experiments for an upcoming international intercomparison of  $^{63}\text{Ni}$ , in which the radioactivity group will participate.

Because these standards were prepared from a stock solution that was originally prepared by NBS in 1968 and again calibrated in 1984, it was possible to study the long-term stability of the solution as well as compare the present calibration to those performed previously. Using the latest available nuclear data, the previous calibrations have been re-evaluated and were found to be in excellent agreement with the present calibration. Furthermore, the data suggest that the standard solution is chemically stable over at least 27 years and allowed the first determination of the half-life by actually following the radioactive decay of  $^{63}\text{Ni}$ .

The results of the experiments carried out during these calibration studies will be described fully in a series of articles currently being prepared for publication.

#### POTENTIAL BONE PAIN PALLIATION RADIOPHARMACEUTICAL $^{117\text{m}}\text{Sn}$ CALIBRATED

The American Cancer Society estimates that about 180 000 new cases of breast cancer and an equal number of cases of prostate cancer are diagnosed each year in the United States. Of these patients, a significant number will most likely develop painful, often crippling, bone metastases. New radiopharmaceuticals are being developed, which have been shown to relieve pain that had been unresponsive to narcotic treatment. As part of an increasingly active program to provide standards and calibration services for radionuclides of medical interest, NIST scientists have performed a calibration of  $^{117\text{m}}\text{Sn}^{+4}$  [diethylenetriamine pentaacetic acid (DTPA)]. Clinical trials using  $^{117\text{m}}\text{Sn}^{+4}$  (DTPA) are currently under way and show great promise. Unlike other palliation radiopharmaceuticals such as  $^{89}\text{Sr}$ ,  $^{186}\text{Re}$ ,  $^{32}\text{P}$ ,  $^{153}\text{Sm}$ , and  $^{131}\text{I}$ , a much higher proportion of the  $^{117\text{m}}\text{Sn}^{+4}$  (DTPA) is taken up directly to the bone surface than in the marrow. This results in a much greater dose to the bone itself relative to the marrow, greatly reducing the toxicity. Furthermore, the radiation from  $^{117\text{m}}\text{Sn}$  decay allows the distribution of the radiopharmaceutical to be readily imaged using a normal gamma camera. The present calibration was carried out with three independent methods and excellent agreement was found among them. Data from the  $\gamma$ -ray measurements allowed the half-life to be determined as  $14.0\text{ d} \pm 0.2\text{ d}$ , confirming the values obtained at the University of Michigan and the University of Illinois in the 1950's, but conflicting

with the Japanese value of  $13.60\text{ d} \pm 0.04\text{ d}$  reported in 1977. This latter value is used commonly in dose calculations and should thus be reconsidered. All of the data from the present calibration will allow accurate measurements of the radiopharmaceutical at several stages, from production to distribution to clinical use.

#### IUPAC SETS WAVENUMBER STANDARDS FOR THE INFRARED

The International Union of Pure and Applied Chemistry (IUPAC) working group on "Unified Wavenumber Standards" recently has issued a set of recommendations for high resolution wavenumber standards for the infrared region of the spectrum. The document provides wavenumber standards for calibration of high-resolution infrared spectrometers where measurements have traditionally been more reproducible than accurate. The most accurate standards are derived from direct frequency measurements made in infrared absorption measurements. These are converted to wavenumber (and wavelength) using the 1983 redefinition of the meter. The spectral lines cited by the group cover the range from about  $4\text{ cm}^{-1}$  to about  $7000\text{ cm}^{-1}$ . The standards will allow much better comparison of measurements made at different laboratories.

A very large share of the measurements cited in the IUPAC recommendations were made at NIST. Many of the recommended values are taken from NIST Special Publication 821, Wavenumber Calibration Tables from Heterodyne Frequency Measurements. These data are available on the Physics Laboratory World Wide Web server.

#### NIST AND NSF COLLABORATE ON LCD RESEARCH

NIST has joined forces with the NSF Center for Advanced Liquid Crystalline Optical Materials (ALCOM) to pursue theory and modeling of materials used in optical displays. LCDs, which are key components of wristwatches, calculators, automobile dashboards, airplane cockpits, and laptop computer screens are largely imported. Currently, the United States has less than 5 % of the world LCD market, even though the technology is an American invention.

ALCOM—one of 25 NSF science and technology centers—is located at Kent State University, and supports liquid crystal research at Case Western Reserve University, Kent State, and the University of Akron. Members of ALCOM's industrial partnership program advise the center on technological issues of importance in the design and fabrication of LC materials, and ALCOM researchers conduct basic R&D to build the knowledge base in this area.

The partnership between ALCOM and NIST brings together researchers at NIST, UCLA, the University of Chicago, the University of Toronto, NYU, and Kent State University in developing a standard suite of theoretical models and computer programs for modeling LC mixtures used in the optical display industry. The models under development couple recently derived free energy expressions with standard simulation methods that have been used to model thermo- dynamic instabilities in alloys and complex fluids, and will be used to predict and analyze mesoscopic morphology arising in polymer-dispersed LC materials during processing. Research resulting from this partnership is anticipated to strengthen the U.S. role in the multibillion dollar international market for liquid crystal displays and related devices. Further details on the group's activities, as well as simulation code and an on-line tutorial, can be found on the NIST Materials Theory Information Web Server at <http://www.ctcms.nist.gov>.

#### **DEVELOPMENT OF A NEW TEMPERATURE MEASUREMENT SENSOR FOR PLASTICS PROCESSING**

NIST scientists have developed a new temperature sensor to measure true plastic resin temperature during processing at high temperatures. Plastic products are manufactured at high temperatures because they are molten and can flow easily into shape forming molds and dies. If the temperature is too high, thermal degradation of the polymer resin can occur, thereby compromising the performance of the plastic product. The process machine temperature is normally lower than resin temperature because of a phenomenon called shear heating, i.e., the flowing plastic dissipates heat due to its viscosity. To measure true resin temperature, NIST scientists have developed an optical sensor based on measuring the fluorescence light from a fluorescent dye mixed with the resin at low concentrations. The sensor employs an optical fiber cable, which is inserted into the process machine where it can view the molten resin. Characteristic changes in the spectrum of the detected fluorescent light are used to determine the temperature. An important milestone in this sensor development was the discovery by NIST scientists of a fluorescent dye that can be used at the high temperatures of resin processing (up to 300 °C). In laboratory tests, the sensor has been used to monitor polymer injection molding for determining the time at which resin solidification occurs. It is now being employed by a private company to monitor temperatures of plastic film processing and to measure shear heating effects. Another private company is collaborating with NIST to develop an extension of the NIST sensor, which will be

used to measure the profile of temperatures across a flowing resin stream. In addition, several instrumentation companies are evaluating the sensor to determine its marketability.

#### **PHASE-FIELD MODELING OF ALLOY SOLIDIFICATION**

NIST researchers have worked together to apply a simulation technique, known as the phase-field method, to solidification. This method has been shown to be a powerful approach to the solution of complicated moving-boundary-value problems. Recent research at NIST has focused on the phase field approach for dendritic solidification of binary alloys, which better represents processes of commercial alloys. Here the final distribution of solute in the solid is highly irregular, which can strongly influence the structure and the materials properties of the solidified alloy.

#### **QUASI-LAUE NEUTRON DIFFRACTION FOR BIOLOGICAL STRUCTURE**

Scientists at NIST and at the University of Maryland have established a collaboration with the European Molecular Biology Laboratory Grenoble Outstation at the Institut Laue-Langevin (ILL) in France to develop techniques for quasi-Laue neutron diffraction. The design of a diffractometer similar to one that is being developed at the ILL, which uses neutron sensitive image plates, is being considered for the Cold Neutron Research Facility at NIST. The quasi-Laue technique, combined with the new liquid hydrogen cold source at the CNRF and the characteristics of guide tubes and new neutron monochromators, promises to increase the rate of data acquisition from crystals of biological macromolecules by a factor of 100. Now experiments that were not possible previously because of the need for large crystals and excessively long data-collection times become feasible. Combined with advances in crystallographic data analysis, this would greatly enhance the importance and application of neutron diffraction in the study of larger protein structures with much smaller ( $\sim 1 \text{ mm}^3$ ) samples.

#### **SIMULATION OF STAIRWELL FIRES**

New York City is experiencing a series of arson fires set in stairwells in high-rise public housing. The fires can ignite the paint on the stairwell walls causing rapid upward fire spread. In partnership with the Fire Administration, NIST has started a study of such fires. The first objective has been to duplicate the spread phenomenon under laboratory conditions. To this end, several models

of one flight of stairs have been built. On Jan. 17, 1996, the fire spread phenomenon was duplicated in a large-scale fire test at NIST. Members of the New York City Fire Department (NYCFD), the New York Housing Authority, and Fox Television News from New York were present to observe the test. In this experiment, the walls and ceiling were covered by 15 coats of oil-based paint, as in New York City. A coat of shellac was applied before the last coat of paint, on one of the two walls, to simulate one technique used in New York to control the graffiti problem; the ignitor was a 200 kW propane fire. Ultimately, the researchers wish to develop a test protocol for paints and coatings that will permit authorities to evaluate materials capable of preventing the spread of stairwell fires.

### **BFRL SUPPORTS MAJOR DEMONSTRATION OF BACnet**

Between 3000 and 4000 people visited the BACnet™ Consortium booth at the International Air Conditioning, Heating, Refrigerating Exposition, Feb. 19-21, 1996, at the Georgia World Congress Center in Atlanta, GA. The consortium, which consists of 17 members and is led by NIST, demonstrated how energy management and control systems from different manufacturers can communicate and “interoperate” using the recently released ANSI/ASHRAE Standard 135-1995. This standard, whose title is “BACnet™, A Data Communication Protocol for Building Automation and Control Systems,” was developed over a period of 9 years and provides the framework for creating and specifying multi-vendor building control systems that interoperate at various levels of functionality.

The demonstration showed how a chiller plant, laboratory, VAV, lighting, and other HVAC system controllers could share data in a peer-to-peer manner. A visitor to the BACnet™ booth could view the status of any participating vendor’s system and make changes to control setpoints from any of four PC-based workstations. Four different local area networks, ISO 8802-3 (Ethernet™), ANSI/ATA 878.1 (ARCNET™), LonTalk™, and MS/TP using EIA-485, were connected through several BACnet™ Routers to provide bi-directional communication between the various BACnet™ speaking devices.

The NIST-led consortium was developed to facilitate the development, prototyping, and interoperability testing of BACnet™ implementations by different control manufacturers and interested third parties. The consortium provides a neutral and noncommercial environment where those interested in implementing the BACnet™ protocol can work together with NIST to perform

testing and debugging in a cooperative manner. As part of the preparation for the exposition demonstration, each participating consortium member went through several stages of testing in the NIST laboratories. Each device was tested individually to verify compliance with the BACnet™ protocol. Devices using the same local area network (LAN) technology were then tested as a group for interoperability. Finally, an internetwork was constructed in the NIST laboratories to match the demonstration environment for testing all of the equipment together.

### **NIST HOSTS DELEGATION FROM THE JAPANESE SCIENCE AND TECHNOLOGY AGENCY**

A delegation from the Japanese Science and Technology Agency (STA) visited NIST as part of a nationwide tour of facilities with strong and visible programs in computational science. STA is in the early phases of building a major program in computational science; this year the program (funded at approximately \$60 million per year) is the highest priority in the agency. A key component of the proposed effort is to create close collaborations with internationally recognized programs in modeling and computational science. The delegation informed NIST that they have extensively used the NIST Guide to Available Mathematical Software (GAMS) system and that they are most interested in new software components that are being developed and released by NIST. Of equal importance are NIST’s contributions in the modeling and simulation of phenomena in materials science. Two members of the delegation gave overviews of their research programs in materials modeling and several members of the delegation toured the NIST computer facilities.

### **NIST SPONSORS SYMPOSIUM ON USABILITY ENGINEERING**

On Feb. 26, 1996, NIST sponsored a Symposium on Usability Engineering: Industry-Government Collaboration for System Effectiveness and Efficiency. The symposium brought together about 100 industry and government representatives to exchange information and strategies for achieving effectiveness, efficiency, and satisfaction in computer-based government systems. Attendees included project development managers, government contractors, procurement officials, analysts and engineers, technical staff and researchers, commercial off-the-shelf (COTS) product vendors, consultants, and policy makers.

In designing new government computer systems and in redesigning legacy systems, industry and government must be aware of the best practices now available to ensure the usability of such systems. Topics covered at the symposium included an introduction to usability engineering, usability trends in government, success stories, costs and benefits, standards and guidelines, industry strategies and practices, special issues for complex systems, and making usability work in the organizations. A post-symposium publication summarizing the event is planned.

#### **NEW REPORT FOCUSES ON VIRTUAL ENVIRONMENTS AND RELATED TECHNOLOGIES FOR HEALTH CARE**

NISTIR 5740, Virtual Environments for Health Care, surveys the state of the art in applications of virtual environments and related health-care technologies. Applications of these technologies are being developed in the following areas: surgical procedures, medical therapy, preventive medicine and patient education, medical education and training, visualization of massive medical databases, skill enhancement and rehabilitation, and architectural design for health-care facilities.

To date, such applications have improved the quality of health care; in the future, substantial cost savings will result. Tools that respond to the needs of present virtual environment systems are being refined or developed. The report concluded that additional large-scale research is necessary in user studies, use of robots for telepresence procedures, enhanced system reality, and improved system functionality.

#### **NEW PUBLICATION SURVEYS OPEN MANAGEMENT APPROACHES FOR DISTRIBUTED SYSTEMS**

NISTIR 5735, Distributed Systems: Survey of Open Management Approaches, presents several alternatives for providing a framework for managing a network of heterogeneous systems. Management services, providing mechanisms to monitor and control a great diversity of components and user interactions with these components, and an integrated approach to assure consistency are now being addressed by standards- development organizations and user consortia. The document overviews some existing approaches proposed by different organizations.

#### **PERFORMANCE MEASUREMENT RESEARCH ADVANCES**

NISTIR 5743, Operating Principles of MultiKron Virtual Counter Performance Instrumentation for MIMD Computers, provides a brief background on performance measurement and describes the features of

the MultiKron virtual counter (MultiKron\_vc) performance instrumentation chip and its processor interface. The report presents an alternative, more cost-effective measurement approach that abolishes the need for a data collection facility by eliminating trace sampling and provides for a large number of resource counters, with some loss of measurement detail.

The MultiKron\_vc chip provides a feature that accomplishes the new technique. Similar in concept to virtual memory, thousands of virtual counters are available but only a small number are real counters that can be active at any one time. Unlike virtual memory, the swapping of counter blocks must currently be handled by the programmer.

#### **STANDARD GENERALIZED MARKUP LANGUAGE (SGML) TEST SUITES EVALUATED**

NISTIR 5762, Standard Generalized Markup Language Test Suite Evaluation Report, describes the methodology used in analyzing an SGML Test Suite and the different types of tests run with the various SGML products available to NIST. SGML defines a set of human-readable codes that are added to electronically prepared documents; it defines the structure of the text and the format in which it is to appear.

As part of the Computer Acquisition and Life-Cycle Support initiative, NIST was tasked to organize an SGML Conformance Testing Service. The first step was to produce a comprehensive test suite by evaluating existing ones. Found to be the most thorough and complete SGML Test Suite available, NIST selected a private company's SGML Test Suite as the basis for its SGML Conformance Testing Service.

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## **Standard Reference Materials**

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#### **MAJOR REVISION OF COATING THICKNESS STANDARD REFERENCE MATERIALS 1357 THROUGH 1364a**

The first revision of the Coating Thickness Standards SRMs 1357 through 1364a is nearing completion and is expected to reach customers before October 1996 and the remaining stock of previous standards is consumed. These standards, developed in the 1960s, consist of pre-configured sets of coupons of fine-grained copper of thicknesses ranging from 2.5  $\mu\text{m}$  to 2 mm, which has been electrodeposited onto low carbon steel substrates. The uniform coatings are then overlaid with a thin protective layer of chromium and the coupon's total coating thickness is certified. They are intended primarily

for use in calibrating coating thickness measurement instruments based on the magnetic induction principle and are used by the organic and inorganic coating industry for the nondestructive measurement of non-magnetic-coatings over magnetic substrates.

Advances in the accuracy of the instruments employed in the laboratory and in the field, coupled with the present policy for reporting uncertainties associated with measurements executed at NIST, required the reevaluation of the production and certification of such standards. The effects of thickness, composition, surface roughness, and mechanical working of the substrate material on the uncertainties associated with the thickness measurement have been determined during the material selection for the production of these standards. Stricter control over the cell geometry and electrode configuration during the electrodeposition stages now yields uniform plates with less than 1 % deviation from the targeted thickness values. In addition, during the certification stage, reproducibility of the positioning of the probe and the distance of the measurement spot from the edges has been increased to minimize edge effects. Uncertainties associated with human operations, calibration curves, probe resolutions, and measurement modes have been modified by increasing the substrate thickness and the size of the measurement area without compromising the uncertainties listed. This allows greater leeway during the selection of the measurement spot by the user. An attempt has been made to tailor the thickness distribution offered within the preconfigured sets by surveying users with respect to the thickness ranges of their present applications and the uncertainties associated with their measurement protocol.

#### **NEW SRM FOR PLASMA-SPRAY POWDERS**

The plasma-spray deposition of yttria-stabilized zirconia coatings is a large and growing industry. These coatings for engine parts permit operation at significantly higher temperatures than would otherwise be possible. An important issue in the reproducibility of plasma-spray coatings is knowledge of the distribution of particle sizes in the starting powder.

Researchers at NIST recently have completed work on a new SRM (No. 1982), an yttria-stabilized-zirconia powder for calibration of particle size distribution (PSD) measurement instruments. This SRM was developed in cooperation with powder producers as well as plasma-spray, instrument, and engine manufacturers. This SRM, with a well-characterized particle size distribution between 10  $\mu\text{m}$  and 150  $\mu\text{m}$ , permits accurate calibration of PSD instruments, and thereby leads to better control of the deposition process.

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## **Standard Reference Data**

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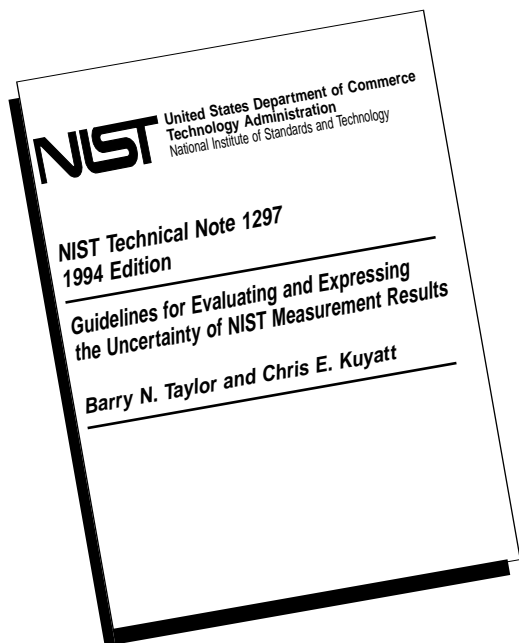
### **NEW AND IMPROVED REFPROP READY FOR MARKET**

A newly expanded version of NIST's popular REFPROP database is now available for engineers designing cooling systems with environmentally friendly refrigerants. REFPROP 5.0 is a computer database of properties for pure and mixed refrigerant fluids. The new version contains data on 43 pure refrigerants and can calculate properties for mixtures with up to five components. New to the database are improved algorithms for calculating fluid mixture properties, and five new refrigerant fluids. NIST provides industry with refrigerant properties to assist in the transition away from chlorofluorocarbon and hydrochlorofluorocarbon refrigerants, which are believed to damage the Earth's protective ozone layer. NIST first introduced REFPROP in 1989 to provide industry with thermophysical and transport properties of promising alternative refrigerants. Version 5.0 of REFPROP is available for \$465. Owners of a previous version may upgrade for \$100. The database requires MS-DOS, PC-DOS 2.1 or higher. A math co-processor is recommended, and a minimum memory of 512 K is required. To order REFPROP 5.0, NIST Standard Reference Database 23, contact the Standard Reference Data Program, Building 820, Rm. 113, NIST, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: (301) 975-0416, e-mail: [srdata@enh.nist.gov](mailto:srdata@enh.nist.gov).





# Evaluating and Expressing the Uncertainty of Measurement Results



Uncertain about expressing measurement uncertainty? Do you need to know how NIST states the uncertainty of its measurement results and how you can implement their internationally accepted method in your own laboratory? Then you need the newly available 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*.

The 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, by Barry N. Taylor and Chris E. Kuyatt is now available.

The 1994 edition of TN 1297 includes a new appendix—Appendix D—which clarifies and gives additional guidance on a number of topics related to measurement uncertainty, including the use of certain terms such as accuracy and precision. Very minor word changes have also been made in a few portions of the text of the 1993 edition in order to recognize the official publication in October 1993 by the International Organization for Standardization (ISO) of the *Guide to the Expression of Uncertainty in Measurement* on which TN 1297 is based. However, the NIST policy on measurement uncertainty, Statements of Uncertainty Associated with Measurement Results, which is reproduced as Appendix C of TN 1297, is unchanged.

It is expected that the 1994 edition of TN 1297 will be even more useful than its immediate predecessor, the 1993 edition, of which 10 000 copies were distributed worldwide.

Those United States readers who wish to delve into the subject of measurement uncertainty in greater depth may purchase a copy of the 100-page ISO *Guide* from the Sales Department of the American National Standards Institute (ANSI), 105-111 South State Street, Hackensack, NJ 07601. Copies may also be purchased from the ISO Central Secretariat, 1 rue de Varembé, Case postale 56, CH-1211 Genève 20, Switzerland.

Single copies of the 20-page TN 1297 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.

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## *Periodical*

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## *Nonperiodicals*

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