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News Briefs

General Developments

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NORTH AMERICAN METROLOGY AND CALIBRATION MEETINGS

Two NIST representatives attended the North American regional meetings on collaboration in metrology, NORAMET, and the Calibration Cooperation, NACC, at the National Research Council in Ottawa, Ontario, Nov. 13, 1996.

NORAMET ensures cooperation among the national measurement institutes in Canada, the United States, and Mexico. Ongoing work of technical groups in all measurement disciplines was reviewed, and plans were established to conduct experiments to assess measurement equivalency among the three national institutes. This work is vital for securing the measurement foundation for international trade and laboratory accreditation based on national systems of measurement services.

NACC works toward mutual recognition of the equivalency of the national calibration laboratory accreditation programs in the three countries. This also will strengthen the foundation for international trade based on the recognition and acceptance of calibration data produced in accredited calibration laboratories. The necessary documents concerning rules of operation and the ground rules upon which assessments of the calibration laboratory accreditation programs in the three countries will be conducted were discussed. The impact of mutual recognition agreements on current and future trade negotiations occurring under free trade agreements, such as NAFTA (North American Free Trade Agreement), was discussed at length.

ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS

During the month of November, NIST recommended an innovative method for producing glass fiber for commercialization to its Department of Energy partner under the Energy-Related Inventions Program.

The new method is a unique hardware design and process for producing glass fiber. The technology will be used in both the fiber glass insulation industry and the composite materials industry where glass fiber is used as a reinforcing member of the composite structures.

NEW NIST REFERENCE ANTENNA REDUCES CALIBRATION ERRORS RESULTING FROM AMBIENT ELECTROMAGNETIC FIELDS

The increasing use of wireless communication products demands ever higher levels of calibration accuracy to support them. For example, manufacturers must be able to delineate precisely the performance characteristics of antennas they employ in-house and those they design into their products. Outdoor calibration test ranges are used frequently, but it has been very difficult to obtain a pure signal from traditional broadband reference antennas used at these ranges, because the signal output contains contributions from the ambient electromagnetic fields of all frequencies present at the calibration site.

Researchers at NIST have developed an improved radiofrequency standard antenna for the calibration system used with the open-area test site by devising a means to discriminate against the out-of-band interference while maintaining the integrity of the signal. The key element of the system is a new radiofrequency electro-optic modulator that converts the radiofrequency signal from the antenna to a lightwave signal that can be piped to the measuring instrumentation via a nonconducting optical fiber. Using the nonconducting fiber

provides a twofold advantage: it preserves the integrity of the signal, and it does not interfere with the electromagnetic field the antenna is measuring. The new modulator is expected to be applicable to calibration systems at other sites than NIST's.

NIST IMPROVES ACCURACY OF NONDESTRUCTIVE METHODS FOR MEASURING DIELECTRIC AND MAGNETIC PROPERTIES OF MATERIALS AT RF/MICROWAVE FREQUENCIES

Applications of newly emerging microwave technologies require that the microwave energy be very accurately focused, directed, and processed using specialized circuitry and phased-array antennas that are generally mounted on a thin layer of polymer or ceramic dielectric, termed a substrate. Usually, efficient and cost-effective design of such high-performance systems requires that the dielectric properties of the substrate materials be known with a relative uncertainty of less than ± 3 %. However, most of the characterization techniques involve preparing a machined sample of the material under test; i.e., they are destructive and unsuitable for use in production-line settings. A very widely used commercially available nondestructive and broadband measurement method involves placing an open-ended coaxial probe in contact with the material under test and deriving its dielectric parameters by measuring the probe admittance by means of an automatic network analyzer. However, measurements performed by NIST researchers and by others on thin substrates have shown consistently that such measurements have a relative uncertainty no less than ± 10 % and generally degrade further with increasing relative permittivity of the material under test. This limit primarily relates to inadequacies in the electromagnetic model used to describe the measurement and solutions to this model based on oversimplified assumptions. The most significant of these is disregard of the "lift-off" factor, defined as the unavoidable presence of an air gap between probe face and material surface. Even where the probe appears to be in good contact with a polished material surface, NIST has shown that an unavoidable air gap of 50 μm to 100 μm has a very significant influence on probe admittance. For some specialized applications, such as the use of a probe for process control in moving assembly lines, lift-off must be included for the measurement to be useful at all.

In efforts to improve significantly the measurement accuracy of the open-ended coaxial probe, NIST researchers have developed full-field solutions (the most accurate) for a much improved model that includes the air gap lift-off; finite layer thickness, with provision for both dielectric and magnetic properties; and the presence of an optional conductive layer (for measuring copper-clad substrates). By deliberately including a small and known probe lift-off in the measurement process through the use of micrometer-equipped probes, measurement uncertainties as small as ± 3 % or less have been demonstrated routinely. The new NIST coaxial probe software also now supports additional measurement options and is compatible with operation on a personal computer.

NIST PROVIDES IMPROVED TECHNIQUE TO CHARACTERIZE LOW-FREQUENCY ANECHOIC CHAMBERS USED TO DEMONSTRATE COMPLIANCE WITH INTERNATIONAL EMI/EMC STANDARDS

By late 1995, rapid mobilization on the part of countries in the European Union with respect to standards for electromagnetic interference and electromagnetic compatibility (EMI/EMC) had created a "preparedness" gap for American manufacturers. NIST has been working actively with industry to diminish this gap in several ways.

As a first step, NIST staff joined with industry leaders to organize and host NIST's 1996 EMI/EMC workshop, which was designed to identify and set priorities for dealing with the gap. Feedback from the workshop has had a major influence on the direction of NIST's EMI/EMC program and has identified specific issues to be addressed. An issue assigned high priority at the workshop was the need to demonstrate that electronic products developed by U.S. firms are in compliance with both domestic and international regulations and requirements for interference and susceptibility. Semi-anechoic and fully anechoic chambers are important and cost-effective tools for demonstrating compliance, but characterization and performance verification of these chambers has been difficult and time consuming. Even so, the number of domestically designed and manufactured low-frequency (20 MHz to 1 GHz) anechoic and semi-anechoic electromagnetic test chambers in use has been increasing steadily.

To meet industry's needs, NIST researchers have developed procedures centering on a new chamber figure of merit based on the decay time of the chamber. This decay time is in turn based on the average reflection coefficient of the energy incident on the chamber walls. Implementation of the new technique avoids labor-intensive measurements and eliminates complex, intensive, time consuming and, therefore, costly numerical analysis by making use of simplifying approximations that can be shown not to affect the utility of the result. It also enables assessment of chamber performance by

means of a single parameter, a significant improvement over existing methods, which have a strong dependence on setup and instrumentation. Details of the work have been published in the IEEE Transactions on Electromagnetic Compatibility, Vol. 38, No. 4, 1996. Still under development is a procedure for relating chamber figure of merit to chamber capability for supporting specific tests.

FINISH MACHINING OF HARDENED STEELS VS GRINDING

Finish machining of hardened steels has been receiving increased attention as an alternative to grinding. Potentially, single-point cutting offers benefits over grinding such as lower equipment costs, shorter setup time, reduced process steps, better surface integrity, and elimination of metal-working fluid. However, tool wear remains a major cost-related barrier. Two NIST researchers have performed studies of finishing performance in turning of hardened powder metallurgy (PM) AISI M50 steel using cubic boron nitride tools. Their data indicate that tool wear and flank wear rate are unusually small and relatively insensitive to cutting conditions. They attribute these findings to the refined transition metal carbides in workpieces which tend to reduce chemical reactivity during cutting and minimize abrasive wear of cutting tools. Surface finish obtained at a fine feed and small depth of cut is in the range of 60 nm to 120 nm Ra. These results suggest that PM steels might be used for machinability enhancement in high-speed finishing hardened die and mold processes.

MEASUREMENTS AND STANDARDS ACTIVITIES IN SUPPORT OF NEW NITRIC OXIDE INHALATION THERAPY FOR NEWBORNS

Pediatric researchers are working under National Institute of Child Health and Development (NICHD) and Food and Drug Administration (FDA) guidelines to evaluate the therapeutic value of inhaled nitric oxide (NO) in newborn patients. When inhaled with oxygen via a ventilator, dilute concentrations (<100 μmol/mol) of NO have shown dramatic results as a pulmonary vasodilator in some newborn patients. However, once NO is mixed with oxygen, spontaneous formation of Nitrogen dioxide (NO_2) occurs. This can cause pulmonary edema and other problems in the respiratory system, especially at NO_2 concentrations above 5 μmol/mol.

For the past 2 years, researchers at NIST have been working with the NICHD to assist pediatric researchers who are evaluating inhaled NO therapy to understand the kinetics of NO_2 formation under conditions expected to be used. The rate of NO_2 formation under these conditions was determined to be 0.137 μmol/mol for each second of dwell time. It was concluded that at anticipated dwell times of < 0.5 s, no significant levels of NO_2 should reach the patient during treatment. Therefore, clinical trials on inhaled nitric oxide (INO) therapy for treatment of persistent pulmonary hypertension in newborn children were initiated and have been completed recently. Based on preliminary results from these trials, it is expected that the FDA will soon approve the use of nitric oxide in inhalation therapy. Because of the potential for adverse effects, levels of NO and NO_2 must be monitored continuously in a typical matrix of > 0.8 mole fraction of oxygen during the therapy. Measurements made with electrochemical sensors and chemiluminescence detectors commonly used for monitoring NO_2 were compared with measurements made using NIST tunable diode laser absorption spectrometry, which can accurately measure NO_2 levels independent of oxygen content at concentrations well below those that trigger adverse effects in newborns. NO_2 electrochemical sensors did not work well in the oxygen-rich matrix, indicating NO_2 concentrations orders of magnitude higher than actually present. Chemiluminescent-based instruments performed better but in some cases registered negative values for NO_2 causing user lack of confidence.

A prototype facility has been established at NIST to allow INO sensor and instrument manufacturers to evaluate the performance of their devices for measuring NO and NO_2 under simulated therapeutic conditions. During a recent workshop held at NIST for pediatric researchers, in the presence of a senior scientist from FDA's Anesthesiology and Respiratory Devices Therapy Panel, technical experts used this facility to test new chemiluminescence monitors and solid-state sensor devices designed to perform in high oxygen environments. The performance of these devices was far superior to those evaluated at NIST a year ago, and NIST is planning additional workshops of this nature to assist the FDA in tackling the tough job of evaluating NO and NO_2 monitoring instrumentation for use in this therapy.

NONCONTACT ANALYSIS OF OXYGENATES IN GASOLINE USING FT-NEAR INFRARED AND FT-RAMAN SPECTROSCOPIES

Oxygenated fuels, blends of gasolines with alcohol or other oxygen-containing materials, are designed to reduce carbon monoxide emissions. To support the enforcement of environmental air regulations, NIST provides eight oxygenated gasoline Standard Reference

Materials (SRMs). These SRMs, provided in 20 mL sealed glass ampoules, contain gasoline and one of four oxygenates [ethanol (ETOH), methyl tert-butyl ether (MTBE), tert-amyl methyl ether (TAME), or ethyl tert-butyl ether (ETBE)] at either the 2.0 % or 2.7 % oxygen mass fraction levels.

The current method used to certify these materials is capillary gas chromatography (GC) with flame ionization detection. Although this separation-based technique provides excellent precision and accuracy, it requires opening the ampoules. As a result, these SRMs must be batch-certified with the expanded uncertainty in their assigned values incorporating an estimate of batch heterogeneity. While the current expanded uncertainty for all these SRMs is an acceptable 0.04 % to 0.06 % mass fraction of oxygen, most of the batch heterogeneity component is attributable to a few "outlier" ampoules. These statistical outliers are thought to result from errors in the flame-sealing procedure and/or sample handling. The expanded uncertainties for all such SRMs could be lowered if each individual SRM ampoule could be characterized and obvious "outliers" discarded from the batch. This requires a nondestructive, rapid, and precise analytical method.

NIST researchers have found that sealed SRM ampoules can be analyzed directly for oxygenate content using Fourier transform-near infrared (FT-NIR) and Fourier transform-Raman spectroscopic methods. Both the Raman and FT-NIR methods are capable of quantifying the oxygenate concentration to within the expanded 95 % confidence interval levels afforded by the GC measurements. Furthermore, such spectroscopic determinations of the oxygenate concentration in individual ampoules can be accomplished in less than 1 min. Of the two spectroscopic methods, NIR gives better estimates (lower standard deviations) of oxygenate concentration and the Raman method is better at identifying the specific oxygenate in unknown data sets.

NIST researchers have constructed and evaluated a prototype instrument that combines the two techniques to maximize their utility for the analysis of such complex mixtures. To do this, they added a fiber-coupled tungsten source to their commercial FT-Raman instrument. Because the FT-Raman instrument uses a 1.06 μm source, the Raman-scattered light is emitted in the 10 000 cm^{-1} to 5000 cm^{-1} (absolute) region. This corresponds to the second overtone and first combination band region used for the analysis in the FT-NIR spectrometer. Thus, the same germanium detector may be used to measure both the Raman and NIR absorbance spectra of a sample. The mass fraction errors of MTBE-in-gasoline samples obtained from the initial combination spectra acquired on the hybrid instrument were about 0.5 %. Work is in progress to extend this technique to the determination of water content in a gasohol SRM, to assist in the certification of a new synthetic fuel SRM, and to determine other functional moieties, such as olefins.

CONTROVERSIES SETTLED OVER FUNDAMENTAL PROPERTIES OF THE HYDROXYMETHYL RADICAL

The highly reactive hydroxymethyl radical (CH_2OH) and cation (CH_2OH^+) play important roles in combustion, atmospheric pollution chemistry, catalysis, and ion processes. Because of their importance, they are also among the most studied unstable species. Despite this intensive study, the thermochemical properties of these species have remained uncertain and controversial. For example, thermochemical functions of CH_2OH calculated from ion data and from kinetic data diverge irreconcilably. Accurate thermochemical functions are required for models of many chemical processes.

By using state-of-the-art spectroscopic measurements and ab initio calculations, NIST chemists have resolved these controversies and reported accurate thermochemical values. This work is reported in the December 18, 1996 issue of the Journal of Physical Chemistry. The key discovery during this research was that hydroxymethyl radicals contain higher molecular symmetry than previously was believed or is deduced intuitively. Such higher symmetry completely changes the rules by which spectroscopic and kinetic data are used during thermochemical calculations.

The higher molecular symmetry of the CH_2OH radical was discovered during experiments conducted at NIST. These experiments used resonance-enhanced multiphoton ionization (REMPI) spectroscopy to observe extremely weak, very low energy vibrational levels of CH_2OH—such levels could exist only if CH_2OH contained higher symmetry. Extensive ab initio calculations were performed on the NIST supercomputer to duplicate these data and confirm the higher molecular symmetry. Using rules dictated by the higher molecular symmetry and the new spectroscopic data, previously reported ion and kinetic data were re-evaluated to obtain thermochemical functions for CH_2OH and CH_2OH^+. Not only are these new functions accurate, but they reconcile the controversies existent between previous evaluations based on ion and kinetic data.

RESEARCH OF NIST SCIENTISTS HIGHLIGHTED ON SEPTEMBER AND OCTOBER COVERS OF *SCANNING*

The research of NIST scientists were featured on the covers of the September and October issues of *SCANNING*. On the September cover (with accompanying article) is a digital x-ray phase map derived from a procedure developed at NIST by a guest worker from the Jozef Stefan Institute, Department of Ceramics, Ljubljana, Slovenia. Phase mapping is used for the identification and interrogation of the chemical relationships of phases in complex high-technology materials. The method was first applied to BiSrCaCuO (BSCCO) high T_c superconductor specimens, which are chemically complex and can have eight or nine phases present. Determining the phase relationships and related phase diagram of this material is difficult but critical to understanding the chemical phase-property relationships. X-ray mapping of the phases in specimens produced under different conditions provides a better understanding of the phase diagram, leading to better methods for producing practical superconducting materials. The procedure digitally maps the five cations—Bi, Pb, Sr, Ca, and Cu—with wavelength spectrometers on an electron microprobe, calculates the concentration of each cation from standards data, and compares the concentration of each element or combination of elements at each pixel with a set of phase rules. The pixels that satisfy a unique phase rule are given a color to label the phase. A graphics interface was developed for adjusting the phase rules to accommodate various uncertainties in the data and observe the changes of the phase labels of the compositional maps. Previous to this development, only multiphase systems of three phases or less could be readily identified with compositional mapping.

The October cover (with accompanying article) depicts an image of machining debris remaining at the bottom of a blind hole after twist drilling in brass using the environmental scanning electron microscope (ESEM). The ESEM is a remarkable variation of a conventional SEM that permits the specimen to reside at pressures six to eight orders of magnitude higher than the conventional SEM, while retaining much of the resolution and analytical functionality. In the conventional SEM, the specimen must be kept under high vacuum, typically 100 μ-Pa. Whereas in the ESEM, the specimen chamber pressure can be in the range 100 Pa to 2500 Pa, which permits examination of high vapor pressure samples as well as in situ experiments involving chemical reactions. The high gas pressure of the ESEM prohibits the use of conventional electron detectors that involve high potentials (e.g., 10 kV) but does enable a new type of ESEM electron detector, the gaseous secondary electron detector (GSED), which is based on modest secondary electron acceleration and collision-ionization cascade amplification. Because the depth of a hole would preclude signal collection, an image such as that shown on the *SCANNING* cover would not be possible with a conventional high vacuum SEM/ detector system while with the ESEM GSED the gas ionization-amplification readily extracted a strong signal. In a series of tests described in the paper, holes as deep as 8 mm with aspect ratios (depth/diameter) of 5:1 were successfully imaged with the ESEM/GSED, including insulating as well as conducting materials. In addition to high-resolution imaging under extreme conditions, the ESEM provides new analytical capabilities to NIST scientists.

SALT DEPOSITION INVESTIGATED IN SUPERCRITICAL WATER OXIDATION REACTORS

In a collaborative effort with the Massachusetts Institute of Technology, NIST personnel are investigating the deposition of sodium sulfate (Na_2SO_4) on the walls of supercritical water oxidation (SCWO) reactors. The results of this work have given new insights to the mechanisms of the salt deposition process in an aqueous supercritical fluid.

SCWO technology shows promise for effective remediation of many toxic wastes. SCWO takes place in water at temperatures and pressures above the critical point of pure water (647 K and 22 MPa). The process brings together water, organic waste and oxidant at high pressures (25 MPa or more) and moderate temperatures (650 K and above) where the organics are completely oxidized to water, carbon dioxide, and molecular nitrogen. However, before the technology can be deployed widely, fouling of reactor surfaces due to scale buildup by salts, which have low solubility in supercritical water, must be understood and controlled. These salts are formed when neutralizing bases are injected into reactors to minimize corrosion of reactor materials. There is scant knowledge of the deposition process as well as the thermophysical properties of aqueous salts at these elevated conditions. However, for limiting cases, the scale buildup in these reactors can be modeled and experiments designed to evaluate the model.

The experiments, conducted at NIST, use a 4 mm OD heated cylinder inserted into the cross flow of a supercritical Na_2SO_4 solution of mass fraction 4 % flowing at about 0.16 g/s for varying amounts of time. The solution impinging on the heated cylinder is preheated to a temperature close to that at which precipitation begins, while the heated cylinder is maintained slightly above this precipitation temperature, thus limiting deposition almost exclusively to the surface of the

cylinder. For exposure times of 600 s or less, the scale buildup is on the order of the diameter of the cylinder. The experimental deposition rate, when interpreted with the model, indicates that the salt is transported to the cylinder in molecular form and nucleates there with little, if any, salt nucleating homogeneously in the bulk flow. Thus, molecular diffusion, not Brownian motion, is the primary transport mechanism. The model further reveals that the thermal diffusivity is comparable or slightly greater than the mass diffusivity of the Na_2SO_4 solution, which suggests gas-like behavior, despite the liquid-like ($\approx$0.6 g/ml) densities at these near-critical conditions. This contrasts sharply with ambient liquids where the relative mass diffusion rates are nearly 2000 times slower. Thus, the experiments and model identify the mechanism of salt deposition, reveal information hitherto lacking on the transport properties of salt solutions, and offer an opportunity to predict and control salt deposition.

SURF II USED FOR PROTEIN ANALYSIS

A NIST scientist has used SURF II (the Synchrotron Ultraviolet Radiation Facility) for the measurement of electroreflectance from metal surfaces with adsorbed proteins. This technique requires the measurement of the change in reflectance induced by a sinusoidally varying applied potential. The bare metal surface has a characteristic reflectance signature in the 250 nm to 400 nm spectral region due to electron plasma oscillations. However, adsorbed species can alter the signal dramatically. This is especially true if the adsorbed molecule is a protein with a metal site that can be reduced and oxidized by electrons from the electrode. The photon absorption tends to be very different in the two redox states, leading to a large reflection modulation amplitude, which can be used to determine electron transfer rates between certain metaloproteins and electrodes.

The NIST Scientist found that synchrotron radiation from SURF II is highly stable, continuously tunable, and results in a greatly improved signal-to-noise ratio over other available UV sources. He observed intrinsic intensity noise at a level of 10^{-5} in the 10 Hz to 100 Hz region, of paramount importance for observing electroreflectance modulations of typical magnitude 1 part in 10^4. These are results obtained in the first experiment on cytochrome-c immobilized on an evaporated gold electrode.

DEVELOPMENT OF NEW INFRARED SOURCE

NIST scientists have collaborated on a project resulting in the development of a new, brighter, infrared (IR) source. The new source yields better signal-to-noise ratios, and therefore higher accuracy, in IR measurements. The source is a stabilized argon arc, which has been characterized in the spectral range of 1 μm to 20 μm. Its radiance was calibrated and found to be approximately equal over much of this range to that of a 10 000 K blackbody. A high-resolution spectrum taken with a Fourier transform IR instrument shows mostly line emission below 5 μm and pure continuum between 5 μm and 20 μm. The stability and geometrical properties of the radiance were determined as well as its dependence on pressure and current. As a result of this project, this source is being used in calibrating IR detectors as well as in projects aimed at advancing IR measurements and technology.

DEEP-UV INDEX-OF-REFRACTION MEASUREMENTS FOR PHOTOLITHOGRAPHY

NIST researchers have completed initial measurements of the index of refraction of fused silica near 193 nm. These measurements are critical in the race to develop photolithographic wafer steppers for future-generation integrated circuit manufacturing. This collaborative project, with MIT Lincoln Laboratory and SEMATECH, seeks to develop the infrastructure required to utilize 193 nm, deep-UV excimer laser emission to form 0.18 μm feature sizes for such products as gigabit memory chips. These results keep industry on track to meet the Semiconductor Industry Association roadmap target date of 2001 for commercial production of these chips.

The NIST researchers have made high accuracy, temperature- and wavelength-dependent index-of-refraction measurements on optical materials that are required for such photolithography. Design engineers require this precision data to achieve the exacting performance requirements of the photolithography tools. To meet the immediate need, the researchers upgraded a precision refractometer, including precisely characterized UV line sources, to enable minimum deviation angle, refractive index measurements that have a relative uncertainty of 1 part in 10^5 with a temperature control of 0.1 °C. For the longer term and for shorter wavelengths, they are developing interferometric methods capable of even higher accuracy.

RAMAN SPECTROSCOPY WORKSHOP ADDRESSES NEEDS IN ADVANCED TECHNOLOGY

A workshop on Raman Spectroscopy in Optical and Material Sciences was held Dec. 10, 1996, as part of the Raman spectroscopy program at NIST. The workshop was devoted to the use of Raman spectroscopy for the understanding and characterization of condensed-phase materials of contemporary importance in technology and industry. Invited speakers from NIST, industry, and government laboratories described research areas in which Raman spectroscopy plays an important role. Present were representatives from the Army Research Laboratory at Ft. Belvoir, the University of Maryland, Howard University, private industry, and NIST.

The eight invited talks were followed by a talk describing the NIST Magneto-Raman Spectroscopy Laboratory including the presentation of the results of current research, and an announcement of the facility's availability as a user laboratory for the NIST staff and other interested parties.

The workshop concluded with a discussion of the current state of Raman spectroscopic research in optical and material sciences, its outlook for the future, and the needs to be met by the user laboratory. The participants stressed the needs for a computer searchable, critically evaluated database of Raman spectral data of organic materials and polymers, and the development of new and improved measurement probes based on fiber optics as well as further developments in Raman imaging techniques.

RADIONUCLIDE SPECIATION STANDARD EXTRACTION METHOD

The remediation of land contaminated with radionuclides from the nuclear power and defense industries is a national concern. However, the behavior of radioactive elements in the environment is fundamentally dependent upon their physico-chemical form, or speciation (e.g., hosted in organic complexes, adsorbed to exchangeable positions, occluded within clay-mineral lattice sites, etc.). No technique currently exists that allows for direct measurement of environmental-level radionuclide speciation in soil or sediment. The most widely applied indirect technique has been sequential chemical extraction techniques designed to chemically fractionate soil and sediment matrices into specifically defined fractions. Although such methods are used widely, no standard method currently exists.

NIST is collaborating with the Florida State University to establish a standard sequential chemical leaching method to characterize environmental soils and sediments. The method will provide selective dissolution of discrete geochemical phases of soil and sediment samples. Results from the application of the method will be used for identifying the primary radionuclide host phases of radiological contaminants and thus can be applied to risk assessments and the development of sound remediation strategies.

Exploratory experiments conducted on a marine sediment indicate that stable element analyses conducted in tandem with radiometric measurements are required to assess phase specificity of selective leaches. Certain chemical reactants and solvents improve the selectivity of the analyses, and certain radioactive elements tend to be associated with particular matrices. The next phase of the collaboration will focus on the optimization of the extraction protocol for extraction efficiency and selectivity. The data from this experimental work will be valuable to interpret the behavior of uranium, plutonium, and other high-priority radionuclides from the geologic components in the NIST natural-matrix SRMs.

FREQUENCY SYNTHESIZERS FOR PRIMARY FREQUENCY STANDARDS

Over the last several years a NIST scientist has developed a state-of-the-art synthesizer design that should prove useful for the next several generations of primary atomic frequency standards. These new generations of standards demand exceptionally low-phase-noise sources for interrogation of the clock transition. This synthesizer exhibits a fractional frequency stability of 10^{-16} at 20 min and 10^{-17} at 1 day. The synthesizer is being used on NIST-7, NIST's primary cesium frequency standard, and the linear ion standard, and it should be more than adequate for the cesium-fountain frequency standards now under development. Copies of the synthesizer have been delivered to other Federal laboratories and to other standards laboratories around the world.

DEMONSTRATION OF A CRYOGENIC LINEAR-ION-TRAP FREQUENCY STANDARD

NIST scientists have developed a cryogenic linear-ion-trap system that can be used to investigate both microwave and optical frequency standards. Presently, the ion used for both standards is $^{199}Hg^{+}$. Trapping of ions eliminates the first-order Doppler shift, and laser cooling reduces the second-order Doppler shift to a very low value. Since observation times can be extremely long, linewidths can be very narrow, leading to high stability even for a small number of ions. For this system, fundamental systematic uncertainties are known to nearly one part in 10^{18} on the optical clock transition. (Of course, other technical problems might limit the performance short of this goal.)

In a preliminary test of the microwave standard, the scientists have demonstrated a frequency stability of 3×10^{-15} 1000 s, but further work should improve this result. The dominant systematic effect appears to be caused by a second-order magnetic field shift due to the presence of currents flowing in the trap electrodes at the trap drive frequency. Efforts are currently under way to reduce this shift and establish a calibration procedure to remove its effects.

QUANTUM STATES OF MOTION OF A TRAPPED ATOM

In a generalization of their earlier "Schrödinger cat" work researchers at NIST have recently reported the creation and full determination of several quantum states of motion of a $^{9}Be^{+}$ ion bound in an rf (Paul) trap. The states were coherently prepared from an ion that was initially laser cooled to the zero-point of motion. They then created states having both classical and nonclassical character, including thermal, number, coherent, squeezed, and "Schrödinger cat" states. Finally, they fully reconstructed the motional state using two novel schemes. One determines the density matrix in the number state basis, and the other determines the Wigner function. These techniques, which are scaleable to several simultaneously trapped ions and to other quantum systems, should allow for well-controlled experiments on decoherence and related phenomena on the quantum-classical borderline.

TELECOMMUNICATIONS SYNCHRONIZATION

NIST is leading efforts to integrate synchronization techniques developed by the telecommunications industry with those developed by the metrology community. As part of this effort, NIST has now sponsored five annual workshops on synchronization, with attendance growing to more than 60 participants per year, mostly from U.S. industry. These workshops grew out of earlier NIST work on synchronization interface standards; industry then asked for assistance in developing more useful timing measures. The rapid success of this venture, along with the rapid acceptance of the results as national and international standards, cemented a working relationship that has stimulated the continuation of the workshop into something that more nearly represents an annual conference on telecommunications synchronization.

In a related project, NIST scientists are developing a system for two-way time transfer in optical-fiber (SONET) links for application to synchronization of network nodes. The system stability has been shown to be better than 100 ps over a period of 4 hours. This work responds to growing international interest in transmitting timing signals for use within the network.

MOLECULAR BEAM EPITAXY CHAMBER COMMISSIONED AT THE COLD NEUTRON RESEARCH FACILITY

A molecular beam epitaxy (MBE) facility custom designed for the specific requirements of neutron scattering recently was commissioned at the NIST Cold Neutron Research Facility (CNRE). It can be used to epitaxially grow single crystal thin films, heterostructures, and superlattices. It also can be used as a protective ultrahigh vacuum environment for both bulk and thin film surface investigations, and as an ultraclean environment for loading interstitials—all while performing in-situ neutron scattering investigations. This chamber will have a wide variety of applications, including studies of surface magnetism and artificial magnetic structures, Si/SiO_2 interface structure, critical phenomena, and hydrogen in materials. It already has been used to study the effects of hydrogen on the magnetic ordering of Fe layers in Fe/V superlattices. While several such chambers are used at synchrotron facilities around the world, this is the only MBE chamber in place at a neutron scattering facility.

To accommodate the many requirements of neutron reflectivity as well as the demands of the experiments to be investigated, the MBE chamber was designed with many unique features. These include large diameter samples ($\approx$75 mm) with temperature control from $\approx$15 K to $\approx$1300 K, a 30 mm flood ion gun for surface cleaning of substrates and samples grown elsewhere, a large cylindrical neutron window that allows both reflectivity measurements and grazing angle neutron diffraction measurements, a unique feedthrough to apply magnetic fields to the sample, and a gas loading system.

Although this chamber was designed to be flexible enough to operate on a wide variety of neutron spectrometers, its primary location will be the NIST polarized neutron reflectometer. This reflectometer, after its recent move to a cold neutron position in the CNRF, has achieved a factor of five increase in intensity over that in the reactor hall—with lower background. Now, reflectivities down to 1×10^{-8}, the best achieved worldwide to date, have been measured. The combination of high reflectivity sensitivity and MBE chamber versatility will allow measurements of totally new phenomena in physics, chemistry, and materials science.

NIST RESEARCH CITED AS SIGNIFICANT CONTRIBUTION TO POLYMER PHYSICS

NIST research on phase separation in ultrathin films made from polymer blends was selected by the American Physical Society (APS) as one of the significant accomplishments in physics for 1996. The NIST research addressed how the miscibility and mobility of polymer blend components and the morphology of the blend films are modified when these films are made very thin. The observations help explain why the properties of thin polymer blend films are often different from bulk polymer materials and may lead to better ways of controlling properties during the manufacturing process. Ultrathin polymer films are important components of advanced technologies such as microelectronics.

The NIST studies on films of polystyrene and polybutadiene on silicon substrates revealed a wide range of new phenomena in phase separation when films were thin enough (< 200 nm) to suppress the normal tendency of the dissimilar polymer components to phase separate into layers transverse to the film surface, as commonly seen in thicker blend films. Optical photographs of the phase separation in these "ultrathin" films had the appearance of the bicontinuous fluid pattern characteristic of phase separation ("spinodal pattern") in the case where the polymers have nearly equal compositions. Atomic force microscopy revealed that the optical patterns arose from undulations of the polymer-air boundary associated with variations of the surface tension within the film. The thicker "ultrathin" films (100 nm) showed a coarsening of the pattern size similar to bulk blends, apart from a change in the coarsen ing rate arising from the thin film geometry. The thinner (20 nm) films, however, exhibited a qualitatively different coarsening kinetics consistent with recent computer simulations of phase separation in ideally two-dimensional fluids. Finally, a 200 nm film was examined and found to give no surface pattern formation over an extended period of time (days), as expected for a film phase separating transverse to the solid substrate.

These NIST experiments revealed novel phase separation processes in ultrathin polymer films that are sensitive to film thickness and the chemical nature of the solid substrate. Many further fundamental questions arise from this research. How is the critical temperature for phase separation altered in ultrathin films? What controls the characteristic length below which the novel surface pattern formation occurs? Can the surface morphology be controlled? An understanding of these questions will advance rational design of ultrathin films.

MONITORING TEXTURE DEVELOPMENT DURING THE ROLLING OF COPPER AND BRASS

A private company has a long tradition of precisely tailoring its sheet metal products to meet user's needs. This involves maintaining careful control over the textures and grain sizes developed during each rolling and annealing step in the production process. Since metallographic examination and x-ray diffraction determination of these microstructural features are time consuming, off-line processes, the company signed a Cooperative Research and Development Agreement with NIST to apply its experience with on-line monitoring of texture and formability in steel to this problem.

NIST scientists combined recent theoretical developments in ultrasonic wave propagation in anisotropic plates with new measurement techniques to develop a rapid method for converting accurate measurements of the velocity of ultrasonic Lamb waves into a mathematical description of the preferred orientations of the grain structure of the sheet (i.e., its texture). By combining commercial instrumentation with carefully designed electromagnetic transducers, a system for monitoring the development of texture as the cast alloys are rolled into tin sheet was designed and made available to the company.

The system also is capable of following the changes in texture as the sheet is annealed to produce the final properties that meet the needs of the ultimate customer. Because the mathematical description of the texture measured by the ultrasonic waves reduces to numerical values for three coefficients, the development of the texture from casting through rolling and annealing to the final product may be traced easily by a curve in the three-dimensional space defined by the three numerical coefficients. Thus, an optimum path for producing a particular product may be developed and monitored during production.

SCANNING ACOUSTIC IMAGING OF STRESS IN MATERIALS

Acoustic microscopy relies on computer analysis of acoustic waves that are radiated into a material and detected using acoustic microscope lenses. Acoustic techniques long have been used to detect cracks and patterns of mechanical stress in materials by measuring the speed of ultrasound waves following different paths. A further development by NIST of this technique, described in a recent issue of Nature (Vol. 384, No. 6604, pp. 52-55), achieves high spatial resolution by exploiting interference between polarized acoustic

waves. The ability to map internal stresses nondestructively will be particularly useful for examining components such as microelectronic devices, engine parts, layered composites that may delaminate under stress, and welds in pressure vessels. Detailed theoretical analysis for many practical deployments is in progress. Further information is available on the World Wide Web (http://www.ctcms.nist.gov/kfrankli/stresses.html). NIST recently was issued two U.S. patents on this technique.

NIST DEVELOPS MEASUREMENT METHOD FOR LEAN FLAMMABILITY LIMITS OF REFRIGERANTS

The refrigeration industry is examining new refrigerants as chlorofluorocarbons are being phased out due to their harmful effect on stratospheric ozone. Some of the new candidates are flammable. While some of these are being used in the pure state, the more flammable chemicals are often blended with nonflammable partners to reduce fire hazard. The current standard test method for measuring the flammability of either pure chemicals or blends is time consuming to operate and has a high degree of uncertainty, especially for weakly flammable refrigerants. Under sponsorship of the Air Conditioning and Refrigeration Technology Institute, two NIST scientists have developed a new, robust, and rapid method for measuring the lean flammability limit of the refrigerant in air. The device consists of two flows of premixed refrigerant and air, aimed at each other. A planar flame forms where they meet. As the fraction of refrigerant is reduced, the flame weakens and then extinguishes. The lean limit is determined as the minimum fraction of refrigerant in the mixtures before the flame goes out. The measurements are performed at different flow velocities and extrapolated to zero velocity, and the resulting value is a basic property of the refrigerant. The uncertainty for difluoro-methane is about 5 %. The findings are being published in Combustion and Flame, the journal of the Combustion Institute.

NIST SCIENTISTS DEVELOP NEW CLASS OF FIRE-RETARDANT MATERIALS

As materials evolve to a higher degree of functionality and safety, new types of formulations are needed. Scientists have been examining novel materials formed by reacting a host organic resin with fine clay particles. At low clay additions (mass fractions of less than 20 %), a polymer layered silicate nanocomposite is formed. NIST scientists working with another government laboratory, have shown that these materials also offer a new means for achieving a high degree of fire retardancy. Reaction of nylon-6 with only 5 % clay results in a product whose peak rate of heat release is reduced by a factor of three. The rate of heat release is the principal property controlling fire growth. The mechanism that results in this significant improvement in fire safety is different from that in simple mixtures and is leading to approaches for other resins.

NIST CO-SPONSORS GREEN BUILDING CONFERENCE

NIST joined with U.S. Green Building Council, the American Institute of Architects, and the San Diego Gas and Electric Company to sponsor the “Third International Green Building Conference.” Green buildings are defined as those designed, constructed, operated, and demolished in ways so as to have a minimum impact on their global neighborhood, and internal environments. The conference, held in San Diego, CA, Nov. 17-19, 1996, brought together experts in the field of green buildings. Approximately 300 people attended this conference, including representatives from industry, academia, and government. International professionals presented the latest information on sustainable practices, materials, and technologies.

A highlight of the conference was a presentation that included a world overview of building-integrated photovoltaic activity with a country-by-country description of component and systems development with selected examples of Solar Electric Architecture. The presentation and 19 others are included in the proceedings from the conference, NIST Special Publication 908, Third International Green Building Conference and Exposition-1996. The proceedings may be obtained by contacting Paula Svincek, ext. 5648.

NIST RESEARCH ADVANCES A STANDARD NEEDED FOR INTERACTIVE CABLE TV

Four private companies have worked with NIST to develop standard specifications for cable TV over hybrid fiber coaxial networks. Working jointly with these companies and other members of the IEEE 802.14 standards group, NIST conducted an unbiased performance evaluation of the media access control (MAC) protocols that had been submitted to the standards group. Researchers implemented the candidate MAC protocols using a commercial network simulation package. The results from this work have been reported to the IEEE 802.14 group, and the software simulation modules have been released to the public. These modules currently are used by several companies as they develop their own standards-conformant MAC protocols.

NEW PUBLICATION DESCRIBES STRUCTURED TESTING METHODOLOGY

NIST Special Publication 500-235, Structured Testing: A Testing Methodology Using the Cyclomatic Complexity Metric, describes the structured testing methodology for software testing, known as basis path testing. Based on the cyclomatic complexity measure, structured testing uses the control flow structure of software to establish path coverage criteria. The resulting test sets provide more thorough testing than statement and branch coverage. The publication also presents extensions of the fundamental structured testing techniques for integration testing and object-oriented systems and describes several related software complexity metrics.

NIST RESEARCHERS CONSIDER DIFFUSE-INTERFACE MODEL FOR FLUID-FLUID SYSTEMS

NISTIR 5887, A Diffuse-Interface Description of Fluid Systems, describes a diffuse-interface model for fluid-fluid systems. In classical models, an interface between two fluids is treated as infinitely thin, or sharp, and is endowed with properties such as surface tension. Diffuse-interface theories replace this sharp interface with continuous variations of an order parameter such as density in a way consistent with microscopic theories of the interface. NIST researchers related the diffuse-interface model to classical, sharp interface models by deriving asymptotically the governing equations and the associated boundary conditions used in the sharp-interface formulation. The diffuse-interface approach is illustrated by modeling internal gravity waves. Results reveal a singularity in the diffuse-interface model in the limit of incompressible perturbations.

WULFFMAN MAKES 3-D IMAGES "CRYSTAL CLEAR"

Wulffman? Despite the unusual moniker, this new NIST product isn't the agency's tribute to Lon Chaney Jr. or a device that grows hair when the moon is full.

Wulffman, an interactive computer program for drawing crystal shapes, was produced by researchers at NIST. The program is designed to help materials scientists quickly visualize three-dimensional faceted crystals and "quasi-crystals."

The program gets its distinctive name from the term "Wulff shape," known to materials scientists as the crystal shape that an element or combination of elements will form naturally because it requires the least amount of energy. Given a crystal's symmetry and surface energies for specific crystal planes. Wulffman constructs an image of the crystal's Wulff shape.

Wulffman's creators hope the program will be useful for designing new materials where crystal shapes affect the material's bulk properties. For example, a major company already is using it to help design reflective paint It also should be useful as a teaching tool. The program is designed to run on Unix workstations. For further information and to download the program, visit the Wulffman website at http://www.ctcms.nist.gov/~ryan/wulffman.

NEW MEASUREMENT CONSORTIUM TO AID INDUSTRY

Engineers and measurement experts at suppliers, manufacturers, and users of ceramics can join a new effort to find agreement on technical measurement methods for ceramic powders—from raw materials to the final product—that can ultimately enhance the competitiveness of the U.S. ceramic processing industry. The Ceramic Processing Characterization Consortium, now being formed by NIST, will work to strengthen the measurement and standards infrastructure for ceramic particulate systems.

The consortium's mission is vital to ceramists because measuring a dense ceramic means thoroughly understanding the physical and chemical characteristics of the powder that made it. This makes ceramics far more dependent than plastics or metals on accurate measurements of raw materials. Because the focus on measurement and standards is a shared concern, results of the consortium will be generic, open, and freely shared with anyone who is interested.

To join the consortium, which has no membership fee, each member's organization must establish a memorandum of understanding with NIST. For more information, contact George Onoda, A256 Materials Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-4489, fax: (301) 990-8729, george.onoda@nist.gov, or visit the CPCC web site at http://www.ceramics.nist.gov/programs/cpcc/index.htm.

NEW METHOD OF DETECTING PIPELINE FLAWS PATENTED

Scientists at NIST have received a patent for a new method of measuring wall thickness and detecting flaws (such as cracks, delamination, scaling, and corrosion) in natural-gas pipelines by using ultrasound. Use of ultrasound inspections is common for oil pipelines but has been less effective for the natural gas variety. Gas is a poor coupling agent for the ultrasonic signals emanating from the transducer to the pipe wall.

The NIST researchers showed that use of wide-band, well-dampened ceramic transducers and high-dynamic-range receiver amplifiers can overcome signal losses. They also demonstrated that by using a diplexer, the same transducer can be used to generate and detect the ultrasonic signals.

"This method is inherently suitable for the task, because it relies on the use of the natural gas as the coupling fluid for transmitting the probing ultrasonic signals into and out of the pipe wall," the patent states.

NIST is working currently with the Southwest Research Institute, under a contract with the Gas Research Institute, to show the practicality of the technology. For a copy of the patent, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, sarabeth@boulder.nist.gov. For technical information, contact Chris Fortunko, Div. 853, NIST, Boulder, CO 80303-3328, (303) 497-3062, fortunko@boulder.nist.gov or Jerry L. Jackson, SWRI, 6220 Culebra Rd., P.O. Drawer 28510, San Antonio, TX 78228, (210) 522-2957, jjackson@swri.edu.

NACLA PROPOSAL ACCEPTED BY U.S. LABORATORY COMMUNITY

Representatives of the nation's conformity assessment community decided at an open forum at NIST on Jan. 7, 1997, to go forward with a proposal to establish the National Council for Laboratory Accreditation (NACLA). NACLA will be a cooperative partnership between the public and private sectors to provide a comprehensive U.S. national laboratory accreditation infrastructure. In the resulting system, the testing and calibration data from a laboratory accredited by a NACLA-recognized body will receive worldwide acceptance based on the laboratory's competence. The open forum, co-sponsored by NIST, ANSI, and ACIL, (formerly the American Council of Independent Laboratories) was attended by more than 350 interested stakeholders from laboratories, accreditors, and those who require accreditation, both from industry and government. Immediate plans call for the election of a balanced, interim board of directors made up of members from the various stakeholders. The board will lay out a proposal for NACLA functions and a permanent structure. These proposals will be reviewed by stakeholders at future public meetings.

ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS

During the month of December 1996, NIST recommended the following two innovative technologies for commercialization to its Department of Energy partner under the Energy-Related Inventions Program: Method for forming thin semiconductor films—a process to manufacture cadmium telluride (CdTe) photovoltaic cells by fusing nano particles of CdTe on a metal or glass support plate; Cyclic microwave treatment of pressed garments—a process for using microwaves to set the permanent press or fire-retardant chemicals in garments.

APPLICATION OF CRYOGENIC DETECTOR TO MASS SPECTROSCOPY SHOWS PROMISE OF GREATLY ENHANCED PERFORMANCE FOR LARGE MOLECULES

NIST researchers in collaboration with researchers from the University of Neuchatel and George Mason University, recently performed initial experiments demonstrating the utility of a NIS (normal-insulator-superconductor) tunnel junction as a molecular detector in a MALDI-TOF (matrix assisted laser desorption and ionization−time-of-flight) mass spectrometer. Conventional MALDI-TOF spectrometers, which use an electron multiplier as the detector, are not useful for analysis of biological macromolecules such as DNA, in part because only a small fraction of high-molecular-weight species are detected. In contrast, the NIS detector, fabricated on a silicon nitride membrane for thermal isolation and cooled to 100 mK, promises nearly 100 % detection efficiency, fast response time, and accurate calorimetric energy measurement even for macromolecules. The detection efficiency and response time assure accuracy in the time-of-flight mass determination, while the calorimetric energy deposited in the detector provides an important check on the ionization state of the molecule. In recent experiments with bovine serum albumin (BSA), having a molecular weight of 66 000 μ, singly and doubly ionized molecules were distinguished easily with an NIS detector having a rise time of about 1 σ. If the full potential of such detectors is realized, MALDI-TOF spectrometers may reduce the time and sample size required for DNA sequencing to a small fraction of that presently required with gel electrophoresis.

NIST DEMONSTRATES ±1 V SNS JOSEPHSON-JUNCTION PROGRAMMABLE VOLTAGE STANDARD ARRAY

Voltage standards that can be quickly switched between levels are needed by U.S. industry for characterizing digital-to-analog (D/A) and analog-to-digital converters. NIST has developed such a programmable voltage standard that will enable fast dc voltage measurements on a microsecond scale. The new circuit has the

equivalent resolution of a 9-bit D/A converter and an accuracy of 30 bits.

The heart of the circuit is an array of 32 768 Josephson junctions. In order to achieve programmability, the array is divided into a binary sequence of segments, in which the least significant bit has 128 junctions and the most significant bit has 16 384 junctions. The voltage across the array is programmed by biasing each segment at one of three constant voltage steps at V = 0 and $\pm Nf/K_J$, where N is the number of junctions in the array segment, f is the microwave signal frequency, and K_J = 483 597.9 GHz/V, the Josephson constant. When this array is driven with a 16.0 GHz microwave signal, it produces 1023 selectable reference voltages in increments of 4.235 mV from –1.085 V to +1.085 V. The voltage steps of the whole array were found to be flat over a current range of about 2 mA.

The circuits are based on a novel superconductor-normal-superconductor (SNS) junction technology developed at NIST. In this technology, a high-resistivity palladium-gold alloy is used as the normal metal barrier sandwiched between two superconducting niobium electrodes. The 2.5 μm × 2.5 μm junctions have an area 1/130 times the area of junctions in the present dc voltage standard. The initial test has shown functionality of the full circuit proving that all 32 768 junctions are working as desired. This demonstrates a level of junction and microwave distribution uniformity about five times better than is typically achieved in the previous technology.

OIDA CALLS FOR NIST ASSISTANCE

In a report previewed at its Annual Forum in Washington, DC, Oct. 17–18, 1996, the Optoelectronics Industry Development Association (OIDA), called for NIST assistance in addressing several problems related to optoelectronics manufacturing. The report summarizes the results of a 6-month-long study of manufacturing infrastructure which focused on four areas of optoelectronics: semiconductor components, passive components, optical storage media, and packaging.

In the semiconductor components area, the OIDA asked for assistance related to the purity and analysis of starting materials and for broad assistance in measurement technology and reference standards. In the passive component area, OIDA asked for assistance in lowering the cost of measurements and for NIST consideration of programs to develop broader functionality in a single set of technologies, to enable large volume applications. For optical storage, it called for NIST to work with media manufacturers to define critical measurement issues and to help in their solution. These are among 15 recommendations contained in the report.

The OIDA has decided that the semiconductor components area should have the highest priority and is preparing to lead discussions among NIST, Sandia National Labs, and a group of manufacturers to develop an action plan.

ELECTRON-BEAM LITHOGRAPHY CAPABILITY AVAILABLE IN GAITHERSBURG

Three NIST scientists have developed and installed an electron-beam lithography (EBL) capability at the NIST Gaithersburg laboratories. This EBL facility is the first available on the Gaithersburg site and will extend nanoscale device fabrication capabilities significantly. EBL capabilities have been available in NIST's Boulder facility for some time.

As implemented in the Gaithersburg facility, EBL gives researchers the ability to define thin-film structures with lateral dimensions below 100 nm. The capability is realized using a commercial scanning electron microscope, together with a separate computer controller, which outputs two voltages to control the lateral position of the electron beam. Together with a CAD software package, this allows definition of the pattern on the computer, followed by a rastering of the electron beam on the substrate.

The researchers have made various test patterns, as well as electrical and optical devices, with typical minimum feature sizes as small as 50 nm. NIST researchers in Gaithersburg will use EBL to fabricate single-electron tunneling devices for a capacitance standard and later perhaps for a current standard. They will also use it to build fine-scale test structures for supporting U.S. advanced semiconductor manufacturing and for related nanostructures research.

NATIONAL ADVANCED MANUFACTURING TESTBED (NAMT) UNVEILED

NIST recently unveiled its new National Advanced Manufacturing Testbed (NAMT). The NAMT is a NIST-hosted and initiated program that is addressing measurements and standards needs associated with information-based manufacturing. In the NAMT, NIST is using a recently developed infrastructure for advanced computing and communications to enable or facilitate a series of manufacturing research projects conducted collaboratively with multiple partners residing at several locations. NAMT projects are characterized by the transfer and use of data, video, and audio information among partner facilities in the performance of leading-edge research across the spectrum of manufacturing technologies.

The existing NAMT technical portfolio consists of four projects that have roughly two dozen industrial, government, and academic partners: (1) nanomanufacturing of atom-based standards, which is focusing on the fabrication, measurement, and dissemination of dimensional reference standards with geometric features based upon atomic lattice properties; (2) machine tool performance models and machine data repositories, which is developing performance-based predictive models for machine tools and coordinate measuring machines that accurately represent real-world manufacturing and measurement capabilities in a virtual environment; (3) characterization, simulation, and remote access of hexapod machines, which is developing tools and methodologies for collaboratively using, characterizing, simulating, and enhancing the performance of a revolutionary new class of parallel-actuated machine tools; and (4) framework for discrete parts manufacturing, which is developing specifications for integrating heterogeneous software applications within distributed manufacturing systems, thus promoting interoperability.

IMPROVED PROCESS CONTROL IN SEMICONDUCTOR PROCESSING

Many semiconductor processes use the thermal mass flowmeter, or mass flow controller (MFC), to deliver known flows of gas to the processing chamber. Many hundreds of these devices are found on a typical fabrication line. Critical semiconductor processes now require relative flow measurement uncertainties of 1 % or less over the range of 10^{-7} to 10^{-3} mol/s. MFCs can provide the wide flow range and are tolerant to the corrosive gases required for semiconductor processing; however, they long have been suspected as having uncertainties much higher than the "few percent" claimed by the manufacturers. As part of the National Semiconductor Metrology Program, scientists at NIST have carefully evaluated the operating characteristics of low-gas-flow MFCs from five major manufacturers to determine if the required uncertainties can be met.

In a calibration of the meters against the NIST primary standard using nitrogen gas, two of the five meters had relative uncertainties of 7 % and 17 %, significantly outside the ascribed uncertainty (± 1 %). The long-term stability of all the meters was within ± 1 % over 1 year, indicating that, with recalibration, all the units would meet the requirements for process control. Although MFCs are used with a variety of gases, most MFCs are calibrated using nitrogen, and gas correction factors are applied to determine the flow with other gases. The meters were calibrated at NIST with several gases, and the correction factors were measured and compared to the manufacturers' reported values. Reported values were in moderately good agreement for helium, argon, and sulfur hexafluoride (–0.7 % to 1.6 % average deviation), while the correction factors for hydrogen and hexafluoroethane were not (10.2 % and 7.7 % average deviation, for all the units). Hence, to achieve the relative uncertainties of 1 % or less required for semiconductor processing, MFCs must be calibrated with the gas to be used at conditions close to the conditions of use. To provide measurement assurance for the reference flow meters used by the manufacturers in the calibration of their meters, NIST has developed a high-repeatability flow transfer standard using an ultrastable laminar flow element, which has a relative uncertainty of 0.07 %. This transfer standard has been used this past year in flow proficiency tests over a flow range of 10^{-6} to 10^{-3} mol/s at four MFC manufacturers and two equipment suppliers' laboratories.

NIST SCIENTIST ELECTED CHAIR OF INTERNATIONAL MANAGEMENT BODY

At the November 1996 annual meeting of the international steering committee of the Network for the Detection of Stratospheric Change (NDSC), a NIST scientist was re-elected committee chair for a new three-year term. The NDSC is a ground-based, long-term international measurement and analysis network designed to make the observations through which changes in the physical and chemical state of the stratosphere can be determined and understood. This dual objective of observation and understanding requires high-precision, state-of-the-art measurements of ozone, as well as a broad range of chemical species and dynamical tracers that influence ozone. On a shorter timescale, the NDSC provides independent calibration for satellite sensors and produces a useful data set for testing and improving stratospheric models. It consists of nearly 50 measurement sites in nearly 20 different countries and has been endorsed by the United Nations Environment Program, by the International Ozone Commission of the International Association of Meteorology and Atmospheric Physics, and by the World Meteorological Organization (WMO) as a major contributor of WMO's Global Ozone Observing System within the framework of its Global Atmosphere Watch. The steering committee is the primary managerial body of the NDSC.

INFRARED ARRAYS FOR FTIR MICROSCOPY

Infrared spectroscopy long has been used to identify chemical species and their environments. Molecules of different species have characteristic spectral absorption features in the 2.5 μm to 11 μm wavelength region, which provides distinctive "fingerprints" for their identity. Coupled with microscopy, spatially resolved infrared spectroscopy is a versatile measurement tool with applications including materials and film characterization, biological research, and medical diagnosis. However, the application of infrared microscopy has been limited by the brightness of infrared sources and by the use of single-element infrared detectors, which necessitate slow rastering of the probed areas to generate images.

Researchers at NIST and National Institutes of Health (NIH) have demonstrated rapid, sensitive spectral imaging in the infrared fingerprint region by attaching NIST's mercury-cadmium-telluride (MCT) infrared detector array to NIH's step-scan Fourier transform infrared microscope. The NIST MCT array is a 256×256 pixel focal-plane detector array, originally designed for DoD projects but now available for civilian applications. An interferometer with a glow-bar source steps its moving mirror through successive positions while the MCT array obtains background-corrected infrared images. In this way, 65 536 time-domain interferograms are obtained, each corresponding to a specific spatial position of the sample. Fourier transforming this massive data set (approximately 100 MB) yields spectrally sensitive images with 10 μ spatial resolution and 16 cm^{-1} spectral resolution over the entire 2.5 μm to 11 μm spectral region. The NIST/NIH team obtained chemical images of inhomogeneous polymer and lipid samples, laminates, and brain tissue, illustrating a wide range of industrial and biomedical applications for this unique apparatus and new imaging technique.

POLYMERS DESIGNED WITH MINIMAL SHRINKAGE DURING POLYMERIZATION

Monomers prepared recently at NIST have attracted the attention of several companies with interests as diverse as dental composite restoratives, lithographic materials, and adhesive coatings. All of these polymer-based applications currently suffer from deficiencies induced by excessive polymerization shrinkage. In the NIST work, a simple method was devised for the preparation of spiro orthocarbonate (SOC) monomers that are characterized by their unusual ability to polymerize with an expansion in volume. The combination of expanding SOC monomers with conventional monomers that contract allows a means to control polymerization shrinkage and its deleterious effects.

The new SOC monomers are actually pre-polymers containing many SOC units repeated along a backbone structure. A cationic double ring-opening polymerization process converts the liquid multi-functional prepolymer into a rigid, three dimensional network with volumetric expansions of 2 % to 3 %.

One of the initial applications of these SOC monomers is as a component of dental composite restorative materials where problems associated with polymerization shrinkage are well documented. The curing contraction results in tooth deflection, which generates pain when the bonding is good and the formation of gaps when the adhesion between the restoration and tooth fails. Copolymerization studies involving SOC prepolymers and traditional methacrylate monomers used in dentistry have been conducted. Results show that practical hybrid composites with extremely low polymerization shrinkage values and good mechanical strength properties can be produced. Meetings have been held with several dental materials manufacturers to discuss the potential of these new monomers.

USING MAGNETOSTRICTION TO MEASURE THE STRENGTH OF STEEL

During the magnetization of a ferromagnetic material such as steel, magnetic domain walls sweep through the material and interact with the same microstructural features that determine its strength. As a result, such measurable properties as Barkhausen noise, coercive force, and differential permeability have been used as nondestructive predictors of the mechanical strength of finished steel products. These magnetic properties are associated with the low field properties of the hysteresis loop and, therefore, can be influenced strongly by the part geometry and extraneous magnetic fields. During recent studies at NIST of a noncontacting method of measuring the magnetostrictive coefficients of high strength low alloy (HSLA) steels at high and intermediate fields, it was observed that the magnetic fields' dependence of the coefficients was sensitive to the development of the precipitates that give these materials their high strengths. Thus, it appears possible to develop a noncontacting and nondestructive probe for predicting the strength of the HSLA steel products currently being produced in large quantities by the steel mills. The method of making the magnetostrictive measurements is based on theoretical and experimental studies performed in the 1970's to explain the role of magnetostriction in the efficiency of electromagnetic transducers (EMATs) when applied to ferromagnetic materials. This efficiency can be deduced from measurements of the enhanced strength of ultrasonic signals produced by EMATs when operated on steels at relatively easily obtained applied fields.

NEW RECORD SETTING VALUES FOR GIANT MAGNETORESISTANCE SPIN VALVES MEASURED

In the few years since they were discovered, giant magnetoresistance (GMR) spin valves have become a hot topic in the computer industry. These ultrathin magnetic multilayer films are being developed by hard disk drive manufacturers as magnetic read-heads with unprecedented sensitivity for reading ultrahigh density hard disks. Another potential application for GMR spin valves is magnetoresistive random access memory (MRAM) chips, which may become a non-volatile replacement for dynamic random access memories. Industrial development is being supported strongly by the Defense Advanced Research Projects Agency. Recent investigations at NIST have discovered that the highest GMR values are not found in films grown in the best vacuum. Some impurities, most notably oxygen atoms, actually increase the GMR by acting as a surfactant during film growth and suppressing defects. By deliberately introducing minute amounts of oxygen into an otherwise extremely clean deposition system, the largest GMR values ever reported for two basic types of spin valves were achieved, 25.8 % for symmetric spin valves, 19 % for bottom spin valves. These NIST accomplishments constitute an important step toward the realization of GMR read-heads and MRAM chips.

OBJECT-ORIENTED FINITE-ELEMENT CODE DEVELOPED FOR MICROMECHANICAL ANALYSES OF MICROSTRUCTURES

Although material microstructures are heralded as the palette from which materials scientists can design and tailor material properties, no general methods exist for evaluating the behavior of a particular microstructure or for relating microstructural images to macroscopic behavior. Toward this objective, an object-oriented finite-element code (OOF) is being developed. OOF is designed to help materials scientists calculate macroscopic properties from microstructural images. The goal is to assign materials properties to all parts of an image and to perform finite-element calculations on the processed image, applying appropriate boundary conditions, temperature changes, distortions, etc. Thus far, OOF has been used to investigate micromechanical properties of several ceramic systems, including effects of glass composition and grain morphology on residual stress and fracture of silicon nitride, effects of porosity on elastic properties of thermal barrier coatings, and residual stress distributions in highly anisotropic ceramics. A beta version of OOF for SGI workstations is available as public domain software on the Internet in a NIST software archive. A link to this archive, as well as a description for using OOF can be found at the WWW address: http://www. ctcms.nist.gov/~wcraig/oof.html.

A portable X-Windows version is under development.

PHOTOVOLTAIC SOLAR WATER HEATING SYSTEM AT GREAT SMOKY MOUNTAINS NATIONAL PARK

A photovoltaic solar water heating system recently was installed to heat water used in the restroom facilities at the main visitors center within the Great Smoky Mountains National Park (GSMNP). Four to five million people visit the GSMNP each year. The system is based upon technology patented by NIST scientists.

This effort is a joint cooperative venture with the Tennessee Valley Authority, NIST, Sevier County Electric System, and the National Park Service. The system is being monitored to learn how well the system performs and to collect solar radiation data for the GSMNP personnel. The system performance data will be used by NIST to verify computer prediction models being developed by the University of Wisconsin. TVA will use the data to document the potential for reduction in energy and peak demand on their utility. The system is being monitored by measuring the energy supplied by the photovoltaic array, the energy supplied by the electrical utility grid, and the volume of water consumed. Data are collected every 10 s and forwarded weekly to NIST for analysis.

For more information on this system, contact Paula Svincek, ext. 5648 or visit the pvsolar page at http://bfrl.nist.gov/863/pvsolar/flyer.html.

APARTMENT BUILDING BURNED TO MEASURE FIRE FIGHTING EFFECTIVENESS

On Jan. 7, 1997, NIST scientists conducted a full-scale fire suppression experiment with an eight-unit apartment building in Mobile, AL. This experiment was conducted in conjunction with the Bureau of Alcohol, Tobacco and Firearms and the Mobile Fire and Rescue Department.

The objective of the experiment was to examine the suppression effectiveness of water and Class A foam solution at flow rates of 475 L/min used in full-scale fire fighting. These results will be compared with the smaller fire suppression experiments conducted by NIST under the sponsorship of the U.S. Fire Administration. The fire suppression experiments conducted during the USFA program involved residential house fires. Unfortunately, even at fire fighting flows as low as 38 L/min the house fires were extinguished easily using plain water. Larger fires were needed to challenge fire fighting equipment sufficiently to measure any differences in performance by different suppressants.

After fire suppression experiments were completed, BAFT agents conducted a post-fire investigation of the building for training purposes. In addition, a camera crew from the public television program, NOVA, filmed the experiment for inclusion in a series on survival technologies. The show is scheduled to be aired during the 1998 season.

NIST HOSTS FIFTH ANNUAL TREC WORKSHOP

On Nov. 20-22, 1996, NIST and the Defense Advanced Research Projects Agency (DARPA) co-sponsored the fifth Text REtrieval Conference (TREC-5) Workshop as part of the TIPSTER Text Program. Started in 1992, the conference series is designed to promote research in information retrieval by providing appropriate test collections, uniform scoring procedures, and a forum for organizations interested in comparing their results. Thirty-eight groups participated, including representatives from nine different countries and 10 companies. All participants used the same test collection that consists of about 2 gigabytes of text (over 1 million documents) and a given set of information needs. Two new retrieval tasks were introduced in the TREC-5 conference: retrieving Chinese documents and retrieving documents that have been corrupted through optical character recognition (OCR) scanning.

TREC-5 proceedings, to be published in the spring, are available on the NIST TREC Web site, http://www-nlpir.nist.gov/trec. Also available are the proceedings from TREC-4, which have been published as NIST Special Publication 500-236, The Fourth Text REtrieval Conference (TREC-4).

MATRIX MARKET EXCHANGE FORMATS PROPOSED

NISTIR 5935, Matrix Market Exchange Formats: Initial Design, proposes elementary ASCII exchange formats for matrices. Specific instances of the format are defined for dense and sparse matrices with real, complex, integer, and pattern entries, with special cases for symmetric, skew-symmetric, and Hermitian matrices.

OPTICAL CHARACTER RECOGNITION (OCR) WORK ADVANCES

NISTIR 5932, Design, Integration, and Evaluation of Form-Based Handprint and OCR Systems, addresses key issues in the design, integration, and evaluation of end-to-end systems for recognizing form-based handprint and cursive handwriting that arise in data entry applications. The applications include recognition of alphanumerals from tax, census, and insurance forms, bank checks, etc. To succeed in these applications, a system should be as accurate as possible and, at the same time, should minimize human intervention to correct that part of the text that machines fail to recognize.

Strong interdependence between the modules of an OCR system argues for an end-to-end system design and evaluation. Design issues such as system training, confidence measures, and the role of correction mechanisms employed by humans need to be considered. NIST researchers recommend a fairly general process for system integration and argue in favor of system-level evaluation. NISTIR 5932 documents several case studies involving OCR systems that have undergone large-scale evaluations, including the NIST form-based recognition system and several commercial systems.

SQL TEST SUITE, VERSION 6.0, AVAILABLE ON THE WEB

The SQL Test Suite, Version 6.0, used to validate commercial SQL products for conformance to SQL standards specified by the International Organization for Standardization, the American National Standards Institute (ANSI), and Federal Information Processing Standards (FIPS), can be downloaded from URL http://www.itl.nist.gov/div897/ctg/ software.htm≠pubsoft and select SQL.

Completed December 31, 1996, the SQL Test Suite, Version 6.0, was developed jointly by NIST, the National Computing Centre Limited (NCC) in the United Kingdom, and Computer Logic R&D in Greece. The European contributions to the test suite were produced under sponsorship from the European Community (EC).

The technical goal of the test suite is to help evaluate conformance of SQL implementations to Entry SQL or Intermediate SQL, as specified in ANSI X3.135-1992 and ISO/IEC 9075:1992, through one or more standard programming language interfaces. The test suite contains new tests to validate the Intermediate SQL level and additional tests to evaluate conformance to FIPS 127-2, Database Language SQL, specification of Transitional SQL, sizing parameters for database constructs and flagging of extensions, as well as X/Open Extensions for features specified in the X/Open CAE Specification. The test suite contains schemas and test programs for Interactive SQL and 10 different programming language test suite types: Embedded C, Embedded COBOL, Embedded Fortran, Embedded

Ada, Embedded Pascal, Module Language C, Module Language COBOL, Module Language Fortran, Module Language Ada, and Module Language Pascal.

NIST HELPS PUT AUTOMATION IN THE DRIVER'S SEAT

Fasten your seatbelts, surrender the steering wheel and cruise control, and let AUTONAV do the driving for you. Automated driving on streets and highways or driver-free reconnaissance over unpaved terrain behind enemy lines are two of the goals motivating work recently begun by a team of German and U.S. researchers.

The collaborators aim to develop a "next generation autonomous vehicle navigation control system." NIST, the Army Research Laboratory and the David Sarnoff Research Center will be exchanging technology with counterparts from Germany's University of Bundeswehr and a unit of a German automotive and aerospace company.

NIST's contributions include its real-time control system architecture, reference-model framework and hardware elements of a control system. The German technologies have been demonstrated in self-guided vans and passenger cars that have logged thousands of kilometers on the country's autobahn.

AUTONAV's goals are to extend the performance and capabilities of these technologies, incorporate and evaluate new ones, and, ultimately, develop a standard platform that manufacturers can use as the basis for the robotic or semiautomated vehicles of the future. For technical information, contact Maris Juberts, B124 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3424, <maris.juberts@nist.gov>.

NEW WAY OFFERED TO BETTER PREDICT CLOCK BEHAVIOR

For those who operate extremely precise clocks and oscillators, a principal task is characterizing the frequency stability of the devices. One recommended method has been to use the two-sample frequency variance known as the Allan variance statistic.

Recent NIST analysis has shown that this statistic has sensitivities and dependencies that make it less accurate for certain types of oscillator behavior and measurement protocols. A new data manipulation, dubbed "TOTAL VARIANCE," has been devised that time-shifts the sampling window. Instead of a limited two-sample measurement, the multisample approach estimates all possible observed frequency deviations over a set period of time. Previously seen estimation errors are reduced by as much as 100-fold.

Use of the "TOTAL VARIANCE" method results in more accurate predictions of the time errors that evolve after a precision clock is synchronized. Details of the new sample variance method are available in a paper, "An Extension of the Allan Variance with Increased Confidence at Long Term." For a copy of paper no. 5-97, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@micf.nist.gov>.

NATIONAL RESOURCE FOR GLOBAL STANDARDS LAUNCHED

The American National Standards Institute announced on Feb. 25, 1997, that its NSSN: A Global Standards Network is officially open for business.

The NSSN (formerly known as the National Standards Systems Network) is a World Wide Web-based, comprehensive source of information on regional, national, and international standards from private-sector organizations and Federal Government agencies. The network links the heterogeneous databases of these groups, giving NSSN users the ability to search multiple standards databases in a few minutes. Examples of standards included are those that set criteria for safety in consumer products, compatibility between computer software packages, and quality of exported products and services (such as the ISO 9000 series of standards). Information on over 100 000 standards—including about 30 000 military specifications—will be in the NSSN database by year's end. The new service is the result of a partnership between ANSI and NIST. The "NSSN Basic" service is free and located at <http://www.nssn.org on the World Wide Web. Users can search for standards by a document number, keyword or developer's name. Links to the WWW pages of specific developers are provided for additional information or ordering. "NSSN Enhanced," available later this year, will be a subscription service with more data about individual standards and an automatic alert to provide immediate notification of standards development and maintenance changes.

NEW GUIDELINES AVAILABLE FOR QUAKE "SHOCK ABSORBERS"

Slide to the left, slide to the right and don't fall down. No, it's not a new dance, but a building with shock absorbers—a feature that could someday save more lives and property in earthquake-prone regions of the United States.

NIST researchers have taken the first step toward making it easier for the construction industry to include

these shock absorbers—more commonly called seismic base isolation systems—in their building plans. NIST has developed performance guidelines for the devices that can be used as standards for reliability and capacity.

Seismic base isolation systems, designed to protect structures of fewer than 10 stories from the dangerous effects of strong ground shaking, isolate a structure from the moving ground by inserting a flexible rubber layer, or Teflon™ steel sliding panel interface, at or near the structure's foundation. The interface isolates the shaking of the ground from the shaking of the structure, minimizing damage.

Up to now, these devices had to be custom designed for each site and individually tested. New testing standards, based on the NIST guidelines, will define minimum levels of reliability and capacity. The sale of "off-the-shelf" isolation systems could then become a reality.

Approval of testing standards by the American Society of Civil Engineers is expected by the end of the year. For technical information, contact Andrew W. Taylor, B158 Technology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6078, <andrew.taylor@nist.gov>.

PARTNERS DRAMATICALLY IMPROVE ELECTRON SCOPE'S VISION

A new scanning electron microscope stage, jointly developed by NIST, a small Massachusetts firm and SEMATECH, will permit chip makers and other SEM users to cover nearly all angles when examining their samples. To be commercialized by the maker of custom accessories for electron microscopes, the new stage doubles the viewing range of typical SEMs and increases the tilt to better than 90° from the horizontal, as compared to the current 60°.

Such improved performance capabilities are expected to increase the utility of SEMs as measurement and research tools. Retrofitted on the SEM that NIST uses for dimensional measurements, the stage enables high-resolution, cross-sectional images of films and substrates. It also benefits measurements of so-called critical dimensions (also known as minimum feature sizes) on integrated circuits and the photomasks used to make them.

NIST has been working with SEMATECH, the consortium of U.S. chip makers and their equipment suppliers, to improve the measurement performance of SEMs and other microscopes used in semiconductor manufacturing. Representatives of several SEMATECH member companies have visited NIST to preview the new stage. For technical information, contact Michael Postek, A347 Technology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2299, <michael.postek@nist.gov>.

DNA DAMAGE/REPAIR COURSE SCHEDULED FOR OCTOBER

NIST, with support from a NATO grant, is organizing an Advanced Study Institute on DNA Damage and Repair to be held Oct. 14-24, 1997, in Antalya, Turkey. A NIST senior scientist who is a well-known expert in DNA damage and repair, is leading the international organizing committee for this high-level tutorial course.

The ASI will cover state-of-the-art knowledge and recent developments in DNA damage and repair, oxygen free radical effects, cellular protection and biological consequences. Presentations will be geared for postdoctoral and higher level scientists.

Approximately 70 applicants will receive support from the NATO grant to participate in the ASI. Scientists who are not eligible for this support but wish to attend should submit an application form, a resume of no more than three pages, a brief justification for participating, a recommendation letter from their supervisor and an abstract of their work. The use of electronic mail for applications is encouraged. The application deadline is June 15, 1997. Applicants will be notified of the decision of the organizing committee by Aug. 1, 1997. For more information and/or application forms, write to Miral Dizdaroglu, A353 Chemistry Building, NIST, Gaithersburg, MD 20899-0001, fax: (301) 330-3447, <miral@nist.gov>.

NIST IMPROVES PYROELECTRIC OPTICAL DETECTOR

Researchers at NIST have developed an improved pyroelectric detector, which, in turn, may improve the performance of products containing these sensors. Such products include burglar alarms, infrared motion detectors, automotive pollution sensors and a variety of military applications that depend on smart sensors.

A pyroelectric detector contains a material that produces a charge when heated. When the material is placed between electrodes and connected to a sensing circuit, a current is generated. These sensors are limited by their acoustic sensitivity, which can trigger false alarms.

The NIST solution (for which a provisional patent has been submitted) is to engineer two or more pyroelectric domains within a single crystal. This significantly

reduces the noise problem, simplifies the construction by using only one piece of material and simplifies the electrode pattern. For paper no. 6-97 describing this development, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, <sarabeth@boulder.nist.gov>.

IMPROVED SOFTWARE SPEEDS PC CHEMICAL ANALYSIS

Researchers who use electron microscopes for x-ray spectrometric analysis will be able to save hours designing experiments with an improved software/database package from NIST and the National Institutes of Health. The Desktop Spectrum Analyzer and X-Ray Database Version 2.5 allows scientists to simulate measurements in an electron microscope. It also helps them predict whether an experiment will produce spectra for accurate analyses without using laboratory trial and error to do so.

Available for Macintosh personal computers since 1992, the new version will operate on a PowerMac or any Macintosh computer with 5 MB of memory and a math co-processor. The database includes more quantitative analysis procedures and improved user interfaces. Prospective buyers can request a free demonstration disk from the NIST Standard Reference Data Program by calling (301) 975-2208. Desktop Spectrum Analyzer and X-Ray Database Version 2.5 is available for $815. An upgrade for the PowerMac is $100, and a 68 kilobyte upgrade is free. Order from the SRDP, Rm. 113, Building 820, Gaithersburg, MD 20899-0001, fax: (301) 926-0416, <srdata@nist.gov>.

NEW PRODUCT RESULTS FROM CENTER'S AID

Throughout the country, centers affiliated with NIST's Manufacturing Extension Partnership are helping America's smaller manufacturers improve their performance and competitive edge. For example, the Connecticut State Technology Extension Program CONN/STEP recently worked with a company, to develop a new gage, which is helping the company expand its production capabilities.

The majority of the company's business is precision machining of round parts for the aerospace and defense industries. But the company's gage for measuring these parts was time consuming and unreliable. With CONN/STEP's assistance, the company developed a new device that could quickly and accurately measure the dimensions of any cylindrical part. Not only did the gage speed up the company's manufacturing process, but the company plans on producing and selling it in the near future. The president says, "Without CONN/STEP's assistance we would not have been able to afford undertaking the massive step from R&D to production.... CONN/STEP helped us accelerate our new product introduction and we look forward to expanding our production capabilities and employment." Other MEP center/client collaborative achievements are chronicled online at <http://www.nist.mep.gov>. Click on "About the MEP," then "Success Stories." For print copies, call the MEP at (301) 975-5020.

COLLABORATION UNDER WAY FOR CONSTRUCTION MATERIALS

The NIST National Voluntary Laboratory Accreditation Program and the International Conference of Building Officials' Evaluation Service Inc. signed a memorandum of understanding (MOU) on Feb. 18, 1997, to work toward a mutual recognition agreement to recognize their respective accreditations of laboratories that test construction materials.

The MRA, which will be based on ISO/IEC Guides 25 and 58 and other appropriate inter-national standards, will help eliminate duplicate accreditations in common areas. Both the MOU and upcoming MRA are examples of the ongoing effort by NIST under the National Technology and Transfer Act to provide leadership in coordinating standards and conformity assessment activities with government bodies—as well as the private sector—to meet the needs of U.S. industry in the global marketplace.

ICBO provides its more than 2300 state and local government members in the south, southeast, southwest, and Atlantic seaboard regions of the United States with a recognized consensus standards program through the ICBO model building code. ICBO ES Inc. has more than 15 accredited laboratories to test products related to the construction industry, with primary emphasis on those products and systems used in the design and construction of buildings. NVLAP has approximately 20 labs accredited in construction materials testing. There are more than 700 domestic and foreign testing and calibration laboratories in the overall NIST/NVLAP program that are accredited in terms of meeting international standards. For more information, contact James L. Cigler, NVLAP, Room 282, Building 820, NIST, Gaithersburg, MD 20899-0001,(301) 975-4016, fax: (301) 926-2884, <nvlap@nist.gov>.

Standard Reference Materials

SRM IS GOLD TO STEEL INDUSTRY FOR 90 YEARS

As NIST scientists well know standard reference materials (SRMs) come and go. Many NIST SRMs change or are discontinued as industry needs change. One notable exception, Standard Reference Material 5m, is celebrating its 90th anniversary of continuous service to the U.S. steel industry.

Recently renewed for the 16th time, NIST's cast iron SRM has changed little since the agency's predecessor, the National Bureau of Standards, adopted it from the American Fundaments Association in 1906. With sales of 50 to 70 units annually, it remains a popular and reliable standard for steel manufacturers. Foundries use SRM 5m to verify the accuracy of their chemical analyses.

In cooperation with the American Society for Testing and Materials, NIST has certified the concentration of 12 elements, among which are carbon, manganese, phosphorous, silicon, and sulfur. Foundries and other analyzing elements in cast iron can purchase a 15 g bottle of SRM 5m for $215. For ordering information, contact the NIST Standard Reference Materials Program, Building 202, Room 204, Gaithersburg, MD 20899-0001. (301) 975-6776, fax: (301) 975-3730, e-mail: srminfo@enh.nist.gov.

Standard Reference Data

GET DATABASE ON STEAM PROPERTIES WHILE IT'S HOT

The NIST Standard Reference Data Program's Version 2.0 of the NIST/ASME properties of steam database is now available. The updated SRD is based on the most recent International Association for the Properties of Water and Steam formulation for general and scientific use of the thermodynamic properties of water.

Developed in conjunction with the American Society of Mechanical Engineers (ASME), the STEAM database is the U.S. implementation of an international standard. Some of the thermophysical properties covered include temperature, pressure, volume, quality, enthalpy, entropy, thermal conductivity, viscosity, dielectric constant, and surface tension.

The database generates tables and plots of property values at specified conditions corresponding to saturation conditions, a fixed property value (such as isotherm) or individual values of two independent ariables. Vapor-liquid, vapor-solid and liquid-solid saturation calculations are available with either temperature or pressure specified. The user can specify which properties to display and which units to use during entering and displaying of data. To order, contact the SRDP, NIST, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: (301) 926-0416, e-mail: srdata@nist.gov. The price is $50 for executable, or $190 for executable and source code (FORTRAN).

SRD UPGRADE OFFERS RAPID ACCESS TO LIGAND STABILITY DATA

Research chemists and chemical engineers can now access stability data for a wide variety of ligands through an upgrade of NIST Standard Reference Database 46, NIST Critically Selected Stability Constants of Metal Complexes.

Ligands, which are atoms or groups of atoms bound to a central atom, interact with hydrogen and other metal ions in water. These interactions are a concern for mining engineers, geologists, manufacturing engineers in controlling industrial processes such as electroplating and finishing, scientists studying the stability of materials in the environment, biologists studying membrane structures, and chemists developing fertilizers and pesticides.

Version 3.0 of the database contains data for more than 5300 ligands, rapid bibliography searching and streamlined commands. The database will run on personal computers with PC or MS-DOS 2.1 or greater and at least nine megabytes free hard disk space. NIST Database 46 is available for $240. Owners of previous versions may upgrade for $150. To order, contact the NIST Standard Reference Data Program, Building 820, Room 113, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: (301) 926-0416, <srdata@nist.gov>. The SRD Products Catalog can be found on-line at <http://www.nist.gov/srd>.

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Calendar

May 5–7, 1997

VACUUM GAUGE CALIBRATION WORKSHOP

Location: National Institute of Standards and Technology
Gaithersburg, MD

Sponsor: NIST.
Audience: U.S. vacuum community/calibration personnel.
Format: Lecture.
Purpose: To provide instruction and hands-on practice in the use and calibration of vacuum groups. Emphasis is on the use of the molecular drag (spinning rotor) gauge.
Topics: Vacuum systems, ion gauges and controllers, spinning rotor gauges, laboratory practice with spinning rotor gauge and review.
Technical Contact: Ana Salazar, NIST, Building 220, Room A55, Gaithersburg, MD 20899-0001, phone: 301/975-4840, fax: 301/208-6962, email: ana.salazar@nist.gov.

May 19–23, 1997

CONFERENCE ON RADIONUCLIDE METROLOGY AND ITS APPLICATIONS-ICRM '97

Location: National Institute of Standards and Technology
Gaithersburg, MD

Sponsors: International Committee for Radionuclide Metrology (ICRM) and NIST.
Audience: Radionuclide metrology community.
Format: Oral and poster presentations and business meetings of the ICRM working groups on radionuclide metrology and its applications.
Purpose: To provide an opportunity for the exchange of information on techniques and applications of radionuclide metrology and to encourage international cooperation in this field.
Topics: Alpha-particle spectrometry, gamma-ray and beta-particle spectrometry, life sciences, low-level measurements, radionuclide metrology techniques (including direct activity measurements), and nuclear decay data (measurements and evaluations).
Technical Contact: J.M.R. Hutchinson, NIST, Building 245, Room C114, Gaithersburg, MD 20899-0001, phone: 301/975-5543, fax: 301/926-7416, email: j.hutchinson@nist.gov, WWW Homepage: http://physics.nist.gov/Divisions/Div846/icrm.html.

June 8–13, 1997

1997 IEEE MTT-S INTERNATIONAL MICROWAVE SYMPOSIUM

Location: Colorado Convention Center
Denver, CO

Sponsors: Institute of Electrical and Electronics Engineers (IEEE) Microwave Theory and Techniques Society and NIST.
Audience: Microwave engineers.
Format: Symposium, workshops, and exhibitions.
Purpose: To present the latest developments in microwave technology.
Topics: Microwave components, power generation, amplification, monolithic technology, millimeter waves, field theory, computer-aided design, measurements, wireless communications, and radio-frequency (rf) technology.
Technical Contacts: Claude Weil, Chair, Electromagnetic Fields Division, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-5305, fax: 303/497-6665, email: weil@boulder.nist.gov. Roger Marks, Vice-Chair, Electromagnetic Fields Division, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-3037, fax: 303/497-7828, email: marks@boulder.nist.gov. Motohisa Kanda, Vice-Chair, Electromagnetic Fields Division, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-5320, fax: 303/497-6665, email: motohisa.kanda@nist.gov, WWW Homepage: http://www.boulder.nist.gov/ims/.

June 9–10, 1997

YEAR 2000 INTERNATIONAL SYMPOSIUM "MASTERING THE MILLENNIUM ROLLOVER"

Location: National Institute of Standards and Technology Gaithersburg, MD

Sponsors: NIST, Data Administration Management Association, National Capitol Region (DAMA NCR), Interagency Working Group on the Year 2000, U.S. Office of Management and Budget, U.S. Federal Aviation Administration, and U.S. Department of Defense.
Audience: International and national government, academia, and industry.
Format: Concurrent sessions, panels, speakers, and vendors.
Purpose: To provide as much information and discussion as possible on this subject in order to assist organizations and international trading partners in dealing with the issue.
Topics: Awareness, assessment, renovation, validation, implementation, and applications.
Technical Contact: Judith Newton, NIST, Building 820, Room 562, Gaithersburg, MD 20899-0001, phone: 301/975-3256, fax: 301/948-6213, email: judith.newton@nist.gov, WWW Homepage: http://speckle.ncsl.nist.gov/~fisher/y2kis0.htm.

June 16–20, 1997

12th ANNUAL CONFERENCE ON COMPUTER ASSURANCE (COMPASS '97)

Location: National Institute of Standards and Technology Gaithersburg, MD

Sponsors: NIST and the Institute of Electrical and Electronics Engineers (IEEE).
Audience: Industry, government, and academia.
Format: General sessions and tutorials.
Purpose: To bring together researchers, developers, and evaluators who work on problems related to specifying, building, and certifying high-assurance computer systems.
Topics: Systems integrity, software safety, and process security.
Technical Contact: Dolores Wallace, NIST, Building 820, Room 517, Gaithersburg, MD 20899-0001, phone: 301/975-3340, fax: 301/926-3696 email: dwallace@nist.gov, WWW Homepage: http://hissa.ncsl.nist.gov/compass/.

June 22-27, 1997

13th SYMPOSIUM ON THERMOPHYSICAL PROPERTIES

Location: University of Colorado at Boulder Boulder, CO

Sponsors: NIST and American Society of Mechanical Engineers.
Audience: International, industrial, governmental, and academic scientists, and engineers.
Format: Plenary and parallel sessions.
Purpose: Theoretical, experimental, and applied aspects of thermophysical properties of gases, liquids and solids will be covered by invited and contributed papers.
Topics: Equilibrium and non-equilibrium thermodynamics, supercritical fluids, refrigerants, aqueous systems, advanced materials, condensed matter at extreme conditions, computer simulations, thin films, polymers, wetting and interfaces with natural gas, complex fluids, process design, optical and acoustic techniques, low gravity, critical behavior, fluid structure and databases.
Technical Contact: William Haynes, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-3247, fax: 303/497-5044, email: haynes@boulder.nist.gov, WWW Homepage: http://www.boulder.nist.gov/div838/symp13.

June 23–24, 1997

TIME AND FREQUENCY SEMINAR-INTRODUCTION LEVEL I

Location: The Broker Inn Boulder, CO

Sponsor: NIST.
Audience: Beginning mathematicians, scientists, engineers, and planners of time and frequency.
Format: Lecture.
Purpose: A variety of modern electronic systems depend critically on precise timing or an ultra-stable frequency reference. The clocks and oscillators in such systems, therefore, must be characterized carefully. This seminar focuses on common methods of measuring and interpreting oscillator and clock performance and how these results affect overall system performance.
Topics: Fundamentals of time and timekeeping, definitions of terms, and measurement methods and analysis techniques in the time domain and frequency.
Technical Contact: David Howe, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-3277, fax: 303/497-6461, email: dhowe@boulder.nist.gov.

June 26–28, 1997

TIME AND FREQUENCY SEMINAR-LEVEL II

Location: The Broker Inn
Boulder, CO

Sponsor: NIST.
Audience: Scientists, engineers, and laboratory technicians involved in the time and frequency system.
Format: Lecture.
Purpose: To learn specialized measurement techniques for quantifying frequency stability and spectral purity of an oscillator. Typical commercial oscillators and atomic frequency standards will be described with explanations for their composite parts. An overview of methods of timekeeping and synchronization will follow.
Topics: Short-term stability, long-term stability, measurement techniques, noise statistics of oscillator signals, special purity measurement, performance and specifications of quartz oscillators, characterization of commercial frequency standards, synchronization in telecom systems, timekeeping and time scales, and GPS and other dissemination services.
Technical Contact: David Howe, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-3277, fax: 303/497-6461, email: dhowe@boulder.nist.gov.

July 14–18, 1997

4th INTERNATIONAL MEETING ON CHEMICAL KINETICS

Location: National Institute of
Standards and Technology
Gaithersburg, MD

Sponsors: NIST, Environmental Protection Agency (EPA), and the National Aeronautics and Space Administration (NASA).
Audience: Scientists.
Format: Lectures and posters.
Purpose: To bring together scientists from different disciplines who use chemical kinetics to explore areas of common interest.
Topics: Free radical thermodynamics, reactions of peroxyl radicals, halogen oxides, and others.
Technical contact: Robert Huie, NIST, Building 222, Room A261, Gaithersburg, MD 20899-0001, phone: 301/975-2559, fax: 301/975-3672, email: robert.huie@nist.gov, WWW Homepage: http://www.nist.gov/cstl/div838/kinet_conf/conference.html.

August 4-6, 1997

WORKSHOP ON ULTRASONIC AND DIELECTRIC CHARACTERIZATION TECHNIQUES FOR SUSPENDED PARTICULATES

Location: National Institute of
Standards and Technology
Gaithersburg, MD

Sponsors: NIST, American Ceramic Society, and American Chemical Society Colloid Division.
Audience: International scientists and engineers.
Format: Invited and submitted plenary lectures.
Purpose: To discuss most recent advances in theory and instrumentation and share experience in specific materials applications for techniques based on high-frequency electric and acoustic fields.
Topics: Electroacoustic spectroscopy, acoustic attenuation spectroscopy, dielectric spectroscopy, concentrated suspensions and emulsions, and powder processing.
Technical Contacts: Dr. Vince Hackley, NIST, Building 223, Room A256, Gaithersburg, MD 20899-0001, phone: 301/975-5790, fax: 301/990-8729, email: vince.hackley@nist.gov. Dr. John Texter, Eastman-Kodak Co., Rochester, NY 14650-2109, phone: 716/477-3019, fax: 716/722-5411, email: texter@kodak.com, WWW Homepage: http://www.ceramics.nist.gov/events/udct/udct.htm

August 11–13, 1997

2ND ANNUAL WIRELESS COMMUNICATIONS CONFERENCE

Location: Regal Harvest House
Boulder, CO

Sponsors: NIST, International Microelectronics and Packaging Society, IEEE Communications Society-Pike's Peak Chapter, IEEE Microwave Theory and Techniques Society, and Institute for Telecommunications Science.
Audience: Commercial wireless industry.
Format: Single track, technical presentations, workshop, panel discussion session, and exhibition.
Purpose: The success of last year's conference demonstrated the appeal of a symposium focused on technological advances in the commercial wireless industry.
Topics: Systems: PCS, cellular, paging, wireless LANs, LMDS, etc.; active components; passive components; packaging; antennas, propagation, compatibility, and interference; and measurements.

Technical Contact: Chairman Roger Marks, NIST, 325 Broadway, Boulder, CO 80303-3328, phone: 303/497-3037, fax: 303/497-7828, email: r.b.marks@ieee.org. Technical Program Chair: Michael S. Heutmaker, Lucent Technologies, P.O. Box 900, RM2-2063, Princeton, NJ 08542-0900, phone: 609/639-3116, fax: 609/639-3197, email: msh@mach.lucent.com. WWW Homepage: http://www.boulder.nist.gov/wcc.

August 11-15, 1997

2ND INTERNATIONAL CONFERENCE ON FIRE RESEARCH AND ENGINEERING

Location: National Institute of Standards and Technology
Gaithersburg, MD

Sponsors: NIST and Society of Fire Protection Engineers.
Audience: Grantees, other agencies, private industry, fire protection engineers, and consultants in the fire research area.
Format: Technical presentations, workshops, and case studies.
Purpose: To bring together leaders in the fire research and fire safety engineering communities from around the world.
Topic: Fire safety applications.
Technical Contact: Nora Jason, NIST, Building 224, Room A252, Gaithersburg, MD 20899-0001, phone: 301/975-6862, fax: 301/975-4052, email: nora.jason@nist.gov.

August 18–22, 1997

INTERNATIONAL CONFERENCE ON FUNDAMENTALS OF FRACTURE

Location: National Institute of Standards and Technology
Gaithersburg, MD

Sponsors: NIST and Institute for Mechanics and Materials (IMM).
Audience: Scientists.
Format: Lecture.
Purpose: The goals of the conference will be to provide a vehicle for the presentation and discussion of current research and future challenges in fracture science. The focus is on the fundamental aspects of fracture in the disciplines of mechanics, metallurgy, ceramics, polymer science, physics and chemistry.
Topics: Fracture in materials.
Technical Contact: Robb Thomson, NIST, Building 223, Room A124, Gaithersburg, MD 20899-0001, phone: 301/975-5665, fax: 301/926-8349, email: robb.thomson@nist.gov, WWW Homepage: http://www.ctcms.nist.gov/~robb/icff.html.

Tutorials-September 22, 1997
Conference-September 23-25, 1997

INTELLIGENT SYSTEMS AND APPLIED SEMIOTICS ’97 A LEARNING PERSPECTIVE (ISAS ’97)

Location: National Institute of Standards and Technology
Gaithersburg, MD

Sponsors: Institute of Electrical and Electronics Engineering, National Science Foundation, Army Research Office, and NIST.
Audience: Leading researchers in the area of intelligent systems, including design and application.
Format: The conference will be organized in three parallel tracks including nine workshops, preceded by a day of relevant tutorials.
Purpose: This meeting is dedicated to applied semiotics and its application in large and complex systems, including intelligent machines. ISAS ’97 will focus on learning processes.
Topics: Learning in large symbolic structures, systems of symbol and knowledge representation emerging from learning processes, and systems and machines designed to acquire knowledge and act intelligently.
Technical Contact: Richard Quintero, NIST, Building 220, Room B124, Gaithersburg, MD 20899-0001, phone: 301/975-3445, fax: 301/998-9688, email: richard.quintero@nist.gov, WWW Homepage: http://isd.cme.nist.gov/proj/semiotics97.

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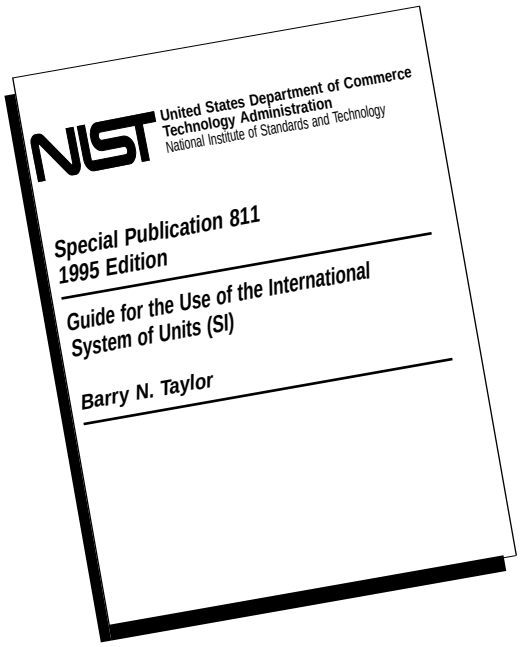
The International System of Units (SI)
The Modern Metric System



Uncertain about the International System of Units (universally abbreviated SI), the modern metric system used throughout the world? Do you need to know the proper way to express the results of measurements and the values of quantities in units of the SI? Do you need to know the NIST policy on the use of the SI? Then you need the 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*.

The 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*, by Barry N. Taylor, is now available.

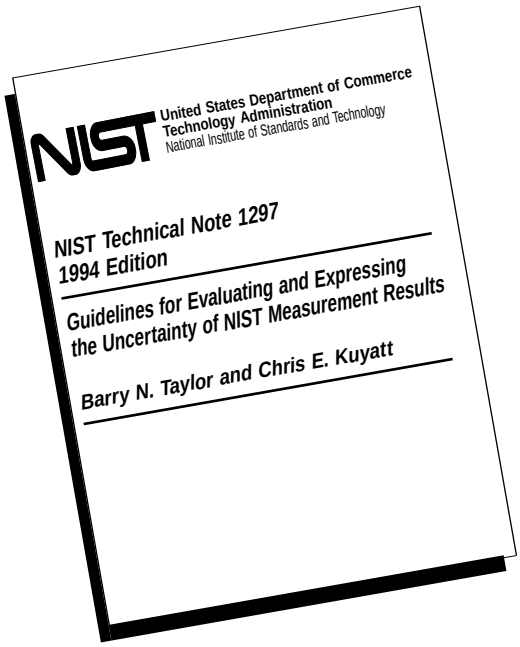
The 1995 edition of SP 811 corrects a number of misprints in the 1991 edition, incorporates a significant amount of additional material intended to answer frequently asked questions concerning the SI and SI usage, and updates the bibliography. The added material includes a check list for reviewing the consistency of written documents with the SI. Some changes in format have also been made in an attempt to improve the ease of use of SP 811.

The topics covered by SP 811 include:

- NIST policy on the use of the SI in NIST publications.
- Classes of SI units, those SI derived units that have special names and symbols, and the SI prefixes that are used to form decimal multiples and submultiples of SI units.
- Those units outside the SI that may be used with the SI and those that may not.
- Rules and style conventions for printing and using quantity symbols, unit symbols, and prefix symbols, and for spelling unit names.
- Rules and style conventions for expressing the results of measurements and the values of quantities.
- Definitions of the SI base units.
- Conversion factors for converting values of quantities expressed in units that are mainly unacceptable for use with the SI to values expressed mainly in units of the SI.
- Rounding numbers and rounding converted numerical values of quantities.

Single copies of the 84-page SP 811 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.

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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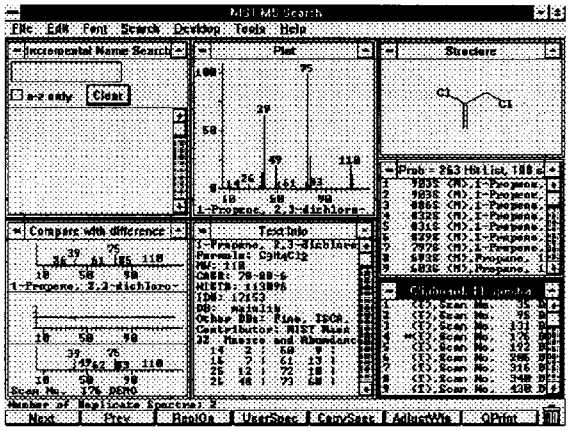

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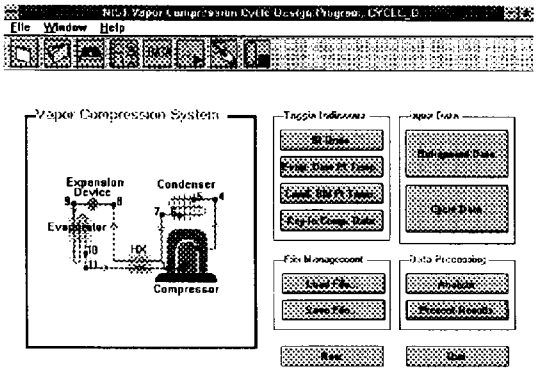

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