CHAPTER SIX

METROLOGY MAKES ROOM FOR INDUSTRIAL PRODUCTIVITY (February 1990–April 1993).

With the change in name reinforcing—on a continual, daily basis—a change in the way that the former National Bureau of Standards was perceived both within and outside the venerable institution, it fell to new leadership to define the myriad of details by which traditional metrology would co-exist with expanded efforts to boost U.S. industrial productivity.

The new legislation made clear that NIST should maintain its historical role as the U.S. authority on standards of measurement. Yet the same legislation called explicitly for emphasis on programs meant for the rescue of American industry in the international economic arena. Managing the balance between these sometimes conflicting goals fell to John Lyons and his senior management staff.

By the time Lyons completed his 3-year term as director, the new orthodoxy of industrial productivity had replaced the standards-and-science orientation of the old National Bureau of Standards. Only time would tell whether the dual roles could co-exist in the same unique institution.

GEORGE HERBERT WALKER BUSH

George Bush was a man of many accomplishments and a Washington insider since 1966. The son of Senator Prescott Bush of Connecticut, George was a decorated Navy pilot in World War II and a graduate of Yale University in 1948. He was an oil-company executive for several years prior to his election to the U.S. House of Representatives in 1966 and 1968, representing Houston, Texas. He was appointed by President Nixon to be Ambassador to the United Nations in 1971 and served as Chairman of the Republican National Committee during Watergate. President Ford posted him as chief of the U.S. Liaison Office in Peking in 1974, and as Director of the Central Intelligence Agency in 1976.

In 1980, Ronald Reagan chose Bush as his Vice-Presidential running mate. Their Republican ticket handily beat the Democratic team of President Jimmy Carter and Vice President Walter F. Mondale, winning 489 of the 537 available electoral votes. The Reagan-Bush ticket won again in 1984, defeating Walter Mondale and Geraldine Ferraro by an even larger margin.

Bush was effective as Reagan's Vice-President, leading the administration's battles on regulatory reform and an anti-drug campaign, assisting the President with crisis management, and serving well as Reagan's surrogate when the President was shot by John Hinckley.

Running for President on his own in 1988, Bush chose Danforth Quayle as his candidate for Vice-President. They faced Michael S. Dukakis of Massachusetts and Lloyd Bentsen of Texas in a campaign nearly devoid of issues save those of personality. The voter turnout on election day fell to 50 % of eligible voters—the lowest in more than 60 years. However, the Bush-Quayle ticket still won easily.

One of Bush's early tasks was to nominate a permanent director of the National Institute of Standards and Technology. This he did, on November 17, 1989; he nominated John W. Lyons, a career scientist and at that time the director of the NIST National Engineering Laboratory.[1]

During his term as America's 41st president, Bush saw the end of the Evil Empire—the Berlin Wall was breached in 1989, and the Union of Soviet Socialist Republics collapsed in 1991 into a loose federation of tentative democracies. The Reagan-Bush policy of militant anti-communism contributed heavily to the termination of the Cold War; Bush gladly undertook the construction of a suitable U.S. stance with respect to post-Cold-War Eastern Europe.

Bush directed the U.S. participation in the Persian Gulf War in early 1991 that successfully liberated Kuwait, overrun by the Iraq military during 1990. Although the U.N. forces stopped short of total victory, the United States, and thus its President, stood tall as the defender of militarily weak nations.

Although he basked in the glory of America's armed might, Bush was required to face the punishing trade and budget deficits that he inherited from his predecessor. He found it increasingly tricky to reduce the deficits while struggling with military cutbacks mandated by the end of the Cold War. His efforts brought on a recession and rising unemployment that persisted throughout his term of office. Bush also presided over the resolution of a financial scandal arising from the insolvency of a number of profligate savings and loan institutions; rescue of the defunct businesses cost the U.S. treasury more than $100 billion.

President Bush chose Robert A. Mosbacher, Sr., a flamboyant Texas oilman, to be Secretary of Commerce in 1988. In 1992, while Bush was campaigning for re-election, he replaced Mosbacher with Barbara H. Franklin, former Commissioner of the Consumer Product Safety Commission (1972-78) and more recently (1979-88) Senior Fellow of the Wharton School of Business at the University of Pennsylvania.

William J. Clinton, Governor of Arkansas, successfully gained the presidential nomination of the Democratic party in 1992. With Albert Gore, Jr. as his running mate, Clinton attacked the Bush-Quayle economic policies vigorously. H. Ross Perot, an independent candidate for President, added spice to an already heated campaign. In an election that attracted the highest voter participation since 1968, the Clinton-Gore team defeated both the Bush-Quayle slate and the Perot-Stockdale ticket.

Clinton's election, preventing a second term for George Bush, would also spell the end of John Lyons' tenure as NIST director.

[1] "Weekly Compilation of Presidential Documents," 25, p. 1774, 1989.

John Winship Lyons

If there was one NIST manager who was at ease with the change of name and modification of purpose as stated in the 1988 revision of the NBS Organic Act, that manager was John Lyons, nominated by President George Bush on November 17, 1989, to be the ninth Director of the agency newly re-named the National Institute of Standards and Technology. Lyons was employed for 20 years by the Monsanto Corporation before joining the staff of NBS in 1973. There he had seen re-organizations, technical re-alignments, and changes in corporate goals on many occasions. He expressed his views on the changes wrought by Public Law 100-418 plainly:

> The Bureau is whatever the Congress says it is. Congress is the Board of Directors and what they say, by definition that is what you are.[2]

Lyons was raised in Boston and graduated from Harvard College with a A.B. degree in chemistry. After 2 years of service in the U.S. Army, he joined the Monsanto Corporation as a chemist. During his service with Monsanto, he obtained M.A. and Ph.D. degrees in physical chemistry from Washington University in St. Louis. While at Monsanto, Lyons investigated the chemistry of phosphorus compounds, the behavior of polyelectrolytes in solution, and rheology.

Participation on an evaluation panel for the NBS fire program led to his joining NBS in October 1973 to head the program.[3] During that year, a report entitled *America Burning,* issued by the National Commission on Fire Prevention, awakened the country to America's deplorable fire-safety record. The United States suffered the highest death rate from fire among all industrial nations, and the worst property-loss rate, too. Spurred by the impact of the report, the 93rd Congress enacted the *Federal Fire Prevention and Control Act of 1974*; it mandated, among other things, the creation of a Center for Fire Research at NBS. Lyons became the first director of the new center.[4]

During the NBS reorganization of 1977-78, Director Ernest Ambler selected Lyons to lead the planning of a new entity, a National Engineering Laboratory, and then to become its first director. The NEL included centers for applied mathematics, electronics and electrical engineering, mechanical engineering and process technology, building technology, fire research, and consumer products. Lyons spent more than a decade working with Ambler and the NEL managers to complete an organization—eventually including a separate chemical engineering unit—that could interact with American industry in all the engineering areas.[5] His leadership in the NBS fire program and in the creation and management of NEL earned him election in the National Academy of Engineering. Ultimately, Lyons was entirely at ease with the idea that NBS could provide direct assistance to bolster U.S. industry in its efforts to adopt the most modern methods of manufacturing.

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[2] John Lyons, Oral History, June 1, 1993.

[3] NBS Admin. Bull. 73-70, October 29, 1973.

[4] See Chapt. 3, The Bureau Gets a New Fire Law and a New Fire Center.

[5] Lyons, Oral History, June 1, 1993.

John W. Lyons was the ninth director of NBS/NIST.

Aware in 1988 that Ernest Ambler intended to retire within months, Lyons hoped to succeed him. A decade of management experience in the engineering/technology area of NBS provided Lyons with a background that matched well the emphasis on technology transfer sought by congressional authors of the 1988 Trade Act.[6]

As noted above, President Bush nominated Lyons for director on November 17, 1989. During his confirmation hearing in February 1990, he was asked about his view of the future for NIST. His response was optimistic:

> In some critical aspects the Institute will be unchanged in the years ahead. We shall still have a substantial core of fundamental research in science and engineering and thereby serve both our internal interests and those of the technical community at large. We shall continue our dedication to excellence in all of our activities. We shall continue to work on the basic physical and chemical standards of measurement on which our National quality assurance systems are based and provide the necessary services to all those seeking to base their work on ours. The Institute will keep up its work in supporting technologies. We shall continue to serve as a crossroads for the technical community, conducting hundreds of conferences, hosting thousands of visitors, and participating in a myriad of external committees and activities. The Institute will remain an open laboratory.

[6] John Lyons, Oral History, June 9, 1993.

The U.S. Senate confirmed the nomination on February 8, 1990.[7] The next day, John Lyons took office as the ninth Director of NBS/NIST.[8]

During his swearing in ceremony, held March 20, 1990, Lyons gave the following view of NIST:

> I am the ninth Director of this great laboratory, the second chemist, and perhaps the first technologist. I am only the second director to have had extensive work experience in industry. Our first priority here at the National Institute of Standards and Technology is to provide technical support to industry. This priority is written down in law. It is also in direct support of Secretary Mosbacher's top priority for the Department of Commerce: namely, to support industry's efforts to improve competitiveness in the global marketplace.
>
> I have enunciated two other top priorities: to support selected programs in public health and safety and to support the scientific and engineering research communities through a program of fundamental research.
>
> There are three related aspects of NIST that are critical to achieving success with the three priorities I have just mentioned. These are excellence, strength of our research base, and relating our rate of growth to industrial demand. Excellence—our reputation for getting it right and getting it right the first time—is our stock in trade. We must not sacrifice this reputation for any objective. Our research base is the foundation on which we build our applied programs and to which we recruit our next generation of staff. It is essential that this research base be maintained at substantial fraction of the total effort and that it do world-class work.
>
> And finally, we must find a way to relate the rate of growth of NIST to that of the industries we serve. These industries are based on advanced technologies that are ever more complex. We find we have to provide more and more services to them. Finding the resources to respond to these needs is a challenge we must somehow meet.

Taking Charge

John Lyons had the same kind of head start on the task of directing the work of NIST that all previous directors save two had enjoyed—he had spent more than a decade as an NBS employee. In addition, his participation in the planning of the 1988 Trade Act gave him a deep understanding of the intentions of Congress for the newly re-created agency.

In testimony during the 1991 Congressional appropriations hearings, Lyons reviewed the agency's progress since NBS became NIST:

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[7] "Lyons New Head of NIST," NIST Research Reports, *NIST Special Publication 783*, May 1990, pp. 5-8.

[8] John W. Lyons, "Welcome and Current View," in *NBS/NIST, A Historical Perspective: A Symposium in Celebration of NIST's Ninetieth Anniversary, March 4, 1991*, Karma A. Beal, Editor, *NIST Special Publication 825*, April 1992, p. 2.

The Omnibus Trade and Competitiveness Act of 1988 established new programs and approaches on a successful foundation at the old National Bureau of Standards. The Act confirms the importance of the existing NBS functions and reaffirms the primary NBS mission to promote the competitiveness of U.S. industry.

Today NIST is a far different laboratory. Over a thousand guest scientists and 88 cooperative research and development agreements with industry and academia (plus 33 in the process of completion) have become part of our lives. We are in the closing stages of our first competition under the Advanced Technology Program. We have three Regional Centers for the Transfer of Manufacturing Technology in the Manufacturing Extension Partnership Program, and we are in the process of selecting two more.

Restructuring NIST

Lyons began to adapt the management of NIST to his vision for the agency as soon as he was nominated as Director.[9] He formed task groups for the purpose of replacing the National Measurement Laboratory and National Engineering Laboratory hierarchies with a management structure that could better focus on newly assigned goals. Despite his early start, however, the new structure did not gain final approval from its many overseers—the Department of Commerce, the Office of Management and Budget, and four congressional committees—until February 1991. In the meantime, many managers were "designated to serve" in their newly assigned positions.

An interim organizational chart issued in July 1990 and a more complete one issued in March 1991 (the latter is shown in part in Appendix K) defined the new structure of NIST. Raymond Kammer continued as Deputy Director until his departure late in 1991 to accept a position as Deputy Under Secretary of Commerce for Oceans and Atmosphere—the chief operating officer for the National Oceanic and Atmospheric Administration. Lyons appointed Samuel Kramer, his long-time planning assistant in the National Engineering Laboratory, to the post of Associate Director of NIST; upon the departure of Deputy Director Kammer, Lyons designated Kramer as Kammer's replacement. Apart from Kramer, the new structure had no Associate Directors.

In accordance with the wishes of Congress, Lyons placed two of NIST's newest entities—the Office of Quality Programs, headed by Curt Reimann, and an Advanced Technology Program (APT), headed initially by Donald R. Johnson, directly under his own supervision. Within a few months Lyons appointed George A. Uriano, a physicist with more than 26 years of service at NBS, to be permanent head of the ATP. It fell to Uriano to manage the large transferred-funds program to develop—on a cost-sharing basis—a variety of high-risk, innovative technologies to enhance the economic growth of American industry.

Within the ATP was created an important unit that contained most of its in-house competence—the directorate of Technology Services, led by Donald R. Johnson. It contained an Office of Standards Services, under Stanley I. Warshaw; an Office of

[9] John Lyons, Oral History, June 9, 1993.

Technology Commercialization, headed by Cary Gravatt; an Office of Measurement Services, under Stanley D. Rasberry; an Office of Technology Evaluation, under George P. Lewett; and an Office of Information Services, headed by Patricia W. Berger.

A directorate of Administration, led by Guy W. Chamberlain, Jr., included a Management and Organization Division, headed by Sharon E. Bisco; an Office of the Comptroller, under John C. McGuffin; a Public Affairs Division, led by Matthew Heyman; a Plant Division, under Jorge R. Urrutia; a Facilities Services Division, headed by Walter J. Rabbitt; an Occupational Health and Safety Division, under Lyman E. Pevey; an Acquisition and Assistance Division, led by Richard E. de la Menardiere; a Boulder Executive Office, under Paige L. Gilbert; and a Boulder Technical Services Division, headed by Henry W. Tyler.

A group of 8 laboratories completed the traditional portions of NBS:

- An Electronics and Electrical Engineering Laboratory, directed by Judson C. French, included an Electricity Division, headed by Oskars Petersons, that was responsible for the standards of electricity; a Semiconductor Electronics Division, under Frank F. Oettinger; a Boulder Electromagnetic Fields Divsion, led by Ramon C. Baird; and an Electromagnetic Technology Division, under Robert A. Kamper, who also functioned as Director of the Boulder Laboratories, reporting directly to Lyons.

- A Manufacturing Engineering Laboratory, directed by John A. Simpson, included a Precision Engineering Division, under Dennis A. Swyt; an Automated Production Technology Division, led by Donald S. Blomquist; a Robot Systems Division, under James S. Albus; a Factory Automation Systems Division, led by Howard M. Bloom; and a Fabrication Technology Division, under Adrian W. Moll.

- A Chemical Science and Technology Laboratory, directed by Harry S. Hertz, included a Biotechnology Division, under Lura J. Powell; a Boulder Chemical Engineering Division, under Larry L. Sparks; a Chemical Kinetics and Thermodynamics Division, under Sharon Lias; an Inorganic Analytical Research Division, led by James R. DeVoe; an Organic Analytical Research Division, under Willie E. May; a Process Measurements Division, headed by Hratch G. Semerjian; a Surface and Microanalysis Science Division, headed by Rance A. Velapoldi; and a Thermophysics Division, led by Richard F. Kayser.

Included within the Biotechnology Division of the Chemical Science and Technology Laboratory was a relatively new unit called Advanced Research in Biotechnology. Like other new technology outreach programs, it was a joint venture for academic, government, and industrial scientists. The program sprang from a desire, shortly after the 1978 Bureau reorganization, to create an effective role for NBS in the biological sciences. Director Ernest Ambler, John Hoffman, and Donald R. Johnson were involved in the effort, although the bulk of the organizational work was accomplished by Johnson.

As a result of visits to industrial biotechnology laboratories, Johnson became convinced that an initiative in biotechnology could be coupled with a long-desired NBS goal to create university classrooms near the NBS Gaithersburg campus.

Drawing on experience gained as a Gaithersburg city planner and a member of the Montgomery County High-Technology Council, Johnson collaborated with Rita Colwell of the University of Maryland—who envisioned the new NBS entity as part of a University of Maryland Biotechnology Institute—to convince NBS management, the state of Maryland, the University of Maryland, a philanthropic trust, and Montgomery County to jointly found a Center for Advanced Research in Biotechnology, eventually housed in its own classroom building.[10] Johnson became its first director.

- A Physics Laboratory, directed by Katherine B. Gebbie. It included an Electron and Optical Physics Division, led by Charles W. Clark; an Atomic Physics Division, under Wolfgang L. Wiese; a Molecular Physics Division, headed by Alphonse Weber; a Radiometric Physics Division, led by Klaus D. Mielenz; a Quantum Metrology Division, headed by Richard D. Deslattes; an Ionizing Radiation Division, led by Randall S. Caswell; a Boulder Time and Frequency Division, under Donald B. Sullivan; a Boulder Quantum Physics Division, under David W. Norcross; and a Radiation Source and Instrumentation Division, under P. H. Debenham.
- A Materials Science and Engineering Laboratory, directed by Lyle H. Schwartz, included an Office of Nondestructive Evaluation, headed by H. Thomas Yolken; a Ceramics Division, under Stephen M. Hsu; a Boulder Materials Reliability Division, led by H. I. McHenry; a Polymers Division, headed by Leslie E. Smith; a Metallurgy Division, under E. Neville Pugh; and a Reactor Radiation Division, headed by J. Michael Rowe.
- The Building and Fire Research Laboratories were combined, despite apparent instructions to the contrary in PL 100-418. Richard N. Wright, veteran in the building-technology area, was named Director, and Jack E. Snell, former director of the Center for Fire Research, was made Deputy Director. Included in the new entity were a Structures Division, under Hai S. Lew; a Building Materials Division, led by Geoffrey J. Frohnsdorff; a Building Environment Division, led by James E. Hill; a Fire Science and Engineering Division, under Andrew Fowell; and a Fire Measurement and Research Division, under Richard G. Gann.
- Two computer-based laboratories completed the NIST structure in March 1991. One, a Computer Systems Laboratory, directed by James H. Burrows, included an Information Systems Engineering Division, under David K. Jefferson; a Systems and Software Technology Division, led by Allen L. Hankinson; a Computer Security Division, under Stuart W. Katske; a Systems and Network Architecture Division, headed by Kevin L. Mills; and an Advanced Systems Division, led by Shukri A. Wakid.

[10] Donald R. Johnson, Oral History, September 8, 1998.

A second computer laboratory, called the Computing and Applied Mathematics Laboratory, directed by Francis E. Sullivan, included an Applied and Computational Mathematics Division, led by Paul T. Boggs; a Statistical Engineering Division, under Robert J. Lundegard; a Scientific Computing Environments Division, headed by Sally E. Howe; A Computer Services Division, headed by Martin R. Shaver; a Computer Systems and Communications Division, under Stephen White; and an Information Systems Division, headed by Patsy B. Saunders.

The new structure, with some 18 office and laboratory directors reporting directly to Lyons, would have been thought unwieldy in many organizations. Nevertheless, it remained in effect during the balance of Lyons' tenure as Director. Lyons recalled later that the communications problem was eased considerably by the ubiquity of electronic mail.

Staffing Changes by Lyons

During his tenure as Director, Lyons, as might be expected, made many changes in staffing. Those assigned at the division level or higher, along with the reference in the NBS Administrative Bulletin (NBS Admin. Bull.) series, follow:

- Ellen M. Dowd, Personnel Officer: Nominated, NBS Admin. Bull. 91-5, April 4, 1991; Confirmed, NBS Admin. Bull. 91-10, July 10, 1991.
- Blaine R. Bateman, Chief, Boulder Chemical Engineering Division: NBS Admin. Bull. 91-11, August 9, 1991.
- Albert C. Parr, Chief, Radiometric Physics Division: NBS Admin. Bull. 91-11, August 9, 1991.
- Radiation Source and Instrumentation Division abolished: NBS Admin. Bull. 91-12, September 30, 1991.
- Allen C. Newell, Chief, Boulder Electromagnetic Fields Division: NBS Admin. Bull. 91-15, October 6, 1991.
- Stephen Freiman, Acting Chief, Ceramics Division: Nominated, NBS Admin. Bull. 91-17, October 6, 1991; Confirmed, NBS Admin. Bull. 93-2, November 1, 1992.
- Bruno Fanconi, Acting Chief, Polymers Division: NBS Admin. Bull. 91-17, November 3, 1991.
- Samuel Kramer, Deputy Director, NIST: NBS Admin. Bull. 92-1, December 29, 1991.
- Harry Hertz, Deputy Director, Office of Quality Programs: NBS Admin. Bull. 92-5, April 5, 1992.
- Hratch G. Semerjian, Director, Chemical Science and Technology Laboratory: NBS Admin. Bull. 92-5, April 5, 1992.

- Marvin A. Bond, Acting Director, Office of Information Services: NBS Admin. Bull. 92-12, October 4, 1992.
- Metric Program, reassigned from the Department of Commerce Technology Administration to NIST Technology Services: NBS Admin. Bull. 92-15, November 13, 1992.
- Paul Vassallo, Director, Office of Information Services: NBS Admin. Bull. 93-2, January 10, 1993.
- Gregory J. Rosasco, Chief, Process Measurement Division: NBS Admin. Bull. 93-2, October 4, 1992.
- Richard D. Suenram, Acting Chief, Molecular Physics Division: NBS Admin. Bull. 93-2, October 4, 1992.
- Joan R. Rosenblatt, Director, Computing and Applied Mathematics Laboratory: Nominated, NBS Admin. Bull. 93-3, February 14, 1993; Confirmed, 93-10, May 2, 1993.
- Chemical Engineering Division abolished: NBS Admin. Bull. 93-9, November 27, 1992.

Strategic Planning

During 1991, John Lyons and his senior managers prepared a 10-year strategic plan for NIST.[11] The plan was based upon three assumptions: that industrial competitiveness would continue to be a top priority for the Department of Commerce; that Commerce would continue as the focus of programs in that area; and that Commerce would support the mission with lively programs to improve industrial technology.

The NIST vision included the following items:

- Doubling of the NIST laboratory budgets.
- Even faster growth for the Advanced Technology Program.
- Modernization of the NIST laboratory facilities.
- Expansion of the Manufacturing Technology Centers program to span state, local Federal, and private-sector technology efforts.
- Tighter coupling between the internal NIST programs in science and engineering and the outreach programs.
- Expansion of the Quality Program to include non-profit organizations in health care and education.
- Addition to U.S. embassies abroad of "Technology Attachés," whose duties would include gathering information on foreign technical projects, as well as the promotion of U.S. technology-based products.

[11] "NIST in the 1990s: The Strategic Outlook," February 1991.

A second management tool prepared by NIST in 1991 was a Research Relationship Handbook, intended to guide NIST staff and potential industrial partners in selecting and establishing collaborative projects. Included in the handbook were:

- Types of organizations to be involved—all U.S. entities.
- Types of activities covered—Cooperative Research and Development Agreements, Proprietary Measurement Agreements, licenses, non-disclosure agreements, guest researchers, and gifts and loans of equipment.
- A "decision tree" for use in selecting appropriate interaction mechanisms.

Personnel Policy

The NBS Authorization Act for Fiscal 1987 designated NBS as the site of a Personnel Management Demonstration Project. Based on a similar project in one of the U.S. Navy research laboratories, the plan was intended to accomplish several goals, partly for NBS and partly for broader use within the Federal government:

- Simplified position classification based on knowledge, skill, and duties.
- Separate career paths—Scientific and Engineering Professional, Scientific and Engineering Technician, Administrative, and Support.
- "Pay banding" into five broad salary ranges within each career path.
- "Pay for Performance," which tied salaries to performance appraisals.
- Direct hiring authority for new employees.

The new personnel policies developed within the Demonstration Project improved the ability of NBS/NIST to attract and retain well-qualified staff in all personnel areas. The length of the hiring process was reduced considerably—by a month or more—improving the NIST competitive edge in the competition for highly qualified candidates.

The flexibility gained by enabling line managers to recommend incentive bonuses and rapid salary increases for deserving employees offered a powerful motivational tool. At the same time, the program reduced the manager's paperwork burden.

The 1990 Report of the Visiting Committee on Advanced Technology

Public Law 100-418 required the Visiting Committee on Advanced Technology (VCAT)—larger and with more responsibility than its predecessor—to meet quarterly rather than annually. This more intense schedule produced a more tightly focused agenda and considerable detail in the VCAT reports.

New VCAT Members

By the time that the VCAT had prepared its 1990 annual report, a new chairman—Arden L. Bement, Vice President for Science and Technology, TRW Corporation—had succeeded William D. Manly. Other new members of the committee were Edward C. Heffron of the Michigan Department of Agriculture; Richard S. Nicholson, Executive

Officer of the American Association for the Advancement of Science; Nam P. Suh, professor of Mechanical Engineering at MIT; and Albert R. C. Westwood, Vice President for Research and Technology at Martin Marietta Corporation. These men succeeded Nolen M. Ellison, president of Cuyahoga Community College; John P. McTague, Vice President for Research at Ford Motors Corporation; and William J. Spencer, Vice President for Research at Xerox Corporation.

NIST Organization

The VCAT expressed admiration for the way in which Kammer and Lyons had revamped the NBS organization to create a NIST that could fulfill the goals mandated by PL 100-418. The committee urged the Department of Commerce and the Congress to note the early success of NIST in raising the level of technology in the U.S. industrial sector, and to solidify the gains by helping NIST to evolve into something like the industrial equivalent of the National Institutes of Health in medicine and the Defense Advanced Research Projects Agency in the military.

Manufacturing Technology Centers

The VCAT visited the three Manufacturing Technology Centers during June 1990 as part of a detailed inquiry into the suitability of the program to assist small- and medium-sized companies in the assimilation of new technology. The committee was pleased with early results; center clients reported technical gains worth a cumulative $75 million during the first 18 months. The committee recommended that the program be expanded to include as many as 12 centers at a time.

Advanced Technology Program

The VCAT also examined the joint-venture technology development and commercialization program initiated as part of the Advanced Technology Program. The program made use of two significant definitions:

- Precompetitive research: research and development up to the point where commercial potential can be evaluated.
- Generic technology: concepts, components, or processes with the potential for generating a broad range of products or manufacturing methods.

The committee recommended several steps for program implementation:

- Include the broadest possible range of American industry in the selection of candidate technologies.
- Refine the technology selection process to include a "discovery" phase to weed out inappropriate ideas generated by the Request for Proposals (some 249 proposals had already been received) and a second, more comprehensive, proposal phase focused on those technologies.

- Refine the program management to develop better interaction between the NIST program and industry, focusing on three to five areas at first, but preparing to expand the program as it succeeded.
- Carefully protect proprietary aspects of companies involved, paying particular attention to intellectual property rights and antitrust liability.

Baldrige Quality Award

The VCAT noted that the Baldrige Quality Award program was drowning in its own success. More than 100,000 copies of the applications guidelines were distributed during 1990 alone. Many firms used the guidelines as self-evaluation tools without regard to potential submission of applications for the prize. Over the 3-year life of the program, it had become extremely successful and, said the committee, needed Federal funds to augment the private monies that thus far had provided its support. Such an increase would permit the addition of staff to process the growing numbers of submissions and requests for information.

NIST Budget

The VCAT noted a distinct improvement in the NIST budget with the Fiscal 1991 appropriation. Funding for laboratory initiatives was paralleled by growth in the allocations for the ATP and the MTC programs.

Cautionary alarms were sounded, however, regarding the state of the NIST physical plant, by then approaching 30 years of age. Critical areas noted were laboratory-space environmental controls (temperature, humidity, vibration, and air filtering), safety systems, and utilities.

NIST Receives an Examination

During Fiscal 1992, the National Research Council Board of Assessment took a long look at the progress made by NIST following passage of the Omnibus Trade and Competitiveness Act of 1988. The Board members liked what they saw, although they made numerous suggestions.

The objectives Congressionally mandated for NIST in 1988 became three in number; to enhance the technical competitiveness of U.S. industry, to maintain the traditional measurement programs, and to solve technical problems of national importance. The Board found that NIST had made excellent progress in meeting its new challenges: "The Board considers conditions and trends at NIST to be better now than at any time in recent history."

In assessing the situation at NIST, the Board used more than 150 technical assessors who visited the 10 NIST operating units. These operating units and their financial support levels from Congress (Approp) and from other Government agenices (OA) are shown in the following table:

NIST Unit	FY91 $M Approp	OA	Total
Electronics and Electrical Engineering Laboratory	20.0	19.3	39.3
Manufacturing Engineering Laboratory	10.0	17.3	27.3
Chemical Science and Technology Laboratory	24.6	19.2	43.8
Physics Laboratory	23.6	13.0	36.6
Materials Science and Engineering Laboratory	32.4	17.2	49.6
Building and Fire Research Laboratory	10.7	10.2	20.9
Computer Systems Laboratory	12.3	14.4	26.7
Computing and Applied Mathematics Laboratory	14.6	5.7	20.3
Technology Services	19.6	13.5	33.1
Director of Administration	----	----	----
Total Support	167.8	129.8	297.6

At the heart of NIST's response to its new challenges was a 10-year master plan, "NIST in the 1990s, the Strategic Outlook," issued by Director John Lyons on February 6, 1991 (see previous section). The plan assumed that the level of U.S. industrial competitiveness would continue to dominate Commerce Department and Congressional thinking over the coming decade, and that Commerce would develop its own program to complement that of NIST. Another assumption was that Congressional funding would reflect its commitment to NIST's goal.

Based on these assumptions, the 10-year plan envisioned a doubling of NIST laboratory budgets and modernization of NIST laboratory facilities; an Advanced Technology Program that would accelerate in order to address more industrial needs; increases in the program budget for the Manufacturing Technology Centers; increased synergism between the intramural NIST technical program—which would help create new technology—and the extramural program—which would help commercialize the new technology and assess new industrial needs; broader scope for the NIST Quality Outreach Program to include non-profit organizations as well as industrial firms; and moves by standards experts to join embassy teams throughout the industrially oriented world to collect and disseminate technical information.

The Board urged that NIST increase the effort to bring industry into fuller participation in strategic planning for the 1990s, and develop measurement techniques for evaluating the success of its program.

The Board noted that funding for the Standard Reference Data and Standard Reference Materials programs had effectively decreased over the previous decade, and that that trend had been deepened by the commitment of NIST resources to the new programs. The Board urged NIST management to reverse that trend, because of the proven value of the SRD and SRM programs to industry.

Fundamental, long-term research also had suffered during recent years, according to the Board. The funding target declared by NIST management for basic work—15 % of each year's appropriation—was a modest one by industry standards. As much as 20 % would have been more in line with industrial practice.

Yet another area that was under-funded, in the view of the Board, was that of building new NIST capabilities through the Competence Building Program. The total of $27 millior spent since 1979 had been too small by at least a factor of two, in the Board's opinion.

Furthermore, the NIST management ran the program as a contest, rather than as an integral part of strategic planning with long-lasting support to fully establish new areas of competence.

The Board report quoted a recent report by the Office of Technology Assessment:[12]

> There is a clear need in the United States for greater attention to standards. In an information-based global economy, where standards are not only employed strategically as marketing tools but also serve to interconnect economic activities, inadequate support for the standard-setting process will have detrimental effects.

Adequacy of NIST's equipment and facilities was another concern expressed in the Board report. In many cases, found the Board's investigators, NIST staff was forced by circumstances to improvise or borrow needed equipment.

The Board suggested that the principles of Total Quality Management be extended at NIST to all levels of the organization.

The report closed with suggestions for new approaches to the challenges facing NIST. Three of these were intended to be applied throughout NIST:

- Utilize specialized management training to facilitate cooperation with industry. If such training is not available, create it.
- Create a "technology transfer organization"—preferably in concert with industry—to facilitate commercialization of new technology.
- Make increasing use of interdisciplinary teams built from various NIST units, perhaps in collaboration with industrial or academic colleagues, to foster technology transfer to industry.

In addition to the suggestions for NIST generally, members of the various panels of the Board made many detailed suggestions that applied to the individual units of NIST listed in the previous table.

The report was powerful in its support of NIST and extremely thorough in the depth of its coverage of the various NIST laboratories.

In 1992 testimony before the Congressional Subcommittee on Technology and Competitiveness, Lyons reinforced the concerns of the Board of Assessment, citing an internal review of the state of the NIST laboratories. He noted that many of the NIST laboratory buildings were no longer equal to modern scientific demands. Such problems as ubiquitous dust—let alone a lack of critical services—rendered some of the laboratories unfit for specialized investigations. Lyons found this to be a double problem: not only did laboratory conditions impair scientific progress, but they made it increasingly difficult to retain some of NIST's most accomplished scientists, who routinely received offers of higher pay and better facilities elsewhere.

[12] "Global Standards: Building Blocks for the Future," *Office of Technology Assessment Report*, 1992, p. 9.

A Politicized Agency?

The Trade Act of 1988 certainly raised the profile of NBS/NIST. The success of the Technology Extension Program, with its Manufacturing Technology Centers; the growth of the Advanced Technology Program; the popularity of the Baldrige Quality Award; and the emphasis on industrial productivity generated in the balance of NIST's laboratories brought the agency more and more to the attention of America's technical establishment. Business and political leaders saw NIST as a leading agency in the technology effort of the Department of Commerce. Further, NIST became a helpful source of funds for research into advanced manufacturing by American companies.

However, with increased attention and increased budgets came unavoidable consequences. One of these appeared when the Clinton administration replaced the Bush team. Instead of treating NIST as NBS and other apolitical agencies had been treated, the new administration considered NIST as one of many departments and agencies whose leadership should be chosen to reflect "correct" thinking on economic and political issues. Despite the obvious successes of NIST during the Bush administration, a Department of Commerce order early in 1993 moved John Lyons to the position of Acting Undersecretary for Technology. Raymond Kammer once again became Acting Director of NIST.[13]

Arati Prabhakar, director of the Microelectronics Technology Office of the Defense Advanced Research Projects Agency, a Defense Department unit, appeared to the administration to be an excellent choice for Director of NIST on account of her experience with an agency that supported many external grants. She was duly nominated for the post of Director, NIST, and confirmed by the U.S. Senate on May 28, 1993.[14]

Under the Clinton administration, the framework created by the Omnibus Trade and Competitiveness Act of 1988 remained essentially intact. In fact, the administration supported substantial funding increases for the Advanced Technology Program and the Manufacturing Extension Center activities.

Technical Work of NIST, 1990-1993

Technology Outreach Programs

Advanced Technology

In mandating the Advanced Technology Program (ATP) for NIST as part of the 1988 Trade Act, Congress sought to make use of the technical sophistication of NIST scientists and engineers to identify promising new industrial ideas. Selected projects would be given Federal support during the crucial period of investigation that necessarily preceded the development of a product, with NIST helping to reduce the high risk inherent in the projects. After considerable discussion between NIST management and the Bush administration, funding became available for the development of "generic, precompetitive" technologies by individual firms or by industry-dominated joint ventures.

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[13] John Lyons, Oral History, June 9, 1993.

[14] NIST Admin. Bull. 93-12, June 4, 1993.

The first person named to head the ATP was Donald R. Johnson, who had participated in planning the program at the request of Ernest Ambler. When the new NIST structure was formally approved on January 4, 1991, however, it included a major unit called Technology Services. Johnson immediately was re-assigned to the position of director, Technology Services, with George A. Uriano succeeding him as director of ATP. Brian C. Belanger was appointed deputy director. The ATP force was never large—numbering but 10 people in 1991—although it administered millions of dollars in contract funds.

Public Law 100-418 limited the amounts of the grants to individual companies to $2 million over a 3-year period. For joint ventures, the grants could be larger, depending on the projects, and they could be continued up to 5 years. The program was intended to be a cost-sharing one, so that all grantees would be seriously committed to their projects.

As NIST gained experience with the ATP concept, Uriano and Belanger made extensive use of industrial representation to identify promising technical areas. They also followed examples from other more-experienced Federal-grant organizations such as the National Institutes of Health and the Defense Advanced Research Projects Agency. Quickly, the NIST ATP reached a workable approach to its responsibility:

- Competitive selection of grantees. Each proposal was examined to see that it conformed to the basic requirements of the program, then given comprehensive reviews by experts in the technical area of the proposal. Further reviews of promising proposals evaluated their potential economic impact and the level of corporate commitment implicit in the presentation. To minimize the conflict of interest that would arise from reviews by industrial personnel, only Governmental and academic scientists and engineers served as technical reviewers; business-related evaluations were performed by industrial consultants who were required to avoid conflicts of interest and to preserve proprietary confidentiality. Proposals were ranked according to published criteria, and awards were based on the ranking.

- Direct support to for-profit firms. Grants were awarded to start-up companies as well as small, medium, and large firms, and to joint ventures. The joint ventures could include universities, governmental organizations, or non-profit groups as sub-contractors or partners.

- Economic goals. The ATP, designed to "rapidly commercialize new technologies," continually maintained as its primary goal the economic success to be derived from its awards. The program supported both new products and lower-cost production methods. By supporting commercial firms, the ATP could rely on strong grantee commitment to financial success.

- Monitoring of performance. ATP managers quickly developed a plan for continuous monitoring of the performance of its grantees. Measurable goals were defined for the critical aspects of each successful proposal.

Independent studies of the ATP undertaken in 1991 showed that the program was quickly bearing fruit. Publication of program criteria provided businesses—particularly smaller firms—with yardsticks by which they could measure their own efforts to bring new technologies to market. The studies detected increased industrial research and development activity, higher productivity, and quicker time-to-market for new products and processes.

Viewpoints of industry on the ATP program were expressed by many individuals and groups. Senior industrial managers provided direct guidance on research priorities. Professional societies and trade associations yielded yet other suggestions. On occasion, NIST sponsored workshops and conferences specifically intended to generate ATP program input from a variety of sources. The file of ATP proposals also provided useful information for program direction.

Brief sketches of successful proposals up to 1993 illustrate the breadth of the ATP:

- Creation of generic computer software for use with motion pictures—to digitally restore, re-format, or enhance sequences of infrared, x-ray, or motion-picture images.
- Design for multiple-axis machine tools of high precision, based on an octahedral frame.
- A low-cost, automated DNA sequencer, incorporating on a single microchip synthetic DNA probes and computer sensor technology.
- High-temperature-superconductor-based thick-film processing technology for radio-frequency communications components.
- Erasable optical disk drive, using an electron trapping optical memory, for high-speed digital video recording.
- Gas-phase cleaning agents for removing surface damage, trace metals, and particles from semiconductor wafers.
- Improved production technology for lasers and light-emitting diodes.
- New technology for designing and analyzing thermoplastic parts in terms of microstructure, geometry, and performance.
- Production technology for ductile, metallic glass ribbon for use in high-power, low-loss electric transformers and motors.

Early in 1995, Lura J. Powell succeeded George Uriano—retiring after more than two decades of service to NBS/NIST—as director of the ATP. Brian Belanger remained as her deputy. As the program expanded, it became necessary to divide responsibility for its various aspects. To this end, Powell created five offices during 1996. These were:

- Economic Assessment, under Rosalie T. Ruegg.
- Information Technology and Applications, under Bettijoyce B. Lide.

- Chemical and Biomedical Technology, under Stanley Abramowitz.
- Materials and Manufacturing Technology, under John P. Gudas.
- Electronics and Photonics, under Brian Belanger.

Still later, an Office of Programs and Information Management was formed under the direction of Alvin H. Sher.

William F. Long of Business Performance Research Associates wrote a critique of the ATP in 1999.[15] His report focused on the first 8 years of the program. During that period, 1990-1998, the ATP received more than 3,500 proposals; 431 of these were selected for funding. Long estimated the total cost of the funded projects to be $2.8 billion. The industrial proposers provided slightly more than half of that sum, with ATP supplying the rest.

As of March 1997, 38 of the projects were completed—final project reports had been submitted and financial and other required data had been supplied to ATP—and another 12 had been terminated prior to completion. Long and his colleagues analyzed the 38 completed projects to assess the significance of the ATP for American industry.

Individual firms—27 with fewer than 500 employees—accounted for 34 of the successful projects. None received more than the $2 million grant from ATP allowed by PL 100-418. Most contributed resources worth as much as the Federal funding; eight firms spent about twice as much of their own funds as the amount of the grant. Of the four joint-venture grants, three received ATP funding of $5 million or less, while one received between $5 million and $10 million.

Almost half of the completed projects involved electronics; the rest were distributed among the areas of computers and communications, biotechnology, energy and environment, manufacturing, materials, and chemicals.

Industry gains from the completed projects could not be assessed accurately because, for most of the projects, the "bottom line" was still under evaluation by the participants. However, estimated returns on project efforts were offered by a few participants:

- Auto Body Consortium, consisting of nearly a dozen industrial and academic entities involved in automobile production, estimated that their "2 mm" project—devoted to reducing assembly-line errors to 2 mm or less—might return $3 billion from quality improvements in American cars. By 1999, the new technology was in use in about half of the U.S. Chrysler and General Motors assembly plants.
- Aastrom Biosciences, involved in a human-stem-cell-production project, projected its return at nearly $1 billion.
- Tissue Engineering, a biotechnology start-up company working toward regeneration of body tissue to avoid the need for artificial replacements, projected return on its project investment at $1 billion or more.

[15] William F. Long, "Advanced Technology Program: Performance of completed projects. Status report No.1," *NIST Special Publication 950-1*, March 1999, 134 pp.

Nearly two dozen of the completed project statements reported that the new technology had already led to new or improved commercial products or processes—some of them anticipated, some serendipitous. Some of the projects received recognition in the form of awards:

- Superconducting wire fabrication and winding techniques, a project of the American Superconductor Corporation, received a 1996 R&D 100 award from *Research and Development* magazine. It also was named a Technology of the Year (1996) by *Industry Week* magazine. Once the ATP project began, the company received support—previously unavailable—from the U.S. Department of Energy.
- Software that recognized handwriting for computer input, developed by Communication Intelligence Corporation, received the "Ease-of-Use Seal" of the Arthritis Foundation in 1997.
- Thallium-barium films for superconducting electronic devices, a project of E.I. du Pont de Nemours & Company, was recognized as one of the "Top Products of 1993" by *Microwaves and RF* magazine.
- Animated three-dimensional representations of the human body, developed by Engineering Animation, Incorporated, received the Smithsonian Award in 1994 for the use of information technology in the field of medicine, given by *Computerworld* magazine. The procedures also received two 1995 awards for animation from the Association of Medical Illustrators and the "Annie" award from the motion picture film industry, as well as a 1996 "25 Technologies of the Year" award from *Industry Week* magazine.
- Robots for transporting medicines in hospitals, a project of HelpMate Robotics, Inc., received the Japan prize in 1997 from the Science and Technology Foundation of Japan.
- Cellular telephone site filters and superconducting ceramics, developed by Illinois Superconductor, Inc., received the *Microwave and RF* magazine award as one of the Top Products of 1996, and the 1997 Corporate Technical Achievement Award of the American Ceramic Society.

The ATP funding received lavish praise from participating companies. Two-thirds of the successful participants testified that their projects would not have been undertaken without the stimulus provided by the program. The others expressed the belief that, without ATP funding, their projects would have been delayed by 18 months to 5 years. Clearly the Advanced Technology Program placed the NIST in the right place at the right time with the right leadership to the benefit of U.S. industry.

Quality Programs

The General Accounting Office did not give praise lightly. Both NIST director John Lyons and Curt W. Reimann, director of Quality programs, were edified, therefore, that a GAO report, written less than three years after the Malcolm Baldrige National Quality Award was established late in 1988, described its selection criteria in glowing terms:[16]

> [It is] the most widely accepted formal definition of what constitutes a total quality management company. In nearly all cases, companies that used total quality management practices achieved better employee relations, higher productivity, greater customer satisfaction, increased market share, and improved profitability.

The Baldrige Award, described in *The Malcolm Baldrige National Quality Award,* Chapt. 4, was an instant success. Its selection criteria, developed in consultation with industry, provided many a company with a useful guide to evaluating its approach to business. The awards themselves lent prestige to the winning firms. Indeed, a letter from Solectron Corporation, a 1991 awardee, to the Secretary of Commerce indicated more tangible benefits as well:

> Since we applied for the Malcolm Baldrige award in 1989, Solectron's sales have increased 316 % and profit has increased 338 % in 3 years.
> We attribute our financial performance a great deal to the improvement we made through the rigorous examination and feedback from the Baldrige application process.

The award guidelines touched upon seven areas of quality in industrial performance:

- Leadership in setting goals for quality and paths to reach them.
- Information and analysis that is timely, reliable, and accessible.
- Strategic quality planning, integrated into the overall company business plan.
- Human resource development and management to maximize employee potential for quality work.
- Management of processes to achieve quality results.
- Evaluation of the quality and trend of company operations.
- Customer focus and satisfaction.

Financial support of the Baldrige award initially was provided by industry, although the NIST staff members who administered the program were supported internally. Eventually, NIST management was successful in obtaining Congressional funding for the program.

[16] "Malcolm Baldrige National Quality Award," pp. 7, 15 in "Guide to NIST," *NIST Special Publication 858,* October 1993.

Quality experts from both the public and private sectors joined in independent reviews of the many applications received each year; those selected as outstanding received on-site visits to flesh out details of their quality programs. All applicants received written summaries of their quality operations.

Curt Reimann remained in charge of the Baldrige award program until 1996, when he retired. He was succeeded by Harry S. Hertz.

Technology Services

Among the most visible of the NIST outreach programs were those adminstered through Technology Services:

- *Manufacturing Technology Centers,* which by 1993 numbered seven, were located in California, Ohio, Kansas, Michigan, New York, South Carolina, and Minnesota. The MTC were set up mainly for the benefit of small- to mid-sized companies.

  Each center tailored its services to the needs of its manufacturing client base, usually offering assistance in the form of individual project engineering, training courses, demonstrations of equipment and procedures, and advice on the selection and use of tools and software. Visits to client plants and on-site advice on installations proved beneficial, as did consultation on management, finances, marketing, and employee training.

  Equipment available for demonstration generally included automated lathes and milling machines, industrial robots, and state-of-the-art measurement instruments.

  The success of the program was measured in terms of progress made by clients of the MTC. The average gains by such firms included an increase in the numbers of employees by 15 %, a 7 % increase in productivity, and an astonishing 34 % increase in sales. The total Federal investment in the MTC—about $18 million from January 1989 to June 1991—resulted in total savings to the client companies of nearly $140 million.

  Richard D. Suenram described several case histories illustrating the variety of assistance provided to client firms by the MTC.[17] The firms discussed ranged in size from 27 employees to 174. Their products and the technologies involved included acrylic residential countertops (computer-controlled routing of one-of-a-kind designs), metallizing of plastic bottle caps (introduction of ultraviolet-light curing, eliminating dust), prosthetic hip and knee joints (computer aided design and manufacturing), high-volume, low-pressure paint spray equipment (plant layout, multi-purpose computing), automated machine tools (multi-purpose computing, automated measurements), surgical-instrument sterilizing equipment (computer-aided design and manufacture), roller bearings (process development and control, measurement), industrial cleaning equipment (computer modeling, videotaping), nuclear magnetic resonance spectrometers (manufacturing process design).

[17] Richard Suenram, "The Manufacturing Technology Centers program: A sampling of individual case histories," *NIST Special Publication 830,* February 1992, 21 pp.

• *State Technology Extension Programs (STEP),* initially known as the Boehlert-Rockefeller Technology Program, provided assistance in developing the infrastructure needed by the various states to assemble their own programs to help smaller manufacturing businesses. The STEP emphasized use of the Manufacturing Technology Center capabilities, local university centers, and community college programs in manufacturing technology.

Among the methods used by the state programs were industrial extension agents to provide advice on the use of new technologies and pilot projects in important sectors of the state's industry. Grants, to be matched dollar for dollar by sponsoring organizations, could be allocated to non-profit institutions as well as state agencies.

During 1993, both the Manufacturing Technology Centers and the State Technology Extension Program became part of a *Manufacturing Extension Partnership Program* (MEPP) mandated by President Clinton. Clinton, recognizing a concept with the potential of benefitting the economy, hoped to see "over 100 manufacturing extension centers nationwide by 1997 to assist manufacturers to modernize their production capability." Philip N. Nanzetta was named head of the MEPP.

• *Office of Standards Services* provided assistance on national and international standards and certification activities. Through the U.S. Interagency Committee on Standards Policy, for which NIST served as the secretariat, the OSS published guidelines for Federal participation in standards creation and laboratory accreditation.

By this time the issue of international industrial standards was critical for U.S. firms participating in international trade. Thousands of U.S. standards affected domestic sales as well. Guiding American businesses through the maze of standards aided them greatly in their efforts to efficiently manufacture and sell their products. In 1992, Stanley I. Warshaw directed the office. Along with his deputy, Walter G. Leight, Warshaw coordinated the work of a *Standards Code and Information* program headed by John L. Donaldson, a *Standards Management* program led by Samuel E. Chappell, a *Weights and Measures* program headed by Carroll S. Brickenkamp, and a *Laboratory Accreditation* program under Albert D. Tholen.

• *Standards Management* administered U.S. participation in the International Organization of Legal Metrology (OIML), as well as the *Voluntary Product Standards* program of the Department of Commerce. Effective representation in OIML of U.S. positions on trade standards was an important part of ensuring fair treatment for American products in foreign markets—OIML involved more than 80 nations in its deliberations.

In the tiny unit, Samuel E. Chappell directed the work of Otto Warnlof, Barbara Meigs, and Rachel R. Phelps, who solicited technical advice from U.S. trade associations, instrument manufacturers, universities, and other government agencies to develop a U.S. viewpoint on OIML draft standards documents. They also assisted NIST scientists who participated in OIML meetings as technical experts.

• *Weights and Measures,* an ancient arm of NBS/NIST, provided an accreditation program for state weights and measures laboratories in mass, length, and volume. In addition, the NIST office, headed in 1992 by Carroll S. Brickenkamp, offered training programs and advice on testing methods.

In a 1993 NIST publication, Georgia L. Harris described the state standards program of the state weights and measures system and identified the capabilities of the member laboratories.[18] Under terms of its 1965 congressional mandate, NIST provided training to state metrologists in NIST testing procedures and conducted a voluntary accreditation program for state weights and measures laboratories.

To qualify for accreditation, state laboratories were required to:

- Maintain resources—laboratories, equipment, and personnel—adequate to perform standards measurements.
- Employ a trained metrologist who could demonstrate use of program test procedures.
- Establish measurement controls, based upon NIST standards, for each area.

The areas of measurement included in the accreditation program were divided into two classifications—tolerance testing and calibration. Tolerance testing, determining whether a given actual standard was sufficiently close to its nominal value, was accomplished in mass and volume. Calibration, the assignment of a value and an uncertainty to a working standard, applied to mass, volume, length, frequency, and temperature.

NBS Handbook 143, *State Weights and Measures Laboratories—Program Handbook,* provided guidance for the state metrologists, including a self-appraisal checklist. Six regional round robin measurement assurance programs conducted by NIST metrologists served to evaluate the accuracy of the state measurements.

Besides the 50 state laboratories, accreditations had been earned by the city of Los Angeles, the Virgin Islands, and the U.S. Department of Agriculture.

The NIST Weights and Measures office also provided leadership for the National Conference of Standards Laboratories (NCSL), an organization devoted to developing and maintaining measurement standards. More than 3000 members of NCSL represented the standards interests of industrial, academic, and governmental organizations. Annual NCSL meetings served as a forum for dissemination of the latest information on standards and for discussion of outstanding standards problems.

• *Laboratory Accreditation,* headed by Albert D. Tholen, monitored the competence and technical qualifications of testing and calibration laboratories operated by either public or private organizations. The National Voluntary Laboratory Accreditation Program offered its services in several areas of testing: product testing, computer networks, construction materials, electromagnetic

[18] Georgia L. Harris, "State weights and measures laboratories: state standards program description and directory," *NIST Special Publication 791,* 1993 Edition, June 1993, 83 pp.

compatibility, energy-efficient lighting, fasteners, metals, telecommunications, and radiation dosimetry. In calibration, applicable areas included electrical quantities, dimensional measurements, radiation, mechanics, thermodynamics, time, and frequency.

- *Standard Reference Data* By 1993, the 30-year-old Standard Reference Data program administered a national network of scientists devoted to the critical examination of data and offered to the public a large group of databases containing the fruits of their labors. In addition, the program continued publication of the *Journal of Physical and Chemical Reference Data.* Malcolm W. Chase directed the program throughout the tenure of John Lyons.

Initially operating under a 1963 request from the Federal Council for Science and Technology to provide reliable data in the physical sciences through a process of critical evaluation, the program received legal status in Public Law 90-396, the *Standard Reference Data Act of 1968* (see Appendix A).

Evaluation programs, conducted in data centers and cooperative projects located at NIST, in universities, and in industrial research centers, assembled sets of reliable data for use in several fields:

- Analytical chemistry.
- Atomic physics.
- Biotechnology.
- Chemical kinetics.
- Materials properties.
- Molecular structure and spectrsocopy.
- Thermodynamics and thermochemistry.
- Thermophysical properties of fluids.

The 1993 SRD catalog listed more than 40 databases available in electronic form—on computer disks, CD-ROMs, magnetic tapes, and in "online" form.[19]

The *Journal of Physical and Chemical Reference Data,* since 1972 published jointly with the American Chemical Society and the American Physical Society, collected critical reviews of data in dozens of scientific areas. Journal articles were prepared by NBS/NIST authors and by experts from academia and industry. In 1993, Jean W. Gallagher became the editor of the journal, succeeding David R. Lide who had edited the magazine from its inception until well after his retirement from NBS in 1988.

- *Standard Reference Materials* In 1993, NIST offered more than 1200 different Standard Reference Materials. Ranging from chemicals certified for their properties to linewidth standards used in the production of integrated circuits, SRMs provided American science and industry with the capability to achieve and verify accuracy in their measurements. From 1991, measurements

[19] "NIST Standard Reference Data Products Catalog 1993," Malcolm W. Chase and Joan C. Sauerwein, editors, *NIST Special Publication 782*, 1993 Edition, January 1993, 79 pp.

were expressed in SI units;[20] for convenience, customary units could also be listed on SRM certificates. Uncertainty statements were required as well. Stanley D. Rasberry directed the program from 1983 until mid-1991, when he was appointed director of Measurement Services. William P. Reed was named as his successor to head OSRM. Thomas E. Gills became director in 1993.

The catalog of Standard Reference Materials offered its units in a variety of categories.[21] One of the oldest lines contained standards based upon chemical composition. These included ferrous and non-ferrous metals, high-purity materials, microanalytic aids, organic and inorganic materials, health-related items, food references, geologic standards, ceramics, and cement. SRMs defined for physical properties covered such fields as thermodynamics, optics, ionic activity, radioactivity, electricity, length measurements, and polymers. Reference materials for engineering touched upon fire research, surface finish, non-destructive evaluation, automatic data processing, and particle sizing.

John K. Taylor, dean of the NBS/NIST staff in 1986 when he retired after 57 years as an analytical chemist, was up-dating a *Handbook for Users of Standard Reference Materials* at the time of his death on March 26, 1992.[22] Nancy M. Trahey brought the handbook to publication. The text offered a bibliography of nearly 200 monographs describing individual SRMs, in addition to discussions of a whole range of other topics related to their use:

- Precision and accuracy.
- Quality assurance.
- SRM concept, production procedures, and certification.
- Methods of use of SRMs in various applications.
- Guidelines for reporting of analytical data.

Electronics and Electrical Engineering Laboratory

Personnel of the Electronics and Electrical Engineering Laboratory (EEEL) provided primary electrical standards and a raft of related services, including support for the Nation's electronic instrumentation, electronic products, electric utility, and semiconductor industries. Their specialties were to be found also in radio-frequency, microwave, and millimeter wave radiation, in superconductivity, magnetism, and optoelectronics.

The staff numbered over 280 permanent employees and nearly 80 guest scientists participating in various programs. The laboratory calibration program provided nearly 40 % of all NIST calibrations.

[20] "The International System of Units (SI)," *NIST Special Publication 330,* Barry N. Taylor, editor, August 1991, 56 pp.

[21] The "NBS/NIST Standard Reference Catalog" was re-issued periodically as *NBS/NIST Special Publication 260* in order to provide an up-to-date listing of available standard samples.

[22] John K. Taylor, "Handbook for Users of Standard Reference Materials," *NIST Special Publication 260-100,* edited by Nancy M. Trahey, February 1993, 102 pp.

Laboratory management was keenly conscious of the importance of close cooperation with industry, academics, and other Federal agencies (see The More Things Change, The More They Remain The Same: Industrial Productivity in Semiconductors—NIST Prefigured), numbering among its clients some of the tiniest firms as well as the largest. EEEL Director Judson French and his senior management staff had learned from experience how to maximize the effectiveness of the EEEL programs in metrology and associated technology, concentrating their efforts where NIST capabilities intersected with industry needs.

Electricity, Oskars Petersons, chief

Quantum-Based Voltage and Resistance Standards

A document entitled *Guidelines for Implementing the New Representations of the Volt and Ohm, Effective January 1, 1990* was published by Norman B. Belecki, Ronald F. Dzuiba, Bruce F. Field, and Barry N. Taylor in 1989.[23] Issuance of the guidelines was the culmination of brilliant technical work that extended over 20 years. Laboratories contributing to final values for the new standards encompassed the earth, bringing a revolutionary change to two of the oldest standards of physical measurement.

In their note, the authors explained to the reader the meaning of the term "practical representation of the volt," which would replace terms such as "laboratory unit of voltage." Side-stepping the stability problems inherent in the electrochemical cell, the international standards community decided to express the standard of voltage through the use of the superconducting Josephson effect. An analogous substitution was made for the resistance standard, replacing the bank of wire resistors used by most nations as the practical embodiment of the ohm with a representation based upon the quantum Hall effect.

From January 1, 1990, the U.S. representation of the volt based on the Josephson effect, "V(NIST-90)," would employ an assigned value of 483 597.9 GHz/V for the Josephson constant $2e/h$. Similarly, the U.S. representation of the ohm based on the quantum Hall effect, Ω(NIST-90), would, from January 1, 1990, employ an assigned value of 25 812.807 Ω for the von Klitzing constant h/e^2. These statements embodied big changes in the technology of electrical standards. We should say "Thank you and goodbye" to the old standards; they had served well.

Electrochemical Cell Calibrations

For a century and a half, electrochemical cells were used as standards of voltage; they had provided NBS with its voltage references from its founding. The history of standard cells at the Bureau was described by Hamer in 1965.[24] The most stable type

[23] Norman B. Belecki, Ronald F. Dzuiba, Bruce F. Field, and Barry N. Taylor, "Guidelines for Implementing the New Representations of the Volt and Ohm, Effective January 1, 1990," *NIST Technical Note 1263, June 1989*, 60 pp.

[24] Walter J. Hamer, "Standard cells, their construction, maintenance, and characteristics," *NBS Monograph 84*, January 1965, 38 pp.

During his tenure at NBS/NIST from 1970 to 2000, Norman B. Belecki led the Electrical Reference Standards group for 25 years. He supervised the change in the electrical standards calibration program from manual to automated measurements and the adoption of primary standards based on quantum phenomena.

of cell, the Weston cadmium cell, dated from 1891.[25] Standard cells were not intended as sources of electric power; therefore, their design differed substantially from such devices as automobile storage batteries or flashlight cells. Enclosed in an H-shaped glass container, each column contained an electrode; the cross-tube contained a saturated cadmium sulfate electrolyte. When carefully prepared and aged and maintained at a constant temperature, a group of such cells could provide a uniform reference voltage (about 1.018 V) within a few microvolts.

Until January 1990, the actual value of the volt was derived from the basic SI units of length, mass, time, and electric current. Even after that date, most users maintained their voltage references in the form of electrochemical cells. The great care needed to maintain Weston cells at constant temperature and to avoid mechanical disturbance made the shipment of these devices for calibration a complex and time-consuming process. In its *Calibration Services Users Guide*, NIST recommended the use of

[25] Forest K. Harris, Electrical Measurements (New York: John Wiley & Sons, 1952), p. 185. The Weston cell is also described in *NBS Monograph 84*, by Hamer.

thermoregulated containers maintained in an upright position for transporting the cells, with a minimum of one week given to stabilizing the cell at NBS, followed by a month of measurements to achieve voltage uncertainties of 1 ppm.[26] Often, the cells were transported to NBS by courier.

Superconductive Volt Standard

Both Weston cells and Thomas resistors provided standards known as *artifacts*—each copy yielding a slightly different value of the intended quantity, depending upon the workmanship exercised by the maker and the care taken by the user.

The prediction in 1962 of a superconductive tunneling effect by Brian Josephson of Cambridge University in England markedly changed the situation for the voltage standard. Josephson received the 1973 Nobel Prize in physics for his theoretical prediction that the flow of electrons between two superconductors connected by a thin insulating layer would be quantized according to the applied voltage. It was found that when a properly prepared Josephson tunnel junction was irradiated with microwaves, its current-voltage curve would exhibit steps at precise, quantized voltages characterized by three quantities; the microwave frequency, an integer denoting the step number, and a Josephson frequency-to-voltage quotient. None of the three numbers involved the identity of the particular Josephson junction, nor even the type of materials from which it was made.

By 1985, a group of scientists from the Boulder Electromagnetic Technology Division—Clark A. Hamilton, Richard L. Kautz, and Frances L. Lloyd—had prepared a practical superconducting voltage standard at 1 V. They accomplished this feat by connecting nearly 1500 Josephson junctions made of niobium and a lead alloy in a series array, then biasing the junctions with microwaves at 72 GHz. Painstaking testing showed that the array could provide a stable reference, despite thermal cycling to room temperatures.[27] Their achievement heralded a new age for voltage standards.

Realizing the implications of the Josephson effect for voltage standards, scientists from national laboratories in Holland, England, the United States (Barry N. Taylor from NBS), and the International Bureau of Weights and Measures (BIPM) in Paris formed a working group within the framework of the BIPM to evaluate the Josephson voltage-to-frequency quotient in units consistent with the International System (SI).[28] In 1988, the International Committee of Weights and Measures suggested that the national laboratories define the value of the quotient to be 483 597.9 GHz/V, with the transition to the new voltage standard to take place on January 1, 1990.[29]

[26] Bruce F. Field, "NBS Measurement Services: Standard Cell Calibrations," *NBS Special Publication 250-24,* October 1987, 52 pp. Also see "NIST Calibration Services Users Guide, 1989," Joe D. Simmons, editor, *NIST Special Publication 250*, January 1989, pp. 114-118.

[27] C. A. Hamilton, R. L. Kautz, R. L. Steiner, and F. L. Lloyd, "Practical Josephson voltage standard at 1 V," *IEEE Electron Device Letters EDL-6,* No. 12, pp. 623-625 (1985).

[28] B. N. Taylor and T. J. Witt, "New international electrical reference standards based on the Josephson and quantum Hall effects," *Metrologia* **26**, 47-62 (1989).

[29] B. N. Taylor, "New internationally adopted reference standards of voltage and resistance," *J. Res. NIST* **94**, No. 2, 95, (1989).

DC Resistance Calibrations

With a similarly venerable history, the NBS standard of dc resistance was embodied in a group of carefully prepared wires. James L. Thomas devised a stable resistance standard for NBS during the 1930s, using bare manganin[30] wire carefully coiled in a stress-free, sealed atmosphere. A group of 1 ohm Thomas-type resistors was found invariant in resistance within 1 ppm over periods of several years.[31] Although manganin wire has a low temperature coefficient of resistivity, standard resistors were maintained as nearly as possible at a constant temperature. Since sudden bumps could induce stress-related changes in the resistance of the wires, careful handling was an important feature of resistance standards measurement.

Quantum-Based Resistance Standards

While evaluation of the Josephson voltage effect was in progress, K. von Klitzing discovered a quantum effect related to measurements of the Hall coefficient in semiconductors.[32] He found that if a high-electron-mobility semiconductor was cooled to a few kelvins above absolute zero and a magnetic field of intensity 10 T was applied to it, the two-dimensional electron gas which the system approximated became quantized. As a result, the plot of Hall voltage (at fixed current) *vs* magnetic field exhibited plateaus of resistance characterized by a constant which, like the Josephson voltage-to-frequency quotient, was independent of material parameters. The BIPM working group happily accepted the task of expressing the so-called "von Klitzing constant" in a value consistent with SI units; following their recommendation, the CIPM selected 25 812.807 Ω as the assigned value during its 1988 meeting.

At NBS, Marvin E. Cage, Ronald F. Dziuba, Bruce F. Field, Thomas E. Kiess, and Craig T. Van Degrift began use of the quantum Hall effect to monitor drift in the resistances of five Thomas resistors of 1 Ω each. The group of resistors provided NBS with its realization of the U.S. legal unit of resistance. Three different Hall-effect devices, all made from gallium arsenide, were used in the study, along with an intermediary reference resistor of 6,453 Ω. The authors found the imprecision of the measurement process to be about 0.05 ppm. Over a 31-month period, the measurements indicated that the resistance of the U.S. legal ohm was decreasing at a rate of (0.05 ± 0.02) ppm per year.[33]

There was no question that new resistance metrology would be adopted at NBS.

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[30] "Manganin" is an alloy containing approximately 84 % copper, 12 % manganese, and 4 % nickel, chosen for its low temperature coefficient of resistivity and its chemical stability.

[31] Harris, op. cit., p. 213.

[32] K. v. Klitzing, G. Dorda, and M. Pepper, *Phys. Rev. Lett.* **45**, 494-497 (1980).

[33] Marvin E. Cage, Ronald F. Dziuba, Bruce F. Field, Thomas E. Kiess, and Craig T. Van Degrift, "Monitoring the U.S. legal unit of resistance via the quantum Hall effect," *IEEE Trans. on Instrum. and Meas. IM-36,* No. 2, 222-225 (1987).

Voltage and Resistance Calibrations

As might have been expected, convincing NBS/NIST customers (and their counterparts in other countries) of the wisdom of accepting a change in the volt and the ohm for any reason—let alone on the basis of quantum physics—was a challenging task as well.[34] Prior to the introduction of the 10 V array discussed below, Clark Hamilton, Charles Burroughs, and Kao Chieh reviewed the operation of the NIST 1 V Josephson-array volt standard, describing the physical structure of the chip on which 1500 junctions were deposited, the mounting of the chip in its cryoprobe, and the use of the array in voltage calibrations.[35]

Generally, calibrations were performed on Zener diodes rather than on Weston cells, because of the possibility of incurring current transients arising from switching within the Josephson array; typically, the uncertainty of the calibration was 0.02 ppm.

As of January 1990, the NIST volt was defined in terms of the Josephson-junction array. The calibration of voltage standards for NIST customers was discussed by Bruce F. Field.[36] An automated system incorporating a carefully chosen set of NIST electrochemical cells was used as the reference comparison. Inevitable drift in the voltage of the cells was monitored through routine comparisons with the Josephson array, using a set of Zener diodes as the intermediary. NIST's primary group of electrochemical cells drifted about 0.3 μV from July 1988 to October 1989, but the continual monitoring of the cell voltages kept the calibration process free of error from that source.

Customers for dc resistance calibrations similarly found the NIST service essentially unchanged by the quantum revolution. The NIST group of 1 Ω reference standard resistors was still used in calibrations, as were the usual working standards in the range $10^{-4}\,\Omega$ to $10^{-6}\,\Omega$.

Stabilizing Electrical Currents

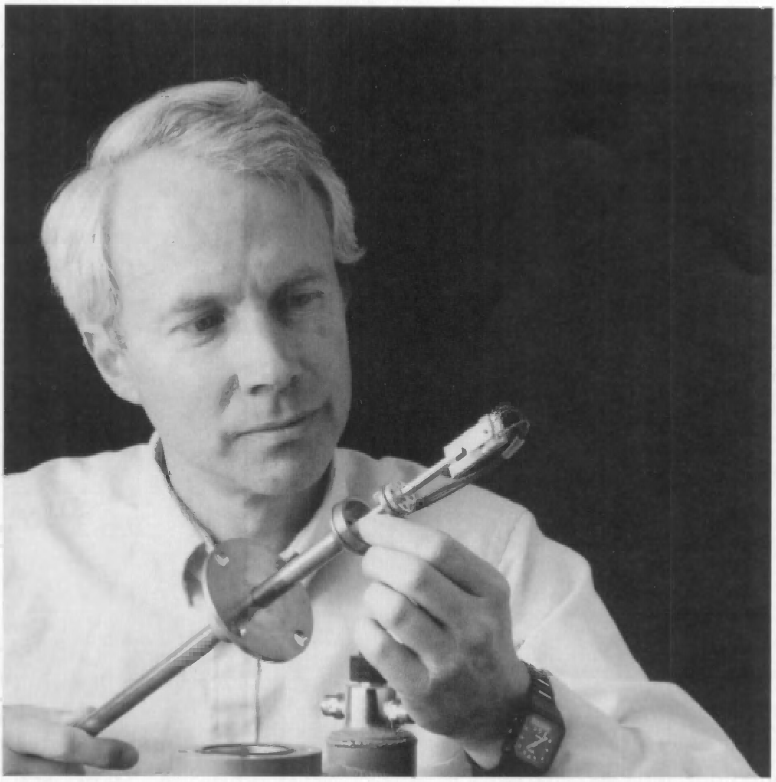
Edwin R. Williams and Weston L. Tew conducted interesting electrical experiments early in the 1990s. The intent of the work was to produce a convenient source of stable electric current. In one case, they were able to reach stability levels as low as one part in 10^9 per hour using the output from a superconducting quantum interference device (SQUID) in an error feedback loop. They monitored a 10 mA test current by enclosing it in a toroidal transformer; variation in the current was detected by the SQUID as a change in magnetic field.[37]

[34] K. B. Jaeger and B. N. Taylor, "U.S. perspective on possible changes in the electrical units," *IEEE Transactions on Instrumentation and Measurement,* Vol. IM-36, No. 2, pp. 672-675, June 1987.

[35] Clark A. Hamilton, Charles Burroughs, and Kao Chieh, "Operation of NIST Josephson array voltage standards," *J. Res. NIST* **95**, No. 3, pp. 219-235 (1990).

[36] Bruce F. Field, "The calibration of dc voltage standards at NIST," *J. Res. NIST* **95**, No. 3, pp. 237-253 (1990).

[37] W. L. Tew and E. R. Williams, "Flux locked current source reference," *IEEE Trans. on Instrum. and Meas.* **42**, No. 2, 186-190 (1993).



NIST electronics engineer Clark A. Hamilton examined the 1 V Josephson series array voltage standard. With about 3000 junctions, these NIST-fabricated integrated circuits are used in standards laboratories around the world.

A second study was performed by Williams and Tew with the collaboration of Cheol-Gi Kim, a guest worker from the Korea Research Institute of Standards and Science, Hitoski Sasaki, a guest worker from the Electrotechnical Laboratory in Tsukuba, Japan, and P. Thomas Olsen and S. Ye, colleagues in the NIST Electricity Division. The work featured the stabilization of a 1 A current using a nuclear magnetic resonance technique.[38] Two pairs of solenoids, co-axial and connected in tandem but magnetically opposing, produced axial fields that were nearly uniform and almost impervious to interference from steady magnetic fields outside the apparatus. The team stabilized the current in the solenoids using nuclear magnetic resonance frequency error signals derived from water samples at the center of each solenoid. Evaluation of the constancy of the test current was accomplished by monitoring the voltage across a series standard resistor with a Zener diode. The current proved stable within 0.1 ppm over an 8-hour period.

[38] C. G. Kim, E. R. Williams, H. Sasaki, W. L. Tew, S. Ye, and P. T. Olsen, "NMR based current/voltage source," *IEEE Trans. on Instrum. and Meas.* **42**, No. 2, 153-156 (1993).

Semiconductor Electronics, Frank F. Oettinger, chief

Semiconductors and Devices

Following passage of the 1988 Trade Act, the EEEL offered "cooperative research opportunities"—chances for outside organizations to work with the NIST staff—on several projects in the semiconductor electronics field. Among these were:

- Silicon characterization.
- Compound semiconductors.
- Microstructures.
- Molecular beam epitaxy.
- Thin film metrology.
- Integrated circuit metrology.
- Fault detection and prevention.
- Power electronics.

In the following paragraphs, work done in some of these projects is briefly discussed.

A Little History

W. Murray Bullis, former chief of the Electron Devices Division, illustrated the development of silicon devices and the integrated circuit industry from its beginnings, when each manufacturer had to produce its own materials, processing technology, and measurement tools. Bullis noted the many lessons learned as the industry matured and a supporting infrastructure arose to provide uniform materials, equipment, and instruments for it.[39]

Analysis of Semiconducting Films

Deane Chandler-Horowitz, Nhan V. Nguyen, Jay F. Marchiando, and Paul M. Amirtharaj collaborated with the Defense Department Advanced Projects Research Agency to analyze amorphous films of silicon carbide grown on silicon substrates. The films were considered for use as mask membranes for x-ray lithography. The Semiconductor Electronics Division team performed ellipsometric measurements on the films—a sensitive technique for the evaluation of thickness and optical properties. A two-layer analytical method provided the best fit to experimental data. In addition to layer thickness, the method was able to detect surface roughness and the presence of silicon and graphite phases.[40]

[39] W. M. Bullis, "Semiconductor measurement technology: evolution of silicon materials characterization; lessons learned for improved manufacturing," *NIST Special Publication 400/92,* July 1993, 46 pp.

[40] D. Chandler-Horowitz, N. V. Nguyen, J. F. Marchiando, and P. M. Amirtharaj, "Metrologic support for the DARPA/NRL-XRL mask program: ellipsometric analyses of SiC thin films on Si," *NISTIR 4860,* January 1993, 21 pp.

Linewidth measurement in x-ray lithography masks became slightly easier with the discovery by Michael T. Postek, Jeremiah R. Lowney, Andras E. Vladar, Robert D. Larrabee, W. J. Kerry, and Egon Marx that satisfactory contrast and good signal-to-noise ratios could be obtained using the transmitted-electron signal in a scanning electron microscope rather than the usual secondary electron signal. In the course of the work, the authors also developed a potential basis for the first SEM-based NIST linewidth standard.[41]

Nanolithography

The use of the scanning tunneling microscope to generate patterns at the nanometer level was demonstrated on III-V semiconductors such as n-doped gallium arsenide by a group that included John A. Dagata, Wing Tsang, Joseph Bennett, Jason M. Schneir, and Howard H. Haray.[42] Molecular beam epitaxy was used to prepare the substrates. They were characterized by time-of-flight secondary-ion mass spectrometry and x-ray photoelectron spectroscopy. The same authors reported a novel method for preparing GaAs substrates with markedly improved topographical and chemical surface uniformity.[43]

X-Ray Absorption

Charles E. Bouldin, G. Bunker, David A. McKeown, Richard A. Forman, and Joseph J. Ritter, of the Semiconductor Electronics Division, demonstrated that the x-ray absorption fine-structure data for the tetrahedral germanium gases $GeCl_4$, GeH_3Cl, and GeH_4 in the range 10 $(nm)^{-1}$ to 30 $(nm)^{-1}$ could be analyzed using a single scattering assumption.[44]

Electromagnetic Fields, Allen C. Newell, chief

Antenna Measurements

During this period, the NIST/Boulder Electromagnetic Fields Division staff developed an automated facility for the measurement of near-field phase and amplitude distributions from test antennas. Using the facility, they could evaluate antenna characteristics, including gain, polarization, and radiation patterns. In addition, they could obtain diagnostic information such as locating faulty elements in phased-array models, making feed adjustments, and finding surface imperfections, and they could calibrate probes.

[41] M. T. Postek, J. R. Lowney, A. E. Vladar, R. D. Larrabee, W. J. Kerry, and E. Marx, "X-ray lithography mask metrology: use of transmitted electrons in an SEM for linewidth measurement," *J. Res. NIST* **98**, No. 4, pp. 415-445 (1993).

[42] J. A. Dagata, W. Tsang, J. Bennett, J. M. Schneir, and H. H. Haray, "Nanolithography on III-V semiconductor surfaces using a scanning tunneling microscope operating in air," *J. Appl. Phys.* **70**, No. 7, 3661-3665 (1991).

[43] J. A. Dagata, W. Tsang, J. Bennett, J. M. Schneir, and H. H. Haray, "Passivation of GaAs surfaces for scanning tunneling microscopy in air," *Appl. Phys. Lett.* **59**, No. 25, 3288-3290 (1991).

[44] C. E. Bouldin, G. Bunker, D. A. McKeown, R. A. Forman, and J. J. Ritter, "Multiple scattering in the x-ray absorption near-edge structure of tetrahedral Ge gases," *Phys. Rev.* **B38**, No. 15, pp. 10816-10819 (1988).

At the same time, Arthur R. Ondrejka and Motohisa Kanda utilized a wideband time-domain reflectometer to evaluate the reflection characteristics of RF and microwave absorbers. The reflectometer consisted of an array of paired antennas, used in a difference mode to remove unwanted signals and thus to improve the instrument sensitivity. Reflection characteristics could be measured over the range 30 MHz to 1000 MHz.[45]

Results were obtained for the initial phase of a NIST study of the alignment of planar phased-array antennas using the merged-spectrum method. An Electromagnetic Fields Division group including Ronald C. Wittmann, Allen C. Newell, Carl F. Stubenrauch, Katherine MacReynolds, and Michael H. Francis developed a theory to support evaluation of the merged-spectrum technique and provided simulation examples to illustrate the calculation of near radiation fields from array and element patterns.[46] The aim of the study was to quantify the effects of errors from uncertain measurements, antenna steering, and a variety of analytic approximations.

A new standard reference source, a spherical dipole, was developed during 1991 for use with automated antenna test systems. Galen H. Koepke, L. D. Driver, Kenneth H. Cavcey, Keith Masterson, Robert T. Johnk, and Motohisa Kanda participated in the project. The instrument had the advantage that it could be characterized theoretically as well as by measurement. The radiated field was generated remotely; connections to the signal generator and control elements were made *via* an optical fiber cable. Initial data showed good agreement between field measurements and theoretical predictions.[47]

Millimeter-Wave Devices

The development of new instrumentation incorporating millimeter waves—wide band satellite communications, short range radar, and control of vehicular traffic, to name three—was traced by Gerome R. Reeve of the Electromagnetic Fields Division. Reeve noted the growth of gallium arsenide based devices to provide less expensive, higher performance circuitry, and the new features of the NIST semiconductor program intended to meet the challenge of the new technology.[48]

Electromagnetic Technology, Robert A. Kamper, chief

A 10 Volt Josephson Reference

A 10 volt reference standard involving thousands of superconducting Josephson junctions—a dream of electronic instrument makers world-wide—first became a reality at the hands of Frances L. Lloyd, Clark A. Hamilton, James A. Beall, D. Go, Ronald

[45] A. R. Ondrejka and M. Kanda, "Time-domain method for measuring the reflection coefficient of microwave absorbers at frequencies below 1 GHz," *Proc. Digest Antennas and Propagation Society Symposium, London, Ontario, June 24-28, 1991* 3, pp. 1656-1659 (1991).

[46] R. C. Wittmann, A. C. Newell, C. F. Stubenrauch, K. MacReynolds, and M. H. Francis, "Simulation of the merged spectrum techique for aligning planar phased-array antennas," *NISTIR 3981*, October 1992, 51 pp.

[47] G. Koepke, L. D. Driver, K. Cavcey, K. Masterson, R. Johnk, and M. Kanda, "Standard spherical dipole source," *NIST Technical Note 1351*, December 1991, 133 pp.

[48] G. R. Reeve, "Millimeter wave metrology at NIST," *Proc. NCSL Workshop and symposium, Albuquerque, New Mexico, August 19-22, 1991*, pp. 183-187.

H. Ono, and Richard E. Harris in the Boulder Electromagnetic Technology Division. A short time later, Hamilton and Lloyd, along with Kao Chieh, a guest scientist from the Sichuan, China, National Institute of Metrology, and Wayne C. Goeke of the Hewlett Packard Company described the operation of such an array.[49] It was composed of nearly 19,000 tiny junctions, 12 μm by 24 μm in area, formed between layers of niobium and a lead-indium-gold alloy separated by Nb_2O_5 insulators on a small chip. Radiation at 70 GHz to 100 GHz generated voltages as high as 12 V dc in the array, a signal suitable for the calibration of Zener diodes and digital voltmeters.

The achievement was a natural consequence of the landmark development described earlier (see Superconductive Volt Standard in this chapter), in which Hamilton, Kautz, and Lloyd produced a 1 V reference using a series array of some 1500 Josephson junctions.

In order to achieve the goal of operating as many as 19,000 junctions within the required specifications, the research team modified the usual fabrication process by improving the lithographic technique and by slight changes in the deposition procedure.

Using the new array to observe a Zener diode over a 5-month period, the group was able to report a drift in the Zener output of about 0.5 ppm with an imprecision less than 0.1 ppm.

Within 2 years, Richard L. Steiner and Robert J. Astalos of the Electricity Division reported the automation of a 10 V calibration system using the new array.[50] They achieved an uncertainty level of 0.02 ppm, using a commercial standard-cell scanner connected to the array, to the Zener references, and to a digital voltmeter. Key to their success were new procedures for using a programmable millimeter wave attenuator and for error checking.

The new system facilitated the calibration of up to 20 Zener diodes per day, along with occasional digital voltmeter calibrations. Self-check features caught infrequent inconsistencies arising from system flaws—some of them so slight as to escape observation by the human operators.

The new array played an important role for the Hewlett-Packard Company, materially advancing its development of a new, high-accuracy multimeter.

High-Temperature Superconductivity

Industrial interest in superconductivity perked up noticeably with the discovery that the phenomenon could exist at unbelievably high temperatures (see High-Temperature Superconductivity, Chapt. 4). No less a personage than President Ronald Reagan touched off the furor with a *Federal Conference on Commercial Applications of Superconductivity* in July 1987. Congress followed the President's lead by including

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[49] Clark A. Hamilton, Frances L. Lloyd, Kao Chieh, and Wayne C. Goeke, "A 10-V Josephson Voltage Standard," *IEEE Trans. on Instrum and Meas.* **38**, No. 2. 314-316 (1989).

[50] Richard L. Steiner and Robert J. Astalos, "Improvements for automating voltage calibrations using a 10-V Josephson array," IEEE Trans. on Instrum. and Meas. 40, No. 2, 321-325 (1991).

high-temperature superconductivity among the subjects to be covered by technical reviews mandated by the Trade and Competitiveness Act of 1988. John W. Lyons was a member of the superconductivity review panel.[51]

NBS immediately became a center for research and information on high-temperature superconductivity.

By 1990, NIST scientists were studying the preparation, analysis, and properties of superconductive materials with transition temperatures as high as 110 K, well above the normal boiling point of liquefied nitrogen—a much less expensive refrigerant than liquefied helium. The NIST program involved major collaborations among scientists from the EEEL and the Materials Science and Technology Laboratory.

A NIST Materials Science group including C. K. Chiang, Winnie Wong Ng, Lawrence P. Cook, Stephen W. Freiman, N. M. Hwang, Mark Vaudin, and Michael D. Hill found that one such material, a cuprate with the improbable formula $Bi_2Sr_2Ca_2Cu_3O_x$ (often given the acronym BSCCO in technical papers), could be synthesized from an amorphous oxide. Melting the constituents in powder form at 1200 °C, the group was able to prepare a ceramic mixture containing the desired compound in crystalline form. They presented a discussion of their preparation methods, along with electrical and magnetic measurements of the superconductor, during a 1990 meeting on Advances in Materials Science and Applications of High Temperature Superconductors.[52]

Another Materials Science group consisting of Lawrence H. Bennett, Marina Turchinskaya, Lyden J. Swartzendruber, A. Roitburd, D. Lundy, Joseph J. Ritter, and Debra L. Kaiser made a detailed study of magnetic flux dynamics in BSCCO and $YBa_2Cu_3O_6$ (often shortened to YBCO or simply 123). These superconductors were Type II, and thus exhibited the general property of allowing magnetic flux to permeate the sample without driving it entirely out of the superconducting state. Bennett and his group studied the way in which the penetrating magnetic flux formed geometric patterns that changed with the magnitude and the frequency of applied magnetic fields.[53]

Structure analysis and magnetic effects in high-temperature superconductor materials could be accomplished readily through the use of neutron diffraction at the NIST reactor. One such investigation was undertaken by a group from Bell Communications Research Company—P. F. Miceli, J. M. Tarascon, P. Barboux, L. H. Greene, B. G. Bagley, G. W. Hull, and M. Giroud—in collaboration with J. J. Rhyne and

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[51] "Report of the National Commission on Superconductivity," *National Commission on Superconductivity*, Washington, D.C., August 7, 1990, 76 pp.

[52] C. K. Chiang, W. Wong-Ng, L. P. Cook, S. W. Freiman, N. M. Hwang, M. Vaudin, and M. D. Hill, "Processing Bi-Pb-Sr-Ca-Cu-O superconductors from amorphous state," pp. 127-136 in *Advances in Materials Science and Applications of High Temperature Superconductors,* NASA Goddard Space Flight Center, 1991.

[53] L. H. Bennett, M. Turchinskaya, L. J. Swartzendruber, A. Roitburd, D. Lundy, J. Ritter, and D. L. Kaiser, "Flux flow and flux dynamics in high-T_c superconductors," pp. 213-229 in *Advances in Materials Science and Applications of High Temperature Superconductors,* NASA Goddard Space Flight Center, 1991.

D. A. Neumann of the Reactor Radiation Division.[54] The authors noted that the high-temperature superconductor $YBa_2Cu_{2.8}Co_{0.2}O_{6+y}$ exhibited either superconductivity at temperatures near 60 K or antiferromagnetism, depending upon details of the composition. They examined in particular the influence of the oxygen concentration in the material, using neutron diffraction to track the magnetic transitions.

Paul Rice and John M. Moreland of the Electromagnetic Technology Division employed an electron tunneling technique known as *Tunneling Stabilized Magnetic Force Microscopy* (TSMFM), a variation of the scanning tunneling microscopy developed at NBS by Russell D. Young (see New Ideas in Physics, Chapt. 3) to examine films of $YBa_2Cu_3O_{7-\delta}$. The tip of the TSMFM was made of a tiny magnetic film that flexed in response to magnetic forces on the test surface. Using it, Rice and Moreland were able to prepare images showing the presence and pattern of magnetic pinning sites on the superconducting films.[55]

One of the first problems to be faced in evaluating the electrical properties of the new superconducting materials was making good electrical contact with samples. When electrical leads were attached to the ceramic superconductors by ordinary methods such as silver epoxy or paint or indium solder, the contact resistance usually was so high as to impair or preclude sensitive measurements. NIST researcher Jack W. Ekin, working with Betty A. Blankenship and Armand J. Panson of the Westinghouse Research and Development Center, created contacts with only about 10^{-8} Ω–cm² resistance by sputter-depositing silver on the sample and annealing it in oxygen at 500 °C for one hour. The new technique reduced the magnitude of the usual contact resistance by about a factor of one million. The U.S. Patent Office issued a patent for the process.[56]

Along with K. Salama and V. Selvamanickam of the Texas Center for Superconductivity at the University of Houston, Ekin used his new electrical contact process to make connections to oriented YBCO crystals prior to measuring the magnitude of their critical current. The new contact method, reducing enormously the heat generated by the test currents, appeared responsible for increasing markedly the maximum attainable current densities.

At 77 K, the measured critical current density reached 8 kA/cm² at 8 T, three times the maximum value achieved in previous experiments. Measurable values of critical current extended to 30 T at the same temperature, well in excess of earlier results.

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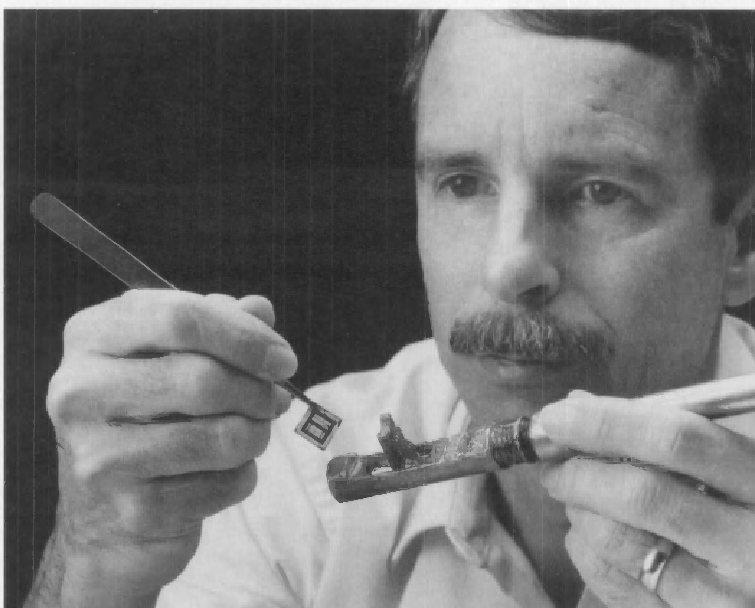
[54] P. F. Miceli, J. M. Tarascon, P. Barboux, L. H. Greene, B. G. Bagley, G. W. Hull, M. Giroud, J. J. Rhyne, and D. A. Neumann, "Magnetic transitions in the system $YBa_2Cu_{2.8}Co_{0.2}O_{6+y}$," *Phys. Rev.* **B39**, No. 16 (Rapid communications), 12375-12378 (1989).

[55] P. Rice and J. Moreland, "Tunneling Stabilized Magnetic Force Microscopy of $YBa_2Cu_3O_{7-\delta}$ films on MgO at 76 K," *IEEE Trans. on Magnetics* **27**, No. 6, 5181-5183 (1991).

[56] J. W. Ekin, A. J. Panson, and B. Blankenship, "Method for making low-resistivity contacts to high T_c superconductors," *Appl. Phys. Lett.* **52**, No. 4, 331-333 (1988). See also Collier Smith, "Low-resistance contacts developed for high-temperature superconductivity," pp. 3, 12 in "NBS Research Reports," *NBS Special Publication 735,* December 1987. See also Collier Smith, "Making Good Contact," p. 11 in NIST Research Reports, *NIST Special Publication 809,* March 1991.

The authors noted the crucial importance of high current density for many applications in superconductivity.[57]

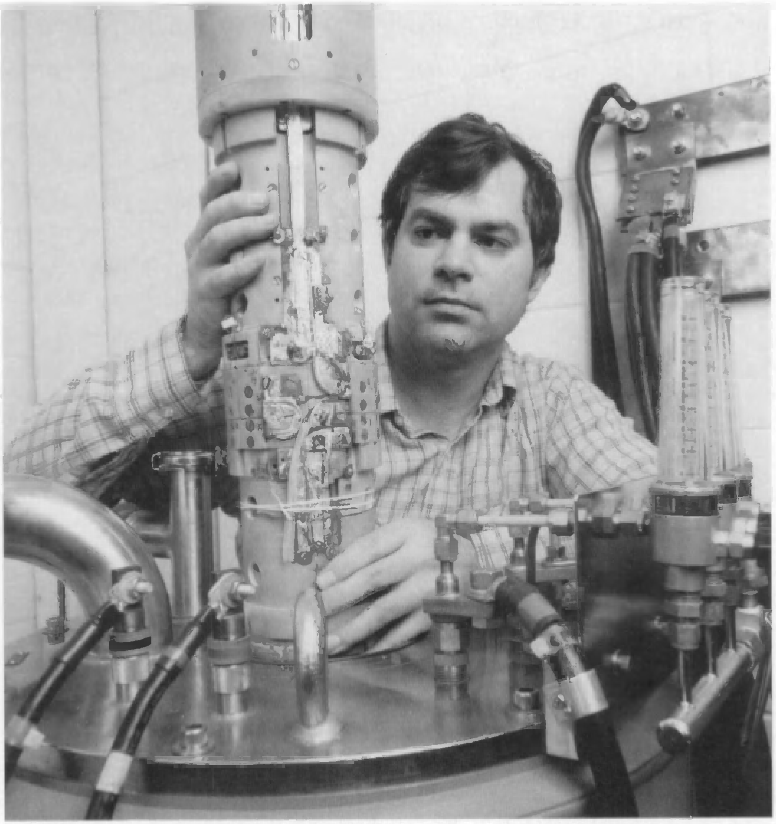
A comparison of methods used for the evaluation of superconducting critical current—the most current that a material could carry without returning to the normal, resistive state—was undertaken by Loren F. Goodrich and Ashok N. Srivastava of the Electromagnetic Technology Division. Noting the variety of available methods to obtain critical current values, the authors outlined several of these techniques, calling attention to the advantages and disadvantages of each.[58]



A method for making improved electrical contacts to high-temperature superconductors was developed and patented by researchers at NIST and the Westinghouse Electric Corporation. Here NIST physicist Jack W. Ekin inserted a sample into a cryogenic test fixture.

[57] J. W. Ekin, K. Salama, and V. Selvamanickam, "High transport current density up to 30 T in bulk $YBa_2Cu_3O_7$ and the critical angle effect," *Appl. Phys. Lett.* **59**, No. 3, 360-362 (1991).

[58] L. F. Goodrich and A. N. Srivastava, "Comparison of transport critical current measurement methods," *Advances in Cryogenic Engineering (Materials)* **38**, 559-566 (1992).



NIST physicist Loren F. Goodrich lowered a test cryostat containing a high-current niobium-titanium superconductor into a high-field magnet for critical current testing.

Robert L. Peterson undertook an analysis of the technical and economic impact of NIST/Boulder superconductivity programs, beginning in late 1990. He accumulated data from 33 companies by means of brief questionnaires, asking whether any of eight programs had benefitted the company, and, if so, in what ways.

Peterson found that high-temperature superconductivity had led to relatively few products by 1992. Half a dozen projects had reached the prototype stage—connectors among semiconductor chips, microwave devices, magnetic bearings and shielding, motors, and infrared detectors, for example, but the economic impact of these projects was minimal.[59]

[59] Robert L. Peterson, "An analysis of the impact on U.S. industry of the NIST/Boulder superconductivity programs: an interim study," *NISTIR 5012*, November 1993, 29 pp.

Manufacturing Engineering Laboratory

The Manufacturing Engineering Laboratory was created during the reorganization approved in January 1991 (see Taking Charge earlier in this chapter). John A. Simpson and his deputy Richard H. F. Jackson founded the laboratory on the marriage of state-of-the-art dimensional measurements and the needs of the U.S. manufacturing industry—the premise that had led, years before, to the Automated Manufacturing Research Facility.[60] Simpson retired in April 1993 after a scientific career that included 45 years' service to NBS/NIST. Robert Hocken, until 1989 chief of the Precision Engineering Division, left NIST to accept an engineering professorship at the University of North Carolina at Charlotte. The rest of the MEL management team remained intact throughout the tenure of John Lyons.

The Manufacturing Engineering Laboratory offered unique facilities and expertise in a variety of technical areas that U.S. industry needed to upgrade its products and processes. The Automated Manufacturing Research Facility continued as a star performer for the laboratory, but scientific competence was highly visible throughout its divisions—Precision Engineering, led by Dennis A. Swyt; Automated Production Technology, under Donald S. Blomquist; Robot Systems, headed by James S. Albus; and Factory Automation Systems, with Howard M. Bloom as chief. The achievements of the scientists and engineers in these divisions were manifold.

Precision Engineering, Dennis A. Swyt, chief

A Molecular Measuring Machine

A major project in NIST's precision engineering area was the development of a measuring machine intended to operate on an atomic scale. E. Clayton Teague, veteran of nearly two decades of experimentation on metallic surfaces, led the project. If all went well, the new instrument, dubbed the Molecular Measuring Machine (M^3), would be able to position itself accurately within a few atomic diameters over an area of about 25 cm^2, and to detect changes in surface topography at the same level. Heart of the M^3 would be a scanning tunneling microscope (STM) to observe surface characteristics, laser interferometry to accurately position the STM probe, and a carefully designed mounting system to provide smooth motion and isolation from the bumpy world surrounding the new tool. By 1993, the noise limit of the M^3 interferometer optics and electronics system was less than 0.1 nm, well within the design specification, although the instrument had not yet achieved overall atomic resolution.

Coordinate Measuring Machines

Under Ralph C. Veale, the dimensional metrology group completed installation of a coordinate measuring machine capable of detecting position at the 1 μm level. Such machines—though not equally capable—were becoming more and more noticeable in

[60] Former Director John Lyons recalled the vital role played by the U.S. Navy and other Federal agencies in funding the AMRF at its inception. Without such aid, the program might well have foundered.

the planning of the manufacturing industries. The group developed several Standard Reference Materials to be used in the testing and calibration of coordinate measuring machines, and collaborated on measuring machine research with a number of industrial firms.

Measuring Surface Roughness

Responding to increased industrial need for smooth surface texture, Jun-Feng Song—in 1991 a guest researcher from the ChangCheng Institute of Metrology and Measurement in Beijing, China—and Theodore V. Vorburger—leader of the division's surface metrology group—reviewed the use of standard reference samples in controlling the quality of product surfaces. They noted disagreements as large as 50 % among measurements made by different methods for profiling surfaces—contact, optical, and electron tunneling techniques. In many such cases, different measuring instruments simply responded to different aspects of the same surface, producing measurement results that varied accordingly. In other cases, significant variation in surface roughness, designated by the symbol R_a, occurred in different areas of the same surface. The two scientists concluded that satisfactory and reproducible manufactured surfaces could be achieved only after reaching a detailed understanding of the desired surface texture and employing reference samples consistent with that texture.[61]

With P. Rubert, of Rubert & Company, Stockport, England, the two researchers compared the R_a values derived from roughness master samples—relatively expensive to produce—with cheaper copies made by electroforming. For the test, they selected two masters with R_a values of 0.028 μm and 0.043 μm and compared measurement results, using a stylus instrument, with electroformed replica surfaces. The agreement between the two surfaces was good—fluctuations in master R_a values repeated on the copies within 1.8 nm. This was good news, as it boded well for more extensive use of the technique with less expensive reference surfaces. Flatness reproduction and hardness was noticeably poorer in the replicas, however.[62]

Vorburger, Joseph Fu, and Russell D. Young adapted a scanning tunneling microscope (STM) for longer-range scanning. They were able to extend its field of view to an area 0.5 mm by 0.5 mm. They accomplished the improvement by mounting the specimen on a traveling stage while holding the STM probe stationary in the plane of the stage. The probe mapped the topological features of the specimen by scanning within a range of 0.008 mm in the direction perpendicular to the plane of the stage.[63]

[61] J.-F. Song and T. V. Vorburger, "Standard reference specimens in quality control of engineering surfaces," *J. Res. NIST* **96**, No. 3, 271-289 (1991).

[62] J.-F. Song, T. V. Vorburger, and P. Rubert, "Comparison between precision roughness master specimens and their electroformed replicas," *Precision Engineering* **14**, No. 2, 84-90 (1992).

[63] J. Fu, R. D. Young, and T. V. Vorburger, "Long-range scanning for scanning tunneling microscopy," *Rev. of Sci. Instrum.* **63**, No. 4, 2200-2205 (1992).

Calibrating Optical Microscopes

Carol F. Vezzetti, Ruth N. Varner, and James E. Potzick completed a family of photomask linewidth standards for use with optical measuring microscopes. An anti-reflecting chromium linewidth standard, SRM 473, joined SRMs 475 and 476 as an optical microscopy reference set. The new standard was expected to be most helpful for measuring linewidths in the range 500 nm to 3000 nm, often required in the production of integrated circuits. Such measurements were difficult with optical microscopes because of the similarity of the linewidth dimension to the wavelength of the light used to make the measurement. The team recommended procedures for use of the standard to minimize edge effects in photomask measurements.[64]

Automated Production Technology, Donald S. Blomquist, chief

Error Compensation in Machine Tools

Researchers in the Automated Production Technology Division made use of the NIST computer-vision capability to help turning-machine operators reduce manufacturing errors in "real time." G. Nobel, M. Alkan Donmez—leader of the sensor systems group—and R. Burton developed a method to compensate for changes in the geometry of a cutting-machine tool while a part was being formed. They accomplished this feat by utilizing the output of a tool-inspection system to determine the magnitude of deviations from the nominal circular shape of the cutting edge. The system relied on visual observation of the tool while it was in use. Feeding the visual output to the control computer, they were able to modify the position of the tool to reduce cutting errors. Inspection of completed parts on a coordinate measuring machine showed substantial improvement in cutting performance.[65]

Somewhat later, Donmez, Kenneth W. Yee, and Bradley Damazo wrote a 50-page dissertation summarizing then-current methods for error compensation in machine tools. The major cutting errors, they said, arose from geometric relationships between the machine and the workpiece, and from heat generated during the cutting process. Geometric errors were caused by unexpected motions of machine elements—carriages, spindles, and work tables—due to imperfect machine construction and misalignments during its assembly. Thermal errors arose from gradients created by tool cutting friction and by drive motors; uneven thermal expansion caused relative displacement of the tool with respect to the workpiece.

The authors presented a generalized approach to error compensation based on the prediction of both geometric and thermally induced errors, using the control software to implement corrections. They identified more than 30 error components, although they pointed out that not all machines would necessarily exhibit all types of error.[66]

[64] C. F. Vezzetti, R. N. Varner, and J. E. Potzick, "Antireflecting-chromium linewidth standard, SRM 473, for calibration of optical microscope measuring systems," *NIST Special Publication 260-119,* September 1992, 52 pp.

[65] G. Nobel, M. A. Donmez, and R. Burton, "Real-time compensation for tool form errors in turning using computer vision," *Department of Energy Report DOE/OR/21584-T1,* 1990, 9 pp.

[66] M. A. Donmez, K. W. Yee, and B. Damazo, "Some guidelines for implementing error compensation on machine tools," *NISTIR 5236,* August 1993, 56 pp.

Robot Systems, James S. Albus, chief

The robotics laboratory of NIST provided a variety of challenges for its staff. A major goal, of course, was to achieve intelligent control of machines, so that they would quickly and efficiently turn out work that consistently met design specifications. To meet this goal, however, required detailed information, both on the machine itself and on its operating environment. Further, the machine had to be equipped with sensors to provide it with real-time monitoring of the process it was performing.

James S. Albus, chief of the Robot Systems Division, led a group devoted to perfecting intelligent machine controls. This was a pivotal study, in the view of its proponents, because the industry-wide lack of a structured, theoretical approach to machine control resulted in relatively unsophisticated applications for robots in manufacturing.

The controls research project emphasized development of a formal theory for machine control, with testing facilities in which matching hardware could be coupled with the new instructions for trial and demonstration.

By 1993, a hierarchical program for machine control was in use in the division. Called the Real-Time Control System (RCS), it could be used in conjunction with job-specific control programs. The RCS program incorporated explicit software subroutines to generate machine actions, process sensor data, and assess the machine world model. Assignments in the program were decomposed by tasks, using generic subprograms to correlate machine motions. John A. Horst and Anthony J. Barbera prepared a discussion of one use of the RCS under joint sponsorship of NIST and Advanced Technology and Research, Incorporated, a local engineering firm. [67]

Another use of the Real-Time Control System hierarchy involved the Advanced Deburring and Chamfering System (ADACS). Keith Stouffer, John L. Michaloski, B. Russell, and Frederick M. Proctor described ADACS in a NIST Interagency Report.[68] In the ADACS, the machine operator was prompted by a graphical display to specify cutting parameters and edges to be chamfered on the part. The program then used the given information to configure and execute a plan for finishing the part. Sensory information was used in the cutting process to correct for small positional errors. The program was found to be successful in controlling the machining of individual parts.

Other efforts in the robotics division specialized in the processing of machine sensory data. A group under Ernest W. Kent focused on vision systems for use in real-time machine control. The team investigated both single-camera and multiple-camera systems, including scanning, non-uniform resolution, and visual input to the machine world model. Much of the sensory work dated back to 1981, when the Robotics Division—under Albus and his deputy Sidney Weiser—led the effort. Weiser, holder of several patents in the field, helped develop the first computer-controlled industrial assembly robot in 1959 while employed in industry.

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[67] J. A. Horst and A. J. Barbera, "Intelligent control system for a cutting operation of a continuous mining machine," *NISTIR 5142*, March 1993, 54 pp.

[68] K. Stouffer, J. L. Michaloski, B. Russell, and F. M. Proctor, "ADACS, an automated system for part finishing," *NISTIR 5171*, April 1993, 14 pp.

Because of the cost of downtime for robotic machines, another group, led by Ronald Lumia, turned its attention to off-line programming of robots. The trouble with off-line machine programming was that the model used to generate the programs almost never matched the actual workshop environment. One approach to the mismatch problem was the extensive use of sensory information to re-form the world view of the robot when it was placed in the workshop; sometimes that technique saved days of setup time.

Yet another effort, led by Kenneth R. Goodwin, sought to develop and validate standard test procedures for measuring robot performance. An industry group, the Robot Industries Association, collaborated in the effort.

The U.S. Army had an understandable interest in mobile robots. Maris Juberts directed the NIST mobile-robot effort, which, besides the Army, included the *Intelligent Vehicle Highway System* program of the Department of Transportation. The Army wanted a standard robot-control program for a variety of labor-saving applications; the DoT wished to develop a vision-based control system for autonomous highway driving. As part of the multi-faceted study, NIST researchers developed a vision-based program for robotic maneuvering on existing roads. Test courses on the NIST/Gaithersburg grounds, on a Maryland highway, and on a closed-loop track at the Montgomery County Police Training Academy were used to demonstrate and evaluate the program. The first tests of the "traveling robot" utilized paint lines on the road for guidance. The tests were completely successful—the system proved feasible even at night.

Factory Automation, Howard M. Bloom, chief

The Factory Automation Division took as its major focus the strengthening of information systems for the manufacturing industry. Information systems helped to streamline manufacturing techniques, reducing the cost of production and enhancing product quality. A crucial first step in establishing the project was to create a standard for the sharing of product data.

One of the first activities of the division became embodied in the *Product Data Exchange Specification*. The PDES was conceived as a non-proprietary, neutral standard for the transmittal of product information among various manufacturing applications. NIST developed a software medium for the manipulation of PDES data; it was described in some detail by Stephen N. Clark. [69]

A medium developed for exchange of PDES product models was a physical file format called STEP—*Standard for the Exchange of Product Model Data.* Clark also presented an early version of STEP.[70]

A whole series of subsequent publications from the division provided details of the maturing standard. The series, mostly published in the *NIST Interagency Report* format, included numbers 4353, 4528, 4538, 4573, 4577, 4612, 4629, 4641, 4683, 4684, and 4685.

[69] S. N. Clark, "NIST PDES toolkit: Technical fundamentals. National PDES testbed report series," *NISTIR 4335*, November 1990, 34 pp.

[70] S. N. Clark, "NIST working form for STEP: National PDES testbed report series," *NISTIR 4351*, November 1990.

The PDES system was applied to the apparel industry with the collaboration of the *Defense Logistics Agency* and the *American Apparel Manufacturing Association*. The short-term goal of the project was to develop a neutral data format for exchanging two-dimensional pattern data between two apparel computer-aided-design systems.

Chemical Science and Technology Laboratory

When NIST was formed from NBS, all chemical standards and research activities were incorporated into the Chemical Science and Technology Laboratory. First director of the CSTL was Harry S. Hertz, a graduate of MIT in analytical chemistry. Hertz joined NBS in 1973; by 1978, he was chief of the Organic Analytical Research Division. During 1992, Hertz became the deputy to Curt W. Reimann in the Office of Quality Programs.

Hratch G. Semerjian succeeded Hertz as CSTL director. Semerjian was trained at Brown University as a mechanical engineer. He came to NBS in 1977. An expert in combustion processes, Semerjian was named chief of the CSTL Process Measurements Division in 1991.

The program in CSTL included a wide variety of research projects gathered into eight divisions devoted to biotechnology, chemical engineering,[71] chemical kinetics and thermodynamics, inorganic analysis, organic analysis, chemical processes, surface and microanalysis, and thermophysics.

In the following pages, we provide brief synopses of projects in each of the CSTL areas.

Biotechnology, Lura J. Powell, chief

Protein Characterization

One of the first problems studied in the biotechnology area was the quantification of proteins. CSTL researchers Susan F. Stone and Rolf L. Zeisler and their colleague Glen E. Gordon, of the Department of Chemistry and Biochemistry at the University of Maryland, utilized the techniques of polyacrylamide gel electrophoresis and neutron activation analysis for the quantification studies. With these tools, they determined the amount of phosphorus associated with separated phosphoproteins by autoradiography and densitometry as a prelude to protein quantification. They found that they could measure quantities of phosphorus at levels as low as 0.2 micrograms.[72]

[71] The Chemical Engineering Division, located on the Boulder campus, consisted of sections devoted to the study of transport processes, systems dynamics, and properties of solids. During 1993, the division was disbanded. The various research groups were reassigned to other CSTL divisions or to the Materials Science and Technology Laboratory.

[72] S. F. Stone, R. L. Zeisler, and G. E. Gordon, "Quantitative determination of proteins using polyacrylamide gel electrophoresis and neutron activation," *Trace Element Analytical Chemistry in Medicine and Biology* 5, pp. 157-166 (1988).

Another early problem tackled by the new biotechnology staff was the development of reliable biosensors. Edith S. Grabbe and Dennis J. Reeder sought to immobilize bioactive species on a substrate without simultaneously destroying its chemical activity. The first step in such a process, it seemed to Grabbe and Reeder, was to determine the structure of immobilized protein films. They decided to employ the technique of total internal reflection fluorescence to characterize layers of the protein immunoglobulin G (IgG) on thin nylon films. The two researchers studied three features of such systems: energy transfer between IgG tagged with fluorescein isothiocyanate and nylon tagged with tetramethylrhodamine isothiocyanate; binding activity, using labelled antigens; and energy transfer between labelled IgG and labelled antigens.[73]

Center for Advanced Research in Biotechnology

The Center for Advanced Research in Biotechnology (CARB) became a reality in 1984, the first major joint venture involving NBS in the biotechnology field (see *Restructuring NIST* in this chapter). As NBS became NIST in 1988, the Center received as its new director Thomas L. Poulos, professor of biochemistry at the University of Maryland. Walter J. Stevens, a veteran of 13 years at NBS as a theoretician in the field of chemical physics, was selected as associate director.

Initial research at the Center focused on five areas:

- Crystal structures of proteins, mainly by x-ray crystallography.
- Structure of proteins in solution, mainly by two-dimensional nuclear magnetic resonance spectroscopy.
- Modeling of protein structures, using a large, dedicated computer.
- Characterization of protein properties, using the methods of physical biochemistry.
- Production of proteins in quantity by means of molecular biology.

Scientists in the Center commenced their studies on the proteins ribonuclease, chymosin, cytochrome P450, and beta-lactamase, working mainly in NIST laboratories until CARB facilities were completed. The NIST scientists studied jointly with collaborators from universities, other government laboratories, and biotechnology firms.[74]

Gary Gilliland and his collaborators Alex Wlodawer of the National Cancer Institute, Joseph Nachman, a guest worker from Isreal, and Evon Windborne of the University of Maryland determined the structure of chymosin, one of the best-known commercial enzymes. The protein, used in cheese production under the name rennin, was composed of 323 amino acids. The group determined its three-dimensional structure,

[73] E. S. Grabbe and D. J. Reeder, "Characterization of adsorbed protein layers on thin nylon films with fluorescence energy transfer between protein and nylon: A method for biosensor analysis," *Proc. 4th European Conf. On the Spectroscopy of Biological Molecules, York, England, September 1-6, 1991*, pp. 321-322.

[74] John S. Makulowich, "Biotech center names top officials," pp. 20-21 in "NIST Research Reports," *NIST Special Publication 743*, October 1988.

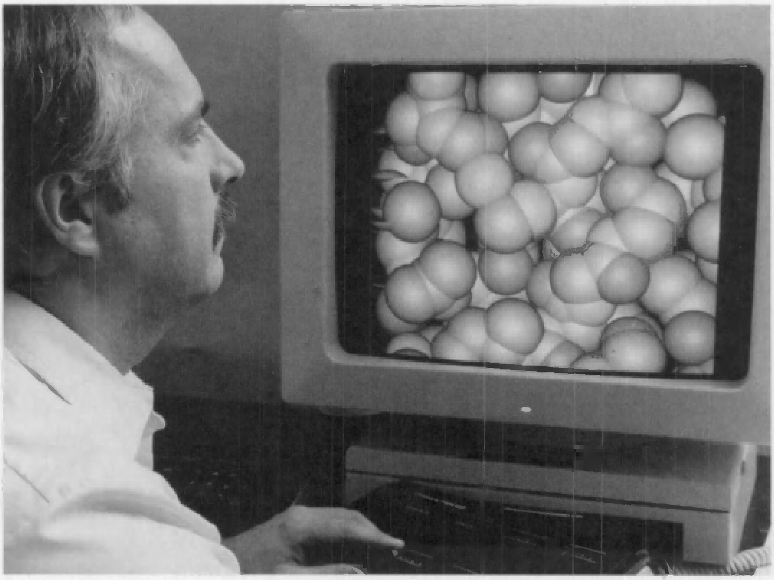
New state-of-the-art laboratory facilities for the Center for Advanced Research in Biotechnology were dedicated in November 1989 in Rockville, Maryland.

mapping the pattern with a resolution of about 0.3 nm. The work was particularly interesting because a form of chymosin was known to occur in human blood, where it played a role in regulating blood pressure.[75]

Acting NIST Director Raymond G. Kammer helped dedicate the first building on the site of the new CARB laboratories on November 29, 1989. Montgomery County, Maryland financed the construction, leasing it to a foundation associated with the University of Maryland. A memorial to Isadore M. Gudelsky, head of a family foundation that gave the 50-acre site for CARB, was dedicated at the same time. The CARB parent organization, the Maryland Biotechnology Institute (MBI) of the University of Maryland, appointed a CARB Board of Overseers headed by Donald R. Johnson of NIST. At that time, the CARB staff numbered 40 people, including 10 principal investigators drawn from NIST and MBI, 12 staff scientists and technicians, 4 postdoctoral fellows, 5 graduate students, and several visiting scientists.[76]

[75] "High-resolution structure for chymosin solved," p. 22 in "NIST Research Reports," *NIST Special Publication 743*, October 1988.

[76] Roger Rensberger, "Biotechnology research facility dedicated," pp. 16-17 in "NIST Research Reports," *NIST Special Publication 770*, January 1990.



NIST research scientist Gary Gilliland studied a model of the structure of the protein chymosin on a workstation screen at the Center for Advanced Research in Biotechnology.

By 1990, a CARB group including Keith McKenney and Prasad Reddy of NIST, and Joel Hoskins, Kalidip Choudhury, and Marc Kantorow of the University of Maryland were studying the sequencing of DNA. McKenney and Reddy focused on *E. coli,* a relatively simple, single-celled bacterium, to discover the mechanisms by which genes transcribed the genetic code into particular proteins and by which DNA molecules replicated themselves.

Chemical Engineering, Larry L. Sparks, chief

Pulse Tube Refrigerators

During the tenure of John Lyons, a group of Boulder researchers, including Wayne Rawlins, Ray Radebaugh—the group leader—Kalidip Chowdhury, James E. Zimmerman, Peter J. Storch, Lori K. Brady, and Peter E. Bradley, conducted extensive studies of cryocoolers. These small, durable devices were useful in the many situations where cooling was necessary in remote locations, or where working temperatures exceeded those provided by liquefied helium or hydrogen gases. Certain infrared detectors required a temperature environment as low as 80 K. Parametric amplifiers used in tracking satellites needed to be kept below 20 K. Cryocoolers also could be found in the semiconductor industry and cooling gamma-ray detectors in space. It was the hope of the Boulder group that cryocoolers would be useful for long-term cooling of high-temperature superconductivity products such as quantum interference devices.

At the Center for Advanced Research in Biotechnology Molecular Biology Laboratory, (in the foreground left to right) University of Maryland chemist Joel Hoskins, graduate student Kalidip Choudhury, NIST molecular biologist Dr. Keith McKenney, and graduate student Marc Kantorow examined the results of a DNA sequencing experiment.

In a 1990 paper, Radebaugh noted the development of pulse tube refrigerators—variations of the Stirling-cycle cooler. He described three types of the devices; basic, resonant, and orifice, emphasizing the evaluation—both theoretically and experimentally—of the cooler efficiency.[77] Along with Peter Storch and James Zimmerman, Radebaugh analyzed the operation of orifice pulse tube refrigerator—a difficult system to model—and compared the analysis with the results of experiments in the Boulder laboratory. Accurate prediction of the refrigeration power remained elusive, although a single-stage version of the device reached a no-load minimum temperature of 60 K, and it could remove 12 W of power at 80 K.[78]

[77] Ray Radebaugh, "A review of pulse tube refrigeration," *Advances in Cryogenic Engineering* **35**, pp. 1191-1205 (1990).

[78] Peter J. Storch, Ray Radebaugh, and James E. Zimmerman, "Analytical model for the refrigeration power of the orifice pulse tube refrigerator," *NIST Technical Note 1343*, December 1990, 86 pp.

In other work on pulse tube refrigerators, Rawlins and Radebaugh studied the performance of the regenerator portion; and Radebaugh, Chowdhury, and Zimmerman examined the effect of pulse frequency variation on the performance. Bradley and Radebaugh studied the effectiveness of regeneration in a three-stage Vuilleumier refrigerator, capable of reaching third-stage temperatures below 20 K.

Space Shuttle Engineering

Boulder cryogenic engineering researchers contributed in several ways to the NASA space shuttle program. In one project, James D. Siegwarth evaluated the performance of vortex shedding cryogenic flowmeters for use with liquid oxygen supplies to the main engines of the shuttle. Siegwarth found that the flowmeters maintained nearly their full design accuracy even though the necessary placement of the instruments was far from ideal.[79]

Siegwarth, Andrew J. Slifka, Larry L. Sparks, D. Chaudhuri, R. Compos, and T. J. Morgan obtained values of the coefficient of friction of Type 440C stainless steel over an extended range of temperature for the benefit of the space shuttle as well. The project was important to NASA as a factor in evaluating the life expectancy of bearings made of type 440C steel to be used in a high-pressure oxygen turbo-pump. In 1990, the group varied the experimental temperature from −140 °C to 350 °C and measured the sliding friction under loads from 1 GPa to 3.6 GPa and at speeds from 0.5 m/s to 2 m/s.[80] During the following year, the measurements were extended to both lower and higher temperatures.[81]

Cryogenic Fluid Flow

A number of projects on fluid flow took place during this period. A method was developed for the production of "slush" hydrogen—a mixture of liquid and solid hydrogen—by James A. Brennan, Roland O. Voth, and Paul R. Ludtke. Simplicity itself, the technique consisted of freezing hydrogen on the inside of a tube by immersing it in liquid helium (boiling point about 4 K, well below the 20 K freezing temperature of hydrogen) and rotating an augur to break up and remove the frozen particles.[82]

[79] J. D. Siegwarth, "Vortex shedding flowmeters for space shuttle main engines," *Proc. Conf. on Advanced Earth-to-Orbit Propulsion Technology, Huntsville, Alabama, May 10-12, 1988* **2**, 429-441.

[80] A. J. Slifka, J. D. Siegwarth, L. L. Sparks, and D. Chaudhuri, "Coefficient of sliding friction of 440C as a function of temperature," *NASA Conference Publication 3092* **1**, pp. 123-134 (1990).

[81] A. J. Slifka, R. Compos, T. J. Morgan, and J. D. Siegwarth, "Tribological behavior of 440C martensitic stainless steel from −184 °C to 750 °C," *Advances in Cryogenic Engineering Materials* **38**, part A, pp. 323-330 (1991).

[82] R. O. Voth, P. R. Ludtke, and J. A. Brennan, "Producing hydrogen slush with a small augur," *NASP Technical Memorandum 1099*, May 1990, 12 pp.

Brennan, Jennifer L. Scott, Charles F. Sindt, and Michael A. Lewis evaluated the effect on accuracy of cryogenic orifice flowmeters exerted by various flow conditioners. Such inserts as tubing bundles, star and Zanker conditioners, and pressure tap locations were considered in an effort to improve industry standards for the installation of orifice-type flowmeters.[83]

Thermophysical Properties of Cryogenic Fluids

An extensive, multi-purpose computer program to facilitate the availability of thermophysical data on cryogenic fluids was offered through the NIST Standard Reference Data Program from 1986.[84] Prepared by Robert D. McCarty, the program was called MIPROPS. In 1992, the program was updated and expanded by James Brennan, Daniel G. Friend, Vincent D. Arp, and McCarty. The revised program employed 32-term equations of state, augmented by equations for the ideal gas specific heat, vapor pressure *vs* temperature, melting pressure vs temperature, saturated liquid and vapor densities vs temperature, and viscosity and thermal conductivity information. In constructing the database, some 90 constants were critically evaluated for each of the 17 fluids listed below:

argon	isobutane	normal butane
carbon dioxide	carbon monoxide	deuterium
ethane	ethylene	helium
normal hydrogen	para hydrogen	methane
nitrogen	nitrogen trifluoride	oxygen
propane	xenon	

The improved program was designed to run on desktop computers, and to provide calculated values of many thermophysical properties over the range of base parameter values.[85]

Chemical Kinetics and Thermodynamics, Sharon G. Lias, chief

Combustion Kinetics

Rate constants for the unimolecular decomposition reactions and the reverse radical combination processes of four simple alkanes—methane, ethane, propane, and isobutane—were determined in 1989 by Wing Tsang.[86] The project was part of a larger program to develop a base of critically evaluated data on chemical reaction rates for combustion, sponsored by the U.S. Department of Energy.

[83] J. L. Scott, C. F. Sindt, M. A. Lewis, and J. A. Brennan, "Effects of flow conditioners and tap location on orifice flowmeter performance," *NIST Technical Note 1352,* October 1991, 73 pp.

[84] R. D. McCarty, "Interactive FORTRAN programs for micro computers to calculate the thermophysical properties of twelve fluids (MIPROPS)," *NBS Technical Note 1097,* 1986.

[85] J. A. Brennan, D. G. Friend, V. D. Arp, and R. D. McCarty, "Computer program for computing the properties of seventeen fluids," *Cryogenics* **32**, No. 2, 212-214 (1992).

[86] W. Tsang, "Rate constants for the decomposition and formation of simple alkanes over extended temperature and pressure ranges," *Combustion and Flame* **78**, No. 1, pp. 71-86 (1989).

The temperature range covered by Tsang's study, 300 K to 1100 K, was more extensive than had previously been attempted in such a work. Consequently, he employed a more complex theoretical treatment than in previous discussions, with specific modifications for particular species. Despite his care, Tsang was not completely satisfied with the results; he was not able to find a reasonable closed form for calculating rate constants that would represent all of the experimental data. In part, the difficulty of describing the data lay in experimental inaccuracies, but more influential in Tsang's view were the complicated dependencies of the rate constants on temperature, pressure, and the nature of secondary ambient species.

For each of the alkanes studied, Tsang provided limiting high-pressure rate expressions for the decomposition and the corresponding recombinations.

In 1991, Tsang and Joseph M. Herron completed an evaluation of a database consisting of kinetic information on a number of elementary reactions involving small polyatomic molecules important to propellant combustion. The work—on which the authors spent a full year—was accomplished under the aegis of the Standard Reference Data System; it was reported in the Journal of Physical and Chemical Reference Data.[87] Both experimental and estimated data were included in the study. Data covered the temperature range from 500 K to 2500 K and the density range from 10^{17} particles per cm^3 to 10^{22} particles per cm^3. Reactions of the following species were considered: H, H_2, H_2O, O, OH, HCHO, CHO, CO, NO, NO_2, HNO, HNO_2, HCN, and N_2O.

Kinetics From Flash Photolysis Experiments

As part of a continuing study of peroxy radicals—important as reactive intermediates in combustion and in atmospheric oxidation of organic compounds—Philippe Dagaut, a guest worker from the CNRS in Orleans, France, and Michael J. Kurylo investigated the absorption spectrum and the reaction kinetics of the neopentylperoxy radical, $(CH_3)_3CCH_2O_2$.

The technique used by the researchers was gas-phase flash photolysis. They determined the absorption cross section at 298 K and 250 nm to be about 5×10^{-18} cm^2/molecule. Coupling flash photolysis with spectroscopic observation of the radical at 250 nm, they also determined the rate constant for self-reaction—chemical reaction of the radical with itself—over the range 228 K to 380 K in low-pressure nitrogen gas.

Theirs was the first spectral analysis of the neopentylperoxy radical in the measured ranges.[88]

Dagaut, Kurylo, and their colleagues Renzhang Liu of NIST and Timothy J. Wallington, a guest worker from the Ford Motor Company, also used a flash photolysis technique—involving resonance fluorescence—to obtain absolute rate constants

[87] W. Tsang and J. M. Herron, "Chemical kinetic data base for propellant combustion. I. Reactions involving NO, NO_2, HNO, HNO_2, HCN, and N_2O," *J. Phys. Chem. Ref. Data* **20**, No. 4, pp. 609-663 (1991).

[88] P. Dagaut and M. J. Kurylo, "Flash photolysis investigation of the gas phase UV absorption spectrum and self-reaction kinetics of the neopentylperoxy radical," *International J. of Chem. Kinetics* **22**, No. 11, 1177-1187 (1990).

for the gas phase reactions of hydroxyl radicals with a series of dioxanes and other cyclic ethers. The authors compared the new results with similar ones obtained for aliphatic ethers, seeking to predict the reaction rates from group reactivity values.[89]

The work was part of a larger program to study trends in the reactivity of hydroxyl radicals in the gas phase. On the basis of their investigations, the authors had developed a scale of reactivity useful for the prediction of rate constants for simple ketones, alcohols, and ethers. The hydroxyl study filled a gap in the literature, significant because of the possible role of the reactions in industrial atmospheric chemistry.

Hydroxide Thermodynamics

New experimental and theoretical data on alkaline earth monohydroxides and dihydroxides caused Malcolm W. Chase and Rhoda D. Levin to re-evaluate the relevant thermodynamic properties. Recent spectroscopic investigations had provided new values for the molecular structure, vibrational frequencies, and electronic energy levels of several hydroxides.

Early studies of magnesium dihydroxide—"milk of magnesia"—and calcium dihydroxide—"hydrated lime"—simply provided information on substances used in the daily lives of American citizens. Later studies of colors in flames led to observations on the monohydroxides of calcium, strontium, and barium. Still later, propellant investigations focused on the monohydroxides of beryllium and magnesium. Gradually, the data were organized as part of joint Army, Navy, and Air Force thermodynamic data compilations.

Chase and Levin brought up to date the information available up to 1989 on molecular structure, entropy, and enthalpy of formation of the monohydroxides of beryllium, magnesium, calcium, strontium, and barium, with emphasis on the gas phase.[90]

Estimating Thermodynamic Properties

Eugene S. Domalski and Elizabeth D. Hearing extended earlier estimation methods, used for calculating gas-phase thermodynamic properties of organic substances, to the liquid and solid phases for a variety of hydrocarbons at room temperature. One scheme in particular, derived by S. W. Benson and his colleagues over a period of years, correlated molecular structure with a corresponding energy contribution to a thermodynamic property, achieving good agreement between calculated and experimentally derived values. Domalski and Hearing used the Benson approach to estimate thermodynamic properties for liquid and solid hydrocarbons at 298.15 K. Among the properties considered were enthalpy of formation, heat capacity, and entropy.

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[89] Philippe Dagaut, Renzhang Liu, Timothy J. Wallington, and Michael J. Kurylo, "Flash photolysis resonance fluorescence investigation of the gas-phase reactions of hydroxyl radicals with cyclic ethers," *J. Phys. Chem.* **94**, No. 5, 1881-1883 (1990).

[90] Malcolm W. Chase and Rhoda D. Levin, "Thermodynamic properties of the alkaline earth hydroxides: a JANAF case history," *High Temperature Science* **26**, pp. 207-214 (1990).

The two researchers made some 1300 comparisons between theory and experimentally derived quantities. The differences were scarcely larger than the uncertainty values often reported with experimental data, demonstrating the versatility of the Benson approach.[91]

In a later investigation, Domalski and Hearing carried the method to the liquid and solid phases of organic compounds containing the elements carbon, hydrogen, oxygen, sulfur, and the halogens. Comparisons between literature values of thermodynamic properties and estimated values were obtained for more than 1500 compounds at 25 °C.[92]

Inorganic Analysis, James R. DeVoe, chief

Analyzing Fluorides

In 1990, fluoride glasses appeared likely to challenge silica fibers as choice materials for conveying messages via optical fibers. Theoretical calculations of the intrinsic power losses at communications wavelengths—about 2 μm—of heavy metal glasses composed of zirconium, barium, lanthanum, aluminum, and sodium fluorides indicated values as low as those of silica. To achieve low intrinsic losses, however, the fluoride glasses had to be nearly free of interfering impurities such as divalent iron, copper, cobalt, nickel, and neodymium—perhaps at the picogram-per-gram level. A second caution was the possible presence of scattering centers such as oxides and oxyfluorides.

In order to prepare fluoride glasses with the extreme purity levels indicated by the calculations, it was crucial that analytical methods be developed to detect specific impurities at the requisite levels. A research team composed of members from NIST—Ellyn S. Beary, Paul J. Paulsen, and T. C. Rains—and from the U.S. Naval Research Laboratory—K. J. Ewing, J. Jaganathan, and I. Aggarwal—devised chemical preparative techniques and selected sensitive analytical instruments that appeared capable of preparing and analyzing fluoride glass samples at the required impurity levels. Following the line of reasoning taken by the group indicates the state of materials preparation and analysis characteristic of that period.

First to be considered was the use of a "clean room" to minimize contamination of the glass blanks by particulate matter in the originating laboratory. Such a facility was available on the NIST Gaithersburg site. Use of quartz or teflon laboratory vessels was preferred over ordinary glassware or plastics. Reagent-grade chemicals were purified with respect to some 37 elements to the part-per-billion level or better.

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[91] E. S. Domalski and E. D. Hearing, "Estimation of the thermodynamic properties of hydrocarbons at 298.15 K," *J. Phys. Chem. Ref. Data* **17**, pp. 1637-1678 (1988).

[92] E. S. Domalski and E. D. Hearing, "Estimation of the thermodynamic properties of C-H-N-O-S-Halogen compounds at 298.15 K," *J. Phys. Chem. Ref. Data* **22**, pp. 805-1159 (1993).

The group discussed methods of glass preparation, emphasizing the care needed to obtain meaningful analytical samples to track the quality of the process.

Various analytical methods were discussed for the project, including atomic absorption spectroscopy, mass spectrometry, neutron activation analysis, and absorption loss spectrometry. In their initial laboratory efforts, the group chose atomic absorption spectroscopy coupled with electrothermal atomization in a graphite furnace (GFAAS).

Detailed examination of the NIST clean room produced measureable amounts—from less than 5 ng/g to 600 ng/g—of iron, cobalt, nickel, and copper in a hood and two ovens, with iron by far the most persistent. Similarly, measureable quantities of the same elements appeared in three NIST "ultra-pure" acids, with iron and nickel the most prevalent. Four lanthanum salts, to be used in glass preparation, yielded ng/g amounts of neodymium impurity.[93]

Clearly, preparation of "pure" fluoride glasses would be a challenge.

Analysis by Mass Spectrometry

Analysis of sulfur in steel was important for a simple reason—the toughness of certain steel formulations was known to increase rapidly as the sulfur content dropped below about 100 ppm. W. Robert Kelly, LeTian Chen—a guest scientist from the Academia Sinica in Beijing, China—John W. Gramlich, and Karen E. Hehn, recognizing the relatively poor accuracy of existing methods, attacked the steel problem using an isotope dilution technique developed at NIST for the determination of sulfur concentration.

Carefully mixing a known amount of ^{34}S with a measured amount of steel samples, the group performed thermal ionization mass spectrometry to compare the $^{34}S/^{32}S$ ratio in the spiked samples with the ratio in the original steel samples. The method proved accurate within about 3 %, considerably better than techniques used previously. An additional benefit was the determination of the sulfur content of four steel Standard Reference Materials with the same accuracy.[94]

X-Ray Analysis Program

Peter A. Pella, B. Cross, and L. Feng—a colleague from China—collaborated on improvements to computer programs for the analysis of x-ray scattering data. Influence coefficient algorithms using fundamental parametric equations for correction of inter-element effects in x-ray analysis were employed both at NIST and in analytical laboratories in China. One of these, a Chinese variation of the Comprehensive

[93] E. S. Beary, P. J. Paulsen, T. C. Rains, K. J. Ewing, J. Jaganathan, and I. Aggarwal, "Approaches to the accurate characterization of high purity metal fluorides and fluoride glasses," *J. Crystal Growth* **106**, 51-60 (1990).

[94] W. Robert Kelly, LeTian Chen, John W. Gramlich, and Karen E. Hehn, "Determination of sulphur in low sulphur steels by isotope dilution thermal ionization mass spectrometry," *Analyst* **115**, 1019-1024 (1990).

Algorithms of Lachance called the FLY-FPM program, served as the basis on which the group developed a new program for use with spectrometers that incorporated both tube and secondary targets for excitation. The new program complemented others in use at NIST.[95]

Ion Exchange Chromatography

One of the analytical techiques in common use at NIST for the characterization of Standard Reference Materials at this time was ion exchange chromatography. The principal use of the method was for the determination of non-metals such as sulfur and chlorine. William F. Koch reviewed newly developed procedures and instrumentation adapted to improve the accuracy and precision of ion chromatography, emphasizing critical features of sample preparation for the analysis of fuels, botanical, and other biological material.[96]

There were many occasions in analytical chemistry when it was important to know the chemical state of a metal rather than just its concentration. Liquid chromatography was useful for those occasions, since it could separate different chemical forms of an element. However, the dilution that accompanied the chromatographic process placed a premium on sensitive detection of the species. Gregory C. Turk, William A. MacCrehan, Katherine S. Epler, and T. C. O'Haver found that laser-enhanced ionization not only was the most sensitive method for use in flame atomic spectroscopy, but it was also well-suited to the liquid chromotographic technique. The group illustrated the method with observations on the trialkyltin compounds.[97]

Organic Analysis, Willie E. May, chief

Analyzing River Sediment

A new Standard Reference Material, SRM 1939, useful in determining the concentrations of trace organic constituents of river sediment, was developed by a group including Richard E. Rebbert, Stephen N. Chesler, F. R. Guenther, Barbara J. Koster, Reenie M. Parris, Michelle M. Schantz, and Stephen A. Wise.[98] Samples for SRM 1939, "Polychlorinated Biphenyls in River Sediment A," were collected from the Hudson River. The new standard joined SRM 1941, "Organics in Marine Sediment"—collected from Baltimore Harbor—in the SRM catalog.

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[95] L. Feng, P. A. Pella, and B. Cross, "Versatile fundamental alphas program for use with either tube or secondary target excitation," *Advances in X-Ray Analysis* **33**, pp. 509-514 (1990).

[96] W. F. Koch, "Ion chromatography and the certification of Standard Reference Materials," *J. Chromatographic Science* **27**, No. 8, pp. 418-421 (1989).

[97] G. C. Turk, W. A. MacCrehan, K. S. Epler, and T. C. O'Haver, "Laser-enhanced ionization as an element-specific detector for liquid chromotography," *Proc. 4th International Symposium on Resonance Ionization Spectroscopy and its Applications, Gaithersburg, MD, April 10-15, 1988*, pp. 327-330 (1989).

[98] Richard E. Rebbert, Stephen N. Chesler, F. R. Guenther, Barbara J. Koster, Reenie M. Parris, Michelle M. Schantz, and Stephen A. Wise, "Preparation and analysis of a river sediment Standard Reference Material for the determination of trace organic constituents," *Fresenius' J. Anal. Chem.* **342**, pp. 30-38 (1992).

The two reference materials were intended for use in marine monitoring projects (SRM 1941) and for cleanup of polychlorinated biphenyl (PCB) spills (SRM 1939). The latter reference featured certified values for three derivatives of PCB and data for some 14 other derivatives. Information was included also on chlorinated pesticides and polyaromatic hydrocarbons present in the samples. The U.S. Environmental Protection Agency supplied bulk samples collected from the Hudson River to NIST for preparation of the standard materials.

Sample preparation for SRM 1939 illustrated the realistic approach to standards that characterized scientific work at NIST. Rather than producing a synthethic river sediment for calibration of environmental monitoring instruments, NIST derived its reference standard from actual river "mud."

Step one consisted of removal from the 55-gallon samples all large debris. The liquid-solid mixture was then blended and progressively sieved to remove all particles larger than about 45 μm. The remaining sediment, about 40 kg, was sterilized by exposure to more than 3 Megarads of ^{60}Co radiation and bottled in 50 g quantities. Uniformity of particle size was ascertained to assure even distribution of PCB contamination.

Four different analytical procedures were employed to determine the amounts of various classes of organic compounds in the reference material. The use of multiple procedures satisfied the SRM certification requirement that at least two independent methods be used to evaluate specified quantities of any certified species.

By the time SRM 1939 joined the more than 1000 other reference materials in the OSRM catalog, it was without peer for the calibration of marine PCB monitors.

Examining Human Plasma

Interest within the biomedical community in the efficacy of an extract of licorice root as a cancer preventative in humans led a NIST research group to develop a new analytical method in 1991. The licorice-root derivative identified as potentially defying cancer was 18β–glycyrrhetinic acid (GRA). Basic to any cancer-related studies of the substance was accurate evaluation of its concentration in human blood plasma. Among the several existing techniques for the determination of GRA concentrations in plasma, none offered a good combination of sensitivity, accuracy, and convenience.

The research team, Jeanice M. Brown-Thomas, Richard G. Christensen, Winfred Malone, and Willie E. May of NIST, and Roland Rieger, a colleague from the University of Ulm in Germany, developed an analytical method based upon the use of high-performance liquid chromatography (HPLC). An important feature of their technique was the preparation of an internal standard, composed of an easily identified GRA acetate derivative. A measured quantity of this substance was added to the plasma samples. Extraction of the GRA and its derivative by the HPLC method was followed by ultraviolet absorbance detection at 248 nm.

The method proved sensitive to concentrations of GRA in blood at the 10 ng/g level; quantitative determinations appeared feasible at the 100 ng/g level with 10 % uncertainty.[99]

Carcinogens in Coal Tar

Coal tar, a common ingredient of urban daily life, was known to harbor many carcinogenic compounds. A Standard Reference Material, SRM 1597, "Complex Mixture of Polycyclic Aromatic Hydrocarbons (PAH) From Coal Tar," was issued by NIST in 1987 to assist in analysis of various samples of coal tar. The NIST development team, Stephen A. Wise, Bruce A. Benner, G. D. Byrd, Stephen N. Chesler, Richard E. Rebbert, and Michelle M. Schantz, had provided certification or other basic information on some 30 polycyclic aromatic compounds in preparing SRM 1597.

It was clearly advantageous to refine the analysis of the reference material, however, to analyze the numerous methyl-substituted PAH compounds. The biological activity of the isomers varied significantly, and certain of the methyl isomers were known to be more active carcinogens than the parent compounds.

To improve NIST understanding of its SRM, Stephen Wise took advantage of an opportunity to collaborate with Philippe Garrigues and Jacqueline Bellocq of the Physico-Chemical Oceanography group at the University of Bordeaux in France, who had some familiarity with the problem compounds. The group used a combination of liquid chromatography and high-resolution Shpol'skii spectroscopy—a low-temperature fluorescence technique—to determine the concentrations of 12 methyl isomers of benzopyrene, some of the most virulent of the known carcinogens. It was the first unambiguous identification and quantification of these substances in a coal tar.[100]

Chemical Processes, Hratch G. Semerjian, chief; succeeded by Gregory J. Rosasco in 1992

A New Scale of Temperature is Adopted, New Thermometry Projects Begin

The *International Temperature Scale of 1990* (ITS-90), the fifth international temperature scale since the Treaty of the Meter in 1875, was adopted by the International Committee of Weights and Measures during its meeting in 1989. Authority for the adoption had been granted by the 18th General Conference of Weights and Measures in 1987.[101] The new scale incorporated the results of numerous NBS/NIST projects in thermometry. Some of these projects were mentioned in Ch. 4, others were of more recent origin. By no means were all of the projects discussed below carried out in the Process Measurements Division—they are gathered here to give coherence to the discussion.

[99] Jeanice M. Brown-Thomas, Richard G. Christensen, Roland Rieger, Winfred Malone, and Willie E. May, "Determination of glycyrrhetinic acid in human plasma by high-performance liquid chromatography," *J. Chromatography* **568**, pp. 232-238 (1991).

[100] Philippe Garrigues, Jacqueline Bellocq, and Stephen A. Wise, "Determination of methylbenzo(a)pyrene isomers in coal tar standard reference materials using liquid chromatography and Shpol'skii spectrometry," *Fresenius J. Anal. Chem.* **336**, 106-110 (1990).

[101] H. Preston-Thomas. "The International Temperature Scale of 1990 (ITS-90)," *Metrologia* **27**, 3-10 (1990).

NIST Contributions to ITS-90

The ITS-90 was defined according to different physical principles in different ranges. Underlying all the ranges were more than a dozen reference temperatures, assigned specific values that were intended to tie the ITS-90 closely to the Kelvin thermodynamic scale. From 20 K to 1000 K, gas thermometry measurements were relied upon to provide approximate Kelvin-scale values for the defining fixed points of the ITS-90; in the range 273 K to 1000 K, the defining gas thermometry was accomplished by Leslie A. Guildner, Robert E. Edsinger, Richard L. Anderson, and James F. Schooley at NBS.

The assigned temperature of the gold freezing point was determined by Klaus D. Mielenz, Robert D. Saunders, Jr., and John B. Shumaker (see below).

Many of the devices and procedures used to create fixed-point reference temperatures in the laboratory originated in part or *in toto* at NBS/NIST. Over many years, fixed-point research was performed by a group led by George T. Furukawa. Other members of the group were William R. Bigge, John L. Riddle, and Earl R. Pfeiffer. Their investigations provided needed data on the triple points of neon, oxygen, argon, mercury, and water, and the freezing points of tin, zinc, and aluminum. Billy W. Mangum and Donald D. Thornton performed similarly successful development of gallium and indium fixed-point devices.

The defining relations in the various portions of ITS-90 were as follows:

- Between 0.65 K and 5.0 K, ITS-90 temperatures were obtained from defined relations between ^{3}He and ^{4}He vapor pressures and temperature.
- Between 3.0 K and 24.6 K (the neon triple point), ITS-90 temperatures were obtained from a gas thermometer, calibrated according to specific instructions.
- Between 13.8 K (the equilibrium hydrogen triple point) and 1235 K (the freezing point of silver), ITS-90 temperatures were obtained from defined relations between calibrated platinum resistance thermometers and temperature.
- Above 1235 K, ITS-90 temperatures were obtained from ratios of blackbody spectral radiances referred to the freezing point of silver, gold, or copper.

For the first time, the international temperature scale did not include a range defined by the electromotive force of thermocouple thermometers. High-temperature versions of the platinum resistance thermometer proved more precise all the way to the silver freezing point.

Temperature of the Freezing Point of Gold

One of the most significant measurements affecting the quality of ITS-90 in comparison to the Kelvin thermodynamic temperature scale was undertaken by Klaus D. Mielenz, Robert D. Saunders, Jr., and John B. Shumaker. In work completed just before the ITS-90 input was finalized, the NIST group offered a first-principles measurement of the freezing point of gold, an especially important reference temperature because it was often used as the basis for radiation thermometry.[102]

[102] Klaus D. Mielenz, Robert D. Saunders, Jr., and John B. Shumaker, "Spectroradiometric determination of the freezing temperature of gold," *J. Res. NIST* **95**, No. 1, 49-67 (1990).

In evaluating the gold-point temperature, the authors used a specially designed heat-pipe blackbody furnace into which they could place sizeable (about 1 kg) gold samples. They also employed a laser-irradiated integrating sphere, a prism-grating double monochromator spectroradiometer, a silicon diode detector, and an electrically calibrated radiometer. They compared the spectral radiances of the gold-point blackbody and the laser-irradiated integrating sphere at three wavelengths, then calculated the gold-point temperature from the Planck law of spectral distribution.

The gold-point freezing temperature determined by Mielenz, Saunders, and Shumaker was 1337.33 K ± 0.34 K. Theirs was the only direct spectroradiometric determination of that temperature; it was immediately incorporated as one of the basic reference temperatures of the new scale.

Calibrations on ITS-90

In order to calibrate thermometers on the new ITS-90, a number of innovations in NIST thermometry were needed:

- Christopher W. Meyer and Martin L. Reilly characterized a standard piston gage for use in calibrations throughout the range 0.6 K to 25 K, where realization of the new scale demanded vapor-pressure measurements of ^{3}He and ^{4}He and classical gas thermometry as well.[103] The authors expected to match the new scale with an imprecision no larger than 10^{-3} K.
- Gregory F. Strouse undertook a series of experiments to evaluate the consequences of non-unique definitions of temperatures in the new scale over the range 84 K to 933 K. He found that the various possible realizations of the scale provided temperature values that were identical within the measurement uncertainties of the standard platinum resistance thermometers.[104]
- Along with his colleagues Billy W. Mangum of NIST and A. I. Pokhodun and N. P. Moiseeva of the D. I. Mendeleyev Institute of Metrology in St. Petersburg, Russia, Gregory Strouse studied the characteristics of new high-temperature platinum resistance thermometers. The objective was to determine the stability of the thermometers during use at high temperatures. Some 26 thermometers of several different configurations were evaluated in the study. The importance of frequent re-calibrations during high-temperature service was clearly demonstrated by the experiments, which showed substantial (as much as 5 °C) drifts in indicated temperatures following long-term exposure to temperatures in the upper reaches of their useful ranges.[105]

[103] Christopher W. Meyer and Martin L. Reilly "Gas dependence of the effective area of the piston gage to be used for the NIST realization of the ITS-90," pp. 133-138 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, Editor.

[104] G. F. Strouse, "Investigation of the ITS-90 subrange inconsistencies for 25.5 Ω SPRTs," pp. 165-168 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor. Further information on the realization of the new scale was contained in the two papers that followed this one in the proceedings.

[105] G. F. Strouse, B. W. Mangum, A. I. Pokhodun, and N. P. Moiseeva, "Investigation of high-temperature platinum resistance thermometers at temperatures up to 962 °C, and, in some cases, 1064 °C," pp. 389-394 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

Rapid-Response Thermometers

The need to measure temperatures quickly in his new pulse-tube refrigerator—discussed earlier in this section—led Ray Radebaugh to collaborate with W. Rawlins and K. D. Timmerhaus of the University of Colorado in the design and test of a new, high-speed thermometer.[106] As a temperature sensor, the authors settled on a platinum-plated tungsten wire only 4 μm in diameter. This probe was sufficiently robust to survive high gas-flow rates, yet it responded to temperature changes with a time constant less than 0.3 ms, providing the needed 30 Hz response time.

For many years there had been a continuing need for accurate thermometry in the hot, harsh environment found inside jet aircraft engines and automobile engines. Such engines generally performed more efficiently, the higher the operating temperature. As the temperature neared the softening temperature of the engine materials, however, there was danger of catastrophic failure. Before he came to NBS in 1973 from the United Aircraft Research Laboratories, Kenneth G. Kreider was well aware of this problem. In 1992, he summarized progress on the use of durable, fast-response thermocouple thermometers for such hostile environments.[107]

For use at temperatures up to 1300 K, thermocouples made in the form of thin (about 2 μm) plates of platinum and platinum-plus-10 % rhodium, protected by various refractory alloys and oxides, proved to be capable of response times of the order of 50 μs, yet also able to survive hostile environments.

A striking new development in high-speed, high-temperature thermometry was proffered in 1983 by Raymond R. Dils of the Thermal Processes Division. Dils utilized single-crystal sapphire to carry radiation from the heated zone of a test chamber to a radiometer, providing temperature values in the range 600 °C to 2000 °C. Dils prepared the "hot" end of the sapphire rod by sputtering on it a refractory metal coating, which approximated a blackbody cavity. Over a period of years, Dils and his colleagues refined the device, achieving speed of response, wide range of temperature, and surprisingly good thermometric accuracy.[108]

For many years, a laser-based method for the determination of temperatures in gases was used to provide information on conditions in harsh environments. Designated by the acronym CARS—Coherent Anti-Stokes Raman Spectroscopy—the technique required only optical access to the gaseous environment.

[106] W. Rawlins, K. D. Timmerhaus, and R. Radebaugh, "Resistance thermometers with fast response for use in rapidly oscillating gas flows," pp. 471-474 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

[107] Kenneth G. Kreider, "Thin-film thermocouples," pp. 643-648 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

[108] R. R. Dils, "High temperature optical fiber thermometer," J. Appl. Phys. 54, No. 3, pp. 1198-1201 (1983). See also R. R. Dils, J. Geist, and M. L. Reilly, "Measurement of the silver freezing point with an optical fiber thermometer: proof of concept," *J. Appl. Phys.* **59**, No. 4, pp. 1005-1012 (1986).

After a decade of work in which they patiently improved the accuracy of the technique at NBS/NIST, Gregory J. Rosasco, Vern E. Bean, and Wilbur S. Hurst proposed in 1990 that diatomic gases could simultaneously serve as primary standards for temperature and pressure in measurements by dynamic methods:[109]

> With modern laser diagnostic techniques, it is possible to characterize the pressure and temperature of a gas at the molecular level. The measurement times for these techniques are such that the response to changes in *T* and *P* is limited only by the fundamental relaxation and transport processes of the molecular system. This provides the basis for a new approach to the calibration of transducers used in the measurement of dynamical *P* and *T*.

The authors stated that the CARS technique permitted them to derive temperature and pressure data from measurements of the optical transitions between atomic or molecular energy levels in the gas under observation. Since the data were generated by nanosecond-length pulses from gas-volume elements of millimeter dimensions, extremely rapid and localized values of temperature and pressure could result.

Rosasco, Bean, and Hurst suggested that they could use the CARS technique to characterize the dynamic temperature and pressure profiles within, for example, a shock tube, with uncertainty levels of perhaps 5 % up to 10^8 Pa in pressure and 1500 K in temperature. These parameters represented entirely new levels of measurement capability for traditional standards.

New Thermocouple Functions

It had long been suspected (see Problems with an Unseasoned Temperature Scale in Ch. 2) that a simple quadratic function could not adequately represent the relation of the emf of standard type S thermocouples to temperature. Exhaustive experimentation demonstrated the need for higher-order reference functions. A wide-ranging collaboration produced the results, involving NIST experimenters George W. Burns, Gregory F. Strouse, and Billy W. Mangum; NIST statisticians M. Carroll Croarkin and William F. Guthrie; and experimentalist colleagues from seven other national laboratories.[110] The new functions consisted of a 9th-degree polynomial for the range –50 °C to 250 °C; another 9th-degree polynomial for the range 250 °C to 1200 °C; a 5th-degree polynomial for the range 1064 °C to 1665 °C; and a 4th-degree polynomial for the range 1664 °C to 1768 °C.

[109] Gregory J. Rosasco, Vern E. Bean, and Wilbur S. Hurst, "A proposed dynamic pressure and temperature primary standard," *J. Res. NIST* **95**, 33-47 (1990).

[110] G. W. Burns, G. F. Strouse, B. W. Mangum, M. C. Croarkin, W. F. Guthrie, P. Marcarino, M. Battuello, H. K. Lee, J. C. Kim, K. S. Gam, C. Rhee, M. Chattle, M. Arai, H. Sakurai, A. I. Pokhodun, N. P. Moiseeva, S. A. Perevalova, M. J. de Groot, Jipei Zhang, Kai Fan, and Shuyuan Wu, "New reference function for platinum-10 % rhodium versus platinum (type S) thermocouples based on the ITS-90," pp. 541-546 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

A type of thermocouple composed of gold vs platinum proved capable of precision at the level of 0.01 °C. NIST colleagues George Burns, Gregory Strouse, and Billy Mangum, in collaboration with B. M. Liu of the Shanghai Institute of Metrological Technology studied the stability of gold-platinum thermocouples and prepared reference functions for them based upon the ITS-90.[111]

Extending ITS-90 to Lower Temperatures

William E. Fogle, Robert J. Soulen, Jr., and Jack H. Colwell created a thermodynamically based temperature scale in the range 0.006 K to 0.65 K using Johnson noise thermometry, the melting curve of ^{3}He, and paramagnetic salt susceptibility.[112] They undertook their study to provide accurate thermometry for world-wide research into the properties of materials at extremely low temperatures—particularly the interesting quantum substance, ^{3}He. To construct the new scale, the authors had to bring known thermometry methods to new stages of perfection.

Expanding on initial experiments led by Robert A. Kamper and James E. Zimmerman[113] (See Thermometry with Superconductors in Ch. 2), Soulen and his colleagues spent a decade perfecting a noise thermometer based upon the use of a resistive superconducting quantum interference detector (R-SQUID). To accomplish this goal required careful circuit design and analysis, and painstaking comparison with other thermometers, including the nuclear orientation thermometer developed by Harvey Marshak (see *Nuclear Orientation Thermometry* in Ch. 4). Ultimately, the noise thermometer was considered to represent thermodynamic temperatures within an uncertainty of 0.1 % over the range 0.01 K to 0.7 K.[114]

In a similar way, the ^{3}He melting curve had to be transformed from a physics experiment into a reliable thermometer—not a primary thermometer, but an extremely useful interpolation device.[115] Using a capacitance cell borrowed from Dennis S. Greywall of the Bell Telephone Research Laboratory, a piston gauge borrowed from the NIST Pressure Group, and careful experimentation involving the R-SQUID noise thermometer mentioned earlier, the authors determined the ^{3}He melting curve (pressure vs temperature) over the range 0.005 K to 0.7 K with an uncertainty estimated at 0.1 %. The curve exhibited a minimum in pressure which the investigators utilized as a fixed point for pressure measurement.

[111] G. W. Burns, G. F. Strouse, B. M. Liu, and B. W. Mangum, "Gold versus platinum thermocouples: performance data and an ITS-90 based reference function," pp. 531-536 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

[112] W. E. Fogle, R. J. Soulen, Jr., and J. H. Colwell, "A new cryogenic temperature scale from 6.3 to 650 mK," pp. 91-96 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

[113] R. A. Kamper and J. E. Zimmerman, "Noise thermometry with the Josephson effect," *J.Appl. Phys.* **42**, 132-136 (1971).

[114] R. J. Soulen, Jr., W. E. Fogle, and J. H. Colwell, "A decade of absolute noise thermometry at NIST using a resistive SQUID," pp. 983-988 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

[115] J. H. Colwell, W. E. Fogle, and R. J. Soulen, Jr., "The ^{3}He melting curve thermometer as a universal temperature transfer standard," pp. 101-106 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

The temperature-dependent susceptibility of the paramagnetic salt cerous magnesium nitrate—prepared in powdered form and with a portion of the cerium atoms replaced by non-paramagnetic lanthanum atoms to extend its useful range to lower temperatures—was used to provide a check on the linearity of the new scale. The authors found the scale to be linear within 0.1 % over its entire range.

The ink was barely dry on the reports of the work before Soulen, Fogle, and Colwell were deeply involved in discussions with scientists at other laboratories on questions of low-temperature physics illuminated by the new scale.

Temperatures from Radiance Measurements

A suggestion that radiance temperatures of refractory metals might serve as useful temperature references in the range 1700 K to 3700 K came from Ared Cezairliyan, Archie P. Miiller, and their colleagues Francisco Righini and Antonio Rosso from the CNR Istituto di Metrologia "G. Colonnetti." The suggestion was based on the results of an exhaustive series of experiments on 10 metals—nickel, iron, palladium, titanium, zirconium, vanadium, niobium, molybdenum, tantalum, and tungsten. The experiments showed that the radiance temperature of each element measured was constant within about 1 K, as long as all measurements were performed at a fixed wavelength. A pronounced wavelength dependence (as much as 0.5 K per nm) was found. A similar dependence was observed for the spectral emissivity of each element.[116]

NBS/NIST Gas Thermometry—An Ending and a Beginning

For a century and a half, thermometrists pursued manometer-based gas thermometry to elucidate the thermodynamic temperature scale. No other fundamental method could match the accuracy of the gas bulb, thermostat, and manometer, operated by an experienced hand over an extended range of temperatures both above and below the ice point. During this period, however, the study of traditional gas thermometry at NBS/NIST came to a halt. To take its place there arose a more modern type of gas thermometry involving acoustical resonances. It was a sea-change in a fundamental measurement area.

The End of Classical Gas Thermometry at NBS/NIST

For decades, classical gas thermometry contributed heavily to the international understanding of the thermodynamic temperature scale. NBS/NIST scientists figured prominently in this effort. With the adoption of the International Temperature Scale of 1990, however, the NIST gas thermometry project came to an end.

Classical gas thermometry was deceptively simple in its principle. The idea was to enclose a gas (the "working gas") in a bulb, measure the pressure of the working gas at some reference temperature, heat (or cool) the bulb to a temperature whose value was to be determined, and to measure the gas pressure at the new temperature. The ratio of temperatures—on the thermodynamic, or Kelvin, scale—was the same as the ratio of the measured pressures, give or take a few corrections.

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[116] A. Cezairliyan, A. P. Miiller, F. Righini, and A. Rosso, "Radiance temperature and normal spectral emissivity of metals at their melting point as possible reference values," pp. 377-382 in *Temperature, Its Measurement and Control in Science and Industry* **6**, 1992, J. F. Schooley, editor.

The principle was so simple that many a thermometrist approached gas thermometry with a light heart and a song on his lips, only to find disaster. It turned out that the two ratios were identical only within strict limits:

- The working gas had to obey the Ideal Gas Law, or deviate from it in a way that could be accounted for with great accuracy. Impurities in the working gas—a common occurence—doomed many an experiment.

- All of the working gas had to feel the same temperature, a difficult constraint whenever the temperature to be evaluated was far from the reference temperature. Gas in the tubing used to fill and empty the bulb and to measure its pressure inevitably passed through a temperature gradient, complicating the analysis of the experiment.

- The manometric pressure measurement had to be highly accurate, a difficult requirement because of the large temperature dependence of the density of mercury—the most common manometric fluid.

The ability to find and evaluate the consequence of each of the hidden, or systematic, errors in gas thermometry determined its success or failure. Over and over again, gas thermometrists felt certain that they had achieved the desired thermodynamic accuracy in their experiments. Just as frequently, subsequent measurements proved them wrong. One experiment would founder on unsuspected impurities in the working gas; another, on imperfect manometry; a third, on poor temperature equilibrium within the measurement chamber.

The National Bureau of Standards entered the gas-thermometry arena in the 1920s. Carl S. Cragoe and T. B. Godfrey, realizing the central importance of manometry to successful gas thermometry, began a project on high-accuracy pressure measurement at that time. C. H. Meyers and R. D. Thompson later made improvements to their design.[117]

Harold F. Stimson, a leader in NBS thermometry for four decades, continued the manometry project, focusing on the use of constant-volume gas thermometers with the pressure of the working gas to be measured using mercury manometry. Stimson settled on the use of gage block end standards to determine the height difference between the lower arm of the manometer and its upper arm. Using these methods and carefully isolating the experiment from thermal disturbances, Stimson achieved estimated uncertainties of ±20 ppm in the density of the mercury, ±10 ppm in the gravitational constant, and ±2 ppm in the end standards.

In the late 1950s, a new team of NBS gas thermometrists was formed. The team leader was Leslie A. Guildner, trained in the science by James A. Beattie at MIT. Guildner's principal Bureau colleagues were Richard L. Anderson and Robert E. Edsinger. This team's destiny was to create the ultimate in gas thermometry, including a mercury manometer with an estimated overall uncertainty of only 1 ppm—arguably the most accurate ever constructed for its range.

[117] L. A. Guildner, H. F. Stimson, R. E. Edsinger, and R. L. Anderson, "An accurate mercury manometer for the NBS gas thermometer," *Metrologia* **6**, No. 1, pp. 1-18 (1970).

Developed on the Bureau's Connecticut Avenue site, the gas thermometry equipment reached its full potential after the move to the new Gaithersburg laboratories. The manometer was installed in a subterranean chamber of the Gaithersburg Physics building, two floors below the main laboratory and one floor below an aggressive air-handling system that regulated and measured the air temperature at the millidegree level. The gas-bulb system could be moved hydraulically from an ice bath to a stirred liquid bath without breaking any gas-handling connections. Four platinum resistance thermometers recorded the temperature of the gas bulb. Some 20 thermocouples measured the temperature distribution along the connecting tube to the manifold that controlled the initial cleanout of the bulb, its filling, and its pressure measurement. No pains were spared to minimize the errors that had deviled earlier experiments, particularly those associated with impurities in the working gas, ^{4}He.

The efforts put into the gas thermometer paid off when the first determinations were made of possible differences between the 1968 temperature scale and the Kelvin thermodynamic scale. Guildner and Edsinger found a difference between the two scales of 0.027 °C at 100 °C, five times larger than the estimated uncertainty of the 1968 scale at that temperature![118] From that moment on, international progress towards a new temperature scale awaited further NBS gas thermometry results.

Before Guildner retired from NBS, he and Edsinger produced gas thermometry results at temperatures as high as 457 °C, and he designed a new oven in which the experiment could be continued to 660 °C. At 457 °C, the 1968 scale appeared too high by 0.08 °C, far beyond previous estimates of its uncertainty with respect to thermodynamic temperatures.[119] Urged by the international thermometry community to continue the NBS project to its ultimate goal despite Guildner's retirement, division management asked James F. Schooley to collaborate with Edsinger to finish the work. Edsinger and Schooley published measurements from 230 °C to 660 °C in 1989.[120] In the region of temperature overlap with the earlier results, they found similar deviations between the IPTS-68 and thermodynamic temperatures, although the magnitude of the deviations appeared to be smaller than those that Guildner and Edsinger had found.

In formulating the International Temperature Scale of 1990, the Consultative Committee for Thermometry chose to use an average of the Guildner-Edsinger and Edsinger-Schooley gas thermometry results as the basis for the new scale in the range 0 °C to 660 °C.[121]

Shortly after completing their measurements on the NBS/NIST gas thermometer, both Edsinger and Schooley retired. The apparatus was never used again.

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[118] L. A. Guildner and R. E. Edsinger, *J. Res. NBS* **77A**, 383 (1973).

[119] L. A. Guildner and R. E. Edsinger, "Deviation of international practical temperatures from thermodynamic temperatures in the temperature range from 273.16 K to 730 K," *J. Res. NBS* **80A**, 703 (1976).

[120] R. E. Edsinger and J. F. Schooley, "Differences between thermodynamic temperature and t(IPTS-68) in the range 230 °C to 660 °C," *Metrologia* **26**, 95-106 (1989). See also "Back to the Basics," pp. 5-6 in "NBS Research Reports," *NBS Special Publication 735,* December 1987.

[121] R. L. Rusby, R. P. Hudson, M. Durieux, J. F. Schooley, P. P. M. Steur, and C. A. Swenson, "Thermodynamic basis of the ITS-90," *Metrologia* **28**, 9-18 (1991).

A New Range for Acoustic Thermometry

Measurements of the speed of sound in purified gases were used to determine thermodynamic temperatures in the cryogenic range for some time. Among others, Harmon Plumb and George Cataland derived a useful scale of temperature from 2 K to 20 K by means of acoustic thermometry in ^{4}He gas (see *Cryogenic Temperature Scales and Cryogenic Physics* in Ch. 1). Beginning in the late 1970s, however, Michael R. Moldover and several colleagues took acoustic thermometry into the range above room temperature, long the exclusive province of classical gas thermometry.

Instead of relying on the accurate measurement of pressure, as had Guildner, Edsinger, and many other thermometrists over a period of more than a century, Moldover sought to measure the acoustic resonance frequencies of an argon-filled sphere that could in principle be used at temperatures well above 0 °C. At vanishingly low pressures of the working gas, the thermodynamic value of a selected temperature could be calculated from the ratio of the speed of sound at that temperature to the speed of sound at a reference temperature, multiplied by the reference temperature value—usually chosen to be the triple-point temperature of water, 273.16 K.

Teaming at various times with colleagues Meyer Waxman, Martin Greenspan, James B. Mehl, J. P. M. Trusler, T. J. Edwards, Richard S. Davis, and M. B. Ewing, Moldover found that he could determine thermodynamic temperatures in this fashion with uncertainties as low as 10^{-3} K. One of the first such temperatures to be determined was the triple-point temperature of gallium.[122] A bonus in the experiments was the re-determination of the gas constant with an uncertainty of less than 2 ppm—smaller than the uncertainty found in previous measurements by a factor of five.[123]

As the spherical resonator was tested and modified to minimize or eliminate systematic problems, it appeared likely to succeed manometer-based gas thermometry as the most reliable thermodynamic thermometer in its operating range of temperature. The transition to a new technology for fundamental thermometry was complete.

Surface Chemistry and Microanalysis, Rance A. Velapoldi, chief

A Festival of Microbeam Analysis

The year 1991 marked the 25th anniversary of the Microbeam Analysis Society, an organization of industrial, academic, and government scientists devoted to the use of the electron microscope and its close relatives in analytical work. NIST staff members were prominent in the MAS: John A. Small served as Secretary to the Executive

[122] M. R. Moldover and J. P. M. Trusler, "Accurate acoustic thermometry I: the triple point of gallium," Metrologia 25, 165-187 (1988). Recently, Moldover, Christopher W. Meyer, S. J. Boyes, and A. R. H. Goodwin reported finding the presence of a "virtual leak"—probably outgassing from sealants—in the spherical resonator that modified the earlier results; this report can be found in *J. Res. NIST* **104**, No. 1, 11-46 (1999).

[123] M. R. Moldover, J. P. M. Trusler, T. J. Edwards, T. J. Mehl, and R. S. Davis, "Measurement of the universal gas constant R using a spherical acoustic resonator," *J. Res. NBS* **93**, No. 3, 85-114 (1988).

Council; Ryna B. Marinenko was one of its directors; an award to outstanding young Society scientists was named for Kurt F. J. Heinrich, by then an Honorary Member; and one of the most recent awards for outstanding authorship had been earned by a group that included Robert L. Myklebust and Dale E. Newbury.

The 1991 technical program included several contributions from NIST, which utilized only a fraction of the instrumental techniques reported during the meeting:

- "AMMS and Raman spectroscopy of metal phthalocyanine particles and films," by Robert A. Fletcher, Joseph A. Bennett, and Edgar S. Etz of NIST, together with S. Hoeft, a chemistry graduate student at Washington University in St. Louis.
- "Micro-Raman characterization of impurity phases in ceramic and thin-film samples of the Y-Ba-Cu-O high T_c superconductor," by Edgar S. Etz, T. D. Schroeder—a NIST guest scientist from Shippensberg University of Pennsylvania—and Winnie Wong-Ng.
- "A controlled-dispersion parallel-wavelength dispersive x-ray spectrometer for electron microscopy," by Charles E. Fiori, Scott A. Wight, and A. D. Romig, Jr., a colleague from the Sandia National Laboratory.
- "Electron probe compositional mapping of particles and samples with irregular surfaces," by John A. Small, David S. Bright, Robert L. Myklebust, and Dale E. Newbury.
- "Analysis of high-T_c bulk superconductors with electron microprobe compositional mapping," by Ryna B. Marinenko.
- "Scanning electron microscopy with polarization analysis: an update," by John Unguris, M. W. Hart, Robert J. Celotta, and Daniel T. Pierce.
- "Monte Carlo electron trajectory simulation of the depth sensitivity of electron backscattering," by Dale E. Newbury and Robert L. Myklebust.

Surface Science

Studies of photoinduced desorption and other chemical reactions at surfaces stemmed in part from their importance to semiconductor and energy-conversion technologies and in part from the insight they provided into surface science. Lee J. Richter, Steven A. Buntin, Daved S. King, and Richard R. Cavanagh sought information on the mechanism involved in the desorption of nitrous oxide from silicon single crystals. Irradiating a (111) silicon surface with a 1064 nm beam from a neodymium-yttrium-aluminum-garnet laser, the authors found evidence that the desorption of NO followed two different mechanisms, depending upon the extent of coverage of the surface.

The experiments were performed in an ultra-high vacuum apparatus, with the pre-cleaned silicon surface held at temperatures in the range 95 K to 100 K. Purified NO gas was introduced through capillaries placed near the crystal surface. Following laser irradiation, desorbed NO was detected by fluorescence of an optical transition in the molecule.

Evidence that the NO desorption mechanism from the (111) silicon surface varied with the extent of initial coverage appeared in a straightforward manner. The laser-induced-fluorescence signal, indicative of the number of NO molecules leaving the surface, varied by more than a factor of four as the initial exposure of the surface to NO was increased by a factor of ten. The authors offered detailed evidence that, at low NO coverage, the desorption involved a complex interaction between the silicon surface and the NO molecule.[124]

A guide to the principles and techniques important to the characterization of surfaces was provided through the publisher Plenum Press, Incorporated, by a group that included prominent present and former NBS/NIST surface scientists—Cedric J. Powell, chief of the division's Surface Spectroscopies and Standards group; Theodore E. Madey, NBS/NIST alumnus (1963-1990) then at Rutgers University; and John T. Yates, Jr., NBS alumnus (1963-1983) then at the University of Pittsburgh. Together with their colleagues Alvin W. Czanderna of the Solar Energy Research Institute of Golden, Colorado, and David M. Hercules of the University of Pittsburgh, the three served as editors of a series of tutorial books on Methods of Surface Characterization.

In "Ion Spectroscopies for Surface Analysis," Powell, Hercules, and Czanderna presented a comparison of the half-dozen major methods by which the compostion of a surface could be analyzed.[125] The techniques discussed were:

- Auger electron spectroscopy (AES).
- Ion scattering spectroscopy (ISS).
- Rutherford backscattering spectroscopy (RBS).
- Secondary ion mass spectrometry (SIMS).
- Secondary neutral mass spectrometry (SNMS).
- X-Ray photoelectron spectroscopy (XPS).

In their discussion, the authors provided comparative information on the types of particles that took part in the techniques, the extent of damage to the surface under investigation, the quantity measured in the techniques, and the depth to which the surfaces were probed. Various features of the analytical methods—factors important for data collection, detection capabilities, strong points and limitations—were provided as well.

[124] Lee J. Richter, Steven A. Buntin, Daved S. King, and Richard R. Cavanagh, "State-resolved studies of the laser-induced desorption of NO from Si(111)7x7: Low coverage results," *J. Chem. Phys.* **96**, No. 3, pp. 2324-2338 (1992).

[125] C. J. Powell, D. M. Hercules, and A. W. Czanderna, "Comparisons of SIMS, SNMS, ISS, RBS, AES, and XPS methods for surface compositional analysis," pp. 417-437 in *Ion Spectroscopies for Surface Analysis* (New York: Plenum Press, 1991).

J. William Gadzuk prepared a discussion that incorporated various aspects of the study of molecular activity at surfaces: photoemission spectroscopy, stimulated desorption, electron energy-loss spectroscopy, and collisions between molecules and surfaces. Gadzuk focused his treatment on molecular-level modeling of time-dependent phenomena at surfaces within the framework of semiclassical wave packet dynamics, building upon earlier theoretical work by E. J. Heller.[126] Details of the modeling formalism were provided to demonstrate that a variety of surface phenomena—adsorbate photoemission line shapes, stimulated desorption distributions, resonance electron energy loss spectroscopy, and molecule surface collisions were the examples given—could be treated in the same way, using time-dependent quantum mechanics.

Thermophysics, Richard F. Kayser, chief

Replacing Chlorofluorocarbons

Scientists at NIST were well aware, during this period, that chlorofluorocarbons (CFCs) had been convicted of poking holes in the earth's protective ozone layer—sunlight freed atomic chlorine from the CFC, chlorine acted essentially catalytically to convert ozone into oxygen, and the sun's most energetic ultraviolet radiation was free to wreak biological havoc on the earth and its creatures (see Chlorofluorocarbons vs the Ozone Layer in Ch. 4). NBS/NIST was asked to help find replacements for CFCs in refrigeration and aerosols, two major uses for the offending substances.

During 1991, Graham Morrison described the NIST program in the acquisition and correlation of data on alternative refrigerants. Experimental measurements discussed by Morrison included the following:

- Saturated liquid and vapor densities and vapor pressures.
- Pressure-volume-temperature relations in the gas phase, using a Burnett apparatus.
- Compressed liquid densities to 6500 kPa.
- Vapor pressure from steady-state boiling.
- Refractive index.
- Surface tension.
- Dielectric properties.
- Ideal gas heat capacity, using the spherical acoustical resonator (described earlier in this section).

[126] J. W. Gadzuk, "Semiclassical way to molecular dynamics at surfaces," *Annual Rev. of Physical Chemistry* **39**, pp. 395-424 (1988).

Refrigerants under study (and their short-hand designations) included:

- 1,1,1,2-Tetrafluoroethane (R134a).
- 1,1-Dichloro-2,2,2-trifluoroethane (R123).
- Pentafluoroethane (R125).
- 1,1,1-Trifluoroethane (R143a).
- 1,1-Difluoroethane (R32).
- 1-Chloro-1,2,2,2-tetrafluoroethane (R124).
- 1,1-Dichloro-1-fluoroethane (R141b).
- 1,1,2,2-Tetrafluoroethane (R134).
- 1,1,2-Trifluoroethane (R143).

Three data correlation schemes were discussed by Morrison. One was based upon a modified hard-sphere equation of state. A second utilized an extension of corresponding states. And a third employed a 90-term Benedict-Webb-Rubin equation.[127]

Mark O. McLinden and David A. Didion also responded to the need for reconsideration of refrigerant working fluids. They presented a discussion of the thermodynamic basis for choosing alternative compounds which emphasized the necessity for tradeoffs among the desirable and undesirable properties of such alternatives.[128] Didion independently pursued the question of the efficiency of heating and air-conditioning equipment with the use of various mixtures of working fluids. Using thermodynamic calculations, Didion provided manufacturers of heat pumps with the opportunity of achieving as much as a 30 % increase in efficiency. He received the Department of Commerce Gold Medal Award in 1987 for his efforts.

Hot Wires for Thermal Conductivity Measurements

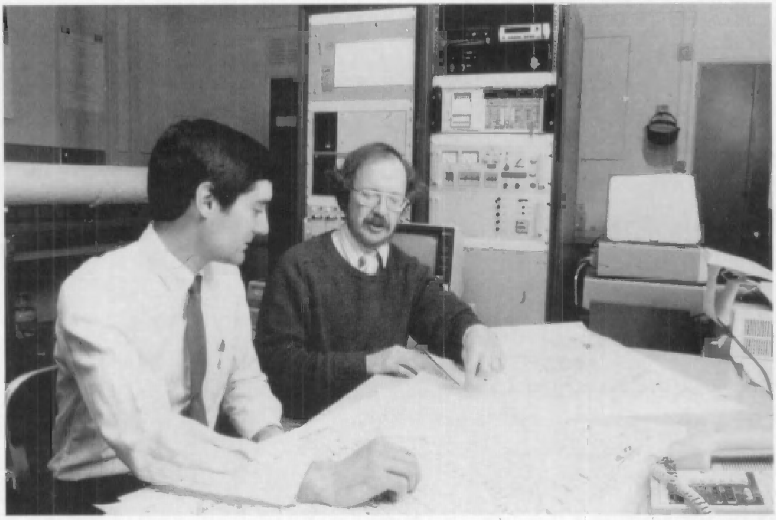
Nitrogen always was an important substance in the technology of cryogenics. As technology became more complex, the level of detail required for cryogenic fluids became more penetrating. Noting a lack of thermal conductivity and heat capacity data below room temperature, Ruth A. Perkins, Hans M. Roder, and Dale G. Friend of Thermophysics/Boulder, in collaboration with C. A. Nieto De Castro of the University of Lisbon, Portugal, undertook a series of measurements to fill some of the gaps.

In 1991, the group reported absolute measurements of the thermal conductivity and thermal diffusivity of nitrogen using a transient hot wire method. The observations, consisting of eight supercritical isotherms, three vapor isotherms, and four liquid isotherms, covered the temperature range from 80 K to 300 K and pressures up to 70 MPa.

Data were obtained with the use of a dedicated microcomputer which controlled two programmable digital voltmeters. The 1500-odd measurements included variation in the power delivered to the hot wire and variation in heating time to verify freedom from

[127] Graham Morrison, "Alternative refrigerant properties measurement and correlation program at NIST," *Pure and Applied Chem.* **63**, No. 10. 1465-1472 (1991).

[128] M. O. McLinden and D. A. Didion, "CFCs (chlorofluorocarbons): is the sky falling, Quest for alternatives," *ASHRAE Journal*, December 1987, pp. 32-42.



As part of the search for alternatives to chlorofluorocarbons, chemical engineer Mark McLinden (left) and physicist Graham Morrison developed property data for refrigerants that do not harm the ozone layer.

identifiable systematic errors and to allow calculation of thermal-difusivity and heat-capacity values with minimum measurement error. The team estimated the uncertainty in thermal conductivity determinations at ±1 % and in thermal diffusivity results at ±5 %, except for a 131 K isotherm near the critical density, where the uncertainties rose to ±3 % and ±10 %, respectively.

Correlating their data with the predictions of kinetic and other theories allowed the group to conclude that observations over a wide range of temperatures and densities would be repaid by better understanding of the thermodynamics of nitrogen.[129]

De Castro, Perkins, and Roder also evaluated the effect of radiative heat transfer in transient hot-wire thermal conductivity measurements during the same period that the work described above was in progress. They used the technique at higher temperatures—up to 548 K—so that the radiative effect would be more pronounced. Measurements on the thermal conductivity of toluene could be fitted well using heat transfer calculations that incorporated participation by the toluene in the radiative transfer process.[130]

[129] R. A. Perkins, H. M. Roder, D. G. Friend, and C. A. Nieto De Castro, "The thermal conductivity and heat capacity of fluid nitrogen," *Physica* **A 173**, pp. 332-362 (1991).

[130] C. A. De Castro, R. A. Perkins, and H. M. Roder, "Radiative heat transfer in transient hot-wire measurements of thermal conductivity," *Intl. J. of Thermophysics* **12**, No. 6, pp. 985-997 (1991).

Flowmeters for Vacuum Standards

Certain low-pressure standards required the creation of accurately known gas flow rates. Two of these concerned measurement of flow through orifices and the measurement of gaseous leak rates. Using one type of NIST standard, a calculable conductance, the operator could produce a known pressure differential by passing a gas through it at a measured flow rate. The NIST conductance standard nominally passed 10 L/s for the range 0.1 Pa and below, requiring a gas-flow reference source in the range 10^{-10} mol/s to 10^{-6} mol/s.

Kenneth E. McCulloh, Charles D. Ehrlich, Frederick G. Long, and Charles R. Tilford developed two constant-pressure piston-displacement flowmeters, either of which could be used for the orifice standard or for leak calibrations. One of the devices employed a piston with sliding seals. Its volume change and the flow rate were derived from the movement of the piston. The other, an all-metal system reaching two decades lower in flowrate, consisted of a welded bellows driven by a hydraulic press.

The group evaluated the uncertainties of the new flowmeters by considering a number of potential systematic errors—uncertainty in the reading of the pressure gage used to measure the pressure in the fixed-pressure reference chamber, volume changes during operation, temperature errors, desorption, and leakage. Over the range of usable flow rates, they estimated the uncertainties at 0.8 % to 2 %.[131]

Membrane Diffusion

Porous membranes came into increasing use during this period for purposes as diverse as blood cleansing, industrial waste recovery, and gas purification. Theorist Rosemary A. McDonald wondered whether one could predict the efficacy of filtration through a given membrane. She examined the process by means of a simple random-walk procedure, using a two-dimensional model for the membrane. She considered two types of membranes—each constructed of two sizes of pores. Spherical molecules, also of two sizes—either pure or mixed 50-50—"walked" through the membranes, sometimes under a simulated pressure head.

McDonald's results were promising in terms of predicting the diffusive nature of her simplified system. She noted, however, ". . . it is important that experimental data be available for comparison with the model results. We hope that this work may stimulate experiments along the lines we have pursued here."[132]

Vapor-Liquid Equilibrium

Increasing interest in the technology of supercritical fluids led to heavy involvement by NIST scientists in reviews of the relevant theory and applications to thermophysical properties. A 600-page text, "Supercritical Fluid Technology: Reviews in Modern

[131] K. E. McCulloh, C. D. Ehrlich, F. G. Long, and C. R. Tilford, "Low range flowmeters for use with vacuum and leak standards," *J. Vac. Sci. and Tech. A-Vacuum Surfaces and Films* **5**, No. 3, pp. 376-381 (1987).

[132] Rosemary A. McDonald, "Modeling macromolecular diffusion through a porous medium," *J. Membrane Science* **68**, 93-106 (1992).

Richard Hyland (left) and Charles Ehrlich compared features of several calibrated leak artifacts mounted to the vacuum manifold of a new primary leak standard. Developed in the mid-1980s, the leak standard initially was used to provide calibrations for helium permeation leaks.

Theory and Applications"[133] was edited by Thomas J. Bruno and James F. Ely of the Thermophysics Division/Boulder. A listing of the titles of chapters written by NIST researchers (six of the 16 chapters in the book) gives a flavor of the topics covered:

[133] CRC Press, Boca Raton, Florida, 1991.

- "Thermodynamics of solutions near the solvent critical point," by Johanna M. H. L. Sengers.
- "Vapor-liquid equilibrium and the modified Leung-Griffiths model," by James C. Rainwater.
- "Thermophysical property data for supercritical extraction design," by Thomas J. Bruno.
- "Properties of carbon dioxide rich mixtures," by Joe W. Magee.
- "Mass transfer in supercritical extraction from solid matrices," by Michael C. Jones.
- "A summary of the patent literature of supercritical fluid technology," by Thomas J. Bruno.

Physics Laboratory

The Physics Laboratory was formed from the Center for Atomic, Molecular, and Optical Physics and the Center for Radiation Research when the new NIST organizational structure was approved in February 1991. The laboratory continued to function much as its predecessors had—as a source of new physics and new instrumentation at the leading edge of physical science. We illustrate the work of the laboratory through examples chosen from each of its eight divisions.

Physics Laboratory Office

An Update on Physical Constants

The Committee on Data for Science and Technology (CODATA), an interdisciplinary arm of the International Council of Scientific Unions, periodically published—through its Task Group on Fundamental Constants—reports summarizing worldwide progress in evaluating the large (some 38 quantities) list of fundamental physical constants.

In 1990, Barry N. Taylor and E. Richard Cohen, a physicist at the Rockwell International Science Center who customarily collaborated with Taylor on the reviews, surveyed recent advances in this field.[134] Major progress had taken place in the evaluation of the Planck constant, the fine-structure constant, and the molar gas constant. Also brought forth since the most recent (1987) CODATA report[135] were new definitions for the volt and the ohm.

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[134] Barry N. Taylor and E. Richard Cohen, "Recommended values of the fundamental physical constants: a status report," *J. Res. NIST* **95**, 497-523 (1990).

[135] E. R. Cohen and B. N. Taylor, *Revs. Mod. Phys.* **59**, 1121 (1987).

It was interesting that the authors could not present new values for the re-determined quantities because to do so would require "nothing less than a new least-squares adjustment" of many of the constants previously publicized. However, they did review the so-called "auxiliary constants"—those quantities for which values were defined by international agreement or else were so accurately known as to not be subject to change by any forseeable experiments. This list included the following constants:

- Speed of light.
- Meter.
- Ratio of the proton mass to the electron mass.
- Atomic masses and mass ratios.
- Rydberg constant.
- Magnetic moment anomalies of the electron and positron.
- Ratios of the magnetic moments of the electron, the proton, and other nucleons.
- The "as-maintained" volt and ohm.
- Acceleration due to gravity.

Taylor and Cohen cited NIST work that contributed to several of these determinations.

Electron and Optical Physics, Charles W. Clark, chief

Images of Ferromagnetism

In 1990, Michael R. Scheinfein, John Unguris, Michael H. Kelley, Daniel T. Pierce, and Robert J. Celotta compared a relatively new measurement method known as SEMPA to other types of instruments that displayed the distribution of magnetization in ferromagnets.

One of the earliest field-imaging methods involved, for example, the use of fine magnetic particles that, when dusted on a magnetic surface, agglomerated in the fringe fields produced at domain walls, thus producing an image that could be viewed with an optical microscope. Another imaging technique, Lorentz microscopy, in which incident electrons were deflected by magnetic fields at the surface of the sample, could be used at low magnification (1000 nm) by beam reflection from thick samples or at high magnification (10 nm) by transmission through samples of thickness less than 300 nm. In general, transmission electron microscopy required the use of thin samples. A newer instrument, the magnetic force microscope, utilized a ferromagnetic tunneling tip to achieve resolution of domain walls at 100 nm.

SEMPA—Scanning Electron Microscopy with Polarization Analysis—achieved 10 nm resolution by detection of the polarization of secondary electrons emitted from ferromagnetic surfaces. Magnetization maps could be obtained from the surfaces of bulk specimens, thin films, and even monolayers of certain types.

In their review article, the NIST group described the individual components of their instrument—a probe forming electron optical column, a spin analyzer, transport optics, and signal-processing electronics—and they outlined the advantages and limitations of the method, including examples of images produced by its use.[136]

Illustrations of the ferromagnetic domains in an iron-silicon crystal surface showed in clear, contrasted detail the domain structure below the 10 nm level, though they were obtained in only a three-minute scan of the sample. Other illustrations portrayed domains on crystalline iron and cobalt surfaces and Permalloy ($Ni_{81}Fe_{19}$) computer memory elements, with similar clarity.

The next order of business for the NIST group was to compare the SEMPA technique with magneto-optical Kerr microscopy (MO), using measurements of surface domain-wall magnetization profiles in a Permalloy film and micromagnetic theory. With the collaboration of P. J. Ryan of Seagate Technology, Inc., they found that the two methods, substantially different in principle, produced similar domain-wall magnetization profiles (though the MO resolution was limited to about 200 nm) and similar analytical results with the use of bulk specimen parameters.[137]

Peeking at Picometers With a Scanning Tunneling Microscope

The electron physics group pursued a goal of refining the resolution of the scanning tunneling microscope (STM) on conductive surfaces. During this period, they developed the capability of routinely observing picometer-sized structures—individual atoms—on metal and semiconductor surfaces, offering seemingly endless possibilities for both science and technology.

In one example of the use of the STM at its best resolving power, Lloyd J. Whitman, Joseph A. Stroscio, Robert A. Dragoset, and Robert J. Celotta presented topographic images of the surface of an InSb (110) crystal face at the atomic level of resolution.[138] The presence of unfilled electron bonds at the surface made InSb an interesting subject for study. In the words of the authors, "... the occupied surface state density, observed when tunneling from the sample to the STM tip, is concentrated on the group-V anion, and the unoccupied state density, observed when tunneling from tip to sample, is on the group-III cation." Both tunneling directions were illustrated in the discussion.

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[136] M. R. Scheinfein, J. Unguris, M. H. Kelley, D. T. Pierce, and R. J. Celotta, "Scanning electron microscopy with polarization analysis (SEMPA)," *Rev. Sci. Instrum.* **61**, No. 10, 2501-2526 (1990).

[137] M. R. Scheinfein, P. J. Ryan, J. Unguris, D. T. Pierce, and R. J. Celotta, "180° surface domain wall magnetization profiles: Comparisons between scanning electron microscopy with polarization analysis measurements, magneto-optical Kerr microscopy measurements and micromagnetic models," *Appl. Phys. Lett.* **57**, No. 17, 1817-1819 (1990).

[138] L. J. Whitman, Joseph A. Stroscio, R. A. Dragoset, and R. J. Celotta, "A scanning tunneling microscopy study of clean and Cs-covered InSb (110)," *J. Vac. Sci. Technol.* **B9**, No. 2, 770-774 (1991).

The authors obtained a clean (110) surface by cleaving the InSb crystal under high vacuum—pressure less than 10^{-8} Pa. Vertical resolution on the surface was typically 2 picometers.

Occasionally, the researchers found defects in the sample surface, which they ascribed to missing Sb atoms or to missing In and Sb pairs. These images, too, were presented in the paper.

A handsome perspective figure was formed by adsorbed Cs atoms, introduced by room-temperature deposition. The preferred modes of arrangement of the adsorbed Cs were easily seen in the image.

X-Ray Optics Characterization Facility

The capability for x-ray optics characterization at the Synchrotron Ultraviolet Research Facility (SURF-II) was described by James R. Roberts, J. Kerner, and Edward B. Saloman.[139] The 300 MeV synchrotron source provided radiation from 5 nm through the visible portion of the spectrum. A turning mirror directed the SURF beam towards a 2.2 m grazing-incidence grating monochromator and thence to a reflectometer, all within a vacuum of about 10^{-7} Pa. A sample manipulator allowed various irradiation angles and directions to be used. This feature allowed, for example, study of the response of the sample to radiation polarization.

The authors illustrated the capability of the x-ray unit with a graph showing the reflectance of a 10-layer molybdenum-silicon sample in the range 12 nm to 17 nm. The reflectance peak of the sample at the second spacing of 13.8 nm was very clearly displayed.

Ideas for improvement of the facility were presented, too. These included better use of the shortest wavelengths of the SURF radiation and improvements in the monochromator, the reflectometer, and the data-acquisition system. A multi-divisional drive to create a new beam line was funded by the Defense Advanced Research Projects Agency in 1991. Development and installation of the new hardware was the responsibility of David L. Ederer, Thomas B. Lucatorto, Robert P. Madden, and Richard N. Watts.

Theories of High-Temperature Superconductivity

The interactions underlying the phenomenon of high-temperature superconductivity were not understood in 1991, although many theorists were investigating the problem. The Bardeen-Cooper-Schrieffer (BCS) theory, so successful in accounting for most details of "ordinary" metallic superconductivity since 1957, appeared to need modifications to fit the newer oxide-superconductor properties.

David R. Penn collaborated with T. W. Barbee and Marvin L. Cohen to explore tenable theories and to explain some of the limitations that any successful theory must meet. Cohen was well-equipped for the task, having earlier predicted the occurrence of superconductivity in semiconducting materials such as $SrTiO_3$, which closely resembled the high-T_c oxides.

[139] J. R. Roberts, J. Kerner, and E. B. Saloman, "Soft x-ray optics characterization on SURF II," *Physica Scripta* **41**, pp. 9-12 (1990).

The authors suggested that simple modifications of the phonon-based BCS and Eliashberg theories might be useful, but that such modifications must accurately represent several experimental parameters of the newer superconductors, including the high superconducting transition temperatures, the relatively small isotope effect, the jump in the heat capacity at the transition temperature, and the ratio of the superconducting energy gap to the transition temperature. Historically, the presence of a strong isotope effect had been taken as evidence for the participation of phonons in the superconductive interaction, whereas the energy gap ratio and the heat capacity anomaly at the transition temperature were used to distinguish between strong-coupling and weak-coupling superconductors.

In their study, the authors focused on the established properties of several high-T_c materials, including the bismuth-strontium-calcium-copper-oxygen system, the yttrium-barium-copper-oxygen system, and the lanthanum-strontium-copper-oxygen system. They restricted their calculations to mechanisms that included at least some phonon participation, possibly in combination with electronic interactions. They pointed out that, for purely electronic mechanisms, the isotope effect would necessarily be insignificant since the ionic lattice mass would make no contribution to the superconductive interaction.

Penn and his colleagues expressed the view that neither a purely phononic nor a purely electronic mechanism was consistent with all of the existing experimental data. They postulated that a combined phonon-electron mechanism should be able to account for all the experimental results on the high-T_c materials, but confessed that, for the moment, no existing theory appeared to fit all of the experimental data.[140]

Atomic Physics, Wolfgang L. Wiese, chief

Calibrating the Hubble Space Spectrograph

Joseph Reader and Craig J. Sansonetti helped NASA calibrate the high-resolution spectrograph of the Hubble Space Telescope during 1991. The wavelength calibration for the spectrograph consisted of an atlas of some 3000 lines of the platinum-neon spectrum in the range 103.2 nm to 410 nm for use with on-board platinum lamps. Reader and Sansonetti measured the lines with a NIST 10.7 m vacuum spectrograph and used photon-counting detection to obtain relative line intensities accurate within about 20 %.

Besides the high-resolution spectrograph, the NIST results were used to calibrate the Hubble's Faint Object Spectrograph, to calibrate other satellite and rocket-borne spectrometers, and for laboratory ultraviolet spectroscopes.

A New Test of Quantum Electrodynamics

Increasingly accurate calculations and experiments in atomic and elementary particle physics provided rigorous testing of the theory of quantum electrodynamics (QED) during this period. Even more stringent tests were possible by examining the Lamb shift in two-electron systems such as helium.

[140] T. W. Barbee, III, Marvin L. Cohen, and David R. Penn, "Experimental constraints on some mechanisms for high-temperature superconductivity," *Phys. Rev.* **B44**, No. 9, 4473-4479 (1991).

Craig J. Sansonetti, John D. Gillaspy, and Chris L. Cromer took up the QED challenge by measuring the energy of the $2^1S–3^1P$ transition in ^4He, using Doppler-free laser spectroscopy.[141] They considered their measurement, 19931.924794 cm^{-1}, accurate within 0.000045 cm^{-1}—an order of magnitude improvement over the uncertainty of previous experiments.

Key to the success of the experiment was the careful use of up-to-date equipment and techniques. The authors created a beam of metastable helium atoms, tightly collimated to reduce the Doppler effect to a value much smaller than the width of the transition. This arrangement gave them access to the 2^1S state of helium. Radiation from a calibrated cw laser intersected the helium beam, exciting helium molecules to the 3^1P state. The process of fitting the metastable depletion curve yielded values for the line center and width. The experiment was repeated so as to generate more than 40 independent values, which averaged to the value quoted.

Comparison with QED theory was made through evaluation of the calculated Lamb shift in the binding energy of the 2^1S state in helium, which was expected to be relatively large in the NIST experiment. The authors found their result to be significantly different from the value predicted on the basis of then-current QED theory.

Atomic Spectroscopy Data

Atomic transition probabilities for the five iron-group elements—scandium, titanium, vanadium, chromium, and manganese—(some 8800 spectral lines in all) were compiled for the *Journal of Physical and Chemical Reference Data* after critical evaluation by Georgia A. Martin, Jeffrey R. Fuhr, and Wolfgang L. Wiese. The data were collated for easy reference. Separate tables were included for each element and stage of ionization, with subdivisions for allowed and forbidden transitions and a subdivision for multiplets. For each line, data included the transition probability for spontaneous emission, line strength, spectroscopic designation, wavelength, statistical weight, and the connecting energy levels.[142]

Wiese also offered a critical evaluation of emission spectroscopic experimental data on the prominent spectral lines of argon I and argon II in an effort to reduce well-known discrepancies of 30 % to 40 % in argon atomic transition probabilities. For the argon I case, the analysis was successful in reducing the discrepancies to ±5 %; Wiese reported only minimal improvement for argon II.[143]

Also during this period, William C. Martin and Wiese embarked on a new project for the benefit of all spectroscopists but especially for scientists working in astronomy and the fusion programs. The work would be incorporated into the Atomic

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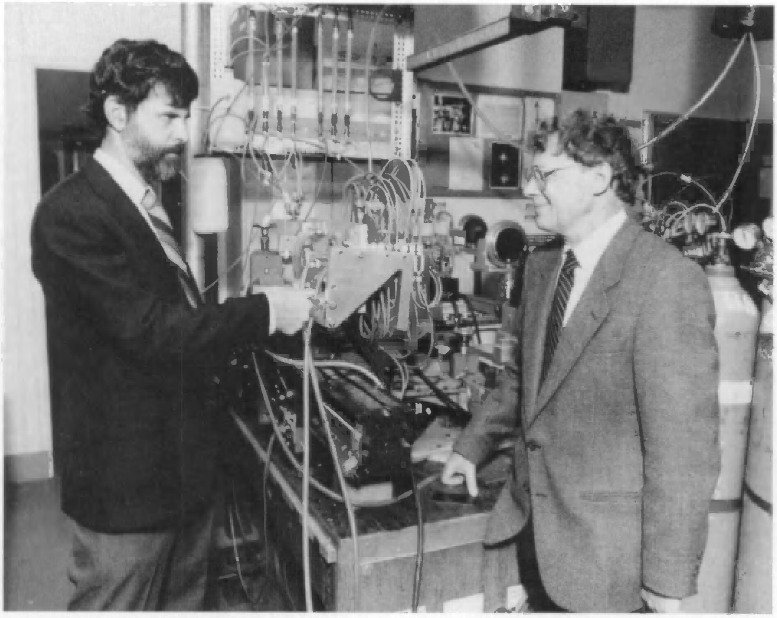
[141] Craig J. Sansonetti, J. D. Gillaspy, and C. L. Cromer, "Precise experimental test of calculated two-electron Lamb shifts in helium," *Phys. Rev. Lett.* **65**, No. 20, pp. 2539-2542 (1990).

[142] G. A. Martin, J. R. Fuhr, and W. L. Wiese, "Atomic transition probabilities scandium through manganese," Suppl No. 3, *J. Phys. Chem. Ref. Data* **17**, 1988, 531 pp.

[143] W. L. Wiese, "Atomic transition probabilities of argon: a continuing challenge to plasma spectroscopy," *J. Quantitative Spectroscopy and Radiative Transfer* **40**, No. 3, 421-427 (1988).



Jeffrey R. Fuhr (left), a physicist in the NIST Atomic Physics Division, demonstrated the wall-stabilized arc to Wolfgang L. Wiese, chief of the division. The arc is an intense source of optical radiation.

Energy Levels Data Center. Their plan was to add to a critically evaluated, digital database for spectroscopy—wavelengths, energy levels, and transition probabilities for atoms and their ions. The database was arranged for easy access by computer. Data could be retrieved in a number of ways—by species or by wavelength range, for example. Wavelengths were included from 0.1 nm to the mm range.

By 1991, spectra of multiply ionized vanadium, cobalt, and chromium and wavelengths and energy levels for aluminum and germanium had been added to the data set.

Ultraviolet Calibration for Semiconductor Processing

Mervyn J. Bridges and James R. Roberts provided special assistance to the semiconductor industry to correct a processing problem. In photolithography—the use of light to create etch patterns on semiconductors—intense ultraviolet (UV) radiation was used in conjunction with photomasks as a precursor to etching the semiconductor surface to form the patterns needed for integrated circuits. Controlling the UV exposure was accomplished by trial and error, however, for lack of adequate UV standards.

The NIST researchers characterized two types of radiometers for SEMATECH, the consortium undertaking development of the new process. Calibration of UV response and the effects of irradiation variables were included in the study, which turned up several unexpected problems with the methods then in use in photolithography. With the collaboration of scientists from the Radiometric Physics Division, Bridges and Roberts also developed a new spectroradiometer for semiconductor work.

Molecular Physics, Alfons Weber, chief

Infrared Spectroscopy of Chemical Complexes

Infrared and microwave spectroscopy of chemical complexes occupied considerable attention in the Molecular Physics Division during this period. A number of different complexes were investigated from as many different points of view.

Both infrared and microwave techniques were used by NIST scientists Gerald T. Fraser, Richard D. Suenram, Frank J. Lovas, Alan S. Pine, Jon T. Hougen, and Walter J. Lafferty in collaboration with J. S. Muenter of the University of Rochester in a study of tunneling in the dimer of acetylene.

One question asked by the group concerned the structure of the dimer and the possible presence of isomers—rarely seen in adiabatic expansion experiments. In the region of the H-C stretching fundamental, an infrared spectrum was obtained with an optothermal molecular-beam color-center laser spectrometer. A pulsed-nozzle Fourier transform microwave spectrometer was used to examine the ground state of the vibrational modes.

The authors interpreted their results as arising from a single isomer which showed internal-rotation tunneling. The wealth of spectral data yielded considerable other molecular information as well.[144]

During the same period, Fraser, Pine, and Suenram teamed with W. A. Kreiner of the University of Ulm, Germany, to obtain the infrared spectrum of the NCH-NH_3 complex. The team was particularly interested in the structure and dynamics of the hydrogen-bonded complex. A molecular-beam apparatus was used to form the complex by adiabatic expansion of a gas mixture of NH_3 and HCN in helium through a 40 μm diameter nozzle. The driving pressure through the nozzle was varied between 100 kPa and 300 kPa. A silicon bolometer cooled by liquid helium served as the detector.

The group obtained a rich spectrum of infrared and microwave transitions for the complex. By the use of applicable theory, these were identified with particular vibrations of the complex, including the "umbrella" mode of NH_3. Discussion of the results focused on a shift of the vibration frequency to lower values than had been seen in other NH_3 complexes.[145]

Pine and Fraser collaborated with M.–L. Junttila of the Helsinki University of Technology in Finland and J. L. Domenech of the Materials Institute in Madrid, Spain, to perform a similar experiment on benzene.[146] Since benzene, with a center of

[144] G. T. Fraser, R. D. Suenram, F. J. Lovas, A. S. Pine, J. T. Hougen. W. J. Lafferty, and J. S. Muenter. "Infrared and microwave investigations of inter-conversion tunneling in the acetylene dimer," *J. Chem. Phys.* **89**, No. 10, pp. 6028-6045 (1988).

[145] G. T. Fraser, A. S. Pine, W. A. Kreiner, and R. D. Suenram, "Optothermal-detected microwave-sideband CO2-laser spectroscopy of NCH-NH3," *Chemical Physics* **156**, pp. 523-531 (1991).

[146] M.–L. Junttila, J. L. Domenech, G. T. Fraser, and A. S. Pine, "Molecular-beam optothermal spectroscopy of the 9.6 μm ν_{14} band of benzene," *Journal of Molecular Spectroscopy* **147**, 513-520 (1991).

symmetry, had no microwave spectrum, it was necessary to determine its ground-state rotational constants indirectly. The authors noted that experimentally determined values of these constants from different laboratories varied by larger amounts than the experimental uncertainties would warrant.

The authors were able to perform collisionless molecular-beam spectroscopy experiments on benzene. They hoped to provide high-quality calibration standards in the range around 10 μm for a variety of purposes. They used laser excitation on a benzene-helium gas vapor that was directed through a flow-controlled nozzle. Doppler shifts were minimized by crossing the laser beam and the molecular beam at nearly a right angle. They recorded spectral transitions in the ν_{14} band of benzene. This information enabled them to determine ground-state molecular constants with improved accuracy, thus reaching their goal of providing a new calibration standard.

Picosecond Energy Transfer on Surfaces

Molecular Physics Division researchers Michael P. Casassa, Edwin J. Heilweil, and John C. Stephenson teamed with John D. Beckerle and Richard R. Cavanagh of the Surface and Microanalysis Division to investigate the ultrafast exchange of energy between adsorbed gases and metallic surfaces.[147] They were able to reach picosecond time resolution by observing with infrared spectroscopy the creation and relaxation of excited states in CO molecules absorbed on a platinum single crystal. An intense picosecond laser pulse at about 2105 cm^{-1} was used to excite a stretching band in CO. Then a much weaker, time-delayed probe pulse monitored the recovery of the surface absorption.

It was the first direct measurement of the time scale of energy transfer in a vibrationally excited, ordered monolayer bound to a metal single-crystal surface. The authors discussed the results in terms of transitions from the excited band of the adsorbed CO molecules to overtone levels.

Orientation Effects in Chemical Reactions

Interest in the effects of orientation on the progress of chemical reactions led Lovas and Suenram to collaborate with scientists from three universities on a study of bimolecular gas-phase reactions. Joining the NIST researchers were C. W. Gillies of Rensselaer Polytechnic Institute, J. Z. Gillies of Union College, and E. Kraka and D. Cremer of the University of Göteborg, Sweden.

The authors took a direct approach to obtaining the orientation of the reactants in a mixture of ethylene and ozone, sampling the reacting process in a pulsed-beam nozzle. Dilute concentrations of ozone and ethylene in argon were separately fed to the high-pressure side of a pulsed solenoid valve, which served both as a flow reactor for the formation of formaldehyde as a reaction product and as a sampling device for study of the reaction. The Van der Waals complexes formed in the pulsed-nozzle expansions of 0.2 ms to 0.4 ms duration, repeated up to 10 times per second, were detected in the Fabry-Perot cavity of a Fourier-transform microwave spectrometer.

[147] J. D. Beckerle, M. P. Casassa, R. R. Cavanagh, E. J. Heilweil, and J. C. Stephenson, "Ultrafast infrared response of adsorbates on metal surfaces: vibrational lifetime of CO/Pt (111)," *Phys. Rev. Lett.* **64**, No. 17, 2090-2093 (1990).

Both microwave data and calculations were used to determine the geometry of the complex, its internal motion, and its stability. The authors were able to deduce the likely orientation of the reactants at large separations along the reaction coordinate and to estimate the stability of the Van der Waals complex involved in the reaction.[148]

Vibrational Spectra of Acetylene Ions

Ionization in planetary atmospheres of simple molecules such as acetylene was known to be an important extraterrestrial chemical process. Partly for that reason, study of acetylene and its fragments attracted the interest of Marilyn E. Jacox and two NIST guest scientists, Daniel Forney and Warren E. Thompson.[149] Earlier experiments in Jacox' laboratory had evolved a new technique for producing molecular ions trapped in solid neon; the infrared spectra of several ions, including CO^{+}_{2}, N_2O^+, and CO^+, were obtained in this fashion. Jacox and her colleagues hoped to use similar experimental methods to study $HCCH^+$.

The authors co-deposited an acetylene-neon mixture at low temperatures—about 5 K—along with neon that had been subjected to a microwave discharge. Absorption spectra of the deposits were obtained using a Fourier-transform interferometer. Further information on the materials was obtained by subjecting them to a variety of visible and ultraviolet irradiations.

Sure enough, the trio of researchers found spectral evidence that permitted the assignment of an infrared absorption to the positively charged acetylene ion, as well as a line attributed to the HCC^- fragment, detected through identification of the known CC-stretch absorption frequency. Their identification of the $HCCH^+$ cation in the neon-matrix experiments led them to postulate that the spectrum shifted strongly as a result of a change in polarizability from an argon matrix to a less-polarizable neon matrix.

In an earlier study involving a similar sampling technique but employing an argon matrix and an argon excitation source, Jacox and W. Bruce Olson had discovered an extremely complex series of absorption bands in the near infrared spectral region. They were able to show that these bands arose in the HCC fragment.[150] Only a few of these bands had been seen in the gas phase, and they corresponded closely with the HCC bands found in solid argon. Daniel Forney, Marilyn Jacox, and Warren Thompson studied these bands later using HCC and DCC trapped in solid neon. They proposed a detailed spectroscopic assignment for the band system.[151]

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[148] C. W. Gillies, J. Z. Gillies, R. D. Suenram, F. J. Lovas, E. Kraka, and D. Cremer, "Van der Waals complexes in 1, 3-dipolar cycloaddition reactions: Ozone-ethylene," *J. Am. Chem. Soc.* **113**, No. 7, pp. 2412-2421 (1991).

[149] Daniel Forney, Marilyn E. Jacox, and Warren E. Thompson, "The vibrational spectra of molecular ions isolated in solid neon: $HCCH^+$ and HCC^-," *Spectroscopy* **153**, 680-691 (1992).

[150] Marilyn E. Jacox and W. Bruce Olson, "The A $^2\Pi$–X $^2\Sigma^+$ transition of HC_2 isolated in solid argon," *J. Chem. Phys.* **86**, No. 6, 3134-3142 (1987).

[151] Daniel Forney, Marilyn E. Jacox, and Warren E. Thompson, "The infrared and near-infrared spectra of HCC and DCC trapped in solid neon," *J. Molecular Spectroscopy* **170**, 178-214 (1995).

Radiometric Physics, Klaus D. Mielenz, chief, succeeded by Albert C. Parr in 1991

Absolute Cryogenic Radiometer

One of the new research tools in the Radiometric Physics Division during this period was an absolute radiometer that employed cryogenic radiation collection and energy evaluation by electrical substitution. The instrument was based upon a development of Martin L. Reilly and Defoe C. Ginnings (see Electrical Substitution Radiometry in Ch. 4) which was brought to the stage of demonstrated high-accuracy radiometry by Terence J. Quinn and John Martin of the National Physical Laboratory in England. Later, a NIST group collaborated with NPL physicists in the development of a similar instrument for the NIST radiometer calibration program. The cryogenic radiometer, operating near 5 K, incorporated smaller and simpler corrections than instruments operating at higher temperatures. Its radiometric measurement uncertainty was estimated at 0.01 %. Jeanne M. Houston operated the instrument for the calibration program.

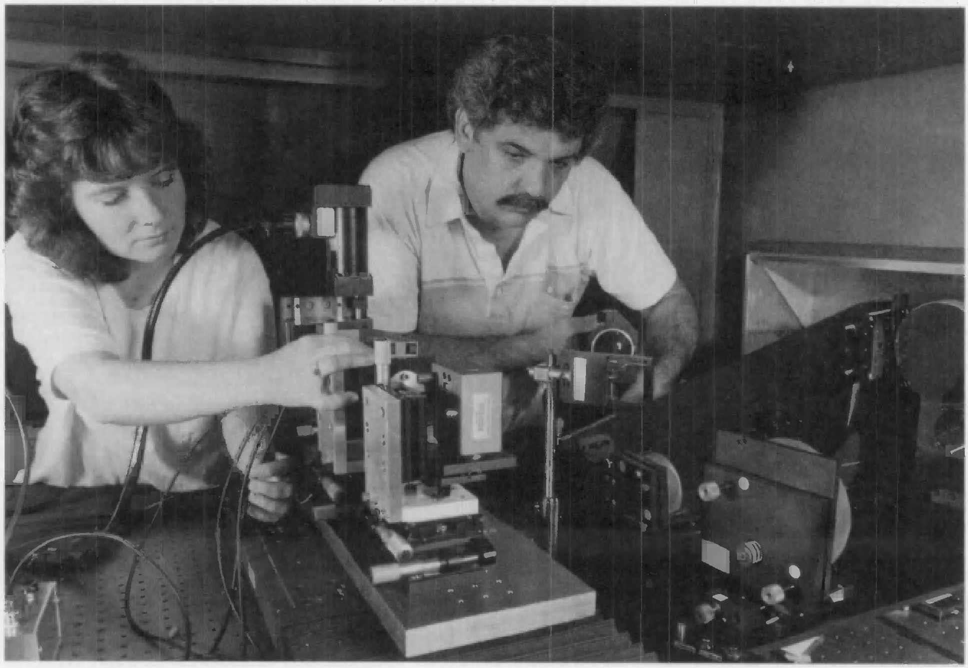
More on Silicon Photodetectors

We described the initial stages of detector-based radiometry in the section entitled *Progress in Optical Radiation Measurements* in Ch. 3. During the tenure of John Lyons, analysis of the response of silicon to radiant energy continued. Jon Geist and Alan Migdall collaborated with Henry Baltes, a guest scientist from the ETH Institute of Quantum Electronics in Zurich, Switzerland, in a detailed study of the silicon absorption coefficient in the spectral range near 760 nm (1.63 eV). They sought to verify or refute evidence obtained in optical density spectra by NIST scientists Richard A. Forman and W. Robert Thurber, working with David E. Aspnes of the Murray Hill, New Jersey, Bell Telephone Laboratories research center, that an indirect transition occurred in silicon at that energy.

For their investigation, Geist, Migdall, and Baltes employed a stabilized ring dye laser to irradiate a 100 μm thick, 2.5 cm diameter wafer of high-purity silicon. The silicon wafer, held at a 45° angle to the laser beam, was backed by a calibrated silicon photodiode, so that accurate measurements could be obtained of its absorption coefficient. They found a smooth absorption spectrum in the range 1.61 eV to 1.65 eV, with no features as large as a few parts in 1000 that would support the existence of a transition at 1.63 eV.

In a study intended to provide the best available accuracy for the silicon absorption coefficient at 633 nm, Geist, Jun-Feng Song, Yun Hsia Wang, and Edward F. Zalewski of NIST, in collaboration with A. Russell Schaefer, a former NIST colleague then at Science Applications International Corporation of San Diego, derived a value of $(3105 \pm 62)\ \text{cm}^{-1}$. Their value was lower by about 15 % than handbook values but they believed it to be more accurate by virtue of improved sample quality and more reliable thickness and transmittance measurements.[152]

[152] Jon Geist, Alan Migdall, and Henry Baltes, "Shape of the silicon absorption coefficient spectrum near 1.63 eV," *Applied Optics* **29**, No. 24, 3548-3554 (1990). See also Jon Geist, A. Russell Schaefer, Jun-Feng Song, Yun Hsia Wang, and Edward F. Zalewski, "An accurate value for the absorption coefficient of silicon at 633 nm," *J. Res. NIST* **95**, No. 5, 549-558 (1990).



On the automated spectral comparator facility, physicists Jeanne M. Houston and Chris L. Cromer aligned the silicon photodiode light traps that were used in photodetector research and calibration.

Low Background Infrared Calibration Facility

The Bureau's Low Background Infrared Calibration Facility (LBIR), discarded in 1985 because of old age and high maintenance costs, was reborn in 1991 at the hands of Raju Datla, Steven Lorentz, and Stephen C. Ebner. Datla designed an infrared monochromator for the spectral calibration of infrared sources—principally blackbody devices, detectors, and optical components. Lorentz developed a laser-based infrared source to provide radiation in the range 2 μm to 25 μm. Ebner was responsible for development of a low background blackbody source.

The Department of Defense contributed funds for the resurrection because of the national need for the calibration capability.

Datla, Ebner, James Proctor, and Albert C. Parr described the new LBIR facility in *NIST Handbook 147*.[153] The facility was placed within a class 10,000 cleanroom 4.3 m by 5.2 m by 3.2 m high, with storage and assembly space outside. A closed-cycle helium refrigerator, built by Donald McDonald and Joseph Sauvageau of the Boulder Electromagnetic Technology Division, provided cooling to 2 K for the instrumentation.

[153] R. U. Datla, S. C. Ebner, J. Proctor, and A. C. Parr, "LBIR Facility User Handbook," *NIST Handbook 147*, June 1991, 23 pp.

A vacuum tank capable of reaching pressures as low as 10^{-7} Pa housed the radiation source to be calibrated, as well as the detector. The standard detector was an absolute cryogenic radiometer (ACR) of the type described earlier in this section. It absorbed more than 99.5 % of all incoming radiation in the spectral range 0.3 μm to 30 μm, providing an overall uncertainty of 1 %. Electrical substitution radiometry was used for the measurement of radiative flux. The ACR could be placed in any of three positions along the optic axis of the system to accommodate sources with different radiant fluxes.

Data collection was performed with a menu-driven computer program that incorporated data files, data reduction, and plotting routines.

Cleanliness, both within the cleanroom and within the vacuum tank, were considered vital. Hydrocarbon contamination of the vacuum tank was especially minimized to preserve the quality of the LBIR optics. Instructions were presented to guide the user in pieparing for clean and accurate calibrations.

Quantum Metrology, Richard D. Deslattes, chief

Lattice Period of Silicon

Richard D. Deslattes and Ernest G. Kessler, Jr. described for the *1990 Conference on Precision Electromagnetic Measurements* the current status of measurements of the lattice spacing in silicon. The discussion necessarily included an update on the division's recently modified x-ray/optical interferometry experiment (XROI).[154]

The XROI experiment, contained in an ion-pumped, thermostated vacuum chamber, was protected from most mechanical vibration by an air spring, a granite support, and an acoustically damped enclosure.[155] Recent additions to the experimental setup included two new laser systems and a revised computer automation capability. One of the lasers, a variable-frequency local oscillator, was monitored continuously by an iodine-stabilized He-Ne laser used as the system reference. The other laser had a fixed frequency; it was used to monitor the trajectory of measurement components.

Measurements of the silicon lattice distance involved excursions of about 280 optical orders, traversed back and forth over periods of 10 h to 20 h. At each measurement point, the x-ray phase was determined by x-ray intensity measurements. The effect on the x-ray phase of pitch and yaw errors was estimated as trajectory curvature.

The primary output data of the XROI-silicon experiment was the rate of change of the x-ray phase with respect to distance covered within the crystal. Uncertainty in the silicon lattice spacing remained at a level of about 0.3 ppm, despite the XROI intrinsic capability for more accurate measurement. With the use of a double-crystal Laue x-ray spectrometer, stated the authors, lattice parameters of silicon crystals could be compared with an uncertainty less than a tenth as large.

[154] Richard D. Deslattes and Ernest G. Kessler, Jr., "Status of a silicon lattice measurement and dissemination exercise," *IEEE Transactions on Instrumentation and Measurement* **40**, No. 2, 92-97 (1991).

[155] The authors noted, however, that noises from nearby traffic and wind outside the building were easily detectable.

Polarized X-Ray Emission Spectroscopy

Capitalizing on earlier studies using synchrotron radiation to excite polarized x rays from molecules in the gas phase, Dennis W. Lindle, Paul L. Cowan, Terrence Jach, Robert E. LaVilla, Richard D. Deslattes, and their colleague R. C. C. Perera of the Lawrence Berkeley Laboratory undertook a detailed investigation of four chlorinated methane compounds—CH_3Cl, CF_3Cl, CF_2Cl_2, and $CFCl_3$.[156]

The group found that they could observe highly polarized molecular valence x-ray fluorescence from each of the test gases. Their experiments made use of x rays from the NIST synchrotron light source that were energy-selected by a large-aperture double-crystal monochromator, providing a well-resolved, intense excitation beam. Ionization chambers monitored both incident and transmitted flux, allowing the team to determine relative photoabsorption cross sections as a function of photon energy for each sample. A Johann-geometry secondary spectrometer analyzed the x-ray emission; use of a silicon crystal in connection with the secondary spectrometer provided polarization analysis of the emitted radiation.

A wealth of information on the x-ray emission properties of the test molecules came from the experiments, leading the researchers to observe that the degree and the direction of x-ray polarization in the chlorinated methanes was sensitive to the initial excitation energy in the core level region, and sensitive as well to the symmetry properties of the valence molecular orbitals involved in the x-ray emission process.

Shortly after completing the experiments described above, Lindle and Cowan, collaborating with Stephen H. Southworth and R. Mayer, found a strong anisotropy in the polarized chlorine K-V x rays emitted following resonant excitation of CF_3Cl gas with a linearly polarized x-ray beam. It was the first such observation.[157]

Previously it had been supposed that observation of anisotropic x-ray emission from molecular crystals and other solids arose from ordering inherent to the crystalline lattice structures; gas-phase samples were expected to exhibit isotropic x-ray emission. But not so, said the authors—excitation by an incident beam could leave an atom or a molecule in an anisotropic state, which then could influence subsequent photon emission.

The authors accomplished their feat in a fashion similar to the experiment described previously. Linearly polarized x rays from the NIST synchrotron light source were used to excite CF_3Cl molecules to energies near the Cl K edge, and the Cl K-V fluorescence emitted normal to the incident beam was analyzed using a curved-crystal spectrometer. The entire Cl K-V spectrum was analyzed at once by a position-sensitive

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[156] D. W. Lindle, P. L. Cowan, T. Jach, R. E. LaVilla, R. D. Deslattes, and R. C. C. Perera, "Polarized x-ray emission studies of methyl chloride and the chlorofluoromethanes," *Phys. Rev.* **A43**, No. 5, 2353-2366 (1991).

[157] S. H. Southworth, D. W. Lindle, R. Mayer, and P. L. Cowan, "Anisotropy of polarized x-ray emission from molecules," *Phys. Rev. Lett.* **67**, No. 9, 1098-1101 (1991).

proportional counter. Angular distribution of the x-ray emission was made possible by rotating the target chamber and the emission spectrometer. Variation in the energy of the exciting beam produced different patterns, interpreted by the researchers as representing the properties of different quantum levels of the target molecule. The authors were able to describe their results in terms of a classical model of the x-ray absorption-emission process.

Ionizing Radiation, Randall S. Caswell, chief

Radon, Invader of Homes

During the 1970s, environmentalists throughout the world became aware that radon gas, mainly in the form of ^{222}Rn, was seeping from the earth into certain dwellings, exposing the occupants to a low-level hazard from radioactivity. NBS quickly developed two approaches to ameliorate the radon problem. One provided a device to evaluate the natural rate of exchange of air between the inside and outside of homes. Owners of homes with rapid air exchange had little reason to fear a buildup of radon within their homes.

The other program involved detection of radon and the creation of standards of measurement for its concentration within homes. The radioisotope ^{222}Rn is a decay product of radioactive ^{226}Ra. The 4 day half-life of ^{222}Rn is short compared to the 1600 year half-life of its radium parent, which occurs naturally in many areas. The ubiquity and striking variability in concentration of radon gas was described simply by Klement:

> Radon is released as a decay product [of ^{226}Ra] from soils, rocks, waters, and building materials. It is also contained in natural gas and other fossil fuels. Radon concentrations vary considerably from place to place and from time to time. [158]

The enormous variability in concentration of terrestrial radon gas, plus the fact that its decay half-life was only 4 days, made it a challenging problem for standards scientists. In June 1989, a 1 day seminar in Braunschweig, Germany, brought together an international group of scientists who shared information about efforts in their own countries to perfect calibration schemes for use in radon detection programs operated by state and local governments. According to J. M. Robin Hutchinson, the favored methods for detection and quantification of radon gas involved ionization-chamber or scintillation-cell measurements of alpha particles emitted by ^{222}Rn samples, and gamma-ray measurements of radon decay products.[159]

[158] Alfred W. Klement, Jr., "Natural sources of environmental radiation," section in *CRC Handbook of Environmental Radiation* (Boca Raton, Florida: CRC Press, Inc. 1982), Alfred W. Klement, Jr., editor, pp. 5-22.

[159] J. M. R. Hutchinson, "Radon measurement standards and calibration," Preface to issue No. 2, *J. Res. NIST* **95**, (1990).

The NIST calibration system was described by R. Collé, Hutchinson, and M. P. Unterweger. The system used as its reference ^{226}Ra sources three Standard Reference Materials consisting of radium solutions of different concentrations. The radon gas generated by the decay of the radium atoms in solution was separated from the solution in a gas-handling system, then the alpha particles resulting from the decay of the radon atoms were detected in pulse ionization chambers. NIST used the ionization chambers preferentially because of their stability.[160]

Perfecting a Radioactive Standard

Because ^{186}Re was under consideration in the 1990s as a therapeutic radioactive isotope, it was important to understand its half-life and its types and levels of radioactive decay. Bert M. Coursey, Jeffrey Cessna, Dale D. Hoppes, Frank J. Schima, and Michael P. Unterweger, along with their colleagues E. Garcia-Torano, A. Grau Malonda, J. M. Los Arcos, and M. T. Martin-Casallo of the Center for the Investigation of Energy and Technology of Madrid, Spain, and D. B. Golas and D. H. Gray of the U.S. Council for Energy Awareness in Washington, DC, undertook this task.[161]

Much was already known about the ^{186}Re decay scheme. The isotope decayed with a half-life of about 89 hours by electron capture to ^{186}W and by beta-emission to ^{186}Os. Radiation associated with the decay included a 1 MeV beta particle, a 940 keV beta particle, and gamma rays of 123 keV and 137 keV.

Using ^{186}Re samples obtained commercially in the form of dilute solutions of sodium perrhenate in saline solution, the group employed a method of $4\pi\beta$ liquid-scintillation efficiency tracing—developed jointly by their two institutions—to standardize the activity of the radionuclide. The team then made use of standard solutions to prepare calibration sources for semiconductor spectrometer measurements of the x-ray and gamma-ray probabilities per decay. They also determined the ^{186}Re half-life and obtained calibration factors for the NIST primary ionization chamber.

Taking account of detectable radioactive impurities in their samples, the group obtained a value for the ^{186}Re half-life of 89.25 ± 0.07 h.

Estimating Absorbed Dose in Food Processing

An important feature of the use of ionizing radiation for the sterilization of foods was the accurate determination of the amount of radiation absorbed during the sterilization process. A multinational group including Marc F. Desrosiers of NIST, G. L. Wilson and D. R. Hutton of the Department of Physics at Monash University in Victoria, Australia, and C. R. Hunter of the Monash Department of Anatomy offered an evaluation method based on electron paramagnetic resonance measurements of irradiated food samples.

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[160] R. Collé, J. M. R. Hutchinson, and M. P. Unterweger, "The NIST primary radon-222 measurement system," *J.Res. NIST* **95**, 155-165 (1990).

[161] B. M. Coursey, J. Cessna, E. Garcia-Torano, D. B. Golas, A. Grau Malonda, D. H. Gray, D. D. Hoppes, J. M. Los Arcos, M. T. Martin-Casallo, F. J. Schima, and M. P. Unterweger, "The standardization and decay scheme of rhenium-186," *Applied Radiation and Isotopes* **42**, No. 9, 865-869 (1991).

Effects from the irradiation of foods were known to show up later in certain analyses by electron paramagnetic resonance (EPR) of bone samples. By re-irradiating samples, one could generate curves of EPR signal amplitude *vs* radiation dosage; extrapolating such curves back to zero signal amplitude then provided an estimate of the initial dose. The EPR signals persisted much longer than the shelf life of the irradiated foods, thus potentially providing a suitable standardized method for evaluating initial doses in the radiation-processing method.

The group focused their efforts on mathematical methods for best analyzing the EPR signal strength-dosage data to provide the most accurate estimates of initial dose. They irradiated samples prepared from ground chicken bones with various measured doses from a ^{137}Cs gamma-ray source. The first irradiation in each case was assumed to be the "initial" dose; subsequent irradiation doses were analyzed to derive experimental estimates of the initial dose.

For initial doses at levels 0.5 kGy and 1.76 kGy, a simple linear plot of dose vs signal amplitude provided acceptable estimates of the initial dose. However, initial doses as high as the legal maximum for chicken processing—8 kGy in certain countries—were estimated less well by the linear approach. Possible complicating features of the method, particularly an unexpected variation in the stablility of the EPR signal amplitude with time after irradiation and a dosage dependence different from linear, were contemplated by the group. In a follow-up article, Desrosiers utilized a response function in which the EPR signal intensity varied with the dose in an exponential manner. This step provided more accurate estimates of initial radiation dosages.[162]

Radiation Transport Theory and Martin J. Berger

During April 1990, a *Symposium on the Physics of Electron Transport* was held at NIST/Gaithersburg in honor of Martin J. Berger, a major contributor to the field during his 36 years at NBS/NIST that ended with his retirement in 1988. The symposium was attended by more than 60 colleagues from North America and Europe. Three sessions held over the 2 days of the symposium featured 8 papers on various aspects of the topic, including five presented by Berger and by four of his long-term co-workers Ugo Fano, Lewis V. Spencer, Stephen M. Seltzer, and Mitio Inokuti.[163]

[162] M. F. Desrosiers, G. L. Wilson, C. R. Hunter, and D. R. Hutton, "Estimation of the absorbed dose in radiation-processed food—1. Test of the EPR response function by a linear regression analysis," *Appl. Radiation and Isotopes* **42**, No. 7, 613-616 (1991). See also M. F. Desrosiers, "Estimation of the absorbed dose in radiation-processed food—2. Test of the EPR response function by an exponential fitting analysis," *Appl. Radiation and Isotopes* **42**, No. 7, 617-619 (1991).

[163] Martin J. Berger, "Differences in the multiple scattering of positrons and electrons," *Appl. Radiation and Isotopes* **42**, No. 10, pp. 905-916 (1991); Stephen M. Seltzer, "Electron-photon Monte Carlo calculations: the ETRAN code," ibid., pp. 917-942; Lewis V. Spencer, "Notes on electron penetration," *ibid.*, pp. 943-952; U. Fano and Ning-Yi Du, "Dissipative polarization by slow electrons," *ibid.*, pp. 975-978; and Mitio Inokuti, "Subexcitation electrons: an appraisal of our understanding," *ibid.*, pp. 979-984.

In a synopsis of Berger's career, Randall S. Caswell and Robert Loevinger noted that Berger had developed a unique approach to photon transport based upon the use of Monte Carlo calculations. Identified by the acronym ETRAN, a computer program telescoped the multitudinous collisions between ionizing particles and complex media into a manageable series of random-walk segments characterized by energy loss and directional changes. The program, initially developed in the 1960s, was, they said, still the most accurate available for transport calculations.

Time and Frequency, Donald B. Sullivan, chief

Rydberg Constant

One of NIST's contributions to the science of fundamental physical constants that has not been discussed elsewhere in this volume came from Howard P. Layer, James C. Bergquist, and their colleagues Ping Zhao, W. Lichten, and Zhi-Xiang Zhou from Yale University.[164] They performed a new determination of the Rydberg constant, providing the value (109 737.315 73 $\pm$ 0.000 03) cm^{-1}. At the heart of their determinations was the wavelength and frequency reference embodied in a $^{127}I_2$-stabilized helium-neon laser.

The experiment accomplished by Layer, Bergquist and their colleagues involved measurement of the 2S-4P Balmer-β line in hydrogen and deuterium. A cw dye laser beam was used to quench the metastable atomic beam through the transition, with the iodine-stabilized laser serving as the frequency and wavelength standard. The experiment yielded four independent measurements of the Rydberg constant (in cm^{-1}): 109 737.315 770; 109 737.315 720; 109 737.315 692; and 109 737.315 741. The four values differed from the average, 109 737.315 731 cm^{-1}, by no more than 4 parts in 10^{10}.

Essays on Time and Frequency

The Institute of Electrical and Electronics Engineers devoted the July 1991 issue of its proceedings to papers on time and frequency. The editors of the special issue were James L. Jesperson and D. Wayne Hanson, veteran staff members of NIST's Time and Frequency division. Among the scientists invited to review progress in various aspects of the subject, we take note of these:

- Norman F. Ramsey, emeritus professor of physics at Harvard University, who received the 1989 Nobel Prize in physics for work basic to the development of atomic time and frequency standards ("The past, present, and future of atomic time and frequency").

- Donald B. Sullivan and Judah Levine of NIST's Time and Frequency division ("Time generation and distribution").

- Wayne M. Itano, also with the NIST Time and Frequency division ("Atomic ion frequency standards").

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[164] Ping Zhao, W. Lichten, Zhi-Xiang Zhou, H. P. Layer, and J. C. Bergquist, "Rydberg constant and fundamental atomic physics," *Phys. Rev.* **A39**, No. 6, pp. 2888-2898 (1989).

- Steven L. Rolston and William D. Phillips of NIST's Atomic Physics division ("Laser-cooled neutral atom frequency standards").[165]
- Jacques Rutman, Deputy Director of the French Committee of the International Union of Radio Science (URSI), and Fred L. Walls, a senior staff member of the Time and Frequency division ("Characterization of frequency stability in precision frequency sources").

We briefly outline 3 of these contributions in the next sections.

Stability of Precise Frequency Sources

Publication of the special issue mentioned above provided a fine opportunity for Fred L. Walls and Jacques Rutman to review progress in high-precision frequency standards over the past three decades. Their discussion focused on the question of stability of frequency sources.

The authors noted the advent of the cesium beam atomic clock in 1955, which led to the commercial manufacture of such time-keepers—cesium beams and optically pumped rubidium clocks—by the thousands. Progress in the quality of oscillators controlled by quartz crystals made them useful, too, in such applications as fundamental metrology, telecommunications, space projects, and commercial broadcasting.

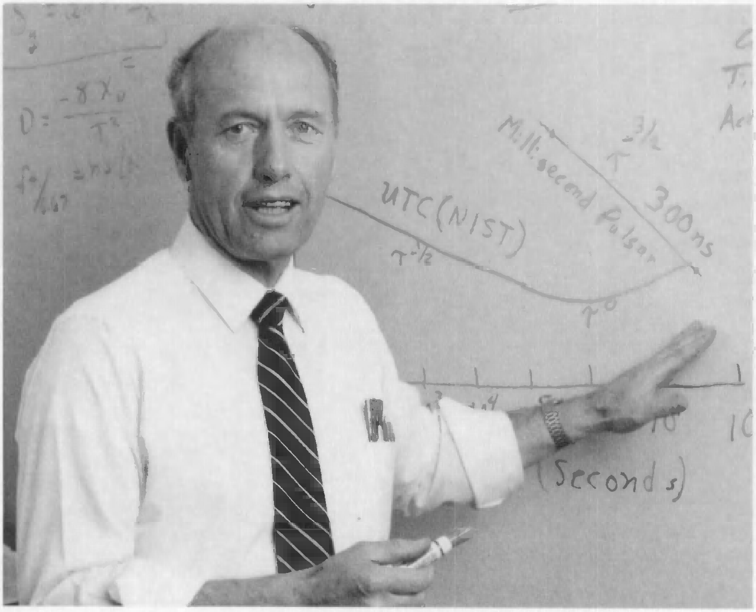
In the early 1960s, the stability of the frequency underlying the chronometric devices was addressed to facilitate comparisons among laboratories and among devices, and to assess the relation between frequency stability and satisfactory performance in specialized applications.

Rutman and Walls pointed out that frequency stability of real oscillators could be characterized only after considering the nature of such devices. For example, their (theoretically) perfect sine-wave outputs were perturbed by random noise or drifts caused by aging or environment. Solution of this problem required development of mathematical models that could account for such deviations from sinusoidal output signals. These models allowed the introduction of tools useful for the characterization of fluctuations—correlation functions, spectral densities, averages, and standard deviations. Quickly, two types of analysis developed: spectral descriptions of phase- and frequency fluctuations in the Fourier frequency domain, and averages of fluctuations in the time domain. NIST's David W. Allan was a pioneer in the use of variances in the study of frequency stability.

The authors brought up to date the progress of stability studies rooted in the concepts they had outlined. They noted, however, that in 1991 there was no single analytical mode that would produce a "best" measure of frequency stability.[166]

[165] This work is discussed in *Sharper Lines from Cooler Atoms* in Ch. 4. Phillips shared the 1998 Nobel Prize in physics for his work on laser cooling.

[166] Jacques Rutman and F. L. Walls, "Characterization of frequency stability in precision frequency sources," *Proc. IEEE* **79**, No. 7, pp. 952-960 (1991).



David W. Allan explained the methods used to measure performance of different clocks and to transfer time worldwide.

Frequency Standards Based on Trapped Ions

Wayne M. Itano described for the special time and frequency issue the progress to date on frequency standards based on the use of trapped ions, a topic discussed elsewhere in this volume. The basic premise in such devices was that the frequency of an oscillator could be locked to a resonance corresponding to a transition between two energy levels of an atomic ion, held in space by a combination of electric and magnetic fields.

A microwave frequency standard that was already a reality in 1991 utilized $^{199}Hg^{+}$. Others under development involved $^{9}Be^{+}$, $^{137}Ba^{+}$, or $^{171}Yb^{+}$. Some studies were then in progress at NIST on the use of narrow-linewidth transitions in single trapped ions for optical frequency standards.

Itano briefly described the Penning trap, which utilized static electric and magnetic fields to hold the ions near the center of the trap, and the Paul trap, involving a radiofrequency potential applied to electrodes of particular geometries. He noted that the accuracy of the frequency derived from trapped ions depended upon phenomena that could shift the resonance frequency of the ions—such factors as collisions with background gas, perturbations caused by external fields, and Doppler shifts. Cooling of the ions provided a useful escape from Doppler effects. Itano called attention to the use of laser cooling for this purpose by himself and David J. Wineland[167] and by Claude Cohen-Tannoudji and William D. Phillips.[168]

Often, Itano pointed out, it was necessary to trade accuracy for stability in trapped-ion frequency standards.

Itano outlined the use of $^{199}Hg^{+}$ in both microwave and radiofrequency standards, he noted NIST work on laser cooling of $^{9}Be^{+}$ ions, and he mentioned other frequency standards based on the probing of a single ion.[169]

Technology of Frequency and Time

Donald B. Sullivan and Judah Levine discussed at length for the IEEE Proceedings the generation and distribution of frequency and time signals using the latest available methods.[170] The two scientists opined that laser manipulation of atoms and satellite time transfer already provided the potential for accuracy that would be superior to existing atomic frequency standards. They pointed out that the stability of a frequency standard was a measure of its ability to remain within specific limits for a given time, whereas its accuracy was based on comparisons with a physical model of the standard.

Frequency standards, according to Sullivan and Levine, jumped in accuracy with the earlier developments of quartz-crystal oscillators and atomic clocks. Quartz oscillators constituted the least expensive class of frequency standards. Increasingly superior in performance—but also increasingly expensive—were rubidium standards, cesium-beam standards, and hydrogen masers. A limit on the performance of these atomic standards, however, was the Doppler effect, arising from their operation at room temperatures or even higher temperatures.

Comparisons of clocks benefitted enormously from the use of satellite methods, which allowed uncertainties as low as 10 ns on a global level, better than typical performance characteristics of individual clocks.

So-called "trapped ion" frequency standards, then under development by David J. Wineland, Robert E. Drullinger, and Fred L. Walls in Boulder, and "trapped neutral atoms," then under development by William D. Phillips and his group in Gaithersburg, offered two major advantages over conventional atomic devices—essentially zero first-order Doppler shifts because of the near-immobility of the ions or atoms, and reduction of transition linewidths because of the availability of long observation times.

Sullivan and Levine described current methods of time transfer as falling into one of three categories; one-way, common-view, and two-way. In the one-way case (typified by the broadcast signals from WWV), a time signal from a source to a user was delayed by the medium through which the signal passed; the delay had to be estimated

[167] D. J. Wineland and W. M. Itano, "Laser cooling," *Physics Today* **40**, pp. 34-40, June 1987.

[168] C. N. Cohen-Tannoudji and W. D. Phillips, "New mechanisms for laser cooling," *Physics Today* **43**, pp. 33-40, October 1990.

[169] Wayne M. Itano, "Atomic ion frequency standards," *Proc. IEEE* **79**, No. 7, pp. 936-941 (1991).

[170] Donald B. Sullivan and Judah Levine, "Time generation and distribution," *Proc. IEEE* **79**, No. 7, pp. 906-914 (1991).

Wayne M. Itano adjusted an external frequency doubler, which was part of a laser setup for the mercury ion clock experiment.

in order to synchronize the user's signal. In the common-view case (typified by the use of the Global Positioning Satellite), both the source and the user compared their clocks to a reference visible to both. The difference between the medium-based delays experienced by the source and the user—often much smaller than either time delay considered by itself—had to be estimated to yield the correct relation between source and user clocks. In the two-way case (for example, two-way broadcasting between the source and the user via a satellite), the source and the user would exchange timing data. In favorable instances such an exchange would allow nearly perfect cancellation of the delay error. Sullivan and Levine estimated best-case accuracy at 100 ns and available stability at 10 ns. They saw the evaluation of time-delay error as the outstanding problem facing progress in time transfer.

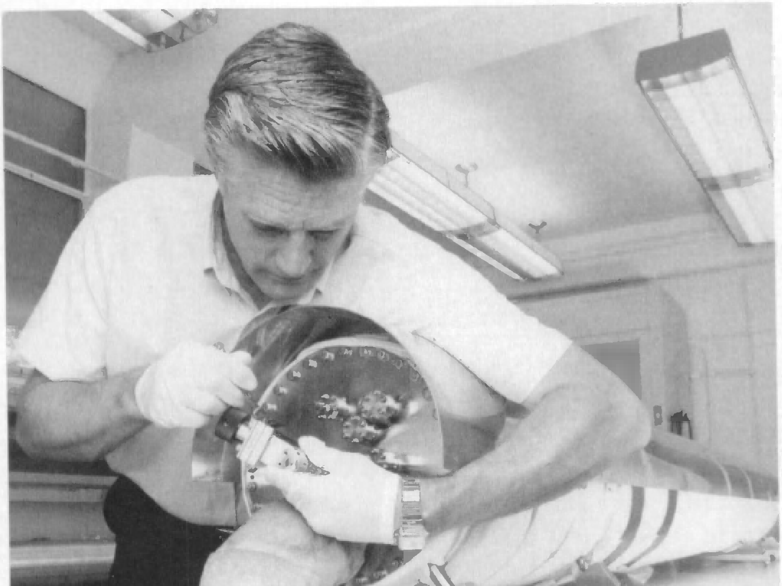
Optically Pumped Cesium Frequency Standard

In 1991, Time and Frequency Division scientists were in the process of developing an optically pumped, cesium-beam primary frequency standard. They were confident that such an instrument would exhibit unusually good stability compared to conventional cesium standards.

The new device was conceived by Robert E. Drullinger, David J. Glaze, John L. Lowe, and Jon H. Shirley. In considering the improvement, they analyzed several systematic uncertainties that afflicted the conventional cesium apparatus: fluorescent light shift; second-order Doppler shift; end-to-end cavity phase shift; Rabi-pulling; cavity-pulling; Majorana effects; RF spectral purity; and magnetic field uniformity.

The new instrument featured a 3 mm diameter atomic beam, derived from a new oven design. The vacuum chamber was 25 cm in diameter and 2.5 m long. Instead of using "off-the-shelf" laser diodes, the authors employed a laser-line-narrowing method based on optical feedback from a high *Q* cavity. They felt that their improvements would yield a line-center accuracy somewhat better than one part in 10^8.

First results were promising, leading the group to predict excellent short-term stability and an overall frequency accuracy of about one part in 10^{14}.[171]



Physicist David J.Glaze adjusted NIST 7, an atomic clock used as an international time standard.

[171] Robert E. Drullinger, David J. Glaze, J. L. Lowe, and Jon H. Shirley, "The NIST optically pumped cesium frequency standard," *IEEE Trans. on Instrum. and Meas.* **40**, No. 2, pp. 162-164 (1991).

Quantum Physics, David W. Norcross, chief

Making New Lasers

Stephen R. Leone, a staff member of both the Quantum Physics division and the Joint Institute of Laboratory Astrophysics, and his JILA colleague Harold C. Miller spent the early 1990s investigating the lasing properties of various systems in an effort to produce lasers for special applications.

With Katsuyoshi Yamasaki of the University of Tokyo and John E. Smedley of Bates College, the two researchers demonstrated that a laser operating between 300 nm and 350 nm could be obtained using the unstable radical sulfur monoxide (SO). Lasing had already been demonstrated in heteronuclear diatomics—NO, IF, and NaK—by pumping energy into the ground state of the molecule and exciting it to a higher level. Such systems, the authors noted, provided excellent opportunities for the study of the influence of the dynamic Stark effect and the stimulated Raman effect on laser processes. In certain cases, the lasers offered high-resolution, Doppler-free spectroscopy as well.

The group knew that the SO radical could be prepared by irradiating sulfur dioxide at 193 nm. They used an argon fluoride excimer laser, irradiating flowing SO_2 for this purpose; it produced SO in the $X^3\Sigma^-$ electronic state. A pumping pulse at 256 nm was obtained by frequency-doubling the visible output of a dye laser. This pulse populated the $B^3\Sigma^-$ state of SO. Emission from the excited SO radicals was observed using monochromator-photomultiplier detection in the 300 nm to 400 nm range. Spontaneous emission appeared in the form of about a dozen lines in that range, and four relatively sharp stimulated emission lines appeared in the range 300 nm to 350 nm.[172]

Miller and Leone also collaborated with Robert L. Pastel of the University of New Mexico and G. D. Hager of Kirtland Air Force Base in Albuquerque to demonstrate lasing at 2.7 μm in atomic bromine. Technical needs for efficient lasers in the mid-infrared spectral range made this work particularly useful.

The authors prepared excited bromine atoms by photolyzing IBr with light at 532 nm. A pump laser—a frequency-doubled neodymium-yttrium-aluminum garnet laser operated at 532 nm—produced laser radiation from the excited bromine atoms with a temporal distribution that varied noticeably with the pump energy. The 2.7 μm laser emission appeared to arise from the $4\,^2P_{1/2}$–$4\,^2P_{3/2}$ transition in atomic bromine. The maximum pump energy conversion efficiency was calculated to be 1.5 %, relatively high for such a process.[173]

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[172] Harold C. Miller, Katsuyoshi Yamasaki, John E. Smedley, and Stephen R. Leone, "An optically pumped ultraviolet laser on SO($B^3\Sigma^-$–$X^3\Sigma^-$)," *Chem. Phys. Lett.* **181**, No. 2, 3, pp. 250-254 (1991).

[173] Robert L. Pastel, G. D. Hager, Harold C. Miller, and Stephen R. Leone, "Efficient Br* laser pumped by frequency-doubled Nd:YAG and electronic-to-vibrational transfer-pumped CO_2 and HCN lasers," *Chem. Phys. Lett.* **183**, No. 6, pp. 565-569 (1991).

Electron-Impact Excitation Cross Sections

A new technique for the measurement of cross sections for the excitation of multiply charged ions by electron impact was put forward by a group of scientists at the Joint Institute for Laboratory Astrophysics late in 1990. The group included Gordon H. Dunn, on the staff of JILA and also the NIST Quantum Physics Division; JILA members E. K. Wahlin and J. S. Thompson; R. A. Phaneuf and D. C. Gregory of the Oak Ridge National Laboratory; and A. C. H. Smith of University College in London.

The authors noted the important role played by highly charged ions in studies of fusion, lasers, and astrophysics, as well as the lack of experimental information on electron-impact excitation of such species.

To help close the gap in that area of physics, the authors developed an apparatus in which electrons were merged with ions in an analyzer involving crossed electric and magnetic fields. After colliding with the ions, the electrons were removed from the stream with a second analyzer and detected with a position-sensitive detector. The detection efficiency approached unity—orders of magnitude higher than conventional fluorescence or differential monochromator techniques.

The new method had the additional advantages of providing absolute cross section values, operating near the critical threshold, and providing data on non-radiating states as well as those that radiated.

The group demonstrated their new method on triply ionized silicon. They chose that species particularly because it resembled sodium and thus could be described with confidence in theoretical terms, thus providing as a bonus an experimental comparison with theory.

The cross section for 3*s* to 3*p* excitation of Si^{+++} by electron impact using the new technique was determined at the 90 % confidence level to be 10^{-15} cm^2 $\pm$20 % at 9 eV electron energy, in good agreement with theoretical predictions.[174]

Spectroscopy from the Hubble Space Telescope

In August 1991, the *Astrophysical Journal Letters* set aside an issue for the first reports of spectroscopy accomplished using the Hubble Space Telescope. The editors dedicated the issue to Lyman Spitzer, known as the "Father of the Hubble Space Telescope." Spitzer, professor of astronomy at Princeton University for some 30 years, had worked towards the deployment of an orbiting astronomical telescope from the end of World War II.

One of the reports in the special issue described the first results obtained with the Goddard High-Resolution Spectrograph (GHRS) as part of the Science Assessment Program for the Hubble Space Telescope. Members of the GHRS team included Jeffrey L. Linsky, a staff member of JILA and NIST; JILA staff scientist Alexander

[174] E. K. Wahlin, J. S. Thompson, G. H. Dunn, R. A. Phaneuf, D. C. Gregory, and A. C. H. Smith, "Electron-impact excitation of Si^{3+} (3*s*–3*p*) using a merged-beam electron-energy-loss technique," *Phys. Rev. Lett.* **66**, No. 2, pp. 157-160 (1991).

Brown; Kenneth G. Carpenter, Richard D. Robinson, Glenn M. Wahlgren, and Thomas B. Ake, of the NASA/Goddard Space Flight Center; Dennis C. Ebbets of the Bell Aerospace Systems Group; and Frederick M. Walter of the State University of New York at Stony Brook.

The report emphasized the types of observations that could not have been obtained without the new equipment, in this case high-precision spectra from the star α Tauri. The authors wrote:

> This [Hubble project] study of α Tau produced the highest quality ultraviolet spectra ever obtained of a cool star. The high dynamic range of the data allowed detection of 25 new emission lines from α Tau, 10 of which do not appear in the solar spectrum. The accurate wavelength calibration led to a confident measurement of flow velocities for most of the spectral lines, but also indicated the need for more precise laboratory wavelengths for ions such as Co II.[175]

It was clear that orbiting astronomical instruments, disparaged in their early days because of a "birth defect" in the Hubble telescope optics, had before them a very useful career.

Linsky was also part of a group utilizing the Very Large Array (VLA), a radio telescope facility of the National Radio Astronomy Observatory, to survey some 26 main-sequence, giant, and supergiant stars in a search for radio emissions at 2 cm and 6 cm wavelengths. Linsky was joined in the search by P. G. Judge of JILA, S. A. Drake of ST Systems Corporation, and M. Elitzur of the University of Kentucky. Within the limits of the VLA sensitivity, the group obtained no signals from the dwarf stars, indicating that such stars emit weakly, if at all, at radio frequencies. Three of the cool stars—two M giants and an infrared carbon star—were identified as radio sources. On the basis of their measurements, the group estimated the temperature of the infrared carbon star, IRC + 10216, as only about 2000 K. [176]

Materials Science and Engineering Laboratory

In 1984, Lyle H. Schwartz came to NBS as director of the Bureau's Center for Materials Science after 20 years with Northwestern University, including service as director of the N. U. Materials Research Center beginning in 1979. The following year, the center became an institute—like the Institute for Computer Sciences and Technology an entity separate from the National Measurement Laboratory and the National Engineering Laboratory. The Institute for Materials Science and Engineering (IMSE) comprised an Office of Non-Destructive Evaluation and five divisions, devoted to studies in inorganic materials, fracture and deformation, polymers, metallurgy, and reactor radiation.

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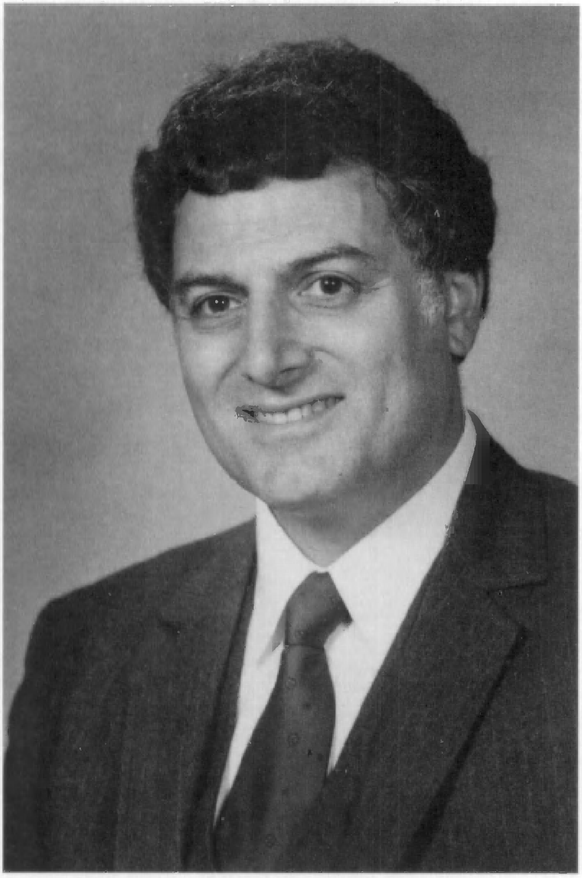
[175] Kenneth G. Carpenter, Richard D. Robinson, Glenn M. Wahlgren, Thomas B. Ake, Dennis C. Ebbets, Jeffrey L. Linsky, Alexander Brown, and Frederick M. Walter, "First results from the Goddard High-Resolution Spectrograph: the chromosphere of α Tauri," *The Astrophys. J.* **377**, L45-L48 (1991).

[176] S. A. Drake, J. L. Linsky, P. G. Judge, and M. Elitzur, "Radio-continuum observations of a variety of cool stars," *The Astronomical Journal* **101**, No. 1, pp. 230-236 (1991).



Lyle H. Schwartz, an expert in the phase transitions in iron alloys, x-ray and neutron diffraction, and catalysis, joined NBS as Director of the Materials Science and Engineering Laboratory in 1984.

As NBS became NIST in 1988, IMSE became the Materials Science and Engineering Laboratory, with the Inorganic Materials and the Fracture and Deformation divisions reconstituted as Ceramics and Materials Reliability, respectively. The Laboratory increased its emphasis on the advanced materials needed for up-to-date manufacturing and construction processes. Research initiatives focused on ceramics, metals, polymers, composites, and superconductors. In mid-1991, the Office of Non-Destructive Evaluation was renamed the Office of Intelligent Processing of Materials.

Office of Intelligent Processing of Materials, H. Thomas Yolken, chief

The OIPM had but three scientists on its staff in 1991; H. Thomas Yolken, John P. Gudas—Yolken's deputy—and George Birnbaum. Theirs was a task in matrix management, to facilitate work in specific technical areas without direct supervisory power over the rsearchers who were involved in the investigations.

The change in name of Yolken's office was indicative of a larger mission—not only to continue to encourage studies of non-destructive testing, but to focus work in technical divisions both within MSEL and elsewhere in NIST on joint materials projects with industrial firms. These latter projects involved the processing of metal powders, modernized steel manufacture, and metallic joining techniques. Companies such as General Electric, United Technology Pratt and Whitney, Crucible Materials Corporation, and Martin Marietta Energy Systems teamed with the Department of Energy and NIST scientists to create the projects.

Ceramics, Stephen M. Hsu, chief; succeeded by Stephen W. Freiman, 1991

Research in ceramics in the 1990s covered a range of topics—processing methods, including the effect of sintering on particle size and shape; surface properties as related to wear and machinability; electro-optic crystals; mechanical properties; phase equilibria; and measurement techniques. A few examples from the work of this division follow.

Standards and Data

In 1991, three new Standard Reference Materials—SRM 1414, a lead-silica glass resistivity reference; SRM 8501, a lubrication oil-oxidation catalyst; and SRM 710a, a soda-lime-silica glass reference—joined more than 70 other standard materials developed and produced by division scientists.

Other standards accomplishments during this period included:

- Development of 25 analytical procedures for the evaluation of the properties of ceramic powders.[177]
- Adoption by the American Society for Testing and Materials of two tests for mechanical properties of ceramics developed by a group led by George D. Quinn.[178]
- Completion of a 350-entry database on material and tribological properties of advanced ceramic materials, by Ronald G. Munro and Edwin F. Begley. Properties data evaluated by Munro and Begley included thermodynamic transport properties, heat capacity, bulk and shear moduli, hardness, fracture strength, and creep properties.[179]

[177] See, for example, S. G. Malghan, "Silicon nitride powders and their processing," pp. 562-576 in *Advanced Materials Processing*, P. Ganguli, editor (Amsterdam: Elsevier Science Publishers B. V., 1991).

[178] G. D. Quinn, et al., "Flexural Strength of Advanced Ceramics at Ambient Temperatures," ASTM Standard C-1161, December 1990.

[179] E. F. Begley and R. G. Munro, "The structural ceramics database," *NISTIR 4601,* 1991.

- Completion of three volumes as part of a joint NIST-American Ceramic Society phase-diagram project involving Helen M. Ondik, Stephen W. Freiman, Mary A. Clevinger, Thomas R. Green, Kimberly M. Kessell, Nils Swanson, and Carla G. Messina. The texts produced were: volume 9 in the series "Phase Diagrams for Ceramists"; a 1991 annual report; and a monograph containing nearly 200 phase diagrams pertaining to high-temperature superconductivity.

Characterization of Ceramic Powders

Robert D. Shull, Joseph J. Ritter, and Lydon J. Swartzendruber were interested in the possibilities of preparing magnetic composites with made-to-order properties. Gelled composites of iron and silica, prepared with iron-containing particles of about 2 nm diameter, had been found to be paramagnetic at room temperatures but likely to exhibit ferromagnetism after certain types of processing. The investigation by the three scientists focused on nanocomposites of silica gel with various concentrations of iron in the range 11 % to 40 %. They determined the iron particle sizes by the use of Mössbauer spectra. After gelling and curing the composites, they heated them in hyrdrogen to reduce the iron, then separated them into two samples for further treatment. One set of samples was re-heated in hydrogen to 770 °C, then annealed for an hour in an ammonia atmosphere at 475 °C. The other set was annealed in ammonia at 375 °C for a day. Mössbauer analysis of the first set of samples showed a marked change in the iron component to a form containing iron silicate with a small admixture of α Fe. Weak ferromagnetism at room temperature strengthened continuously on cooling to 10 K, with no apparent phase change. Evidently the short ammonia anneal had no effect on these samples.

The second set of samples exhibited superparamagnetism and an indication of magnetic spin-glass behavior. The magnetic moment per iron atom was enhanced by factors of three to four by the lengthy, medium-temperature ammonia annealing.[180]

Facilitating Crack Detection With Electron Microscopy

Understanding the mechanics of cracks in ceramics required the observation of the crack area during the initiation of stress on samples. To facilitate the use of the scanning electron microscope (SEM) in crack investigations, Brian R. Lawn and James F. Kelly, in collaboration with their colleague Jürgen Rödel of Lehigh University, designed and built a new testing device.

Designed for use with disks about 10 cm in diameter, the device incorporated a piezoelectric force actuator connected by pivot arms to either side of the test disk, which was notched in the crack-test area. The entire device was housed in the sample chamber of an SEM. Actuating voltage to the piezoelectric stressor could be pre-programmed or externally controlled. The SEM in use allowed videotaping of the entire test procedure.

[180] R. D. Shull, J. J. Ritter, and L. J. Swartzendruber, "Change in magnetic state of Fe + silica gel nanocomposites due to low temperature treatment in ammonia," *J. Appl. Phys.* **69**, No. 8, 5144-5146 (1991).

The researchers used alumina and soda-lime glass test disks to illustrate the capability of the new device. "Starter" cracks were initiated in the disks using standard procedures. They found that crack extension generally occurred in a discontinuous fashion between grains. Active grain bridges appeared at intervals over the entire length of the cracks; these were easily seen in illustrations provided in the report. The authors analyzed the crack growth quantitatively to evaluate the toughness of the specimens used in the tests.[181]

Machining Ceramics

The cost of machining ceramic materials in the 1990s occasionally accounted for 90 % of the total cost of highly precise components, in part because of inadvertent damage during the machining process. Said Jahanmir, Lewis K. Ives, Arthur W. Ruff, and Marshall B. Peterson set about to study the state of ceramic machining in America and, if possible, to identify research areas which could lead to significant improvements. Their efforts, recorded in a NIST Special Publication, included literature searches, visits to industrial firms involved in machining ceramics, and the products of a NIST workshop on the topic held in September 1990.[182]

What did they find? First of all, they found that, indeed, the cost of machining was the primary impediment to the introduction of advanced ceramic parts into industrial goods—more so than the admittedly high costs associated with raw materials, processing, and quality control. They also found a serious gap in the store of data on machining methods for ceramics, a fault that tended to keep machining costs high. They identified rapid machining of advanced ceramics as unknown territory that needed careful investigation, along with techniques for the assessment of damage to ceramic components in machining and for automated machining.

The authors recommended a broad program of research to address the roadblocks slowing the adoption of advanced ceramic materials as routine industrial tools:

- Optimization of the grinding process.
- Chemically assisted machining.
- Grinding wheels for machining of ceramics.
- Direct damage assessment methods.
- Non-destructive methods for machining damage evaluation.
- Sensors for real-time surface finish and damage detection.
- Standard Reference Materials for use in tool calibration and evaluating machined parts.
- Post-machining damage remediation.
- Development of high-strength, machinable ceramics.
- Automated systems for high-volume production.

[181] Jürgen Rödel, James F. Kelly, and Brian R. Lawn, "In situ measurements of bridged crack interfaces in the scanning electron microscope," *J. Am. Ceram. Soc.* **73**, No. 11, 3313-3318 (1990).

[182] S. Jahanmir, L. K. Ives, A. W. Ruff, and M. B. Peterson, "Ceramic machining: Assessment of current practice and research needs in the United States," *NIST Special Publication 834*, June 1992, 102 pp.

Properties of Alumina

A group including Gabrielle G. Long, Susan Krueger, R. A. Gerhardt, and R. A. Page used the Small Angle Neutron Scattering facility at the NIST reactor to examine the evolution of pore microstructure in glassy silica and polycrystalline alumina as a function of sintering. They showed that the two major sintering mechanisms, viscous flow and diffusion, led to similar surface-porosity levels.[183] Their work led to improvec models for ceramics processing and better predictability for the microstructure of ceramics.

Another group, including Steven J. Bennison, J. Rödel, S. Lathabai, P. Chantikul, and Brian R. Lawn, also investigated the properties of alumina. Focusing on the toughness of the ceramic, which they assumed derived from the interlocking of grains to stop the progression of cracks, they developed a formalism to describe the origins of ceramic toughness. Many features were considered—the strength of internal boundaries, internal stress levels, and other details of alumina microstructure. Scanning electron microscopy observations provided information on many degradation mechanisms, leading to the conclusion that alumina ceramics could be processed to order for various properties.[184]

Creep in Ceramics

Sheldon M. Wiederhorn, D. F. Carroll, and D. Ellis Roberts developed an experimental technique for evaluating the phenomenon of creep in ceramics. The method utilized laser extensometry to follow the displacement of markers attached to the test sections of the samples, which were shaped like dog bones. The tensile fixture featured hot grips and could be used to temperatures as high as 1500 °C.[185]

Materials Reliability, Harry I. McHenry, chief

The Materials Reliability Division was located in the NIST/Boulder laboratories. Its major focus was placed on advanced materials. Division staff developed measurement techniques for process control, for nondestructive evaluation, and for reliability assessment. Long experience in topics such as cryogenics made the scientists aware of the importance of carefully preparing materials for potentially hostile environments in particular applications.

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[183] G. G. Long, S. Krueger, R. A. Gerhardt, and R. A. Page, "Small Angle Neutron Scattering characterization of processing/microstructure relationships in the sintering of crystalline and glassy ceramics," *J. Materials Res.* **6**, No. 12, 2706-2715 (1991).

[184] S. J. Bennison, J. Rödel, S. Lathabai, P. Chantikul, and B. R. Lawn, "Microstructure, toughness curves and mechanical properties of alumina ceramics," *Toughening Mechanisms in Quasi-Brittle Materials*, pp. 209-233, 1991.

[185] D. F. Carroll, S. M. Wiederhorn, and D. E. Roberts, "Technique for tensile creep testing of ceramics," *J. Amer. Ceram. Soc.* **72**, No. 9, 1610-1614 (1989).

Diagnostics With Ultrasound

An MSEL group that included Raymond E. Schramm, Alfred V. Clark, Jr., Stephen R. Schaps, Robert C. Reno, Gerald V. Blessing, and Todd J. McGuire employed ultrasound for a variety of diagnostic purposes in materials science. One development, accomplished with the collaboration of Dragan V. Mitraković of the University of Belgrade, Yossef Cohen of the Nuclear Research Center in Israel, and Peter J. Schull of The Johns Hopkins University, was the detection of defects in the wheels of railroad cars. By embedding a device called an electromagnetic-acoustical sensor in the train tracks, the group found that they could identify wheels with unusual stress conditions or unsatisfactory metallurgical texture—both properties likely to lead to failure of the wheel. The device emitted short bursts of horizontal shear waves and detected anomalies in the echoes.[186]

A prototype residual stress measurement system was delivered during 1991 to the American Association of Railroads for evaluation in its testing center.

In collaboration with the Ford Motor Company, with Robert B. Thompson and Y. Li of the Center for Nondestructive Evaluation at Iowa State University, and with D. Matlock of the Colorado School of Mines, the research group also compared ultrasound with older, mechanical methods of testing sheet steel for its texture and formability. They found an ultrasonic measurement technique that accurately predicted the critical properties of the steel, potentially leading to on-line measurements to replace slower, more expensive mechanical testing in the laboratory.[187]

The group also developed electromagnetic probes used in characterizing dielectric materials. A low-frequency capacitance probe could evaluate the density of certain ceramics within 0.2 %.

Nondestructive Testing of Composites

Christopher M. Fortunko, deputy division chief, led a research group dedicated to improving experimental methods for characterization of composites. The group, which included Dale W. Fitting, V. K. Tewary of the Ohio State University, E. Jensen of the Colorado School of Mines, L. J. Bond of the University of Colorado, and M. Renken of Valparaiso University, designed, built, and tested a high-pressure, gas-coupled acoustic microscope capable of 10 μm resolution on silicon wafers. They also produced an acoustic array for the testing of glass-epoxy composites. The gas-coupled microscope operated at frequencies from 10 MHz to 30 MHz and at pressures up to 10 MPa.

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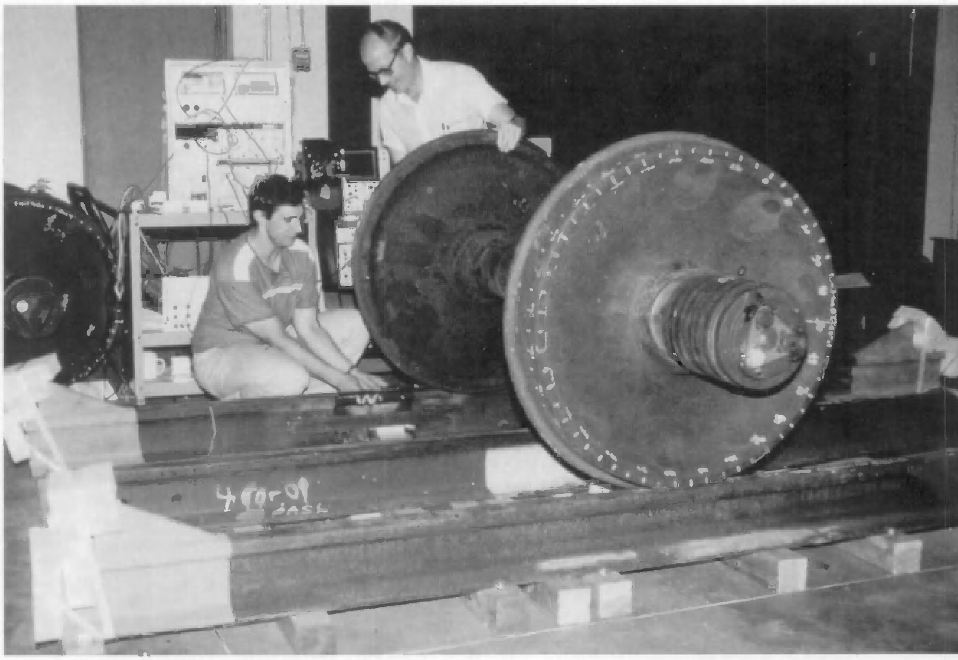
[186] Raymond E. Schramm, Alfred V. Clark, Jr., Dragan V. Mitraković, Stephen R. Schaps, and Todd J. McGuire, "Residual stress detection in railroad wheels: an ultrasonic system using EMATs," *NISTIR 3968*, May 1991, 47 pp. See also Raymond E. Schramm, Alfred V. Clark, Jr., Dragan V. Mitraković, Yossef Cohen, Peter J. Schull, and Stephen R. Schaps, "Tread crack detection in railroad wheels: an ultrasonic system using EMATs," *NISTIR 3967*, May 1991, 69 pp.

[187] A. V. Clark, R. B. Thompson, Y. Li, R. C. Reno, G. V. Blessing, D. V. Mitraković, R. E. Schramm, and D. Matlock, "Ultrasonic measurement of sheet steel texture and formability; comparison with neutron diffraction and mechanical measurements," *Res. in Nondestruct. Eval.* **2**, 239-257 (1990).



In 1990, Dragan Mitraković (left), a guest researcher from the University of Belgrade, and NIST physicist Raymond E. Schramm inspected railroad wheels using an Electromagnetic Acoustic Transducer (EMAT) sensor mounted in a typical rail.

A particular challenge for the group was insuring that a composite material was suited to its intended application. This task involved such measurements as quantifying the fiber volume fraction, the fiber orientation, porosity, and quality of bonding between matrix and reinforcing materials in composites. The evaluations were undertaken through the measurement of elastic wave velocities. Absolute phase velocities, for example, could be observed in the determination of elastic moduli.

Cryogenic Properties of Materials

A group led by Harry I. McHenry investigated the behavior of materials at low temperatures. One project, sponsored by the U.S. Air Force Systems Command, provided data on the mechanical properties of aluminum-lithium alloys under consideration for use in the Advanced Launch System (ALS). This was a detailed project. It addressed grain sizes and morphology of samples, hardness, tensile strength, stress-strain relations, and fracture toughness of four different alloy compositions. Richard P. Reed, Patrick T. Purtscher, Nancy J. Simon, Joseph D. McColskey, Robert P. Walsh, John R. Berger, Elizabeth S. Drexler, and Raymond L. Santoyo all contributed to the research.

The materials were envisaged for use in storage of cryogenic fluids. The evaluation project was a textbook case in fitting the material to the application—the reaction of the alloys to stresses caused by a combination of mechanical forces and large temperature excursions had to be predicted. The potential for tank leakage or breakage had to be anticipated, and measurements were needed to guide the choice of optimum composition and configuration of construction materials.

The group found that two prime candidates for the ALS program, Al-Li alloys 2090-T81 and WL049-T851, showed 10 % to 20 % higher tensile yield strength and ultimate strength than the other alloys. Toughness in the alloys, however, proved to depend strongly on the direction of the stress with respect to the orientation of the composites. Many of the measurements addressed fracture toughness and issues relating to delamination of the composites under a variety of stresses. Recommendations were included for further detailed testing of the alloys under actual service conditions.[188]

Oxide Superconductors

Hassel M. Ledbetter and Ming Lei, a colleague from the Institute of Metal Research in Shenyang, China, prepared a comprehensive study of elastic constants and related properties of oxides and oxide superconductors in 1991. Theirs was a contribution to the investigation of high-temperature superconductivity—in part their own measurements and in part the testing of models to provide guidance to others.

The two scientists measured polycrystalline elastic constants by the use of a megahertz-frequency pulse-echo method in the range 4 K to room temperature, employing measured mass density data and a model developed by Ledbetter and S. K. Datta to correct the measurements for voids in the samples. They compared the elastic moduli of $YBa_2Cu_3O_7$ (YBCO) with those of the related perovskites $BaTiO_3$ and $SrTiO_3$ and found good agreement with calculations based on the Born ionic model. They also derived a value of 423 K for the Debye temperature of YBCO, in good agreement with the value obtained from heat capacity data, 440 K.

From these measurements and similar ones on related high-temperature-superconductor materials, Ledbetter and Lei were able to offer conclusions regarding the valence of copper in the compounds and the electron-hole distribution. [189]

Polymers, Leslie E. Smith, chief

Major industrial partners of the Polymers Division included resin producers, processors of polymeric materials, and end-users of polymer-based products. Division scientists assisted these technical partners in understanding the scientific basis for polymer technology, and they helped to provide measurement methods and standards as well.

[188] R. P. Reed, P. T. Purtscher, N. J. Simon, J. D. McColskey, R. P. Walsh, J. R. Berger, E. S. Drexler, and R. L. Santoyo, "Aluminum alloys for ALS cryogenic tanks: comparative measurements of cryogenic mechanicl properties of Al-Li alloys and alloy 2219," *NISTIR 3979,* February 1993, 137 pp.

[189] Ming Lei and Hassel Ledbetter, "Oxides and oxide superconductors: elastic and related properties," *NISTIR 3980,* August 1991, 92 pp., 249 references.

Polymer Vehicles for Printing Inks

An interesting Polymers Division project involved the development of polymeric vehicles for use in various printing processes. In one case, three different types of polymer resins were prepared for trial as potential vehicles for inks to be used in printing by the intaglio technique—in which a plate is engraved or otherwise gouged and inked, and the printing paper is then pressed into the inked depressions. The most successful vehicle, according to Brian Dickens, Barry J. Bauer, and William R. Blair, was found in a family of alkyds based on pentaerythritol, sebacic acid, linseed oil fatty acids, and succinic anhydride. These alkyds formed a group of high-molecular-weight, low-viscosity materials that air-dried well and resisted subsequent wetting with various solvents.[190]

Thermodynamics of Polystyrenes

Questions about the thermodynamics of phase separation in polystyrenes intrigued Robert M. Briber and Barry J. Bauer. They knew that polymeric cross-linking might change the free energy of a given system and lead to phase separation. However, they wanted to study the thermodynamics of single-phase blends of cross-linked and linear polystyrene. Accordingly, they prepared samples containing linear protonated polystyrene and about 1 % of cross-linked deuterated polystyrene. These they analyzed in the small-angle neutron scattering facility at the NIST nuclear reactor.[191]

The two researchers obtained scattering results using an incident neutron beam of wavelength 0.1 nm with a dispersion of 25 %. From these data, they derived static structure factors for the samples as functions of the scattering vectors. They used classical elasticity theory combined with estimates of the free energy of mixing to calculate the zero-angle scattering as a function of the density of the cross links.

Shear Stress and Fluorescence Anisotropy

A new experiment in which measurements of the anisotropy of fluorescence was used to monitor shear-induced molecular orientation in polymer systems was undertaken by Anthony J. Bur, Robert E. Lowry, Steven C. Roth, Charles L. Thomas, and Francis W. Wang early in 1991.

In general, fluorescence anisotropy was expected to depend on the molecular orientation, on the decay time of the fluorescence, and on the rotational relaxation of the probe. The authors provided optical instrumentation for a cone and plate rheometer,

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[190] Brian Dickens, Barry J. Bauer, and William R. Blair, "Cylinder wipe air-drying intaglio ink vehicles for U.S. currency inks," *NISTIR 4498*, January 1991, 23 pp.

[191] Robert M. Briber and Barry J. Bauer, "Small-angle neutron scattering of blends of cross-linked and linear polystyrene," *Macromolecules* **24**, No. 8, 1899-1904 (1991).

and they synthesized a polymeric fluorescent probe molecule consisting of polybutadiene tagged in the center of the principal polymer chain with anthracene. The tagged polybutadiene was expected to participate in the entanglement network of the host matrix of polybutadiene, reflecting the host orientation under applied stress.[192]

The authors observed the anisotropy as a function of shear rate and shear stress for 5 % and 50 % plasticized specimens. They found that the samples displayed non-Newtonian behavior, and they obtained values for the sample relaxation times. They expected the new type of measurement to open windows into the stress-induced behavior of polymers.

Modeling Tooth Decay

Three American Dental Association research associates, using the facilities of the Paffenbarger Research Center at NIST, contributed to the development of mathematical models to describe dental caries. Their study, published in the NIST Journal of Research, expressed the demineralization of teeth in terms of diffusion and dissolution. The three, Thomas M. Gregory, Laurence C. Chow, and Clifton M. Carey, noted the typical progress of decay in teeth: a thin outer layer of enamel in relatively good condition, a deeper layer consisting of partly demineralized enamel, and an advancing front of demineralization where acids diffused into the sound portion of the tooth and dissolved mineral from it.

Other studies had indicated that tooth mineral dissolved faster than the product ions could be transported from the site. Thus, a diffusion-controlled process appeared to be at work. The authors introduced into the modeling process an interaction between the permselective diffusion of ions and the dissolution of tooth mineral. They predicted the existence of concentration gradients in the calcium and phosphate ions, owing to gradients in the electrochemical potentials and differences in the permselectivity of the diffusion barrier. Instead of the concentration ratio 5/3 for Ca/P, as was found in healthy tooth enamel, the loss of these ions to the outer layer of the tooth was limited by the (complex) diffusion process.[193]

Despite the limitations of their assumptions—for example, that the barrier between the decaying portion of the tooth and its outer layer was infinitely thin—the authors found that their results agreed well with available experimental data.

[192] Anthony J. Bur, Robert E. Lowry, Steven C. Roth, Charles L. Thomas, and Francis W. Wang, "Observations of shear-induced molecular orientation in a polymer melt using fluorescence anisotropy measurements," *Macromolecules* **24**, No. 12, pp. 3715-3717 (Communications to the Editor) (1991).

[193] T. M. Gregory, L. C. Chow, and C. M. Carey, "A mathematical model for dental caries: a coupled dissolution-diffusion process," *J. Res. NIST* **96**, No. 5, 593-604 (1991).

Metallurgy, E. Neville Pugh, chief

Stress Corrosion Cracking

In a collaboration between Richard E. Ricker and James L. Fink of NIST and A. K. Vasudevan of the Office of Naval Research, a study was made of the importance of precipitates in grain boundaries to the stress corrosion cracking of aluminum alloyed with small quantities of lithium, manganese, and copper. There were so many factors that were known to influence stress-corrosion cracking in precipitation-hardened alloys that the real challenge was to devise experiments in which one or two factors could be isolated for study.

The group chose to hold constant the matrix precipitates of a binary aluminum-lithium alloy. In turn, this technique held constant the yield strength. Then they could vary the size and volume percentage of grain-boundary precipitates. They also varied the matrix precipitate size and distribution with the grain-boundary precipitate size in a ternary aluminum-lithium-copper alloy so as to keep its yield strength constant at the same level as the binary alloy.

Using a variety of heat treatments and aging, the authors measured the strain-to-failure in aggressive environments—half-molar deaerated sodium chloride, and a brew prepared by adding to the sodium chloride solution pinches of sodium bicarbonate, sodium carbonate, and lithium chloride. These data they compared to similar measurements taken in a nitrogen-gas reference environment. At relatively low values of strain-to-failure in nitrogen, equal values of strain-to-failure occurred in the aggressive environments. At higher values in nitrogen, however, the aggressive-environment strain-to-failure values tapered off noticeably. Moreover, although both the binary alloy and the ternary alloy showed similar deviations, the effect on the binary was significantly more pronounced.[194]

The experiments furnished evidence that the type, size and density of grain-boundary precipitates strongly increased the susceptibility of aluminum-lithium alloys to stress corrosion cracking.

Graphite Fiber Composites in Electrolysis

George L. Cahen, Jr. and Glenn E. Stoner of the University of Virginia knew how to purge seawater of certain micro-organisms in 1990—this they could accomplish by ac electrolysis. But they wanted to know how graphite fiber-polymer composites would perform as electrodes in this type of system. To find out, they teamed with Gery R. Stafford of NIST. The group was optimistic, since graphite displayed good electrical conductivity, good chemical resistance, and generated a volatile oxidation product that consumed no electrical energy upon repolarization. But how would the composites perform?

Preliminary screening of candidate composites turned up some promising materials and some spectacular failures. Clearly a systematic study was needed.

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[194] R. E. Ricker, J. L. Fink, and A. K. Vasudevan, "On the stress corrosion cracking of Al-Li alloys: the role of grain boundary precipitates," *Metallurgical Transactions* **22A**, 264-267 (1991).

The authors prepared test electrodes, backed with nickel electroplating and embedded in a polyester resin so as to expose only a 4 cm by 0.25 cm face to the electrolysis solution, containing 2 % dehydrated seawater. A variety of electrical-current settings was used with random-oriented, parallel, and perpendicular fiber composites. Water absorption tests were performed on the composites, and voltage-time curves were obtained at fixed current densities, using the composite as the anode.[195]

Typically, after a period of time that varied with the composite, the measured potential at fixed current density would rise sharply for each material. Anodes with parallel fibers failed quickly, with the composite simply falling apart. Anodes with only the tips of fibers exposed to the solution lasted the longest.

Composites made with polyphenylene sulfide appeared to survive substantially better than those based on nylon, epoxy-butadiene, polyester, or epoxy.

The authors concluded that degradation of the cells occurred almost entirely at the anode, with loss of electrical contact to the conductive fibers. In the case of polyphenylene sulfide-fiber composites, integrity of the composite electrode could be expected to last for about 100 hours.

Laser Vaporization Mass Spectrometry

A dearth of reliable information on the behavior of materials at temperatures beyond 2000 K prompted Peter K. Schenck, David W. Bonnell, and John W. Hastie to investigate the coupling of laser vaporization and high-pressure molecular beam mass spectrometry as a means of obtaining thermochemical data on refractory materials.

Stripped to its bare essentials, the method chosen by the group was simple; blast the surface of a test sample with a powerful pulse of laser radiation, then peek at the debris as it whizzed through the mass filter of a mass spectrometer. However, estimating the temperatures reached on the surfaces of the samples and determining which chemical reactions might have given rise to the observed species was not so simple.

The authors tried the new technique on two types of materials; graphite, and the high-temperature superconductor, $YBa_2Cu_3O_x$ (YBCO). They used a neodymium-yttrium aluminum garnet laser pulsed at 20 Hz as the energy source. The laser delivered from 10 mJ to 40 mJ of energy in 10 ns at a wavelength of 532 nm to a spot on the sample that was about 0.25 mm in diameter, producing temperatures estimated from the time of flight of the vapor plume to be as high as 8000 K.

The mass spectrometer operated in a tank evacuated by a high-conductance (about 2000 L/s) pump, allowing free flow of the vapor plume. The group collected mass data with a time resolution less than 0.1 ms. By moving the samples under the laser beam, the authors could obtain time-resolved mass data averaged over many laser pulses. Alternatively, they could integrate time-gated spectral scans.

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[195] Gery R. Stafford, George L. Cahen, Jr., and Glenn E. Stoner, "Graphite fiber-polymer matrix composites as electrolysis electrodes," *J. Electrochem. Soc.* **138**, No. 2, 425-430 (1991).

The graphite targets yielded both neutral species and ions tentatively identified as C_1, C_3, C_1^+, C_2^+, C_3^+, and C_5^+ under direct bombardment of the samples by the laser. The YBCO samples produced a rich menu of molecular fragments: Cu, Cu^+, CuO^+, O_2, O_2^+, H_2O, CO_2, BaO, YO, CuBa, and YCu.

The authors declared the new technique an unqualified success. Both equilibrium and nonequilibrium effects were found, and the vapor plumes generated by the laser appeared to be nonhomogeneous both in space and in time; yet the method was productive and the results were subject to reasonble interpretation.[196]

Reactor Radiation, J. Michael Rowe, chief

The Reactor Radiation Division (RRD) was built on the concept of service. Possessing one of the few nuclear reactor research facilities in the Nation—with accompanying instruments that were unique—division personnel were keenly aware of the need to provide reactor run time for those scientists with experiments that required use of the facility. In some cases—both inside NIST and beyond its borders—scientific visitors could plan and conduct their own experiments with only minimal assistance from the division staff. There were plenty of requests for instrument time, however, in which the expertise existing among the staff was needed to make a fruitful experimental plan and carry it out.

The division explicitly welcomed visitors who needed only run time as well as those who desired to collaborate scientifically with its staff. In addition, division scientists maintained their own materials research programs involving the use of neutrons, and they continually looked to develop and maintain state-of-the-art instruments to take best advantage of the NIST reactor.

We have already described many experiments performed at the NIST reactor by personnel of other divisions, often in collaboration with one or more RRD colleagues. Specialized instruments associated with the reactor included neutron spectrometers, reflectometers, and diffractometers serving the thermal column, and a like set of devices—plus a small-angle neutron scattering capability—facilitating experiments that used the cold neutron source.

High-T_c Superconductors and Phonons?

A question asked immediately after the discovery of high-temperature superconductivity was: Does the origin of high-temperature superconductivity involve the crystal lattice, as the Bardeen-Cooper-Schrieffer theory indicated for conventional (low-temperature) superconductors?

There seemed to be as many answers to that question as there were high-T_c materials. Certain materials with T_c values near 30 K—lanthanum-strontium cuprate and barium-potassium bismuthate, for example—showed substantial isotope effects, indicating that the lattice indeed played a role in the superconductive interaction. Other materials, particularly those with transition temperatures above 77 K, showed no isotope effect at all.

[196] P. K. Schenck, D. W. Bonnell, and J. W. Hastie, "Laser vaporization mass spectrometry of refractory materials: graphite and $YBa_2Cu_3O_x$," *J. Vac. Sci. Technol.* A7, No. 3, pp. 1745-1749 (1989).

A team of investigators including Jeffrey W. Lynn, Dan A. Neumann, and John J. Rush of the Reactor division and I. W. Sumarlin, J. L. Peng, and Z. Y. Li of the University of Maryland, aware that inelastic neutron scattering provided good insight into lattice resonance modes, or phonons, conducted a series of such measurements on a 43 g sample of polycrystalline $Nd_{1.85}Ce_{0.15}CuO_4$ (NCCO).

In the range of neutron energy up to 100 meV, the group found structure that they attributed to generalized phonon density of states peaks at 13 meV, 51 meV, and 65 meV. Tucked away in the results they also discovered smaller phonon structure. Comparing their results with those obtained in superconducting tunneling experiments, they found agreement at 14 meV, 32 meV, and 40 meV, indicating that, indeed, phonons did play a part in the superconductive interaction in NCCO.[197]

In the meantime, Russell C. Casella extended his earlier discussion of high-temperature superconductivity to account for anisotropy observed in electron tunneling experiments in $Bi_2Sr_2CaCu_2O_8$ (BSCCO). Previously, Casella had proposed a multiple-energy-gap explanation for the dependence of T_c in certain compounds containing layers of CuO_2 upon the number of layers.

In tunneling measurements on single-crystal BSCCO, the ratio of the superconducting energy gap to the transition temperature had been evaluated as 6.2 ± 0.3 when the current was injected in the *ab* plane and as 3.3 ± 0.5 when the current followed the *c* axis. Casella employed a model in which double intermediate boson exchange took place between fermions in two bands. The consequences of his model were consistent with current experimental results and allowed for specific experimental tests.[198]

Vibrational Modes in Fullerene

R. L. Cappelletti spent a portion of his sabbatical leave from Ohio University at NIST, investigating fullerite, an interesting molecule with the formula C_{60}. In the work, he collaborated with John R. D. Copley and William A. Kamitakahara of NIST and with Fang Li, J. S. Lannin, and D. Ramage of Penn State University.

The shape of the C_{60} molecule and its relatives was nearly spherical, with all atoms on the surface, forming cage-like structures known as "Buckey balls" or "Buckminster Fullerene." Some 46 distinct vibrational frequencies were possible for the C_{60} structure. Previous observations of the molecule by infrared and Raman spectrsocopy were limited to only 14 of the frequencies, but all participated in neutron scattering.

The team purified a 640 g polycrystalline sample of C_{60} and conducted scattering measurements using a modified three-axis spectrometer at the NIST reactor. The sample was held at 10 K during the 6-day run.

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[197] J. W. Lynn, I. W. Sumarlin, D. A. Neumann, J. J. Rush, J. L. Peng, and Z. Y. Li, "Phonon density of states and superconductivity in $Nd_{1.85}Ce_{0.15}CuO_4$," *Phys. Rev. Lett.* **66**, No. 7, 919-922 (1991).

[198] R. C. Casella, "A theoretical model for the tunneling-gap anisotropy observed in layered copper-oxide high-temperature superconductors," *Solid State Commun.* **78**, No. 5, pp. 377-379 (1991).

The neutron scattering spectrum, taken from 0 meV to 240 meV, was complex. It included three prominent peaks associated with vibrational density-of-states measurements in graphite, along with other structure attributed to the infrared-active and Raman-active modes as well as other vibrational modes predicted by theory. In certain respects, however, the data were not in accord with available theoretical predictions, leading to the suggestion that theoretical treatments might be improved on the basis of this type of measurement.[199]

Low-Temperature Motion of Protons in Scandium

A neutron-scattering experiment on purified, single-crystal scandium provided a diverse group of scientists with new information on the motion of hydrogen within the scandium lattice. Norman F. Berk, John J. Rush, and Terrence J. Udovic of NIST collaborated on the project with I. S. Anderson of the Paul Scherrer Institute of Switzerland, R. G. Barnes of Iowa State University, A. Magerl of the Laue-Langevin Institute of France, and D. Richter of the Institute for Research in Solid State and Nuclear Science in West Germany.

The group knew that hydrogen, when dissolved in hexagonal-close-packed rare-earth metals, displayed unusual short-range ordering, with consequences that were noticeable in certain physical properties of the material. They hoped to excite a "hopping" mode of hydrogen within the scandium lattice that would reveal to them some details about the localized motion.

Accordingly they loaded one single crystal of scandium with 5 % to 20 % concentrations of hydrogen, left a second crystal unhydrogenated, and subjected both to neutron irradiation at the Laue-Langevin Institute in Grenoble, France. The difference spectra, obtained at temperatures of 300 K, 125 K, 100 K, 70 K, and 50 K showed a sharp spectral line whose width surprisingly exhibited a noticeable minimum near 100 K. Below that temperature, the linewidth increased approximately as $1/T$, the first such observation seen in the system.

The authors took the results as an indication of nonadiabatic behavior in the hydrogen motion. The data also indicated extremely rapid hopping rates, more than 10^{10} per second. They discussed their results in terms of the locations of the hydrogen atoms in the scandium lattice and the energetics of the hydrogen motion.[200]

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[199] R. L. Cappelletti, J. R. D. Copley, W. A. Kamitakahara, Fang Li, J. S. Lannin, and D. Ramage, "Neutron measurements of intramolecular vibrational modes in C_{60}," *Phys. Rev. Lett.* **66**, No. 25, pp. 3261-3264 (1991).

[200] I. S. Anderson, N. F. Berk, J. J. Rush, T. J. Udovic, R. G. Barnes, A. Magerl, and D. Richter, "Rapid low-temperature hopping of hydrogen in a pure metal: the ScHx system," *Phys. Rev. Lett.* 65, No. 12, pp. 1439-1442 (1990).

Building and Fire Research Laboratory

The building research and fire research organizations were separate within NBS from 1974, when the Federal Fire Prevention and Control Act directed the Bureau to create a Center for Fire Research (see A Hectic Decade For NBS Fire Science in Ch. 4), until Director John Lyons established the new NIST organization in 1991. At that time, Richard N. Wright was appointed director and Jack E. Snell was made his deputy.

Structures, Hai S. Lew, chief

Abating Earthquake Hazards

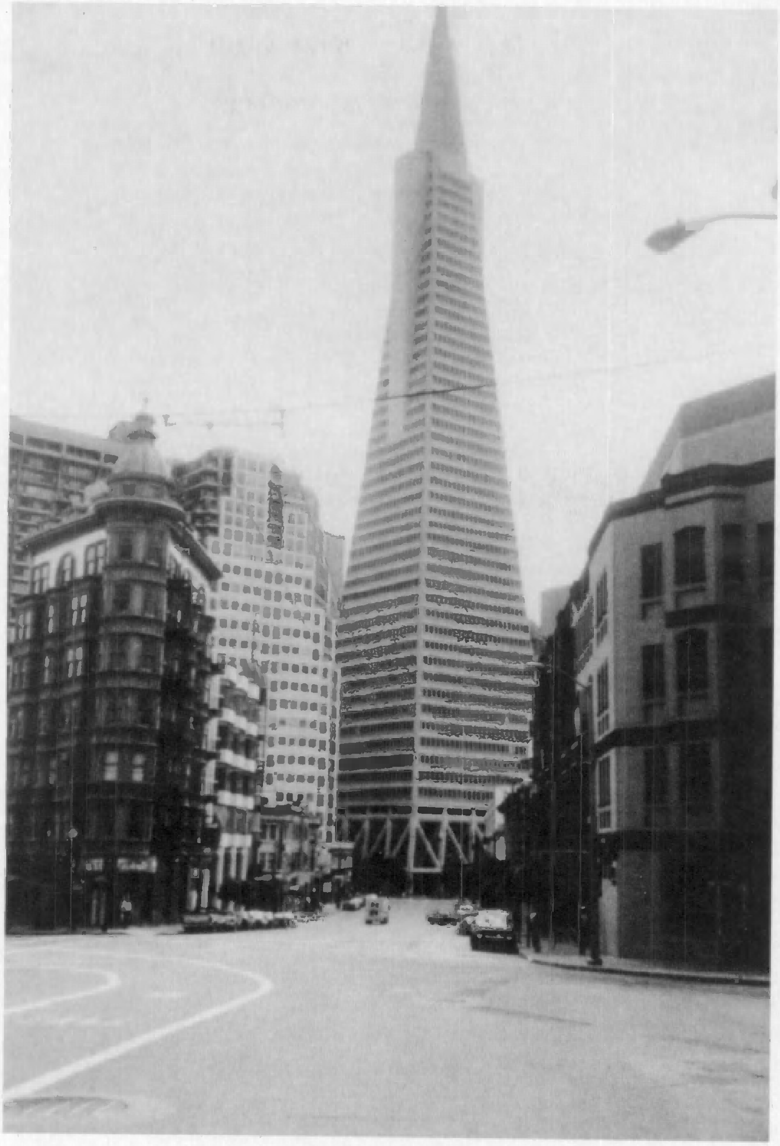
On October 17, 1989, the "Loma Prieta" earthquake shook the San Francisco Bay area. The epicenter of the quake was located about 90 km southeast of the city along the San Andreas fault line. Because of the "shaky" history of the Bay area, many buildings had been instrumented with accelerometers to record their motion in response to earthquakes. Thus the Loma Prieta event provided NIST with an excellent opportunity to compare the recorded responses to the strong shaking of the earthquake with building-response predictions derived from low-level vibrations. Richard D. Marshall and Long T. Phan of NIST collaborated with Mehmed Çelebi of the U.S. Geological Survey on the project.

The team studied five buildings from the earthquake region. These were selected on the basis of several criteria:

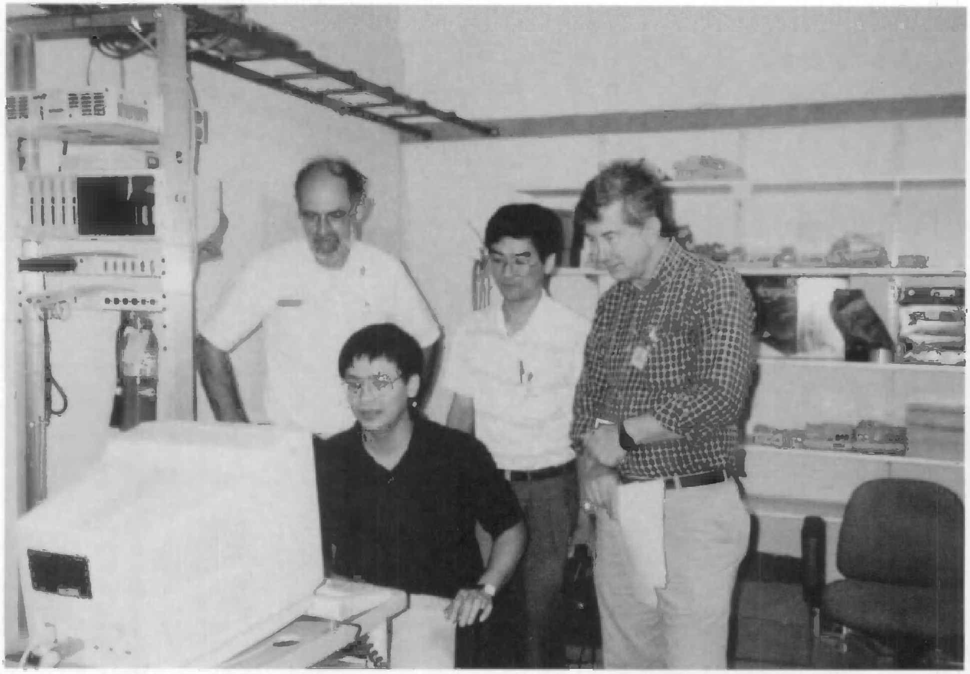
- Availability of accelerometer records showing strong motion as a result of the earthquake.
- No visible signs of structural damage from any earthquake.
- Accessibility to the building for ambient vibration studies.
- Availability of detailed structural drawings and knowledge of soil conditions at the building site.
- Adherence of the building to current construction codes.
- Contribution to the variety of construction materials and methods desired in the investigation.
- Degree of complexity in the analytical model to be used to describe the building response to earthquakes.

Two organizations had instrumented buildings in the area with force balance accelerometers: the California Division of Mines and Geology, through its "Strong Motion Instrumentation" program; and the U.S. Geological Survey, through its Engineering Seismology Branch. The accelerometers produced time histories of velocity and displacement at various positions within each building.

NIST Structures Division engineers H. S. Lew and Nicholas J. Carino investigated structural damage to the two-tiered Nimitz Freeway, which collapsed during the 1989 Loma Prieta Earthquake.



View to the southeast of the landmark Transamerica building in San Francisco, 97 km from the epicenter of the Loma Prieta earthquake. The Transamerica was built on a 9-ft thick concrete mat foundation with no piles. It was instrumented by the U.S. Geological Survey.



NIST scientists Richard D. Marshall (left) and Long T. Phan (seated) monitor real-time measurements of accelerations of a building resulting from ambient vibrations, along with Chang H. Hyun (standing, center) of the Korea Institute of Nuclear Safety and Mehmed Çelebi of the U.S. Geological Survey.

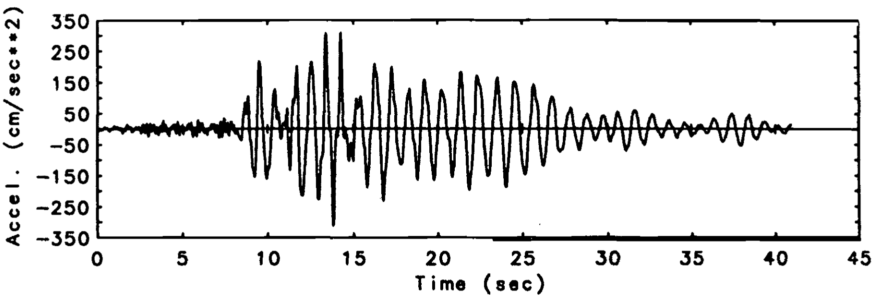
The five buildings chosen for the study were the 13-story California State University Administration Building at Hayward, the 12-story Santa Clara County Office Building at San Jose, a 6-story commercial office building in San Bruno, the 49-story Transamerica Building in San Francisco, and the 30-story Pacific Park Plaza in Emeryville.

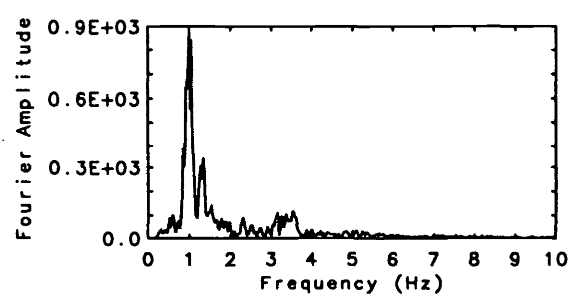
Once the required structural and Loma Prieta records were in hand, the team conducted ambient vibration measurements in each building, using its existing accelerometer array to record building response to ordinary, everyday vibration—mostly from the effects of wind.

Both the recorded strong-motion data and the ambient vibration responses were analyzed to identify relevant dynamic characteristics of each building; then the two sets of analyses were compared.

The team found that, with suitable signal conditioning, the force-balance accelerometers produced reliable ambient vibration measurements. This finding would allow the use of the accelerometers to identify the main modes of building vibration. Several other conclusions were reached as well; these related to details of the building construction and to the nature of the soils on which the buildings were constructed.[201]

[201] R. D. Marshall, Long T. Phan, and M. Çelebi, "Measurements of structural response characteristics of full-scale buildings: comparison of results from strong-motion and ambient vibration records," *NISTIR 4884*, October 1992, 74 pp.





(Top figure) Acceleration-time graph in the east-west direction at the center of the roof of a 6-story office building in San Bruno, California during the Loma Prieta earthquake. (Bottom figure) Fourier spectrum derived from the top figure, showing the major vibrational frequencies of the building.

As a consequence of the Loma Prieta earthquake, President Bush issued Executive Order 12699 on January 5, 1990. Entitled "Seismic Safety of Federal and Federally Assisted or Regulated New Building Construction," the order was intended to reduce risks to the lives of occupants of buildings owned by the Federal government and to persons who would be affected by failure of such buildings in earthquakes. The order helped secure new building codes in earthquake-prone areas.

"The future of lifeline earthquake engineering depends, in large part, upon our ability to develop and communicate new concepts and ideas for design, construction, operation, and post-event restoration of lifelines." So read the Foreword to the *Proceedings of the 4th U.S.-Japan Workshop on Earthquake Disaster Prevention for Lifeline Systems,* held in Los Angeles from August 19-21, 1991. Robert D. Dikkers of NIST's Structures division served on the organizing committee for the workshop, which featured contributions from earthquake scientists from many organizations in Japan and America.

Dikkers offered to the workshop participants a discussion of the U.S. response to the *National Earthquake Hazards Reduction Act of 1977* (PL 95-124)—updated by Congress in 1990—which requested the Federal Emergency Management Agency to oversee the formulation of a plan aimed at design and construction standards for lifelines. In the context of the Act, lifelines included electrical power, gas and liquid fuel, telecommunications, transportation, and water and sewer.

The 1990 reauthorization of PL 95-124 directed FEMA to consult with NIST on the formulation of an action plan to create standards for the preservation of lifelines. Dikkers and his colleagues from industry, government, and academia collaborated to create a comprehensive response to that request.[202]

Locating Reinforcing Bars in Concrete

An important step in evaluating the load capacity of an existing reinforced concrete structure was the capability of locating the number and configuration of the steel rods used to strengthen the concrete. Particularly under the types of stresses arising in earthquakes were the details of the reinforcing significant. For years, sensing devices had been available to detect the presence of reinforcing rods and their locations within the concrete in a nondestructive fashion, but no U.S. industrial standards existed for these so-called "covermeters" when Nicholas J. Carino set about to characterize them in 1992.

Carino conducted experiments comparing the capabilities of two types of covermeters available commercially—one based on measurements of magnetic reluctance and the other on eddy current observations. He examined the relationships between meter reading and "cover thickness," the thickness of concrete covering the reinforcing rod, and the change in reading occasioned by a change in rod diameter. He also investigated the limits for useful response of the meters to multiple bars, to ends of bars, and to spliced bars.

In his report, Carino offered simple descriptions of the operation of the two types of covermeters, summarized the results of his many tests, made recommendations for developing standard test methods for the meters, and presented suggestions for enhancing the effectiveness of then-existing devices and for improvements in the design of such equipment.[203]

Building Materials, Geoffrey J. Frohnsdorff, chief

Detecting Surface Defects

In certain industries—for example, automobiles and appliances—a perfect or near-perfect surface finish was vital to the commercial value of the manufactured item. Unfortunately, lumps and craters were the constant companions of coatings that

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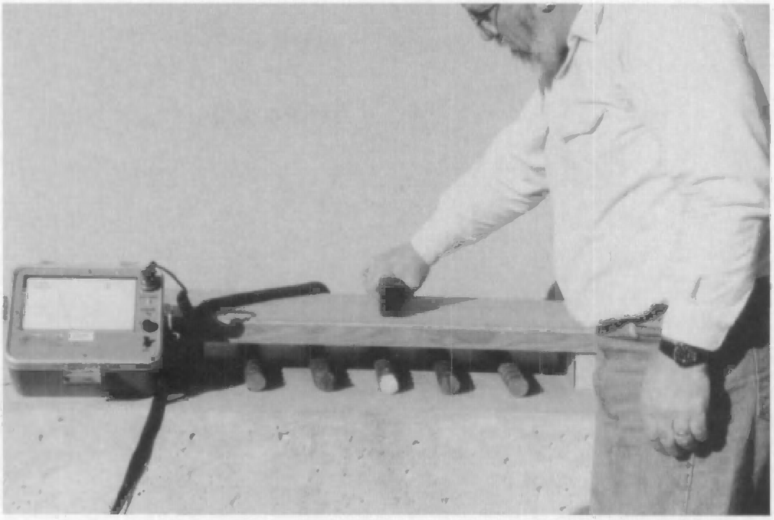
[202] Robert D. Dikkers, "Development of seismic design and construction standards for lifelines," *NIST Special Publication 840*, August 1992, 481 pp.

[203] Nicholas J. Carino, "Performance of electromagnetic covermeters for nondestructive assessment of steel reinforcement," *NISTIR 4988*, December 1992, 127 pp.



NIST Structures Division engineer Nicholas J. Carino operated a covermeter for locating reinforcing rods in reinforced concrete. The device operated by detecting magnetic reluctance.

satisfied the criterion of minimal volatile organic content, and these defects were hard to spot. Ordinary visual inspection proved unsatisfactory, and profiling styli were unrealistic in time and expense. However, Dale P. Bentz and Jonathon W. Martin adapted a thermographic method that was up to the task.

Relying on previous experience with thermography, the two scientists developed a theory for use of the technique for coating-defect detection, then devised a method to automatically survey the surface finish of test samples. A key element of the method was to create a temperature gradient of 20 °C to 30 °C between the coating and its environment. A thermographic camera then could be coupled with a computer-driven image processor for digitization of the surface appearance. Surface defects were readily apparent in the thermographic images, primarily because of the difference in coating thickness at the defect. The method was attractive—it was quick, simple, and automatic.[204]

Evaluating Roofing Materials

A research effort conducted by the U.S. Army Construction Engineering Research Laboratories, attempting to identify types of roofing that could be easily installed, durable, and low in maintenance, was materially aided by personnel from NIST. D. M. Bailey, Walter J. Rossiter, and James F. Seiler assisted Army personnel in the evaluation of three types of bitumen-based roofing at Fort Polk, Louisiana. The properties of the roofing materials were tested prior to installation on one of the buildings; at the end of each year for 3 years, the roofs were inspected for obvious damage and test samples were taken from the roofs for comparison with the original samples.

[204] D. P. Bentz and J. W. Martin, "Thermographic imaging of surface finish defects in coatings on metal substrates," *Materials Evaluation* **50**, No. 2, pp. 242-246 (1992).

The NIST team found the roofing materials to be satisfactory in terms of wear on the building and to be usefully durable as well.[205]

Predicting Service Life of Concrete

Very costly decisions on the repair or replacement of concrete structures relied upon evaluations by inspectors of the serviceability of the structure. In order to place such evaluations on a sounder basis, James R. Clifton of NIST and James M. Pommersheim of Bucknell University collaborated on a study of the methods involved in lifetime predictions.

Most evaluative methods assumed that corrosion was the principal cause of degradation of concrete structures. To predict the remaining useful life, the inspector typically would examine the concrete to determine its present condition, then seek the cause of any perceived decay. Extrapolating the progress of the degradation, the inspector would then estimate the time when the structure would reach a stage where repair or replacement would become necessary.

Both modeling and measurements were used in forecasting the life of concrete. Both approaches assumed that degradation would follow a known path: an initiation period during which attacking ions would slowly penetrate the concrete to reach its reinforcing steel members and commence the process of chemical decay; and a propagation period during which the chemical decay would progress until the concrete structure lost its strength. The authors summarized the steps recorded by various writers in modeling the corrosion process, as well as polarization resistance measurements used to monitor the growth of corrosion currents.

By conducting accelerated degradation tests, the inspecting authority could evaluate various corrosion routes, determine rate-controlling steps, and obtain estimates of useful lifetimes. The authors noted that such testing, while expensive, could well be justified to improve useful-life predictions of expensive concrete structures.[206]

Building Environments, James E. Hill, chief

Thermal Bridges in Buildings

The building industry was well aware that certain features in the construction of buildings provided easy paths for the flow of heat energy between the inside of the building and the outside. These energy pathways were known as "thermal bridges." They included such items as ceiling fasteners in built-up roofs, metal studs in insulated masonry walls, steel casings at window frames, the joints between the roof and the side wall, and the joints between the floors of the building and its side walls. In every case, they provided a kind of "short circuit" of material with relatively high thermal

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[205] D. M. Bailey, W. J. Rossiter, and J. F. Seiler, Jr., "Three-year field test summary for experimental modified bitumen roofing at Fort Polk, LA," *FM-93/05*, December 1992, 34 pp.

[206] James R. Clifton and James M. Pommersheim, "Methods for predicting remaining life of concrete structures," *NISTIR 4954*, November 1992, 24 pp.

conductivity that reduced the effectiveness of the insulation installed in the building envelope. Steady-state analysis of thermal bridges typically put the increase in energy transmission on their account at 10 % to 20 %. Douglas M. Burch, George N. Walton, and Betty A. Licitra of NIST collaborated with John E. Seem of Johnson Controls Corporation to show that a time-dependent analysis of thermal bridges raised the steady-state estimates substantially.

For their study, the authors configured a modest two-story office building constructed according to current practice. Then they calculated the conduction transfer function for the building without considering the thermal bridges. Finally, they repeated the calculation using a two-dimensional, finite-difference analysis to evaluate the dynamic heat-flow contribution of the thermal bridges. The overall heat transfer coefficient through the outer envelope of the building increased by an impressive 33 % when the contributions of the thermal bridges were included in the analysis.[207]

Improved Buoys for Water Safety

In the early 1990s, the U.S. Coast Guard—as a correspondent with the International Association of Lighthouse Authorities (IALA)—was concerned that the imperfect base of knowledge of visibility of navigational buoys was hampering the adoption of improved international standards for their shapes and color-coding. The IALA suggested that standard shapes and colored markings could identify the port and starboard limits for channels, the deepest water in an area, especially dangerous or especially safe areas, and special features of particular locations. The trouble was, nobody was sure how well any particular designs could be seen by boaters. To help the Coast Guard evaluate the effectiveness of some of the designs before they invested significant effort and expense in new buoys, Belinda L. Collins and Philip A. Sanders reviewed available research on buoy visibility and undertook experiments to enhance the state of knowledge on the topic.

In a *NIST Interim Report,* Collins and Sanders reviewed existing IALA standards for buoys, including type, shape, color, and geometrical topmarks. They also reviewed the lore surrounding the use of buoys for daytime navigation—problems caused by tides, marine growths, sun-fading, open-air deterioration, and color-blindness in navigators.

As their own contribution to the visibility question, Collins and Sanders hit upon the photography of 8 % scale models of test buoys, followed by evaluation of the images by a panel of observers. Ten different buoy configurations were prepared by Robert E. Stachon of the Groton, Connecticut, Coast Guard facility. Rosemary Porterfield and James R. McMahon, officials at Black Hills Regional Park, a facility of the Maryland-National Capital Park and Planning Commission, made their 200 hectare lake available for the photography. Some 13 slides were prepared for each scale model to simulate views from typical boating distances of 0.2 km to 4 km, and in a variety of lighting and background conditions.

[207] D. M. Burch, J. E. Seem, G. N. Walton, and B. A. Licitra, "Dynamic evaluation of thermal bridges in a typical office building," *ASHRAE Transactions: Research 98, Part 1*, pp. 291-304 (1992).

The resulting photographic slides were first shown to 17 observers denoted as "naive," then again to 10 trained observers at Coast Guard Headquarters in Washington. The observers determined the distances at which the scale buoys could be identified under the various experimental conditions, relying on shape, color, and topmarks.

Interpretation of the results required careful analysis, which the authors detailed in their report. Their conclusions included the following:

- The presence of topmarks did not increase the visibility of the buoys tested for distances beyond 0.8 km—color was substantially easier to recognize than topmarks when viewed with front lighting.
- In back lighting and twilight conditions, visibility decreased substantially, with or without topmarks.
- Red colors were visible at greater distances than greens.
- Painted bands in contrasting colors appeared to be effective visual aids for buoy recognition. [208]

New Lamps for Old

The introduction of new, more compact fluorescent lamps for commercial and domestic use in the United States and Canada brought longer lamp life, reduced energy use, and lower operating costs. These benefits were most welcome. However, the new lamps also brought questions about their performance compared to the older lamps they replaced—such concerns as their response to extremes of heat and cold, sensitivity to lamp position and mechanical shock, and visibility of flicker.

In conjunction with Michael J. Ouellette of the National Research Council (NRC) of Canada, Belinda L. Collins and Stephen J. Treado of NIST began in 1992 to resolve the outstanding uncertainties regarding the performance of the compact lamps. The trio of scientists decided first to test lamp response to variation in ambient temperature.

In a large NIST environmental chamber, 12 three-lamp sets of various types of the compact fluorescents and a control set of incandescent lamps were subjected to six different temperatures in the range 45 °C to −18 °C. Each lamp had been operated for 100 hours at the NRC for pre-conditioning. Photocells positioned near each lamp monitored luminous output and flicker. Extensive measurements were made by the group—of time-to-ignition, light output vs time for each lamp at each temperature, power use of each lamp, flicker characteristics, and harmonic distortion.

The authors found that the temperature of the space in which the lamps operated generally affected their performance in a big way—in some cases, the lamps failed to operate, in others the light output dropped to 10 % of the value at 25 °C. A notable exception were lamps with outer glass enclosures, which performed well at low temperatures. On the other hand, the fluorescent lamps were noticeably more efficient than the incandescent lamps when measured near 25 °C.[209]

[208] Belinda L. Collins and Philip A. Sanders, "Evaluation of the visibility of buoys and topmarks," *NISTIR 4756*, March 1992, 68 pp.

[209] Belinda L. Collins, Stephen J. Treado, and Michael J. Ouellette, "Evaluation of compact fluorescent lamp performance at different ambient temperatures," *NISTIR 4935*, December 1992, 47 pp.

Moisture Accumulation Under Roofs

Department of Housing and Urban Development officials thought they might have a problem with moisture condensation in manufactured homes. A 1984 survey of several dozen manufactured homes throughout the United States turned up moisture problems in about one-third of the cases. A later survey in Canada showed similar results.

Why should moisture particularly concern residents of manufactured homes? For one thing, HUD did not require ventilation of the space below the roof to the outside air, as many conventional homes possessed. For another, manufactured homes generally were smaller than conventional homes built for the same number of occupants, thus concentrating the moisture generated by normal household activities into a smaller space; in cold weather, unavoidable exchange of air between the living space of the home and its roof cavity carried the moist air into contact with the cold roof, resulting in increased humidity and, perhaps, accumulation of water.

At the request of HUD, Douglas M. Burch of NIST fired up his computer and analyzed the problem. He assumed a layered roof structure for which he sought the time-dependent moisture content. Other assumptions included:

- Moisture transfer was driven by temperature and humidity gradients.
- Moisture transfer was one-dimensional.
- Heat transfer was temperature independent.

Burch prepared a computer program—which he called MOIST—to solve the problem, using standard heat-and moisture-transfer equations. Among many other findings, a significant one was the effect of indoor humidity levels on the moisture content of roof fibers. In cold climates, the calculations indicated that fiber saturation would occur above about 40 % relative humidity during the three coldest winter months if no roof-cavity ventilation to the outdoors was available. Another calculation showed that, in hot, humid climates, outdoor ventilation of the roof cavity could allow the sub-roof humidity to exceed 80 %, favoring the growth of molds and mildew.

Burch's prescription for manufactured homes?

1. In cold climates, install vapor barriers in the ceilings, seal all openings in the ceilings, and vent the roof cavity to the outdoors.

2. In hot, humid climates, do not ventilate the roof cavity to the outdoors.[210]

Fire Safety, Andrew J. Fowell, chief

Predicting Fire Danger: HAZARD I

On March 25, 1990, 87 people died in a fire in the "Happyland Social Club" in the Bronx, New York. Evidence found on the scene indicated that the fatal blaze was the result of arson—gasoline appeared to have been poured and lighted near the club

[210] Douglas M. Burch, "Controlling moisture in the roof cavities of manufactured housing," *NISTIR 4916*, November 1992, 43 pp.

entrance, dooming many club patrons to death from smoke inhalation or from burns. A few days later, the New York City Fire Department asked NIST to help identify the reasons why the fire resulted in so many deaths; what changes on the scene could have reduced the death toll?

NIST was ready for the NYFD request with a computerized program that could evaluate structure fires on the basis of the nature of the structure, the types of fire safety equipment on hand, and the progress of the conflagration. The program was called the HAZARD I Fire Hazard Assessment Model. Quickly, a NIST fire research team collected on-site information on the circumstances surrounding the fire for analysis with HAZARD I. On the basis of the analysis, Richard W. Bukowski and Robert C. Spetzler prepared a report for the NYFD which they later summarized in a journal article.

The authors used HAZARD I to process the data gathered at the fire site and to predict the progress of the fire. The prediction matched closely the descriptions given by witnesses to the blaze, including the elapsed time until conditions in the club became inconsistent with life. The program also identified two key points regarding the high mortality rate from the fire:

- No automatic sprinklers were operating in the building—such equipment would have stopped or severely restricted the loss of life.
- The use of non-combustible finishes on the inner surfaces of the building would have denied the fire the fuel it needed to build to the magnitude that took so many lives.

Bukowski and Spetzler pointed out that HAZARD I, with its relatively simple calculational tools such as algebraic equations and simple models, could effectively complement the more complex zone and field models already in use in fire safety engineering. It could identify factors leading to high fire-death tolls and point to strategies for avoiding similar fires in the many social clubs operating within large cities.[211]

Bukowski also presented a strategy for the development and implementation of performance-based fire codes for international use. His presentation took place during a forum for International Cooperation on Fire Research held in Sydney, Australia, October 18-20, 1992. The first step in the process, according to Bukowski, was agreement on a common set of goals, followed by agreement on suitable predictive tools. Ultimately, the procedure could be expected to lead to estimation of the performance of any building under the stress of fire.[212]

Evacuating the Handicapped

Rising concern within the U.S. General Services Administration for the safety of handicapped persons in burning buildings led to consideration by NIST researchers of the feasibility of using elevators to evacuate such individuals. In most countries, elevators were shut down during fire emergencies. By 1992, the NIST team, including

[211] R. W. Bukowski and R. C. Spetzler, "Analysis of the Happyland Social Club fire with HAZARD I," *J. Fire Prot. Eng.* **4**, No. 4, 117-131 (1992).

[212] R. W. Bukowski, "Toward the goal of a performance fire code," *Fire and Materials* **15**, No. 4, 175-180 (1991).

John H. Klote, Daniel M. Alvord, Bernard M. Levin, and N.E. Groner, completed a report to GSA that addressed the installation of elevator systems in buildings, design changes needed to make them available during fires, and human factors affecting their use. The study indicated that re-designed elevators could be of considerable help both in fires and in other types of emergencies.[213]

Fire Science, Richard G. Gann, chief

One of the greatest challenges to fire researchers was the need to place fire tests on a solid scientific footing and to develop computer models to reduce the amount of large-scale testing. A fully furnished room presents a very large number of potential ignition scenarios and hence a similar mumber of fire-growth alternatives.

The tradition of fire testing was to devise simple bench-top tests. Since these could not generally be related to actual fire conditions, the technique grew to incorporate full-scale models of rooms or buildings, which then were burned to obtain one-time data.

Beginning in the 1970s, computer modeling began to be feasible. The HAZARD I and CFAST models developed at NBS incorporated material properties, design data, and testing results, including the use of such devices as the cone calorimeter—developed at the Bureau.

Fire Danger and Heat Release Rate

During the 1990 meeting of the Fire Retardant Chemicals Association, Vytenis Babrauskas and Richard D. Peacock presented evidence, based upon years of correlating damage caused by fires with the various data characterizing them, that the single most important factor in assessing fire hazards was the heat release rate.

To support their assertion, the two scientists cited case histories illustrating that, although toxic gases were the primary cause of death in fires, it was heat release rate, not the toxicity of the product gases, that was the best predictor of fire hazard. Such other observables as delays in ignition time proved to be of only minor importance.[214]

Fire at Sea

Fire damage control, important everywhere, was crucial on board combat ships in wartime. Simple survival, let alone successful effort in battle, depended upon freedom from crippling fires.

During 1989, a study team including NIST's Emil Braun, Darren L. Lowe, and Walter W. Jones, collaborators Patricia Tatem and Jean Bailey of the Naval Research Laboratory in Washington, DC and Richard Carey of the David Taylor Naval Ship Research and Development Center in Annapolis, Maryland, conducted a series of fire tests on the USS Shadwell, a decommissioned Naval vessel, to help improve shipboard fire control procedures.

213 J. H. Klote, D. M. Alvord, B. M. Levin, and N. E. Groner, "Feasibility and design considerations of emergency evacuation by elevators," *NISTIR 4870,* February 1992, 126 pp.

214 V. Babrauskas and R. D. Peacock, "Heat release rate: the single most important variable in fire hazard," *Fire Safety Journal* **18**, No. 3, 255-272, 1992.

Four full-scale tests were performed on the Shadwell, using two different fuels, diesel oil and polyethylene beads, and two different ventilation systems. The results were analyzed to determine fuel mass loss, progress of the fires in the burn compartments, and changing conditions in compartments separated from the burn areas. In the burn compartments, a load platform tracked the fuel mass, thermocouple thermometers tracked the fire progress, and gas analyzers monitored O_2, CO, and CO_2. Gas and smoke concentrations were observed in two passageways and on the main deck. The effects of opening or closing doors and vents connecting various areas were examined. A smoke ejection system was set up to remove combustion products from surrounding areas. The data showed that within 6 to 7 minutes, the ejection system, coupled with the provision of adequate ventilation of test passageways and compartments, removed enough of the combustion products to remove the threat to life.

A NIST model for fire growth and smoke transport, known as CFAST,[215] was used to simulate the fires and predict the temperature distributions and the effect of ventilation on the fire progress and smoke concentrations.

Results of the experiments indicated that sealing the compartments containing the fires and ventilating adjoining areas would provide the most effective control of the fire. The authors also found that the CFAST program successfully predicted the shipboard environment under fire conditions.[216]

A New Tool for Flame Studies

Nelson P. Bryner, Cecilia D. Richards, and William M. Pitts published a description in 1992 of a new instrument designed to give unprecedented measurement access to flame jets and plumes. The device, a Rayleigh light scattering facility that combined laser diagnostics with a cylindrical "clean room" test space of diameter 2.4 m and height 2.4 m, gave the authors the ability to examine the process of mixing in free jets and buoyant plumes.

Key to the success of the new device was the minimization of interference from glare and Mie scattering by the suppression of background light and dust particles in the test cylinder. For the first time, the facility permitted scattering measurements to be made in momentum-driven flows.[217]

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[215] W. W. Jones and G. P. Forney, *NIST Tech Note 1283* (1990).

[216] Emil Braun, Darren L. Lowe, Walter W. Jones, Patricia Tatem, Jean Bailey, and Richard Carey, "Comparison of full scale fire tests and a computer fire model of several smoke ejection experiments," *NISTIR 4961*, November 1992, 43 pp.

[217] N. P. Bryner, C. D. Richards, and W. M. Pitts, "Rayleigh light scattering facility for the investigation of free jets and plumes," *Rev. Sci. Instrum.* **63**, No. 7, 3629-3635 (1992).

Fire Hazards from Burning Furniture

The leading cause of residential fire deaths in the late 1980s was fire in upholstered furniture. From 1983 to 1987, one death in four came from that source. A majority of those fires originated from smoking materials, either pipes or cigarettes. Disturbed by the uncertainties in statistical data regarding the relation between the hazard created by a fire and the manner of ignition, Thomas G. Cleary, Thomas J. Ohlemiller, and Kay M. Villa designed an experiment to further examine the fire danger in upholstered furniture.

The fire scientists subjected a set of upholstered chairs made from five different combinations of foam and fabric to five types of ignition source:

- Burning cigarettes.
- Small flames such as a burning match might create.
- An incandescent lamp, typical of a reading aid.
- A space heater, producing primarily radiative energy.
- A large flame source such as an arsonist might choose.

The experiment was conducted in a furniture calorimeter, which captured the plume from the burning chairs; measurements included heat release rate, oxygen usage and rate of production of gaseous CO and CO_2, and rate of change of the chair mass.

The authors realized the limitations of their experimental procedure; they could not hope to duplicate the myriad types of fire leading to the death statistics. However, they were confident that heat release rate would best correspond to the fire hazard associated with a particular type of burning. They also relied upon HAZARD I, a methodology developed at NIST over the course of many years of fire research.

Of the total of 25 combinations of chair type and ignition source, 15 resulted in ignition and burning of the chair. Only one chair type was ignited by the lamp, only one by the cigarette. The "match" ignited three types of chair. Predictably, the space heater and the gas burner ignited all the test chairs. The progress of the burning—time to ignition and time to peak heat release—varied with both chair type and ignition source.

Using the HAZARD I modeling program, the authors assessed the likely fatality rate in their tests. To use the program, it was necessary for them to specify a number of conditions accompanying the fire: the type of home involved; the location of the chair; the age, mobility, and location in the dwelling of the victim; the time of day; and the presence or absence of a smoke detector. The rate and magnitude of heat release and the rate and nature of release of CO and CO_2 constituted the measured input data.

It is significant that the HAZARD I program predicted no deaths in the NIST experiment if a working smoke detector was in use. Without a smoke detector to warn the occupants of the simulated dwelling, about 45 deaths would have occurred to the 60 people—in family groups of baby, mother, father, and grandparent—involved in the 15 fires. The "arson-type" burner would have claimed 20 of the victims, and the space heater another dozen. The "match" and the "reading lamp" together would have killed another 13 occupants. Only the cigarette, relatively ineffective as an ignition source in this experiment, would have spared any humans occupying the fictitious home.[218]

A New Particulate Generator

Seeking the development of an effective tool for the study of small particulates in flames, a research team including Thomas G. Cleary, George W. Mulholland, Lewis K. Ives, and Robert A. Fletcher of NIST and J. W. Gentry of the University of Maryland used the tools of transmission electron microscopy (TEM) and laser microprobe mass spectroscopy (LAMMS) to investigate the particles produced in combustion aerosols.

For their study, the team adapted a laminar diffusion burner developed at NIST by Robert J. Santoro, Hratch G. Semerjian, and R. A. Dobbins in 1983. They used acetylene as the fuel, operating the burner at sufficiently low fuel flow rates that no visible sooting occurred.

Examination of the burner flame with the NIST TEM capability showed particle sizes clustered tightly around 7 nm. Observations with the LAMMS technique indicated the presence of carbonaceous materials as well as phosphorus and sulfur compounds originating in impurities in the acetylene. The authors stated that theirs was the first known steady-state generation of essentially monosized ultrafine aerosols in a diffusion flame.[219]

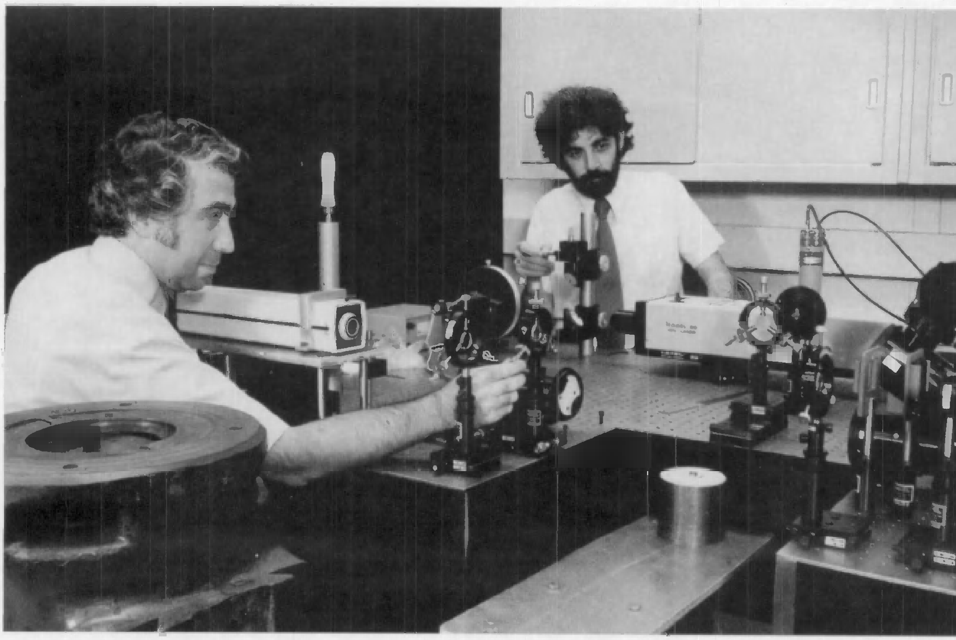
Computer Systems Laboratory

Much of the work of the Computer Systems Laboratory during this period was rooted in legislation—the *Brooks Act, Public Law 89-306*; the *Computer Security Act of 1987*, PL 100-235; and, of course, PL 100-418, the legislation that transformed NBS into NIST. The reach of the laboratory effort extended deep into America's computer-based technical structure, creating standards, guidelines, and test methods for computer systems and networks and providing assistance to both government and industrial information-system workers.

Under the leadership of director James H. Burrows, a hallmark of the laboratory was its role the development of the concept of "open systems," which allowed computer users to purchase and install computèr equipment from a variety of sources without the fear of component incompatiblity that could hamper or entirely frustrate system efficiency. This role was buttressed by a program of research on computer security, software engineering, data management, and new types of systems. During Fiscal 1991, the laboratory enjoyed direct congressional funding of $12 million, augmented by $14 million in reimbursable funds—mostly from other Federal agencies.

[218] Thomas G. Cleary, Thomas J. Ohlemiller, and Kay M. Villa, "The influence of ignition source on the flaming fire hazard of upholstered furniture," *NISTIR 4847,* June 1992, 64 pp.

[219] T. G. Cleary, G. W. Mulholland, L. K. Ives, R. A. Fletcher, and J. W. Gentry, "Ultrafine combustion aerosol generator," *Aerosol Science and Technology* **16**, pp. 166-170 (1992).



Hratch G. Semerjian (left) and Robert J. Santoro developed a technique called optical tomography for taking cross-sectional "pictures" of mixed gas or liquid flow systems. The technique used an infrared laser and an array of optical detectors to determine the type and concentration of specific molecules in a given flow system.

Information Systems Engineering, David K. Jefferson, chief

The responsibilities of the Information Systems Engineering staff lay primarily in the areas of data administration, the technology of data management, testing of computer graphics, and the validation of software standards.

In 1989, Federal Information Processing Standard (FIPS) 156 was adopted. This standard was based on the Information Resource Dictionary System (IRDS), part of the standards library of the American National Standards Institute. Division personnel, led by Alan H. Goldfine, prepared test methods and techniques to evaluate use of the IRDS. Along with similar efforts in regard to the database language SQL, NIST rendered valuable assistance with the implementation of these information-management tools. Compliance with the requirements of FIPS 127—Database Language SQL—was eased by NIST's continuing development of test suites for SQL. During 1991 alone, 16 licenses were issued for use of the test suites.

FIPS 120, the Graphical Kernel System, required that implementations be tested prior to availability for Federal procurement. Some 9 copies of the *Programmer's Hierarchical Interactive Graphics System Validation Test Suite* were distributed by NIST during 1991, under the direction of Mark W. Skall.

The testing of programming language compilers for conformance to FIPS standards, too, continued to occupy division staff members. Several languages were included in the NIST work, among them COBOL, Fortran, Pascal, Ada, and MUMPS.

The ISE division played an important role in a Department of Defense program known as CALS—Computer-Aided Acquisition and Logistical Support. The program, begun in 1985, was intended to accelerate the development and deployment of both weapons systems and commercial products. In June 1991, Sharon J. Kemmerer provided a synopsis of NIST activities in support of the CALS program. These activities took place in several areas: electronic data interchange; graphics; document standards; raster compression; data management; security; and data communication. In each of the activity areas, Kemmerer noted the progress achieved during 1990. Many of the activities included contributions to military or civilian standards; others took the form of consultation and research work. Additionally, NIST served as a focal point for information receipt and distribution with regard to CALS.[220]

Systems and Software Technology, Allen L. Hankinson, chief, succeeded by Roger J. Martin, 1992

In April 1991, the SST division defined a framework for open systems in a broad range of Federal software systems applications—information technology services, protocols, interfaces, and data formats. The discussion was contained in *NIST Special Publication 500-187* entitled "Application Portability Profile (APP): The U.S. Government's Open Systems Environment Profile OSE/1 Version 1.0." Known as the APP Guide, the document rapidly gained wide acceptance in industrial information technology activities as well.

The division also supported the FIPS 151-1 POSIX protocol with conformance testing, facilitating the portability of applications software. Seven POSIX testing laboratories were accredited under the National Voluntary Laboratory Accreditation Program during 1991.

A third division initiative operated in the realm of open-systems standards for multimedia computer-based interactive training software. With the collaboration of other Federal agencies, NIST sought to create an environment in which such multimedia software would become available as commercial, off-the-shelf products.

Computer Security, Stuart W. Katzke, chief

A direct response to the Computer Security Act of 1987 could be seen in a joint NIST-National Security Agency project report published in September 1990. Instructed by the act to receive and review Federal security plans for unclassified but sensitive information, the two agencies immediately initiated the Computer Security and Privacy Plans (CSPP) review project. Dennis M. Gilbert coordinated the preparation of a report summarizing the results of the first year's work on the project.[221]

[220] Sharon J. Kemmerer, "NIST support of the CALS program; 1990 synopsis," *NISTIR 4609*, June 1991, 19 pp.

[221] Dennis M. Gilbert, "1989 Computer Security and Privacy Plans (CSPP) review project: a first-year Federal response to the Computer Security Act of 1987," *NISTIR 4409*, September 1990, 170 pp.

During the first year, the review team examined some 1500 plans from 63 civilian agencies and another 27,000 plans from over 400 Defense agencies. The team found considerable uncertainty among the agencies regarding the most effective means to provide for secure computer operations; thus, team recommendations focused on education. Each agency was urged to consider the types of loss that compromise of its system could entail, and to investigate the methods needed to prevent such loss. NIST plans for the second year included visits to the agencies by NIST, NSA, or Office of Management and Budget staff members for on-the-spot guidance. NIST also planned to develop model standardized specifications and language for agency use in contracting for assistance from security specialists.

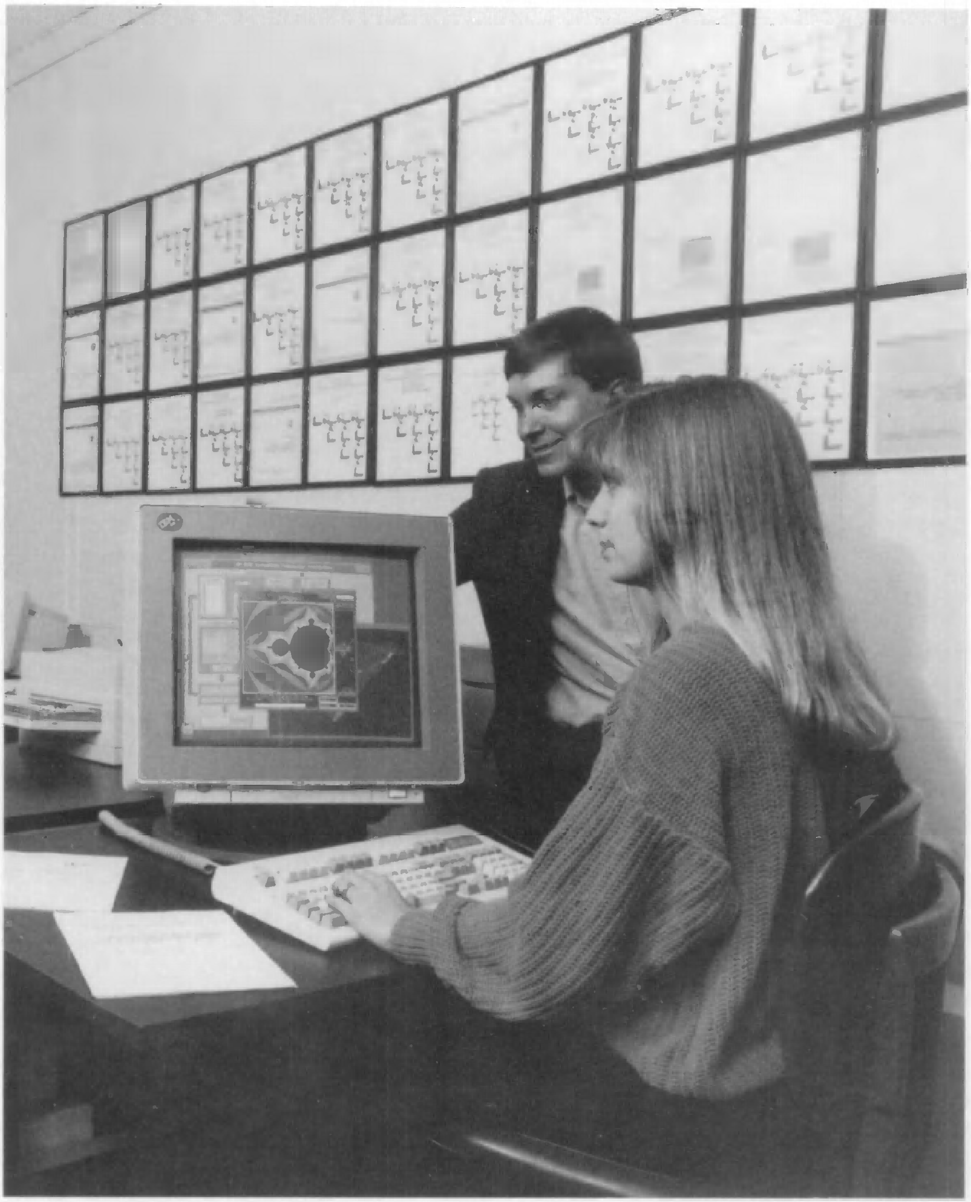
It was clear to review team members that Federal computer security would continue to present a hazard for some time to come.

In its efforts to provide guidance and support to Federal agencies and industry in computer security, the division proposed in August 1991 that a FIPS be issued on the subject of digital signature standards for use by Federal agencies. In the proposed standard, a digital signature algorithm would be based on a public key. The digital signature standard would use the public key to verify to a recipient the integrity of data and the identity of the data source.

A revision of FIPS 140 was initiated to help a great variety of organizations to use the latest equipment to establish the physical and logical security requirements for the design and manufacture of data encryption standard equipment. The new revision was expected to form a framework within which cryptographic standards would be incorporated into new products.

During this period, the Computer Systems Laboratory initiated a new series of publications on computer security. Each report became part of *NIST Special Publication 800.* First in the series was a retrospective bibliography describing computer security in the 1980s, compiled by Rein Turn and edited by Lawrence E. Bassham. It was followed by "Public-key cryptography," by James R. Nechvatal, and "Establishing a computer security incident response capability," by John P. Wack.

Special Publication 800-7, issued in July of 1994 by a research team including Robert H. Bagwill, John F. Barkley, Jr., Lisa J. Carnahan, Shu-Jen H. Chang, David R. Kuhn, Paul Markovitz, Anastase Nakassis, Karen J. Olsen, Michael L. Ransom, and John P. Wack, contained a discussion of computer security in open systems. The 280-page document described the Federal response to threats of disruption to the *Public Switched Network,* which provided telecommunications for national security and emergency preparedness. It offered answers to programmers in the network for the question "How do I build security into software based on open system platforms?" The POSIX open system environment was described, with its security interfaces and mechanisms; also the X Window system, a network-transparent graphical user interface system, and the database language SQL. In each case, security considerations were discussed. A long section in the publication described further procedures to be used in achieving a secure system.



NIST computer scientist Karen Olsen and computer specialist Robert Bagwill worked to improve standards for open systems environments to allow greater compatibility among software systems.

Systems and Network Architecture, Kevin L. Mills, chief

In the Systems and Network Architecture division, NIST researchers worked with industry and other government agencies in three primary areas. In one of these areas, a division group headed by Frances Nielsen helped to establish standards for exchanging information on network management, emphasizing solutions to problems of communication among heterogeneous management systems. Methods utilized for this purpose included definitions for protocols needed for the exchange of management information, assembling proper formats for information under exchange, and preparing protocols for the support of the management function.

As an example of information exchange, we note the work of Paul Markovitz in 1991 on behalf of the Internal Revenue Service. Markovitz discussed the principles of electronic data interchange (EDI) as it applied to invoices, purchase orders, and other business information exchanged between companies. Use of electronic means for such exchange provided a quicker and more accurate set of transactions between trading partners.

Markovitz pointed out that electronic messages in the EDI system were not intended for humans, but were to be interpreted by computers in terms of inventory updates, shipping orders, billing, and the like. Many standard formats for EDI were available at that time—the three most widely used were known as X12, EDIFACT, and UN/TDI. The X12 family of formats was approved by the American National Standards Institute. A message handling system (MHS) provided a necessary interconnecting protocol to permit X12 messages to be exchanged between companies with incompatible computer systems. The balance of the 34-page report published by Markovitz consisted of a discussion of the nature of the MHS system and EDI.[222]

In another area, a division group under the leadership of Michael L. Ransom promoted the standardization and commercialization of networking technology. Activities in this area included standardization of the techniques for distribution and management of encryption keys, the design of a new protocol useful in negotiation of security services, facilitating the convergence of network-addressing methods, and standardizing algorithms involved in multicast transport and routing.

Yet a third area of work, led by J. P. Favreau, had as its goal the advancement of tools for editing, compiling, and interpreting computer communications protocols in order to automate their execution.

Advanced Systems, Shukri A. Wakid, chief

Personnel of the Advanced Systems division worked in the areas of parallel processing, data storage, distributed systems, and automated recognition.

[222] Paul Markovitz, "Electronic data interchange in message handling systems," *NISTIR 4608,* June 1991, 34 pp.

A group of scientists in the Systems and Network Architecture Division developed Open Systems Interconnection standards to facilitate the integration of computer components built by different manufacturers into efficient systems. Standing, from left to right, were Michael Anzenberger, Michael Wallace, and group leader Jerry Mulvenna. Seated were Deborah Tang and Paul Markovitz.

A primary activity, headed by David Su, involved the *Integrated Services Digital Network*. ISDN standards under development combined voice, data, text, and image communications within a single network connection. NIST helped develop the measurement capabilities and testbed facilities needed for conformance testing for the standards, as well as performance measurement techniques. An example of this activity was an overview of ISDN conformance testing prepared by Leslie A. Collica, Kathleen M. Roberts, and David Su; in a few pages, they outlined the scope of ISDN conformance testing, they described several test suites for as many layers of ISDN, they noted guidelines for conformance testing, and they provided information on test suite selection.[223]

[223] Leslie A. Collica, Kathleen M. Roberts, and David Su, "Overview of Integrated Services Digital Network conformance testing," *NIST Special Publication 823-1*, March 1992, 11 pp.

A study performed by William E. Burr examined the potential for voice, video, and data networks based on the use of optical fibers. Optical fibers, with the capability of information transfer by light waves, offered an enormous bandwidth relative to copper lines, and thus the possibility of carrying a correspondingly larger volume of information. Burr noted that the lagging development of fast, low-cost switching for optical fiber transmission had delayed the full utilization of its potential benefits. Burr speculated on the architectures that might be used in future high-bandwidth integrated services networks for voice, video, and data applications, comparing them with then-current schemes.[224]

Another group, led by Gordon E. Lyon, looked to enhance the evaluation and use of the most modern computers by the Federal government. Their activities ranged from the characterization of new computer architectures to the identification of improved capability for the automatic processing of data, to the study of programming methods that promised standardization among different classes of computer architecture.

An example of the characterization of state-of-the-art computer analysis was a study published by Robert D. Snelick in 1991. Snelick wished to examine the ratio of time spent by parallel processors in communication to the time spent in actual calculation. He noted that loosely coupled multiprocessors synchronized their operations and shared data through the exchange of explicit messages; thus the speed of communication was crucial to optimizing the performance of such machines.

A process Snelick called time dilation allowed him to vary the ratio of communication time to computational time for hypercube applications. The time dilation technique offered better detail on these features than previous methods of system analysis. Snelick employed as a test program a ring-type benchmark, in which a synthetic ring was created with a number of logical nodes or processes. Each node originated a given number of messages and also processed all other messages passing through it. When a particular message reached its originating node, it was removed from the network. When all messages programmed into the ring were processed, the program was complete. Message reception was acknowledged within the ring to prevent buffer overflow.

Snelick also used a random communication model benchmark in his tests, as well as a mesh model. He analyzed the results of the tests, finding that the time dilation technique provided an accurate method for investigating the performance of loosely coupled machine applications and, ultimately, improving the capabilities of programmers to better utilize their computing equipment.[225]

Yet a third team worked in the area of optical disks. Under the direction of Dana S. Grubb, group personnel stayed abreast of developments in optical disks, preparing test methods for conformance to both national and international standards.

David S. Pallett headed a group that was involved in collecting speech-recognition databases for distribution to dozens of organizations interested in the use of speech to direct the operation of computers.

[224] William E. Burr, "Architectures for future multigigabit lightwave networks," *NISTIR 90-4240*, February 1990, 64 pp.

[225] Robert D. Snelick, "Performance evaluation of hypercube applications: using a global clock and time dilation," *NISTIR 4630*, July 1991, 21 pp.

Leader of the NIST Automated Recognition Group David S. Pallett (left) and computer scientist James Hieronymus discussed a computer program designed to recognize vowels in their phonetic context.

Image recognition, too, received the attention of the Advanced Systems division. Research led by Charles L. Wilson was directed to the development of methods for evaluating image quality, efficiency of data storage, and systems used in optical character recognition. Among the projects touched by the work were fingerprint recognition and automation of personnel form completion.

Computing and Applied Mathematics Laboratory

The Computing and Applied Mathematics Laboratory staff was composed primarily of mathematicians and statisticians. Formed decades earlier as the Applied Mathematics Division, whose staff gave much-appreciated assistance on the interpretation of scientific data to researchers and calibration personnel alike, the division built as well a program of independent research. By 1991, under the leadership of Francis E. Sullivan, former chair of the Mathematics Department at the Catholic University of America, the new CAML program included mathematics, statistics, scientific computing, graphics generation, data manipulation, and parallel processing. Following the 1993 departure from NIST of Sullivan to engage in supercomputer research, NIST Director John Lyons appointed Joan R. Rosenblatt, veteran of nearly 40 years at NBS/NIST, to become laboratory director.

Applied and Computational Mathematics, Paul T. Boggs, chief

Parallel computing received the attention of a group under the leadership of Isabel Beichl. Realizing that algorithms and computer codes developed for data processing on single machines provided little increase in efficiency when transferred to parallel computers, the group conducted research on algorithms specifically for use in parallel processing. Particular problems addressed in the work were computational geometry and random processes.[226]

Work in the solution of computational geometry problems using parallel processing was discussed by Beichl and Sullivan in a 1991 paper. The authors noted the relative speed of progress in the use of parallel processing to solve problems in which iteration was known to be convergent—not the case with computational geometry problems. They outlined a new algorithm for triangulation to exploit the use of Single Instruction Multiple Data Stream computers.

Other groups in the division attacked problems in analytical approximation, particularly in non-linear mechanics calculations; mathematical modeling; optimization and computational geometry; and accurate and robust software for scientific computing.

Statistical Engineering, Robert J. Lundegard, chief

The Statistical Engineering Division staff supported experimental design as it had for decades. The division also developed strategies for data acquisition, graphical and numerical analysis, modeling of results, and estimation of experimental uncertainties for scientific, technical, and calibration personnel of NIST and its industrial collaborators.

An example of the work of the division during this period was a discussion of an ancient topic made new by changing events in science—uncertainty and accuracy in physical measurements—by Harry H. Ku. Ku outlined the historical scientific use of the term "uncertainty" to include "random" and "systematic" components. Random components could be treated statistically, whereas systematic components influenced all measurements leading to the reported results. Experimenters typically estimated bounds for the magnitudes of systematic uncertainties, added them linearly, and reported them separately from random error estimates.

In 1978, the International Bureau of Weights and Measures (BIPM) issued questionnaires to the national laboratories, seeking information and opinions on the treatment of uncertainties. In 1981, the BIPM issued a set of five rules for the combination of experimental uncertainties. In these rules, new categories replaced "random" and "systematic":

- Two categories for the components of experimental uncertainty were to be identified as "A"—those that were evaluated by statistical means—and "B"—those evaluated by other means. Detailed reports of all measurements were to include a complete list of the components, specifying the method used for each quantity. It was stressed that the new categories did not necessarily correspond to "random" or "systematic" definitions.

[226] I. Beichl and F. Sullivan, "Parallelizing computational geometry: first steps," *Siam News* **24**, No. 6, pp. 14-16 (1991).

- Category A components were to be characterized by the estimated variances s_i^2, (or the estimated standard deviations s_i) and the number of degrees of freedom, ν_i.

- Category B components were to be characterized by quantities u_j^2, which might be considered as approximations to the corresponding variances.

- The combined uncertainty was to be characterized by the numerical value obtained by applying the usual method for the combination of variances, with the use of standard deviations.

- If, for particular applications, it was necessary to multiply the combined uncertainty by a factor to obtain an overall uncertainty, the multiplying factor had to be stated.

Ku gave his own view of the new rules, pronouncing them "liberal" in potentially underestimating the overall uncertainty of a given measurement because of the "root-sum-of-squares" treatment of the non-statistical uncertainty components; in comparison, the older uncertainty treatments were "conservative," potentially overestimating the overall uncertainty because of the linear treatment of systematic uncertainties.

Ku suggested that the best justification for the new method of calculating uncertaintites should be the uniformity that it would give to the treatment of data. He also suggested that the new treatment, minimizing the effect of non-statistical uncertainties, was a healthy change in international metrology, since it more clearly delineated differences in metrological results among the various laboratories that sought an accurate measure of a physical quantity. The older definition of uncertainty, he wrote, was more suited to industrial calibration laboratories, where elucidation of the reproducibility of a set of measurements, not its fundamental accuracy, was the goal.[227]

Scientific Computing Environments, Sally E. Howe, chief

Early in the period, Darcy P. Barnett and David K. Kahaner described a computer program designed to solve ordinary differential equations, a common problem for scientists. The two called their program PLOD; it attempted to simulate human reasoning in the solution of such problems. [228]

One of the strong programs that developed in the Scientific Computing Environments division was the use of graphical display techniques for the presentation of results in physics and chemistry. Also under exploration was the manipulation of dynamic objects in scientific research and in automated design and manufacturing systems.

[227] Harry H. Ku, "Uncertainty and accuracy in physical measurement," *NIST Special Publication 805,* December 1990, 9 pp.

[228] D. Barnett and D. Kahaner, "Experiences with an expert system for ODEs," *Mathematics and Computers in Simulation* 31, Nos. 4-5, pp. 315-323 (1989).

An interesting illustration of imaging techniques concerned the elucidation of the structure of "quasicrystals"—the icosahedral phase formed by rapid solidification of an aluminum-manganese alloy (see A "Renaissance" Scientist Comes to NBS in Ch. 4). At that time, no complete description of the atomic structure for the icosahedral phase had yet been found. In the investigation, division scientist Howland A. Fowler collaborated with A. J. Melmed—at that time in the private sector, but for many years a member of the NBS/NIST Surface Science Division—and H. B. Elswijk, a young scientist working for Philips Research Laboratories in Holland.

Starting with a field ion microscope image of the icosahedral phase showing two-fold, three-fold, and five-fold poles, the authors attempted to duplicate the experimental image by computer simulation. Beginning with the cubic phase, they gradually increased the size of the unit cell, introducing a manganese sublattice forming a collection of Mackay icosahedra decorated with aluminum atoms. As the unit cell size was increased from an edge length of 1.3 nm to 3.3 nm, the cubic features became weaker in comparison to pentagonal features. It was their view that the simulation experiment might point to an improved understanding of the interesting new structure.[229]

The division also carried the responsibility for maintaining the central NIST computing facility, as well as investigating the use of new computational hardware, software, and communications techniques.

THE MORE THINGS CHANGE, THE MORE THEY REMAIN THE SAME: INDUSTRIAL PRODUCTIVITY IN SEMICONDUCTORS—NIST PREFIGURED

In 1988, the National Bureau of Standards, an institution devoted to the creation of standards of measurement and to solving technical problems afflicting America, changed into the National Institute of Standards and Technology, an institution devoted to solving technical problems afflicting America and to the creation of standards of measurement. "A new institution, and not a better one, either," opined more than a few old-time employees. "A better institution, but hardly new," responded others.

One of the most cogent pieces of evidence indicating that only a change in emphasis—not a change in actual practices—occurred at NBS in 1988 could be found in a tale told later by an NBS/NIST senior manager who was on the scene for a long time. We are indebted to Judson C. French, who recently completed a half-century of service to NBS/NIST, for the details of a 1995 presentation to its Standards Alumni Association. His story, "Development of the NBS/NIST Semiconductor Program" is told from the point of view of a physicist who opened a long Bureau career with investigations of microwave gas tubes and semiconductor materials and devices shortly after World War II.[230]

[229] A. J. Melmed, H. B. Elswijk, and H. A. Fowler, "Field-ion microscope image simulations for icosahedral Al-Mn," *Colloque de Physique*, Supplement to Journal de Physique, 50, Colloque No. C8, November 1989, pp. C8-259 to C8-263.

[230] Judson C. French, "The NIST Electronics and Electrical Engineering Laboratory and the development of its semiconductor program," *NISTIR 6507* (2000).

The National Bureau of Standards had a difficult time keeping a scientific force to assist in the birth of transistorized electronics at mid-century. One effort, in the proximity fuze laboratory, was transferred to the U.S. Army along with the laboratory itself. A second effort began in 1955, when $30,000 of precious Bureau funds were apportioned to three sections in the Electricity Division—$10,000 each for work under Franklin Montgomery on transistor-based instrumentation, under Augustus Shapiro on transistor reliability, and under Charles Marsden on transistor physics. The transistor work was to be performed by French. The expertise developed in Montgomery's section disappeared when the general instrumentation project—of which it was a part—was disbanded. The reliability work left NBS with the rest of a U.S. Navy project of which it was a part.

French, working in the Bureau's Electron Tube Laboratory, digested William Shockley's first writings on transistors,[231] then began visits to Federal and industrial laboratories to find common needs in the transistor field. Soon, industry-wide problems surfaced in attempts to characterize semiconductor materials and devices. Such investigations suited NBS just fine, so French and his early colleagues William Keery and Marvin Phillips concentrated their efforts in those areas.

Visits to the American Society for Testing and Materials and to the Electronic Industries Association were similarly rewarding; both organizations were well aware of member complaints on the unreliability of semiconductor measurements.[232] From the plethora of problems, the NBS group selected semiconductor resistivity and second breakdown in transistors. Each was addressed successfully at NBS and the results were well-received by the growing semiconductor industry.

In the case of the resistivity of silicon—the most basic of measurements for electron device design and manufacture—measurement discrepancies were entirely too large to permit effective control of manufacturing processes or materials purchases. Rejecting the destructive two-probe measurement technique then believed by the industry to be necessary for accurate results, the NBS team, after visits with colleagues in industrial laboratories, developed a non-destructive, four-lead test involving commercially available instrumentation and precise procedures. The principal scientist in this project was Lydon J. Swartzendruber. The new techniques reduced measurement imprecision by more than a factor of ten.

French and his team took their new ideas to the ASTM for study by the semiconductor industry. Once the Bureau scientists convinced their industrial colleagues, through interlaboratory trials under ASTM oversight, that reproducible results could be

[231] William Shockley, *Holes and electrons: an introduction to the physics of transistors* (Murray Hill, NJ: Bell Telephone Laboratories, 1949), based on lectures at the Bell Laboratories. See also William Shockley, *Electrons and holes in semiconductors, with applications to transistor electronics* (New York: Van Nostrand, 1950).

[232] Memorandum, J. C. French to R. D. Huntoon, Deputy Director NBS, March 9, 1960: "Minutes of a meeting of representatives of the Joint Electron Device Engineering Council and NBS on October 1, 1959. See also

Letter, R. D. Huntoon to Virgil Graham, Chairman, Joint Electron Device Engineering Council and Associate director, EIA Engineering Department, June 1, 1960.

achieved only by the introduction of improved temperature control, more careful measurements, and better procedures, the NBS methods were quickly accepted.

Five industrial semiconductor measurement standards on silicon resistivity resulted from the NBS work, as well as several Standard Reference Materials.

In this one project undertaken in the mid-1950s, the NBS research team demonstrated the kind of close interaction with industry that Congress mandated in its 1988 Trade Act legislation—to select projects with high industry priority and a good match to the NBS measurement standards mission, and to involve industry directly in making quick use of the newly developed technology. Then, as later, NBS scientists found easy access to industrial laboratories because of their unique positions as objective scientific collaborators.

With the advice and assistance of the semiconductor industry, the NBS team conducted a benefit-cost study on the resistivity project.[233] The study showed economic benefits of more than $30 million in marketplace transactions as a result of the work—more than 100 times the cost of the project—and even more savings in manufacturing costs.

Although considerable time has passed since they were first developed, semiconductor resistivity SRMs recently were purchased from NIST by as many as 180 companies. A vice president of a process control systems firm stated, "the availability of resistivity SRMs was a significant factor in successfully developing and selling the $50 million installed base of these systems."[234]

Clearly, the NBS semiconductor research prefigured the later insistence that Bureau programs should be guided by industry needs.

In the second-breakdown project, for which Harry Schafft was the principal investigator, an even more startling development took place. During a visit to NBS by William Shockley, co-inventor of the transistor, the Bureau group reported to him experimental results that contradicted his theory of second breakdown. Convinced by the thoroughness of the NBS investigation, Shockley modified his theory to take account of the new results. The NBS characterization principles became widely applied in the industry.

As the NBS work in semiconductors became better known, more frequent requests for assistance were heard from industry and government alike. Under French's direction, NBS developed a joint program with funding and participation from other Federal agencies, attacking problems considered important by both industry and the agencies. Quarterly progress reports from the joint semiconductor project were requested by as many as 2000 representatives of organizations in industry, government, and academia.

[233] Judson C. French, "Improvement in the precision of measurement of electrical resistivity of single crystal silicon: a benefit-cost analysis," *NBS Electron Devices Section Internal Report 807*, September 20, 1967.

[234] Letter, Winthrop A. Baylies, President, The Baytech Group, to James Ehrstein, NBS, June 22, 1989. See also Letter, Winthrop A Baylies, Vice President/Marketing, ADE Corporation, to Malcolm Baldrige, Secretary of Commerce, November 25, 1981.

Passage of the Mansfield Act in 1973 seemed to doom the joint-agency program—no Department of Defense agency would be allowed to fund generic technologies. In the nick of time, however, the DoD Advanced Research Projects Agency commissioned NBS to undertake a multi-year, multi-million-dollar project to improve the performance and reliability of integrated circuits. Selection of NBS for the project was a direct result of the reputation for effective service gained by Bureau researchers.

The ARPA project accomplished more than the salvation of the NBS semiconductor research program; it also initiated a pattern of external contracting by NBS—using Bureau funds to pay for research to be done at universities and private companies. W. Murray Bullis, Alvin H. Sher, and Joseph A. Coleman led the NBS contract-monitor group.

The experience of the NBS contract monitors—who found that painstaking analysis and measurements were not part of the technical culture everywhere, and that the process of effectively monitoring technical aspects of contract research was both laborious and time-consuming—was not an entirely happy experience for the Bureau scientists. Yet, during the 6-year life of the ARPA program, some 30 contracts were let, some 240 report documents were prepared, and some $11 million was spent productively.[235] Most pleasing was a letter of praise for the NBS work, sent to the Secretary of Commerce by the ARPA director.[236] The letter stated that the program had met its objectives, industry had benefitted directly, and dramatic improvements had been realized in the performance and reliability of devices built for DoD use, saving millions of dollars for Defense agencies. As a consequence, the director predicted that his agency and the military services would expand their programs of joint DoD-DoC projects.

One of the ARPA program projects involved wire bonds to semiconductors. Unpredictable wire bond failures had imperiled the reliability of certain weapons. George G. Harman led the Bureau research effort to ameliorate the problem, visiting industrial laboratories and factories where bonding work was performed and then commencing NBS laboratory work with the help of existing (commercial) bonding equipment used for the DoD devices. Quickly, Harman found unexpected sensitivity in the bonding equipment to temperature variations and to vibrations, even those of small amplitude.

Harman's work led to better understanding of the physics of bonding, to new bonding procedures, and to improved equipment. Productivity in bonded circuit manufacture improved by as much as 35 times, and the improved quality permitted the use of circuit elements with as many as 500 bonds each. Failure of bonds in field systems ceased to be observed. Once again, NBS had anticipated the change to NIST.

[235] W. M. Bullis, "Advancement of reliability, processing, and automation for integrated circuits with the National Bureau of Standards (ARPA/IC/NBS)," *NBSIR 81-2224*, March 1981.

[236] Letter, Robert R. Fossum, Director, Advanced Research Projects Agency, to Juanita M. Kreps, Secretary of Commerce, April 17, 1979.

As the ARPA program drew to a close, NBS commissioned an "impact study" by the Charles River Associates to assess the value of the Bureau work to the semiconductor industry.[237] Industry officials were happy to testify to the importance of the NBS effort. Specific benefits cited included improved product reliability, improved production, lower costs, increased ability to meet customer specifications—even new directions for company research.

The Federal Government's financial stringency in the period almost caused the demise of the semiconductor project at the conclusion of the ARPA program, although new funds for the program had been appropriated by Congress. These would have replaced the ARPA support, but they were to be cut by the Office of Management and Budget. However, the perceived benefits of the program led to a successful industry-wide effort to make clear how important and appropriate it was for NBS to conduct such collaborative work with its own resources serving as a strong basis.[238] The program was saved and has continued to this day.

The semiconductor program provided direct benefits to industry through a continuous process of reaching outside of NBS for program guidance and evaluation. In that process it paralleled other programs described in this chapter—programs motivated to emphasize industrial productivity largely by the language of the 1988 Trade Act. The semiconductor program was not alone among NBS disciplines in initiating its policy of outreach decades prior to passage of the Trade Act—the fire research, building research, cryogenic engineering, time and frequency, and electromagentics programs, in particular, spring to mind immediately. But French's semiconductor progam serves admirably to demonstrate that no new practice for NBS as an institution was created when it became NIST in 1988. Rather, a culture already present in the agency was prescribed for its entire structure; a culture which believed that NIST should provide direct practical assistance to industry where NBS was uniquely positioned to respond, with that assistance backed up by the fundamental research needed to provide a solid foundation.

As NIST settles into its expanded role in the Nation's technical life, it remains to be seen whether the agency will retain the capability for fundamental work in physics and chemistry that characterized its first century of existence—the capability that made it instantly successful in its new role.

[237] Planning Report No. 8, "Productivity impacts of NBS R&D: a case study of the semiconductor technology program," Summary Volume, prepared for Planning Office, NBS; CRA Report No. 493, Charles River Associates, Inc., June 1981.

[238] Letter, T. D. Hinkelman, Executive Director, Semiconductor Industry Association, to David Stockman, Director, Office of Management and Budget, December 14, 1981.