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NBS

TECHNICAL HIGHLIGHTS

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UNITED STATES DEPARTMENT OF COMMERCE

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U.S. NATIONAL BUREAU OF STANDARDS

Lewis M. Branscomb, *Director*

1969 Technical Highlights of the

National Bureau of Standards

Institute for Basic Standards

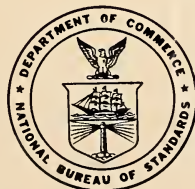
Institute for Applied Technology

Institute for Materials Research

Center for Radiation Research

Center for Computer Sciences and Technology

Annual Report, Fiscal Year 1969



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ERRATA TO ACCOMPANY NATIONAL BUREAU OF STANDARDS
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REPORT 1969

- p. 33, picture caption should refer to the electrostatic (not "electromagnetic") field within a Kerr cell.
- p. 76, line 10, should have identified the research project sponsor as the Army Research Organization -- Durham (ARO-D, not "AQDP").
- p. 85, line 6, the figure after the = sign should be 4.04362 (not 4.0362).
- p. 88, line 9, the term at the beginning of the line should be (d001).
- p. 88, line 24, delete "a thermal stability to temperatures of the order of 500°C" and add "increased thermal stability".
- p. 88, line 26, should have said -40°C, not -50°C.
- p. 107, line 11, delete d and make word "ethane".
- p. 117, line 26, should refer to "2054°" rather than "2053°".
- p. 117, line 27, should give the standard deviation as $\pm 6^{\circ}\text{C}$, not $\pm 4^{\circ}\text{C}$.

FOREWORD

This report on technical highlights of the National Bureau of Standards is valedictory in nature. The report covers fiscal year 1969, July 1, 1968 to June 30, 1969. Allen V. Astin, director of the Bureau for 17 years, retired on August 29, 1969.

As Dr. Astin's successor, I am well aware that the progress evident in this and in the preceding 16 annual volumes reflects the determining influence of a scientific administrator whose leadership strengthened U.S. science and technology in an era of tremendous change. Building upon Dr. Astin's solid contribution, we hope in the years ahead to draw inspiration from his work and to rise to future challenges in a similar spirit.

Lewis M. Branscomb, *Director*

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INTRODUCTION

Staff and Organizational Changes

The National Bureau of Standards has continuing responsibility as the Nation's central measurement laboratory under terms of the Organic Act which created it in 1901 and through subsequent amendments to the Act. In addition, as a result of changing technology and through special legislative acts, new and expanding activities have been undertaken over the years. Changes in Bureau programs and activities have, not surprisingly, required redirection of administrative and management emphasis.

Significant changes of the past fiscal year are discussed below. In each case, the change was designed to make the Bureau more responsive to needs of the commercial, scientific, and industrial groups which it serves.

New Deputy Director Named

Dr. Lawrence M. Kushner was named Deputy Director of NBS in May, 1969. He was previously the Director of the NBS Institute for Applied Technology.

Office of the Associate Director for Information Programs Established

The major organizational units at NBS that gather, analyze, and distribute technical information have been combined under an Associate Director for Information Programs. The new arrangement brings together related information activities that were previously managed independently. It permits improved coordination, common policy interpretation, and centralized and coordinated program planning, budgeting, and evaluation.

The organizational components included in the new office are the Office of Standard Reference Data, the Clearinghouse for Federal Scientific and Technical Information, the Office of Technical Information and Publications, the Library Division, the Office of Public Information and the Office of International Relations.

The information activities of the new office range from those of the Bureau's library, which primarily serves Bureau employees, to those of the Clearinghouse for Federal Scientific and Technical Information, which is responsible for disseminating technical information generated throughout the Federal Government.

Dr. Edward L. Brady, formerly Chief of the Office of Standard Reference Data, has been appointed Associate Director for Information Programs.

Dr. Brady came to NBS in 1963 to serve as Chief of the Office of Standard Reference Data.

Reorganization of the Center for Computer Sciences and Technology

The Center for Computer Sciences and Technology, established in 1966 in response to the Brooks Bill, has been made a separate organizational entity reporting to the Director of the Bureau. The purpose of the Center is to develop standards and conduct research in the field of automatic data processing and to provide technical services to other agencies for improving the cost-effectiveness of Federal programs in the selection, acquisition, and use of automatic data-processing equipment.

The Director of the Center, Dr. H. R. J. Grosch, received his B.S. and Ph.D. degrees from the University of Michigan. In 1945 he began his career in the data processing field with International Business Machines Corporation. In 1951 and 1952 he did logical design research at M.I.T. and later went on to manage project DEACON at General Electric's Center for Advanced Studies at Santa Barbara. From 1959 through 1965 he served as a consultant to major computer and information handling business concerns in the United States and Europe. Dr. Grosch has written articles on such diverse topics as celestial mechanics, lens design, numerical analysis, and the installation of computer operations. For four years he was a contributing editor of the journal, *Datamation*.

Advanced-Research Teamwork With Universities

Major moves toward increased cooperation with the Washington area's institutions of higher learning were made when the National Bureau of Standards entered into agreements with the University of Maryland and George Washington University to facilitate joint efforts in advanced scientific research. The agreements are a continuation of NBS efforts in the field of University-Government cooperation consistent with a White House memorandum in 1965 which encouraged sharing of laboratory facilities. In particular, these agreements are

designed to encourage contributions to the Nation's research and development from young scientists early in their careers.

The participating universities and NBS are providing for greater use of the Bureau's personnel and facilities in furthering graduate training and research, focusing university competency more strongly on work geared to national goals, and establishing machinery for close collaboration between each university and NBS in selected joint programs. Under the agreements, every effort is being made to attract distinguished scientists to limited-term appointments furthering the going projects, either at the Bureau or at the universities. University-related activities engaged in by NBS staff are considered part of the Bureau's normal activities, and activities of university staff and students on joint projects at NBS are regarded as part of normal university responsibilities. Supplementing the general agreement between the University of Maryland and NBS, a specific memorandum of understanding has been adopted, providing for a Cooperative Program for Advanced Materials Research, teaming the Bureau's Institute for Materials Research with the University of Maryland's Center of Materials Research.

Progress in Equal Employment Opportunities

During fiscal year 1969 several steps were taken to improve the NBS Equal Employment Opportunity Program. Among the steps were the strengthening of the EEO Committee, the establishment of an Affirmative Action Plan for promoting equal employment opportunity, and a vigorous information program, highlighted by meetings of the Director with the entire NBS staff explaining the Bureau's EEO program and enforcement procedures for non-discrimination rules. The Affirmative Action Plan defined short-term and long-term objectives for the program, pinpointed responsibility for the achievement of the objectives, and set target dates for their achievement. Objectives include improved training and job enrichment for minority employees, and monitoring efforts to assure adequate representation of qualified minority employees on promotion certificates and training class rolls.

The Education Committee established a subcommittee to improve training opportunities for disadvantaged employees. Following the subcommittee's report, an adult basic education program has been planned to start in September, 1970, to prepare employees for technician courses. A new position is planned for a full-time counselor on career development.

Transfer of NBS-CSA Test Development Division

For several years NBS has operated a testing laboratory for the General Services Administration to develop new test methods and

run tests on specific commodities purchased by the Federal Supply Service. In keeping with the general NBS policy of developing and disseminating measurement expertise as widely as possible throughout other government agencies and private enterprise, the laboratory has been strengthened and transferred to the General Services Administration. The transfer of the laboratory is appropriate, since GSA has responsibility for qualified product lists and has the legislative authority to run a laboratory for testing products against standards.

Programs Affecting National Policy and Consumer Problems

Metric System Study

Since the August 9, 1968 enactment of Public Law 90-472 authorizing a Department of Commerce appraisal of "the advantages and disadvantages of increased use of the metric system in the United States," the Bureau's exploration of the question in cooperation with other groups has indicated that the study will be concluded on time and that it will be possible to provide reasonable estimates for some of the costs of any major U.S. changeover to the metric system.

The basis for estimated costs to firms has been laid by looking at potential costs as they would accrue if metric measurements were to be applied to new or redesigned products, stressing the concept of planning a transition—if there is to be a transition—at an optimal rate, without going back and changing all the drawings that are in the files. This method, it is felt, avoids the exaggerations and "astronomical" figures which would result from a doctrinaire approach based on an all-or-nothing plunge into metric measures throughout U.S. science and technology.

Specialists conducting the NBS study have relied on the invaluable orientation guide devised by the American National Standards Institute for companies making metric studies. In this approach costs are being estimated on the assumption that metric specifications will be used only for new, uniquely designed major components or end products requiring new special tooling—only after new metric standard parts and materials are readily available at reasonable cost. This can be accomplished on an optimum schedule as present product designs become obsolete, on a timetable compatible with marketplace requirements and normal tool obsolescence. In some areas—for example, the railroad and oilfield industries—changes in existing equipment do not appear to be warranted.

Several questionnaires are being developed by NBS to aid in the study. Companies which have already looked closely into the advantages and disadvantages of increased metric use are being queried, as are other companies which have not attacked the problem in detail. A general questionnaire will be sent to both groups and their answers will

be compared. Large Federal agencies will be asked by the Bureau to gage the probable effects that “going metric” would have on their areas of responsibility. The Department of Defense, in particular, has a study team working intensively on the question of what the impact would be in the area of national security.

A series of NBS conferences is being planned to gather further data and to provide an opportunity for representatives from the various sectors of the economy to present their points of view.

An advisory panel has been organized to provide constructive assistance to the NBS Study Group. Members of the panel, which numbers almost 50, were selected from a broad cross-section of the economy.

Great Britain’s experience, and that of other nonmetric countries undertaking a changeover, is being analyzed in the NBS study to see what the United States can learn from their accomplishments and mistakes.

The Bureau expects to present to the Secretary of Commerce a detailed report on both domestic and international implications of this issue—whether positive or negative—in order that the Secretary may recommend a course of action to the Congress.

Automotive Safety

An agreement between the Department of Commerce and the Department of Transportation was extended to continue the National Bureau of Standards Office of Vehicle Systems Research as an objective third-party resource to the National Highway Safety Bureau.

One of OVSR’s major activities during the year was working on a uniform quality-grading system for tires to allow for an informed choice by consumers. Objectives are to establish standard test methods for several important tire properties, define quality levels for each property, and develop a means of conveying the information to the consumer.

Broader Protection from Flammable Fabrics

Procedures for carrying out the Secretary’s responsibilities under new and amended sections of the Flammable Fabrics Act were published in the *Federal Register* of October 1, 1968. They included rules for developing flammability standards by a four-step process.

The NBS Fabric Flammability Section initiated research on the transfer of heat from burning fabrics and related materials, of hazards resulting from ignition of interior furnishings (beds and upholstered chairs), and development of a statistical sampling technique for investigation of deaths, injuries, and economic losses.

After analyzing data from Consumers Union, the Department of Health, Education and Welfare, and other sources, NBS recommended to the Assistant Secretary of Commerce for Science and Technology

that he issue findings that there may be need for new or amended standards for both clothing and carpets and rugs. Accordingly, on October 23, 1968, notices were published in the *Federal Register* that new or amended flammability standards or other regulations, including labeling, may be needed for wearing apparel. On December 3, 1968, a similar notice was published, applying to carpets and rugs.

The National Advisory Committee for the Flammable Fabrics Act was appointed, effective January 1, 1969. The committee's 17 members—representing manufacturers, distributors, and the consuming public—were selected by the Secretary of Commerce from among more than 50 nominations. The committee is expected to be particularly helpful in giving guidance on priorities for efforts both in test-method development and in research.

The National Bureau of Standards organized a Symposium on the Measurement of Flammability. Several hundred attendees heard representatives of industry, government, universities, and the medical professions speak on the importance of heat, flame spread, toxic products, and ignition characteristics of wearing apparel, mattresses, drapes, blankets, linens, furniture, and other items.

CENTER FOR COMPUTER SCIENCES AND TECHNOLOGY

In the quarter-century since the electronic computer made its debut in America, the National Bureau of Standards has been a leader in the development of what is now the fastest growing technology in the world. Greatly expanded since the forties, when early NBS research on computers was a partial function of the Ordnance Development Division and the National Applied Mathematics Laboratories, the Bureau's program in this field today is carried on by the Center for Computer Sciences and Technology under a Congressional mandate embodied in the 1965 Brooks Bill (Public Law 89-306).

This law and supporting policy guidance from the Bureau of the Budget assigns to NBS the responsibility for:

- providing guidance in the promulgation of hardware and software standards, both for industry-wide voluntary standards and Federal standards
- providing other Government agencies technical assistance and consultation in both hardware and software areas in the efficient use of computers
- promoting training in the various areas of computer applications
- providing information services related to computer technology
- providing computer services—both use of equipment and programming—to other Government agencies

and

- engaging in exploratory research.

History of the Program

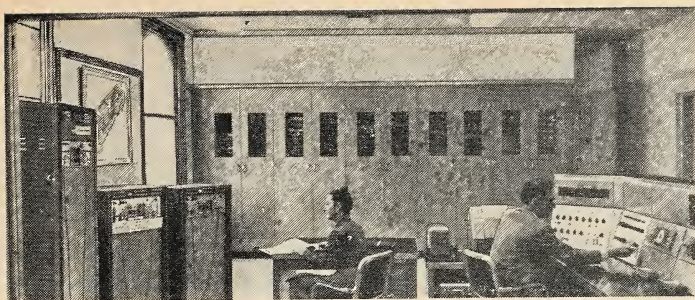
NBS involvement in electronic computing machines began as a part of the Bureau's program of technical assistance to other agencies. In 1946, two of them asked NBS to provide specifications and deal with manufacturers in the procurement of electronic computing machines. For the Bureau of the Census, NBS contracted with Eckert and Mauchley's Electronic Control Company for a UNIVAC; for the Office of Naval Research, the Bureau contracted with the Raytheon Corporation for a RAYDAC. Then the Bureau's own laboratories became fully occupied with a two-year program for the development of improved components for digital computers, carried out under sponsorship of the Office of the Chief of Ordnance, Department of the Army.

The NBS Computers

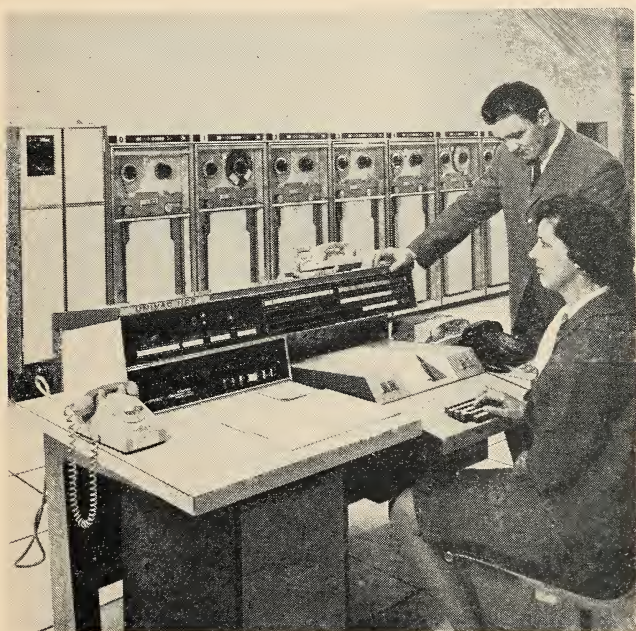
Work on the first UNIVAC followed an uncertain timetable because of unforeseen technological and contractual difficulties; delivery was made to the Census Bureau in 1951 after completion of extensive acceptance tests designed by Dr. Edward Cannon and Mrs. Ida Rhodes of the NBS Applied Mathematics Division.

These were also the years of Project SCOOP (Scientific Computation of Optimum Programs), a pioneering effort to apply scientific principles to large-scale problems of military management and administration. The Air Force's Office of Air Comptroller had requested that an NBS contract be amended to include a UNIVAC for Air Force program planning applications under the project, but delays in completion of both university and commercial machines led to a request for a "stopgap" installation. The Air Force asked the Bureau to launch a crash development program for a modest system designated as the NBS Interim Computer, renamed SEAC (Standards Eastern Automatic Computer). In the summer of 1948, NBS explored possibilities of designing and constructing a machine with sufficient power for general use and yet simple enough to be constructed quickly. To supervise SEAC's construction, Samuel N. Alexander, then chief of the Electronics Division's electronic computer section, recruited from among von Neuman's group at Princeton a young engineer and inventor named Ralph J. Slutz. Working with some 30 engineers and technicians, Slutz implemented the logical design of Dr. Samuel Lubkin, formerly of the University of Pennsylvania's Moore School of Electrical Engineering. When it became evident that the proposed SEAC computer would be the only equipment available to NBS and collaborating Government agencies for at least two years, the modest "interim" objectives were reconsidered. Dr. Slutz and his team made improvements and additions to Dr. Lubkin's original design and SEAC went into production in May 1950, just 20 months after its inception. It was the first general-purpose, internally sequenced electronic computer in operation in the United States, and provided the initial high-speed computing support to Project SCOOP until its UNIVAC was delivered in 1952.

In addition to the UNIVAC's for Project SCOOP and the Census Bureau, a third UNIVAC for the Army Map Service was contracted for by the Bureau. Another computer, SWAC (Standards Western Automatic Computer), was being built at the Institute for Numerical Analysis, a section of the NBS Applied Mathematics Division, at Los Angeles. Operative early in 1952, SWAC differed from SEAC in using a parallel mode of operation. Its primary memory was composed of Williams tube units, enabling all digits of a number to be placed in memory or transferred simultaneously and arithmetic operations to be performed in parallel. SWAC's development was



SEAC, NBS's first computer, was completed in 1950. For the fourteen years of its useful life, SEAC was used by dozens of other government agencies; portions of this computer are now preserved in the Smithsonian Institution.



The NBS 1108/UNIVAC facility also serves as a service center for government and is available to all government agencies. Many users are served by computer terminals, some as far away as 2,000 miles. This modern facility is able to deliver results more than 1,000 times as fast as the pioneer SEAC.

jointly sponsored by the Air Force's Office of Air Research, from interest in the performance of the machine itself, and the Office of Naval Research, from interest in programming and mathematical research. SWAC was used in the solution of many aircraft problems by several Federal agencies; it was ultimately turned over to the University of California at Los Angeles, where it remained in use for 15 years.

While working on SEAC and investigating the possible creation of other machines, Bureau specialists decided that SEAC's dynamic circuitry could lend itself to considerable electronic standardization. This in turn provided a basis for a repetitive physical configuration and the assembly of high-level pulse circuitry.

The combined efforts of R. P. Witt and R. D. Elbourn produced a package design which, with some refinements, was used in two computing machines that were essentially modified copies of SEAC—FLAC at the Air Force Missile Test Center and MIDAC at the Willow Run Research Center, University of Michigan.

NBS revised this package design to incorporate etched circuit techniques. Such packages were used in constructing DYSEAC, designed by A. Leiner, W. Notz, L. Smith, and A. Weinberger and completed in 1954. This project was sponsored by the Research and Development Board for the evaluation of various organization and engineering innovations, among them the location of the equipment in a trailer for purposes of mobility. DYSEAC was used by the Signal Corps at White Sands, New Mexico.

Combining digital and analog techniques, the Bureau solved several unique problems, such as the development (for the Weather Bureau and the Atomic Energy Commission) of a special-purpose computer for predicting the radioactive fallout pattern for a 250-mile radius from the detonation point of a nuclear device.

Recognition of the need for a multi-processor stimulated the design of the Pilot Data Processor in the late fifties. Pilot combined a central processor, a satellite processor, and an input-output format controller. Used for experimental information processing in the early sixties, it was not considered feasible to move the one-of-a-kind vacuum-tube machine with its limited software to the new site when NBS shifted its headquarters from Washington to Gaithersburg, Maryland.

Typical of new NBS projects in computer development to assist other agencies were the AMOS IV (Automatic Meteorological Observation Station), designed to serve as the central element of an automatic system to process data for the Weather Bureau, and ACCESS (Automatic Computer-Controlled Electronic Scanning System) for handling information on the Nation's resources.

Research in Computer Components

Originally sponsored at NBS by the Chief of Ordnance, Department of the Army, research on computer components gained impetus when basic components of the first UNIVAC for the Census Bureau had to be evaluated for performance and reliability. As specifications were being written for the UNIVAC, some research efforts were directed to the mechanical aspects of components such as tape drives and keyboards.

Jacob Rabinow was heavily involved in this work, and as his group began to experiment with magnetic recorders using wire and tape, he invented the magnetic particle clutch in 1947. At the request of Army Ordnance, the Rabinow team initiated the design of a large-capacity magnetic memory which resulted in the notched-disc file in the early fifties.

Efforts were concentrated on developing basic components, including storage devices, input and output equipment, and associated specialized electron tubes for such computing functions as gating, switching, delaying signals, interval timing, and pulse shaping. Two prototype high-speed memory units were developed, one using cathode ray tubes and the other diodes and capacitors as the storage units.

One of the devices developed during this period was the magnetic wire cartridge, a simple input-output device that recorded on and read electrical pulses from magnetic wire. This was used in all of the automatic electronic computing systems designed and developed by NBS: SEAC, SWAC, DYSEAC, and the Pilot Data Processor, as well as in SEAC's two offspring, MIDAC at the University of Michigan and FLAC at the Air Force Missile Test Center.

Analog-digital techniques were used also in the design and implementation by J. P. Nigro of NBS and his team of a man-machine systems simulator for the Wright Air Development Center and the Federal Aviation Agency. Resulting techniques were employed in the early sixties for designing MAGIC I and MAGIC II, cathode-ray display devices used with light pens as manipulative controls in conjunction with a computer in applications such as computer-aided design, electronic circuit organization, file information retrieval, linguistics, and others requiring computer graphics in the handling of two-dimensional information.

Electro-mechanical problems associated with the transport and rapid start-stop features of the Rapid Selector engaged NBS attention as a result of Dr. Vannevar Bush's interest in this microfilm machine, which was able to search large amounts of data and retrieve particular sections by means of codes recorded on the film. At the same time, the Bureau continued research on the magnetic tape-handling device and the high-speed card punch.

A corollary development was FOSDIC (Film Optical Scanning Device for Input to Computers) for the Census Bureau. FOSDIC, using optical, mechanical, and electronic techniques, scanned microfilm of hand-marked census forms and recorded the data on magnetic tape for input to a computer. So useful was FOSDIC for scanning forms and graphic material that advanced and more flexible versions were later devised not only for the Census Bureau but also for the Weather Bureau and the Office of Emergency Planning.

After FOSDIC the next step—accomplished through another of Jacob Rabinow's inventions—was the development of OCR (Optical Character Reading) facilities, and an experimental model of a Best-Match Reading Machine emerged in the early fifties. This machine, demonstrated in 1954, is now on exhibition at the Smithsonian Institution.

Computer Applications

Mathematicians in this country and abroad were attracted to the use of the powerful tools represented by SEAC and SWAC. A large group of numerical analysis specialists was built up around each computer, devoting their efforts primarily to the development of linear programming techniques and the restructuring of equations for greater ease in handling by the machines.

SEAC's first solution to a real problem was marked by the May 9, 1950 successful run of the program for tracing skew rays through an optical lens system. This was programmed by Ethel Marden of the NBS Applied Mathematics Division. Mrs. Marden, with Otto Steiner and Ira C. Diehm, represented the entire programming staff for SEAC at that time, but additional personnel were recruited at a fairly rapid pace from the Bureau's mathematics staff, and a series of training courses in computer work was undertaken for NBS and other Government employees. Outside personnel—including employees of other Government agencies, university staff members, and prominent foreign mathematicians—were frequently detailed to the Bureau for training.

In addition to several optical problems, early applications were concerned with heat flow, analysis of crystal structures, and simulations related to wind tunnel designs, airfoil characteristics under various conditions, smog-producing factors, and statistical analyses. There was no lack of mathematical and scientific problems, both from within the Bureau and from other agencies, and they were of remarkable variety.

Concentrated attention was given to two areas of mathematics:

- Numerical analysis for investigation of series approximations and other numerical approximation techniques tailored for computer handling of mathematics (some of this work helped to

provide a basis for revised mathematics curricula at all three levels of the educational system).

- Algebra, matrix manipulation, and linear programming techniques (with applications for purposes of aircraft deployment and maintenance during the Korean war, in bid evaluation for the Army Quartermaster Corps, and in other fields).

In the early fifties, after SEAC made the first contributions to H-bomb design calculations, Dr. Allen V. Astin, NBS director, received a letter of commendation from the Atomic Energy Commission.

Programming research and mathematical research for computers advanced together at NBS. The Computation Laboratory, under Dr. Joseph Levin, developed a library of subroutines, including double precision and floating-point operations, and some of the pioneering software such as "interpretive" programs. Joseph Wegstein devised a simple assembly language for SEAC in the early fifties. He chaired the international committee that developed the ALGOL language and was the first chairman of the committee that later developed the COBOL language.

Early work on management information systems included studies for the Social Security Administration, the Treasury Department's savings bond division, and the Navy's Bureau of Supplies and Accounts. In the mid-fifties there were investigations of common processing problems such as sorting and file management. Ida Rhodes developed a combined sorting and file-merging program using push-down store techniques. For the Public Housing Administration a method of generating reports from raw data, based on automatic error-detection and logical-consistency checking techniques, was developed.

Work on pattern recognition, pictorial data processing, chemical structure searching, and natural language processing also began in the mid-fifties. It gained impetus when, upon recommendation of the Bush Committee, a collaborative research program with the U.S. Patent Office was established in November 1954. Element-by-element chemical structure search (Ray and Kirsch) and HAYSTAC, a searching system capable of multiple tracings of variable constituents of nodes of chemical structure representations (Marden), were demonstrated before 1958.

Interest in optical character recognition (OCR) began as an important possibility for computer input. NBS supported work on a demonstration model of an electronic version of Rabinow's Diamond Ordnance Fuse Laboratory "First Reader" and surveyed OCR possibilities for the Social Security Administration, the Rome Air Development Center (RADC), and the Army Signal Corps, among others. Participation in OCR standardization efforts of the United States of America Standards Institute (then the American Standards Association) also began in the formative years of these efforts.

The RADC project involved general problems of pattern recognition for which a programmable scanner (Kirsch, Cahn, Ray, and Urban, 1957) was developed. It was applied to studies of local operations on pictorial data (Thomas), to continuing analyses of biological tissue data (Kirsch) and of micrographs of metallurgical specimens (Moore).

Kirsch and Rankin in 1955 began taking a linguistic approach to pictorial data-processing, now continued by Rankin. The fifties were also notable for work on analysis of electrocardiograms in cooperation with the Veterans Administration (Marden) and traffic simulation projects (Stark).

Among NBS contributions to natural language processing in connection with information selection, storage and retrieval systems were the development of a small-scale question-answering or fact-retrieval system (Stevens), the development of a KWIC-indexing system (Stevens), and experiments in automatic classification or indexing (Stevens and Urban).

DYSEAC, designed in 1951-54 by Leiner, Notz, Smith, and Weinberger, was interconnected to SEAC to bring about a time-sharing, multiprogramming and multiprocessor capability. This mobile computer was used in conjunction with radar scanning and on-line display equipment to demonstrate air traffic control applications, and the interconnection of SEAC and DYSEAC was made in April 1954 in order to demonstrate multiprocessing.

Ever since 1950, when the Bureau first was asked to advise the Post Office Department on automation, NBS has cooperated in the effort to provide increasingly sophisticated tools for carrying on this mammoth operation. The Bureau's advice and assistance has covered the overall improvement of mail-handling, including mail-sorting codes and the design and selection of equipment ranging from keyboards and automatic mail sorters to automated post offices.

After the Bureau acquired an IBM 704 in 1957, SEAC continued to be used solely as a research facility for equipment and programs until its retirement in April 1964. The 704, later followed by a 7090, a 7094, and a UNIVAC 1108, served not only NBS needs but also those of other agencies which either did not have computers or were not fully equipped in this area.

With the move toward larger and more complex equipment, NBS computer work took new directions:

- greater emphasis on sophisticated software essential to efficient functioning of larger computers
- broader applications in data processing, complex file manipulation, pictorial data processing, library functions and document handling, large operations research problems, and others

- development of language and other user aids to increase programmers' efficiency
- standardization.



This microfilm rapid reader-copier, designed and built by the Bureau for the National Library of Medicine, is an example of services provided to other government agencies by the NBS Center for Computer Sciences and Technology.

Establishment of the Center

In response to the new responsibilities given it under the Brooks Bill, the National Bureau of Standards brought together several units in 1966 to form the nucleus of a new organization called the Center for Computer Sciences and Technology.

The structure of the CCST reflects the nature of the additional responsibilities given to NBS and the broadened scope of its role in connection with computer operations in government. The underlying theme of that role calls for the CCST to improve the effectiveness and efficiency of the Government's use of computers. To that end emphasis has been focused primarily on standards and on providing technical advice and assistance to other agencies through computer systems design and development and through directed research in critical areas. Other functions include the operation of a computer service center for government and the provision of information on activities and personnel in the computer technology.

The first director of the CCST was Mr. Norman J. Ream; he was followed by the present director, Dr. H. R. J. Grosch.

Recent Activity

During fiscal year 1969 the Center broadened its contributions to computer sciences and technology as employed both in the Federal Government and in the private sector. Among steps taken were:

- Establishment of an official publication medium, the Federal Information Processing Standards Publications (FIPS PUB), for information relating to standards adopted and promulgated under the law. Nine FIPS PUBS were developed in fiscal 1969.
- Implementation of the Federal Standard Code for Information Interchange and related media standards, through detailed instructions issued by Secretary of Commerce Maurice H. Stans to heads of Federal departments and agencies. The Code defines a set of 128 characters commonly used in information processing and communications. Among other things, instructions are provided for new installations, replacements of computers, augmentation of existing configurations, interchange situations, and ADP/telecommunication interfaces.
- Development of a new unrecorded reference tape standard, in cooperation with an NBS research associate sponsored by the International Business Machines Corporation. This work on amplitude reference magnetic computer tapes was undertaken at the request of both national and international magnetic-tape

standards bodies. The General Services Administration will use the resulting secondary tape standards for calibration of qualification test equipment.

- Launching of a program, in cooperation with the industry, to develop a standard unrecorded magnetic reference surface for ready interchange of magnetic disc packs among equipments of different makes and models.
- Establishment of a new Federal Information Processing Standards Coordinating and Advisory Committee, with members representing the Bureau of the Budget, General Services Administration, Department of Defense, Atomic Energy Commission, and National Aeronautics and Space Administration. The new committee works through five inter-agency task groups on Transition to Federal ADP Standards.
- Federal adoption of a voluntary commercial standard for the COBOL programming language, approved and published—with the active participation of CCST staff—by the United States of America Standards Institute (now American National Standards Institute). This will facilitate the interchange of computer applications, programmed in COBOL, among Federal agencies. Work is continuing on the development of evaluation and validation routines, a necessary adjunct to Federal adoption of the commercial COBOL standard.
- Intensification of measurements automation research and development, emphasizing functional modularity and flexible stored-program control—concepts which not only help to serve the needs of the Bureau's own laboratories but also appear with increasing frequency in commercially available instruments for making physical and electrical measurements and collecting, recording, and processing experimental data.
- Technical assistance in cost-saving, efficiency-promoting operations such as the application of digital computers and ADP technology to geophysical data-processing, fingerprint processing and identification systems, computer-controlled calibration of jet-fuel engine pumps, and development of a general microscope input for a computer (used at the National Institutes of Health).
- Development of "anticipation techniques" and appraisal of factors influencing optimum design of man-machine systems, as part of a point study in cooperation with the National Aeronautics and Space Administration's Electronic Research Center. The new techniques are being applied in continuing experimental activity involving display and time-sharing equipment in NBS laboratories.

- Development and study of hardware and software techniques for interconnecting various computer processors and peripheral equipment. The NBS-designed-and-constructed MAGIC II display terminal has been interfaced to the MOBIDIC B Time-Shared Computer Research Facility and several smaller devices have been attached to MAGIC II and MOBIDIC.
- Implementation of a general-purpose system for storing data and programs on disc files within the NBS CORD Time-Sharing System. The system employs novel techniques for improving access to files stored on disc and for improving the reliability of file storage.
- Utilization of the NBS CORD Time-Sharing System and the new disc file storage mechanism to help implement an information storage and retrieval system for data describing a specialized collection of documents in the on-line systems and computer display areas. Besides being a useful tool to support the research program, this retrieval system is being used to study tradeoffs between manual and automatic search of a specialized data base with respect to the efficient use of manpower and equipment.
- Implementation, within the NBS CORD Time-Sharing System, of a conversational version of a language developed at Bell Telephone Laboratories for efficient list-processing. This system, designated CL6, incorporates compilation, execution, and debugging facilities in a flexible manner within a highly interactive supervisory system.
- Development of a graphical assembler for generating display processor code on the larger time-sharing system, and implementation of a graphical debugging system to minimize the time necessary for obtaining operational programs for display applications, taking into account the advantages of using the graphical display during the debugging process.
- Further development of a class of program generators that will enable an instructor to produce a Computer Assisted Instruction (CAI) course as the result of an interactive process; and continuation of studies on the generation of "generators" in order to explore the possibility of using this approach to produce computer programs of all types.
- Investigation of a programming language that (1) is a dialect of a standard programming language, hence uses an existing compiler; (2) permits content addressing, eliminating the need of a user to be concerned with the mechanics of search or indexing, and (3) permits dynamic creation of files for intermediate output or cross-reference indexes.

- Application of two-dimensional linguistic techniques—used earlier by NBS for development of grammars from Chinese ideograms—to the analysis of other means of communication such as chemical-structure diagrams. Sponsored by the National Institutes of Health, the research is expected to extend the linguistics discipline and provide easier means of interfacing with a computer in the area of graphics.
- Experimentation with an information retrieval system for pharmacological data, designed for the National Institutes of Health, with special consideration given to the construction of numerous special-purpose files containing a variety of information, including chemical structures. Such a system would help scientists to draw significant correlations of data for determining the effects of chemical substances on the body.
- Expanded design work for management and intelligence information systems applicable to tasks performed by the Agricultural Research Service, the Public Health Service, and law enforcement agencies.
- Increased use of digital computers for automating experimental and research work in NBS materials and basic standards laboratories. Computer-controlled data acquisition and control of experiments are now being widely applied to improve experimental accuracy and reproducibility, reduce errors and repetitive labor, more tightly control parameters, permit very high data rates and very lengthy experiment time, and increase experiment throughput.
- Collaboration with technical societies and professional organizations to develop improved means for exchanging information recorded on magnetic tape, with emphasis on arrangements for sharing the task of descriptive cataloging and recording data elements in advance of the exchange of tapes.
- Drafting of a proposed plan for preparing a roster of sources of computer programs and documentation, and publication of a report embodying a "Recommendation for Formats for Computer Program Catalogs," based on an evaluation of existing formats and the information that should be available about a program in order to decide on its applicability to the solution of a specific problem.
- Initiation of a research project for extending the classification scheme for computer information. This is being carried on with the advice and support of the American Patent Law Association's subcommittee on computer information classification, in conjunction with the Association for Computing Machinery.
- Testing and evaluation of a simplified single-fingerprint identification system designed to allow the computer code for a finger-

print to be easily written and sent to any central computer for a report on the probable identity of a suspect, amnesia victim, or corpse.

- Experimentation under the CCST's natural language processing program for purposes of improving information selection, storage, and retrieval systems; developing and using automatic classification and indexing techniques; applying selective recall techniques to both associational files and natural language texts; and developing on-line search strategies for text processing.
- Completion of selective literature reviews on Information Acquisition Sensing and Input, Information Processing Storage and Output, Overall Systems Design Consideration, and Privacy, Confidentiality and Security.
- Provision of data-processing services during fiscal 1969 to 60 organizations including 10 executive departments, the Executive Office of the President, and 15 agencies of the Government, the Congress, and State and local governments, quasi-government organizations, and universities. Assistance in evaluating and selecting automatic data-processing resources was given to the U.S. Civil Service Commission, U.S. Army TACFIRE headquarters, Commerce Department officials concerned with automated personnel systems, the Equal Employment Opportunity Commission, and the Harry Diamond Laboratories.
- Development of operating software to handle remote access from keyboard terminals, expected to permit remote job and data entry from Model 33 or Model 35 Teletypes and to make possible future support of such applications as text editing, remote file inquiry, and conversational compilers.
- Installation of a "Graphical Display System" software package, developed at the University of California, allowing the user to generate a graphical display from his source program by using a standard set of simple specifications. The display can then be directed, by simple control card changes, to any of the available display devices.

INSTITUTE FOR BASIC STANDARDS

The primary responsibility of the Institute for Basic Standards (IBS) is to provide the central basis for a complete and consistent national system of physical measurement, coordinating that system with those of other nations, and furnishing the essential services leading to accurate and uniform physical measurements throughout the United States. The IBS staff of 900 includes 528 scientists and engineers, 200 at the Ph.D. level, in facilities at Gaithersburg, Md., and Boulder, Colo. In addition to these primary locations, specialized activities are carried out at Fort Collins, Colorado; Arcata, California; Maui, Hawaii; and an abandoned gold mine in the Colorado Rockies. The broad nature of the IBS responsibility requires work at many levels of technical sophistication; however, the critical contributions in measurement generally lie at the frontiers of science and engineering.

PHYSICAL QUANTITIES

The international system of measurement is based upon six quantities: mass, length, time, temperature, electric current, and luminous intensity. All other physical quantities can be derived from these six. The United States and most of the other nations base their local systems upon these, but since it is impractical to make all measurements directly in terms of the base units, standards for the other physical quantities have been developed which are consistent with them.

In a high technology society, the needs for better standards and means of measurement—more precise, stable, and easily disseminated—increase continually. Some of the current research in this area is described below.

International Base Units

Length

Dynamic Distortion of Laser Rods Using Holography—Holographic interferometry methods, and high speed photography have been combined to investigate the distortions of solid state laser rods

caused by optical pumping, lasing and subsequent cooling. In this technique, the optical wavefront generated by passing continuous laser light through a passive rod is recorded in a hologram. If the wavefront then changes, interference fringes are produced by the hologram which interferes the recorded wavefront with the new one. Distortions of the active rod create rapidly moving fringes which are photographed using a high speed camera. The films thus obtained can be evaluated at a much slower speed and analysis of the distortion is readily made. Ruby rods have been tested using this method and a convex distortion has been observed at several pumping levels. The information obtained by this method will make possible the manufacture of laser rods which are optically flat in the active state.

Measurement of the Internal Diameter of Small Holes—The development of a new system for measuring internal diameters of small bore tubing utilizing capacitance techniques constitutes a significant advance in metrology, as well as contributing directly to the accuracy of the NBS gas thermometry program. The technique permits measurements at any position inside a tube up to 1 meter from its end, and can be used for tubes having diameters as small as 0.5 mm to an accuracy of approximately $\frac{1}{4} \mu$ in diameter. It has been used to measure the "dead space" that is part of the gas thermometer volume.

Saturated Absorption by Neon Inside a 6328 Å Laser—The stability and reproducibility of a 6328 Å He-Ne laser locked to the saturated absorption peak of a pure neon discharge inside the cavity are being studied. In the He-Ne: Ne system however the pressure induced shift in the gain curve results in the situation where the absorption peak (due to the low pressure cell) does not occur at the maximum of the tuning curve. The frequency output of this laser is therefore expected to be sensitive to excitation conditions. It has been found that by utilizing the isotope Ne²² in the absorption cell and a mixture of about 8 percent Ne²⁰ and 92 percent Ne²² as the neon component of the gain cell the maximum of the gain curve can be shifted to the same frequency as that of the absorption peak. This is expected to increase greatly its usefulness as a wave-length standard.

High-Accuracy Laser—Since the invention of lasers, it has been assumed that they would eventually lead to an accurate standard of length. This has now been accomplished by stabilizing a 3.39 μ m helium-neon laser to the center of a methane infrared transition. The method used is known as "saturated absorption." The laser beam passes through a cell containing methane vapor at low pressure, and it partially saturates the methane transition so that the absorption coefficient is reduced. If the laser beam is tuned even one part in a billion away from the methane transition frequency, the saturation is substantially less and a greater amount of the laser light is absorbed. The resulting narrow peak in the transmitted laser light is used to

automatically keep the laser adjusted to the proper frequency. The shift with methane pressure has been shown to be very small. Two independent devices have been built, and their frequencies agreed to one part in a hundred billion. Such highly stabilized lasers may be used as the basis for a new definition of the meter which would be over 300 times more accurate than the present definition.

Time and Frequency

World-Wide Standard Frequency and Time Signal System—NBS has traditionally provided strong support to the International Radio Consultative Committee (CCIR). This organization's Study Group VII (currently chaired by a senior NBS scientist) met in Boulder, Colo., in July of 1968 to discuss standard frequency and time signals. At the suggestion of the United States delegation, an international working party was set up to assure more active coordination on the important question of improving the worldwide coordinate time system, UTC. Reports initiated chiefly by the United States delegation led to international recognition of the conceptual distinction between time interval measurement (metric time) and coordinate time systems and intervals. There was also recognition of the challenge posed by space and aircraft navigators who need a widespread synchornized timing system of high precision.

Advances in Satellite Timing Techniques—New experimental statelite time dissemination experiments were conducted. The experiments utilized a synchronous satellite, positioned over the Galapagos Islands, designed to cover the Americas as well as some ocean regions. Primary design considerations were economy, simplicity, and versatility. A "listen only" technique was employed, eliminating the need for transmitting capabilities at user stations. In the first phase of the experiment, the basic timing format was tested; results indicate that the user can easily obtain an accuracy of 100 microseconds and that with more care 10 microseconds may be obtained. Voice announcements will be added at a later date.

TV Time-Frequency Dissemination Studies—The use of television to disseminate time and frequency has been investigated. An initial study made use of a local TV station to synchronize the NBS WWV master clock at Fort Collins, Colo., with the NBS time scale maintained at Boulder, Colo. TV synchronization pulses were monitored and the difference in their arrival time at the two locations was noted. With this information and accurate knowledge of the difference in propagation delay time between the two locations and the TV station, the time difference between the two clocks was obtained. Recent work between Boulder and Cheyenne, Wyoming, indicates that this method can be used to synchronize clocks to a few tenths of a microsecond. Besides its use in clock synchronization, television is a valuable

source of frequency information. Measurements made at Boulder on a 3.58 MHz color subcarrier originating in New York City have demonstrated that precision of a few parts in 10^{11} may be obtained for 15-minute averaging times. Since the major networks use rubidium frequency standards in the generation of the color subcarriers, it appears that there is very little degradation of the frequency information over the 4000-mile microwave path between Boulder and New York. An important aspect of these results is that the accuracies are orders of magnitude better than can be obtained with the WWV high-frequency broadcasts and yet they can be obtained essentially for the price of a TV set.

Improved Stability and Accuracy of the NBS Frequency Standard—As a result of improvements in the stability and reliability of the NBS-III cesium beam frequency standard, a much improved calibration precision has been obtained for measurement periods from several hours to several weeks. A relative fractional precision was found to be 1.1 parts in 10^{13} for σ ($N=2$, $\tau=1$ day, $T=7$ days), based on two months of evaluation of NBS-III versus a commercial cesium beam frequency standard. An accuracy of 1.5 parts in 10^{12} (3σ) is tentatively assigned to the improved NBS-III, based upon the most recent evaluation.

Improved Timing Using LF and VLF—The usefulness of low frequency and very low frequency broadcasts has been improved by a careful analysis of phase propagation noise. Negative correlation of phase fluctuations was found between WWVL (20 kHz, Fort Collins, Colo.) and WWVB (60 kHz, Fort Collins) as monitored in California. Taking advantage of the negative correlation gave a factor-of-three improvement in timing. A similar improvement was obtained in a study of NSS (21.4 kHz, Maryland) and WWVL (20 kHz, Colorado). In this case a positive correlation of the phase fluctuations was found. In addition, the spectral densities of phase fluctuations of the NSS-WWVL paths and of the two driving oscillators were determined. The corresponding optimum filter was applied to the data; when used simultaneously with the positive correlation information, a factor-of-ten improvement in timing was achieved.

Completion of NBS-X4 Cesium Beam Tube—Construction of the NBS-X4 beam tube has been completed. Preliminary measurements indicate that the design goal of a figure of merit of 100 has been realized. (Commercial cesium beams have a figure of merit around 1-4. NBS-III currently operates in the range 5-10.) The beam tube was jointly constructed by NBS and the Hewlett-Packard Company, and will be combined with NBS-built electronics to form a much improved NBS frequency standard with a stability ten times greater than NBS-III.

Time Scale Coordination with U.S. Naval Observatory—On about

1 October 1968, the universal coordinated time (UTC) scales maintained at NBS and the U.S. Naval Observatory were in coincidence. At zero hours UTC on that date, the rate of UTC(NBS) was raised 4 parts in 10^{13} and the rate of UTC(USNO) was lowered 4 parts in 10^{13} in order that the divergence of these time scales would be at a much slower rate. Portable clock measurements have indicated time synchronization of the two scales to within ± 5 microseconds, and agreements between USNO and NBS will guarantee that both are maintained within this tolerance. The United States thus has, for the first time, a single coordinated time scale jointly maintained by USNO and NBS.

Temperature

Stability Improvement in Pyrometer Strip Lamps—The major limitation in the calibration of tungsten strip lamps used in optical pyrometry and radiometry is the instability of the lamps themselves. A theoretical model has been developed which explains the major features of these instabilities in terms of the changes that would be expected to occur in the tungsten and lamp envelope due to temperature changes or prolonged heating at one temperature. This model has been used as a guide for the construction and heat treatment of 22 strip lamps. Of these, six exhibit a long-term drift of 0.01°C in 100 hours, which is a factor of ten less than the best lamps previously available; however, short-term temperature cycling of these lamps produces changes varying between 0.05 and 0.1°C . Although it may not be possible to understand and reduce these cycling effects, the lamps will improve significantly the accuracy of high-precision optical pyrometers.

Vapor Pressures of the Neons—The vapor pressures of Ne of natural isotopic composition, ^{20}Ne and ^{22}Ne were determined with respect to the NBS-1955 temperature scale from their triple points to their normal boiling points (about 14.5 to 27 K). The results for natural neon were used by the Comité Consultatif de Thermométrie in its assignment of the neon normal boiling point as a defining mixed temperature of the International Practical Temperature Scale of 1968. The measurements for the pure isotopes are about twice as precise as those for the natural mixture, where complete isotopic equilibrium possibly does not exist. Because of the possible variation in the isotopic composition of natural neon (90.92% ^{20}Ne , 0.257% ^{21}Ne , 8.82% ^{22}Ne) the most abundant pure isotope, ^{20}Ne , is a more practical thermometric standard.

Superconductor Thermometric Fixed-Points—A program whose aims are the extension to lower temperatures of the International Practical Temperature Scale and the development of simple, accurate thermometry techniques has commenced with the examination of the

reproducibility on cycling of the superconduction transition breadths of lead, indium, tin, aluminum, gallium, zinc, and cadmium as measured by magnetic induction. The transition breadths have been compared with the temperature-dependent resistance of doped germanium, and preliminary results indicate reproducibility in the range of a fraction of a μK (both the doped germanium resistivity and the superconducting transitions).

A-C Thermometer Bridge—An improved resistance thermometer bridge has been developed. A frequency of 400 Hz was chosen for the bridge so that optimum performance could be obtained using commercially available operational amplifiers and decade voltage dividers. The bridge resolution is about 20 μK when used to measure a standard 25 Ω platinum thermometer at 0°C with a thermometer current of 0.001 A. The bridge enables one to determine the ratio of an unknown resistor to a standard resistor. The bridge ratio is stable and has such high accuracy that the measurement accuracy is governed by the resolution of the bridge and the accuracy of the standard resistor. To measure the usual standard platinum resistance thermometer on the new bridge, additional connections are required—a pair of coaxial leads, which may be as long as 16 meters without affecting the accuracy. Values of resistance determined by this bridge agree with values determined by a high quality DC bridge within the accuracy of the measurements. However, by contrast with DC bridges based on resistor networks, the ratios are stable without thermostating and do not drift. No lead reversals or auxiliary balances are required with this system, so that continuous recording of resistance versus time may readily be made.

High Temperature Thermocouples—The development (supported by NASA) of reliable high temperature thermocouple probes, which are being sought for use in the design of nuclear reactors and thermionic devices, requires an understanding of the thermocouple behavior at very high temperatures in carefully controlled environments. Facilities were constructed for investigating performance of doped W-3 percent Re and undoped W-25 percent Re thermocouple wires at temperatures of 2400 K and above when the thermocouple wire is subjected to high vacuum, argon and hydrogen environments. The changes in calibration of W-3 percent Re versus W-25 percent Re thermocouples were studied as a function of exposure time in these environments for time periods of 1 to 1000 hours. In addition, metallographic and chemical analyses were performed to characterize material behavior.

The results in vacuum confirm previous suppositions that evaporative loss of rhenium from the alloys at temperatures above 2200 K prevents their use as stable thermocouple elements. Stability with time in the carefully characterized argon and hydrogen environments was confirmed.

One of the more interesting by-products of this work was the discovery of extreme ductibility in the recrystallized doped alloy after heating in the vacuum environment.

New Temperature Scale—NBS is calibrating thermometers on the International Practical Temperature Scale of 1968, which was adopted by the International Committee on Weights and Measures at its meeting in 1968. The Scale replaces the International Practical Temperature Scale of 1948 (amended edition of 1960).

Electric Current

Gyromagnetic Ratio of Protons in Water-Surveillance of the NBS Ampere—The precession frequency of protons in a magnetic field provides a convenient reference for the electrical standards; however, fluctuations in the earth's magnetic field are a source of interference. A self oscillating rubidium magnetometer was set up at a distance from the proton sample, and phase-locked to a constant frequency source. The derived correction signal was used to maintain one component of earth's magnetic field essentially constant at both magnetometer and proton sample. Continuous detection of the precession frequency by the method of nuclear induction was found to result in improved convenience and reliability. By using the method of nuclear induction and compensation for changes in earth's magnetic field, the center of the proton resonance line can be located with an accuracy of about 1 part in 10^7 .

Fundamental Physical Constants

Measurement of Optical Frequencies—Modulation of a helium-neon laser with a 10 GHz microwave frequency has produced sidebands, $\nu \pm \omega$ (where ν is the laser frequency and ω is the microwave modulation frequency) yielding about 10^{12} photons per second in each sideband for a 1 watt microwave excitation. With the addition of an optical spatial filter, a Fabry-Perot cavity and servo systems for locking the laser frequency to the Fabry-Perot and the Fabry-Perot to the microwave frequency, the laser frequency has been locked to the microwave frequency with a precision, $\delta\omega/\omega$, of about 1 part in 10^8 . It is hoped that the continuation of these experiments will yield the velocity of light to the accuracy approaching 1 part in 10^8 (known at present to 1 part in 10^6) and that of light frequencies to 1 part in 10^9 . The latter can be utilized in problems of standardization of length.

Mechanical Quantities

Microphone Calibration at Infrasonic Frequencies—As a result of research done at NBS over a period of several years, it is now possible to calibrate laboratory standard microphones at frequencies down to

about 1 Hz with good accuracy, and a calibration service will be offered. This service should be valuable to those concerned with the measurement of low frequency noises, of which the sonic boom is a prime example.

Precise Measurement in the Medium Vacuum Range—Accurate measurements in the medium vacuum range provide a firm basis for extension of measurement reliability into the higher vacuum ranges. These also satisfy a number of technical and scientific needs such as calibration of precision air data computers for high altitude flight and for atomic collision cross-section studies. Ultrastable vacuum environments and a precision oil manometer allowed absolute measurements with an uncertainty of 1.2×10^{-4} torr + 6 parts in 10^5 of the reading for the range 1×10^{-4} to several torr. These procedures have now been extended with precision mercury columns as reference for inter-comparison with the oil manometer and for calibration of certain transfer gages. Calibration uncertainty in the higher range now appears to be about 4×10^{-3} torr + 4 parts in 10^5 of the reading to 25 torr.

Hydraulic Research in the United States, 1968—A new compilation, summarizing hydraulic research being conducted in university, government, and in industrial laboratories in the United States and Canada was issued as NBS Special Publication 316. This issue summarizes 1,690 research projects reported by 212 laboratories. Compilations of this nature are very useful in promoting contacts among researchers in similar areas and in preventing costly duplication of effort.

Self-Calibration Procedure for the High Vacuum Range—A major difficulty in measurements at high vacuums is the lack of a convenient method for in situ self-calibration of working gages and of a suitable stable-transfer procedure. Fixed-point processes would be helpful. A method has been under study utilizing the dissociation of metallic nitrides. Results for barium nitride show dissociation pressures ranging from 2×10^{-5} torr at a temperature of 740 K to 0.7 torr at 1150 K. The slope of the $\ln p$ versus $1/T$ plot corresponds to an enthalpy of reaction of 175 kJ/mol N_2 , which is consistent with dissociation into the subnitride, Ba_2N , and nitrogen gas. The rate of approach to steady-state pressure is approximately logarithmic with steady values reached in time of about one hour. The steady-state values of pressures are dependent upon the direction of approach in pressure with the $\ln p$ versus $1/T$ plot parallel but slightly displaced. Pressures are reproducible at present to within 10 percent. Present results suggest that such a technique may have application in vacuum calibration. Work toward improved precision and extension to lower pressures is in progress.

Automated Vibration Calibration Facility—A facility capable of calibrating piezoelectric vibration pickups automatically has been

developed. It incorporates a small digital computer which controls the measurement in addition to acquiring the data. The facility makes it possible to collect considerably more data points than before, and to perform statistical comparison of the data with the previous calibration history of the pickup. The calibration at three times the number of frequencies can be done in one-third of the time needed before.

Improved Transfer Standard Accelerometer—An improved transfer standard accelerometer has been developed for the calibration of working vibration pickups in or near field locations. These units will improve field calibrations by permitting the examination of the several motional components to which the pickups are subjected, thereby avoiding a common source of error.

The transfer standard is unique in two respects relative to commercial units: (1) Instead of a metal housing, it has a ceramic housing which provides high stiffness, strength, electrical isolation, and substantial weight reduction. (2) Rather than a single pickup for each acceleration axis, the new unit has three reference pickups mounted symmetrically about the vertical axis. They make possible a reliable monitoring of relative motion with respect to phase and amplitude of points over the shaker table surface. Other design features help to reduce loading effects and to produce a high resonant frequency, thereby increasing the usefulness of the device. The range of operation is 10 Hz to 12 kHz at acceleration levels to 100 g and with loads to 50 g.

Strain Measuring Standard for High-Temperature Use—An extensometer for use as a laboratory strain standard which employs a gas-cooled variable capacitor was designed and constructed for the U.S. Air Force Systems Command. Tests showed it to have a resolution of 0.3 μm length change over a range of more than 2mm and to operate to at least 1400 °C in a normal atmosphere. A knowledge of structural strength at elevated temperatures is of urgent importance in the development of gas turbines, rocket engines, missile and aircraft frames exposed to aerodynamic heating, and components of nuclear reactors.

Misalignment Detector for Axial Loading Fatigue Machines—The alinement of the grips of an axial-load fatigue machine has a marked influence on the fatigue life of specimens tested in it. It is not possible to measure these misalignments by ordinary means of linear measurement. To accomplish the purpose, a compliant element equipped with strain gages has been developed which is attached to the machine heads in parallel with the test specimen. As the specimen is cycled slowly through its loading excursion, the detector senses the minute deflections due to undesirable lateral forces. If these lateral deflections are found to be within allowable limits, the detector may be removed and the specimen tested in the normal manner.

Internal Wave Generation—Under sponsorship of the Office of Naval Research, a study was made of the internal waves created by the steady horizontal motion of a sphere through a liquid having a uniform rate of density increase with depth. A new theory was developed for this three-dimensional problem which closely approximates the experimental results. Most of the previous work in this area has been restricted to analysis in only two dimensions. Wooden spheres, having radii from 2.4 to 4 cm, were towed to speeds up to 5 cm per second through fluids having total density differences of 0.9 to 6.7 percent over 50 cm of depth. This permitted a wide variation in flow parameters without excessive turbulent wake formation and mixing. The vertical fluid movements, which were only a few millimeters, were measured by a wire probe.

In addition to contributing to the understanding of internal wave generation in the oceans, the new theory will be useful to meteorologists studying the generation and effects of lee waves behind mountains. These are of particular concern to the aviation community, contributing to the dangerous phenomenon known as clear air turbulence (CAT).

Small-Scale Structure of Turbulence—In collaboration with the Naval Ship Research and Development Center, and with the partial support of the Atomic Energy Commission, an experimental investigation of the small-scale structure of a turbulent field was carried out by extending the methods of digital analysis to the measurement of turbulent velocity derivatives. The statistical behavior of these derivatives was studied in the nearly isotropic decaying turbulence field generated by a 1-inch square mesh grid in the NBS 4½-foot wind tunnel. Higher-order correlations for velocity gradients up to the eighth order, at two stages of decay, and for a limited change in Reynolds numbers have been determined. Markedly different from the turbulent velocities, the higher-even-order correlations of velocity gradients clearly evidence the departure from a two-dimensional Gaussian probability distribution, and more clearly exhibit similarity with a characteristic length or time scale. The results require that current views as to the intermittent character of the small-scale structure, at least for the Reynolds number range of the present investigation, have to be carefully evaluated. It is expected that investigations of this type, under laboratory-controlled conditions, can play an important role in the understanding of the turbulence structure and provide insight into closely related problems associated, for example, with atmospheric turbulence.

Heated-Air Adiabatic Saturation Psychrometer—A humidity-measuring instrument, based on the heated-air adiabatic saturation principle, and suitable for use in both laboratory and field, has been designed and constructed. With it, the moisture content of atmospheric

air can be measured over a mixing ratio range of 0 to 50 g of water vapor per kilogram of dry air; in terms of dew point, this range is -40 to $+40$ °C. When compared with the NBS two-pressure humidity generator over a mixing ratio range of about 2 to 19 g/kg, the mean difference was 0.046 g/kg and the maximum difference was 0.12 g/kg; the corresponding differences in dew point were 0.08 °C and 0.30 °C, respectively. The instrument is portable and self-contained, requires a 110-volt, 60-hertz, 2.5-ampere power source, and a barometer. It samples test gas at a gas-flow rate of 4 liters per minute.

Electrical Quantities—D.C. and Low Frequency

Current Transformer Calibration—A compact and versatile measurement system for calibrating current transformers at power frequencies has been developed. The system based on the current comparator principle, permits calibration of all ratios normally encountered (to a maximum of about 12000/5), and features the capability of testing transformers at operating currents up to four-times-rated. It provides those laboratories concerned with power and energy measurements with a method for calibrating their current transformers to improved accuracies with minimum recourse to the NBS facility. For transformer ratios up to 1200/5, the measurement accuracy is within 5 ppm for both the ratio factor and phase angle. Calibrations at ratios greater than 1200/5 are limited by the NBS calibration accuracy of standard transformers, but can be typically made to 50 ppm or better.

High Voltage Capacitance Bridge—A high-voltage capacitance bridge based on the current comparator principle has been constructed. In this bridge a three-winding current transformer or current comparator forms the ratio arms, offering unequaled accuracy, stability, and sensitivity. In contrast with conventional high-voltage bridges, the dissipated power is very small and does not affect the accuracy when capacitors of high volt-ampere rating are measured. With this bridge, it is possible to calibrate a broad range of capacitors: from 100 pF to several thousand μ F. The voltage range of the bridge is from 100 to 200,000 V, the upper limit depending only on the availability of a high-voltage capacitor. The direct reading accuracy with which two capacitors can be compared is within 2 ppm for small ratios and within 10 ppm for the largest ratios ($2 \times 10^6:1$). The instrument is designed to calibrate those items which cannot be accommodated on existing NBS low-voltage bridges: high-voltage capacitors and power factor standards used by the manufacturers of high-voltage equipment.

International Comparison of Capacitance Standards—Another comparison circuit of three 10 pF fused silica capacitors has been completed, this time at laboratories in the Union of South Africa,

France, and at the International Bureau of Weights and Measures. Measurements made at NBS show that the capacitors continue to remain stable to about one part in 10^7 .

Improved Techniques for Audio Frequency Admittance Measurements—A number of bridges suitable for comparing four-terminal-pair admittance standards, including a so-called quad bridge for comparing resistance with capacitive reactance has been developed. Many simultaneous null conditions must be realized with these bridges, but suitable techniques have been developed for achieving the required conditions quickly. A reevaluation of the defining conditions for four-pair standards, and the development of techniques for assessing the errors due to insufficient suppression of ground currents with coaxial chokes, have made it possible to reduce the measurement errors below one part in 10^9 .

New Base of Reference for the Volt Adopted—On January 1, 1969, the United States, along with eight other countries, on recommendations of the International Committee on Weights and Measures, adopted a new base of reference for the volt. In the United States the new base differs from the old base, used prior to January 1, 1969, by 8.4 parts per million. This means that the electromotive force (emf) obtained for a standard cell is now higher than that obtained on the old base of reference by a factor of 1.0000084. The new value represents a better measurement of voltage in terms of the theoretical unit of emf derived from the basic mechanical units of length (meter), mass (kilogram), and time (second). This change came about as a result of absolute measurements made on the ohm and ampere since January 1, 1948, when the International Committee on Weights and Measures recommended a conversion from the "international" electrical units to the "absolute" system.

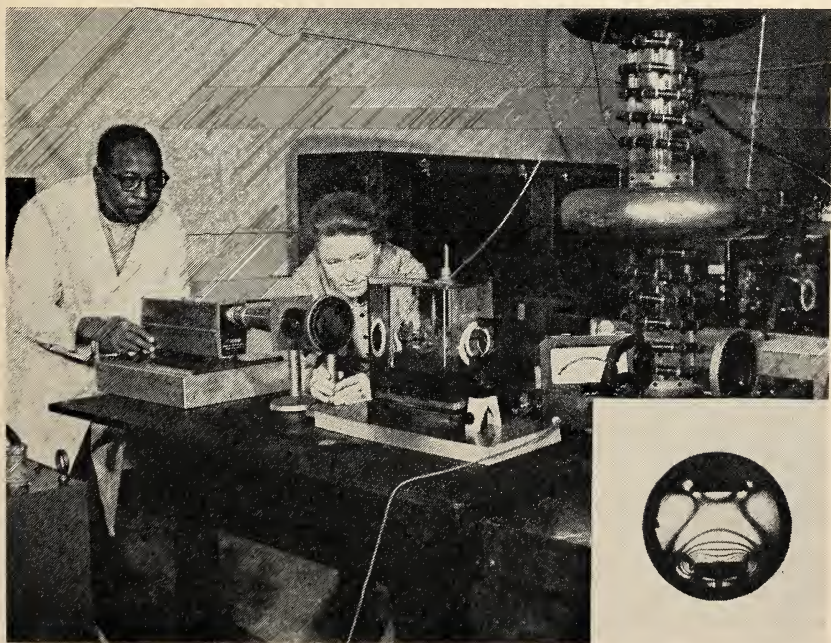
Comparisons of the Unit of Electromotive Force at BIPM, NRC, and NPL—International comparisons of the unit of electromotive force are made approximately every three years by the Bureau International des Poids et Mesures (BIPM). Each country participating in the experiment sends a group of saturated standard cells to BIPM, and on the basis of measurements made at each national laboratory and BIPM, differences between the units of emf maintained by the various countries are determined. This comparison usually requires 6 to 8 months to complete. In cooperation with BIPM, the National Research Council of Canada, and the National Physical Laboratory of Great Britain, an experiment was conducted in which a group of saturated standard cells in a temperature-controlled enclosure was shipped to the various laboratories. A complete experiment between NBS and the participating laboratory required only 3 to 6 weeks; the two intercomparisons between NBS and BIPM showed excellent agreement between the "rapid" and conventional methods. The means

of the two rapid intercomparisons differed by 0.2 ppm from the conventional method. Additionally, this excellent agreement serves to verify the adjustments made to the NBS unit on January 1, 1969.

Inductive Voltage Divider—Aerospace industries require high accuracy in the voltage ratio standards used for calibration of guidance and control equipment. Commercially available inductive voltage dividers suffer from several limitations, the most serious being the nonindependence of the corrections to one decade with respect to the setting of the others. Several single-decade inductive voltage dividers have been designed, constructed, and cascaded. The results show that it is entirely feasible to construct multidecade dividers having overall errors two orders of magnitude smaller than the presently available ones. The single-decade units utilize the multistage voltage transformer principle and an impedance compensating network, and show output-to-input voltage ratio errors of approximately 1 part in 10^9 .

Precise EMF's from the Microwave Josephson Effect—Instrumentation has been developed for the precise comparison of standard cell voltages with the emf's produced by microwave Josephson junctions operating at a temperature of two kelvins. These junctions act as very precise frequency-to-voltage converters making possible the translation of standards of radio frequency into independent, nonelectrochemical sources of d-c electromotive force.

High-Intensity Electric Field Measurements—An electro-optical technique which utilizes laser light for analysis of high-intensity



The electromagnetic field within a Kerr cell is rendered visible through use of polarized laser light. A typical fringe pattern is shown in the insert.

electrostatic fields has been developed and used for the measurement of distorted fields obtained when high direct voltages (to 90 kV) are applied to nitrobenzene-filled Kerr cells. The field can be mapped from fringes produced by the Kerr effect. This technique, which was developed to permit calibrations of Kerr pulse-measuring systems, offers several advantages over conventional field-mapping techniques, including the following: (1) the two-dimensional visual images, similar to those achieved in photoelastic mechanical stress analysis, permit direct observation of the field distribution, thereby enabling immediate detection of regions of high electrical stress; (2) space-resolved measurements of field strength and potential are obtained from a single photograph; (3) measurement resolution increases with the magnitude of the field; and (4) a laser source allows simplicity in the optical system. It is anticipated that this technique will also be useful in electrical stress and breakdown studies, in high-intensity field-mapping experiments, and in investigating the behavior of many dielectric liquids.

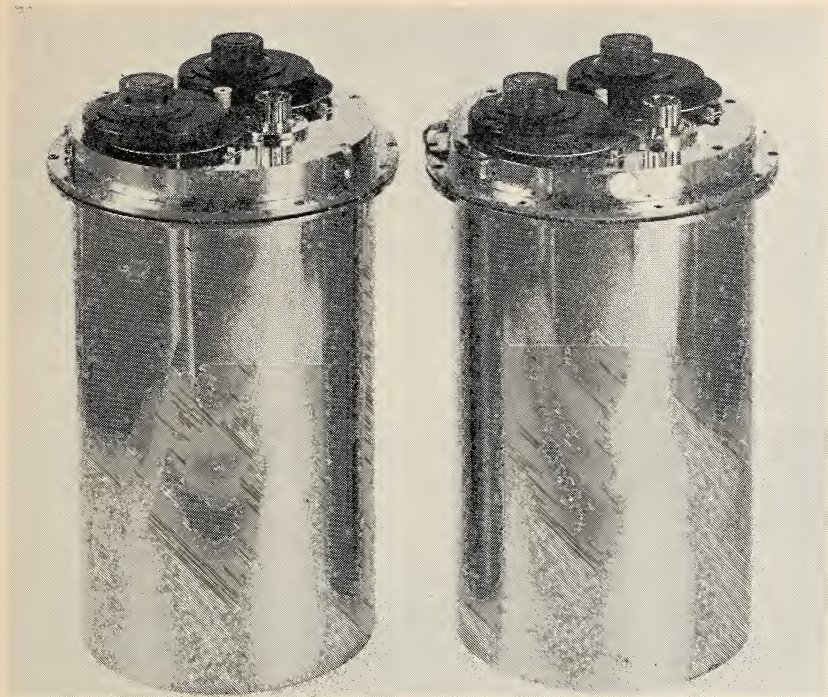
High-Voltage Pulse Measurements System—A system which generates and measures high-voltage pulses (rise-time $\approx 1 \mu\text{s}$, duration $\approx 4 \mu\text{s}$) has been designed and constructed. This system, which received partial support from the Atomic Energy Commission through the Sandia Corporation of Albuquerque, New Mexico, is equipped for time-resolved, simultaneous, voltage divider and Kerr electro-optical measurements of pulses peaking between 5 and 300 kV.

Electrical Quantities—Radio Frequency

High-Frequency Region

Design Data for Impedance Standards—NBS uses quarter-wavelength short-circuited sections of waveguide as reference standards for calibrating microwave impedance standards. These waveguides can be accurately evaluated and have the additional advantage that losses at the place of connection are negligible. In order to make these reference standards more available to other laboratories, design data have now been published. The data consist of a series of computer-calculated curves of return loss versus frequency for each of the standard sizes of rectangular waveguide and coaxial line presently in use. The curves are normalized to a conductivity of 10^7 mhos/meter and can be read to ± 2 percent, which gives sufficient accuracy for many purposes. In case greater accuracy is required, formulas are given for the calculation of return loss. Using these data, any interested laboratory can design and construct accurate impedance standards and improve the accuracy of their impedance measurements.

Primary Standard Coaxial Noise Generators—Two precision, coaxial, thermal noise generators were designed and constructed for use as primary standards of noise power in the high-frequency range.



Precision coaxial thermal noise generators for use as primary noise power standards at frequencies from 3 to 100 MHz.

At present both operate at either 30 MHz or 60 MHz, but they can be modified to operate at any frequency between 3 and 100 MHz. One generator, using a bath of liquid nitrogen, has a noise temperature of approximately 76 K with an uncertainty of 0.10 K. The other, using a bath of heated fluorochemical liquid, has a noise temperature that is adjustable between 300 K and 500 K with an uncertainty of 0.15 K. These standards are used to measure high frequency random noise sources having temperatures between 75 K and 30,000 K.

Baseband Pulse Transmission—Frequency and time domain analyses of normally conducting coaxial cables at low temperatures have been performed. The anomalous and classical skin effects were considered by employing the Reuter and Sondheimer anomalous skin effect theory. Frequency and time domain response versus temperature (4 to 300 K) has been predicted for six miniature commercial coaxial cables and one Nb-Pb superconductive cable maintained in the normal state. Good agreement was obtained with available time domain data, both directly and by application of Fourier transform techniques. The methods developed in this work provide a means for computing transmission characteristics in the presence of temperature gradients along a coaxial cable such as would be encountered between a cryogenic environment

and ambient space. From this information, pulse distortion in the cable can be predicted. Similarly, this technique can be used as a means for pulse shaping to predetermined requirements.

Improved Pulse Power Meters—NBS has been instrumental in the recent commercial development and manufacture of three improved RF pulse power meters. The Bureau's role consisted mainly of pointing out the need to industry and providing technical consultation. The uncertainty limit in all three instruments is 5 percent (0.25 dB) as compared to 10 percent previously. The need for improved accuracy in RF pulse power measurements has resulted from recent changes in DoD and FAA specifications for Airborne Tactical Air Navigation (TACAN) and radar altimeter equipment. These changes were designed to improve the accuracy and reliability of airborne systems vital to the safe operation of military and civilian aircraft. The new meters have been evaluated at NBS and are available for purchase on the open market.

Microwave Region

Development of Microwave Noise Standards—A new mechanism for describing and evaluating performance parameters in microwave systems has been developed. This description is based on "power equations" instead of microwave circuitry. As suggested by the terminology, the fundamental parameter is the net power flowing across the terminal surface; thus, the new technique requires only a single scalar parameter, while providing a systematic and unified approach to the mismatch error problem. It also includes simplified techniques for evaluating mismatch corrections, and because the description is based on "terminal invariant" parameters, the uniform waveguide and precision connector requirement is eliminated for an important class of practical measurement problems.

Development of Millimeter Wave Standards—The conventional microwave approach to component design begins to break down at millimeter wavelengths because of difficulties in achieving precise dimensions and tolerances and because of unacceptable losses and limited power-handling capabilities. It is currently thought that development of standards at these wavelengths will require the use of electronic components based on quasi-optical principles. As part of a new NBS effort in the area, a circular polarizer and a turnstile junction were built and evaluated. Their design was based on an optical principle not previously applied to components at millimeter wavelengths. Successful operation of the components has broadened the base of technology available for the development of millimeter wave standards.

Precision Microwave Power Measuring System—Power is a fundamental quantity in microwave equipment design and testing. Highly accurate measurements can now be made with new d-c substitution techniques using a bolometric detector in a bridge arrangement. This

system is an improvement over the original self-balancing d-c bridge developed at NBS in 1956 and used extensively by standards laboratories throughout the country. The new system will find even wider acceptance because it permits improved accuracy at the field level. In addition, there is a reduction of cost by a factor of seven, and reduction of weight by a factor of twenty. The system, which is completely solid state, also functions as a precision power leveller, a 0–10 V differential voltmeter, and an ultrastable 0–10 V power supply. This combination of features permits the measurement of r-f power levels of the order of 10 mW without accessory slidewire potentiometers or digital voltmeters.

Josephson-Effect Studies—Experiments were undertaken to determine the maximum frequency at which Josephson-effect oscillations could be observed in superconducting point contacts. Earlier work in this field was limited to photon energies below or near the energy gap of the superconductor, since it was generally believed that significantly higher frequencies would not be observable. Quite to the contrary, oscillations were detected at frequencies as high as 8200 GHz—a photon energy of approximately 12 times the energy gap. These results are significant both for the theory of superconductivity and because they indicate that Josephson junctions may be useful as detectors, harmonic generators, mixers and spectrum analyzers in the infrared, where few other coherent devices are available.

Photometric and Radiometric Quantities

Effect of Lowering Size and Brightness on Color Discrimination—The ability to discriminate between colors becomes poorer when the brightness is lowered or the size of the target is decreased. The loss in color discriminability is more marked for blue-yellow differences than for red-green differences. This phenomenon has long been known in color signalling, such as in the railroading and lighthouse services, where it is generally understood that purple, blue and yellow can not safely be used for signal lights that must be identified at a distance. As the distance from the observer to the target is increased, the visual size of the target decreases, and a loss in color discriminability results. A study has been made of the relation between size and brightness for a given loss in discriminability. This study indicates that decreases in size of target can be compensated by increase in brightness. Conversely, low levels of brightness can be compensated by an appropriate increase in target size. The appropriate increases were also determined and were found to be systematic.

Theory of Perceived Size of Color Differences—Because of industrial need of a mathematical basis for setting color tolerances, several approximate measures of perceived size of color differences have been developed empirically. One of these is based on distance on the CIE

1960 UCS diagram, so-called because it was recommended by the International Commission on Illumination (CIE) in 1960 as yielding an approach to uniform chromaticity spacing. Current studies at NBS have shown that this diagram can be derived from the second stage of the Müller theory of vision. This second stage treats normal vision as a combination of protanopic (yellow-blue) vision with tritanopic (red-green) vision. This view implies that the true measure of color difference is not distance on the CIE 1960 UCS diagram, but rather the angles between the protanopic confusion lines on this diagram for amount of yellow-blue difference, and those between tritanopic for red-green. This theoretical measure checks out well for differences between the various colors of the spectrum, and is being checked now for other colors.

The Ideal Lovibond Color System—The Lovibond System of red, yellow, and blue glasses has been used for many years for the grading of transparent materials. The color scales of the system were calibrated in arbitrary units with the three units related in such a way that for daylight illumination a combination of one unit each of the red, yellow, and blue glasses results in an approximately neutral filter. In 1962, ideal units of the three filters were defined and colorimetric coordinates were determined, in accord with the 1931 (International Commission on Illumination) standard observer and coordinate system, for multiples of the unit filters and various two-filter combinations of them. These 1962 determinations were made only for tungsten illumination of the filters (Illuminant A). Calculations of the chromaticity coordinates have now been made of the same combinations of the unit filters, for daylight illumination (Illuminant C). In 1960 and in 1964, the CIE recommended the use of two transformations of the original 1931 coordinate system yielding improved uniformity of spacing. The chromaticity data of the Lovibond System have now been computed for both Illuminants A and C, in terms of both the 1960 (u, v)- and the 1964 (U^*, V^*, W^*)-transformations of the 1931 (X, Y, Z)-coordinate system. These tabulations enable convenient expression of uniform color tolerances in terms of Lovibond glasses.

Spectroradiometric Photometry—A procedure has made it possible for the first time to calibrate with reasonable accuracy the lumen output of a light source of any spectral distribution by means of incandescent standards. The method is based on a comparison of flux from the lamp to be tested with that from an incandescent standard at each wavelength interval of the visible spectrum. A set of 40 W cool-white fluorescent lamps was calibrated with this instrument and made available to the lamp industry through a commercial testing laboratory. An industry-wide intercomparison of 40 W cool-white and daylight fluorescent lamps was set up and preliminary results are now available. Although substantial progress has been made toward con-

sistent spectroradiometric measurements throughout the industry, further work will be necessary before the measurement system can be considered completely satisfactory.

Standard Detectors for the Vacuum Ultraviolet—The investigation of the day-to-day reproducibility of the photoelectron yield of several materials—Au, W, Al_2O_3 on Al, Co, Al—suitable for use as the cathode of a windowless diode has continued. Detectors of this type could serve as standards for wavelengths less than 105 nm, where durable window materials do not exist. Preliminary results indicate that certain materials— Al_2O_3 on Al, Au—under prescribed conditions of measurement and storage, exhibit reproducibilities of photoelectron yield of better than 5 percent. Windowed, evacuated diodes are being investigated for use at wavelengths longer than 105 nm. Early results indicate excellent stability of calibration—with reproducibility better than 5 percent—is possible in this region.

Laser Power Measurements—Using a laser calorimeter with a timed shutter (developed at NBS for pulsed laser energy measurement), techniques have been developed to measure the average power of CW lasers in the wavelength range of 450.0 to 1060.0 nm. CW power-measuring devices may be intercompared with a ± 4.0 percent accuracy by using comparison techniques similar to pulsed energy measurements. Also, a power meter was developed which is capable of measuring the power of a 200 W CW CO_2 laser with an accuracy of ± 4 percent. The laser beam is absorbed in a hollow, blackened copper cone through which water flows at a known rate. The measured temperature rise and the rate of flow give the power of the laser. The range of the meter was estimated to be from 1 W to well over 10 kW; however, NBS at present does not possess lasers with powers to test the full range of the meters.

PHYSICAL PROPERTIES

Approximately half of the activity within the Institute for Basic Standards is related to the measurement of physical properties of well-defined substances. The rapid growth of the physical sciences has provided scientists with many new techniques for measuring “old” physical properties and has made accessible many new ones. With new techniques it has been possible to increase the precision of measurement, provide the scientific community with a greater range of reference data, and characterize certain processes more fully. Examples of these activities, drawn from current work, are given below.

Atomic and Molecular Properties

Radio Line Spectra of OH—During January and February of 1968, precision measurements were made, of the radio line spectra of

two excited states of the OH molecule, at the request of Harvard radio astronomers. Current theories of the excitation conditions of interstellar OH gas involve these two states, and the Harvard astronomers were about to make a sky search for the corresponding line radiations, using the large radio telescope at the National Radio Astronomical Observatory at Greenbank, West Virginia. Provided with laboratory measurements accurate to within 1 ppm, the Harvard scientists tuned their telescope to the correct frequencies and were able to detect faint OH signals in a single night of observation. Without the NBS measurements, the search might have failed or might have required a great deal more telescope time and expense.

Departure from Equilibrium in a Nitrogen Arc—Although previous studies of sources similar to the atmospheric nitrogen arc had not revealed departures from local thermodynamic equilibrium (LTE), the availability of improved experimental techniques made the reexamination of this source attractive. Absolute intensity measurements were made on various atomic and molecular spectroscopic features produced in an atmospheric-pressure nitrogen arc operating at temperatures from 5,000 to 15,000 K. The relationships among the intensities of these spectral features were then compared with the corresponding relationships among the upper-state populations in LTE in order to obtain optical transition probabilities and reveal departures from equilibrium. The data from the N I and N II lines were consistent with the existence of LTE above 11,000 K and recently published transition probabilities. On the other hand, *no* choice of transition probabilities for N⁺₂ or N₂ bands resulted in agreement between the experimental data and LTE calculations below 10,000 K.

Arc Measurement of Some Ar II Optical Transition Probabilities—In recent years, many efforts have been made to measure or calculate transition probabilities and related quantities for the strong lines in the visible spectrum of Ar II. While two recent sets of lifetime measurements show good agreement with each other, results derived from theoretical calculations and emission measurements exhibit disagreement as large as a factor of two. Emission measurements leading to the determination of more than 100 Ar II transition probabilities were made in the hope of reducing the uncertainty attached to some of these values. Measurements were made in a wall-stabilized, atmospheric-pressure argon arc and transition probabilities obtained using the generalized Fowler-Milne technique. Transition probabilities, having an estimated uncertainty of 10 percent for the best cases, for all lines of the $4s-4p$ and $3d-4p$ Ar II arrays lying below 7200 Å. Where comparison with lifetime measurements was possible, the agreement was satisfactory.

Vibration-Rotation Hamiltonian for Triatomic Molecules—Two conventional theoretical formalisms exist for the vibration-rotation

problem in triatomic molecules: one appropriate for linear molecules, having four vibrational and two rotational degrees of freedom; the other appropriate for bent molecules, having three vibrational and three rotational degrees of freedom. In the present work, a single, unified formalism was developed, applicable to both cases. The unified formalism has two vibrational and three rotational degrees of freedom. The remaining degree of freedom, corresponding to the bending vibration, is singled out for special treatment. Among other things, an exact quantum mechanical vibration-rotation Hamiltonian was derived; a slightly modified FG matrix formalism for the stretching vibrations was introduced; and the dependence of the rotational constant on the bending vibrational quantum number was determined. The formalism was applied to experimental data for HCN/DCN , $\text{H}_2\text{O/D}_2\text{O}$, and CsOH/CsOD . It displayed distinct advantages in the HCN/DCN case.

Atomic Spectra—New energy levels have been found for atoms or ions of Tc, W, Cl, Y, La, Pr, Pm, Tm, and Th. These theoretical calculations have aided in the interpretation of many of these new levels, as well as in electron configurations in K, Cu, Zn, Ag, Cd, and Tl. Such analyses of optical spectra are useful for interpreting astronomical and plasma spectra, predicting thermodynamic and chemical properties, and for solid state and atomic physics.

Accurate Reference Wavelengths in the Optical Region—New measurements and application of the Ritz principle have yielded 1,570 improved wavelengths in the thorium spectrum from 2600 Å to 13,000 Å. The uncertainty ranges from 0.0002 Å for the shorter wavelengths to 0.002 Å at the long wavelengths. Similar work gave over 500 wavelengths for krypton 86 (the spectrum containing the primary standard of length), accurate to ± 0.0001 Å at the short wavelengths (3400 Å) and to ± 0.01 Å at the longest wavelengths ($4\mu\text{m}$). A list of almost 2,200 vacuum-ultraviolet wavelengths accurate to ± 0.002 Å or better has been compiled for the region 300–2000 Å. This list contains many lines (belonging to frequently observed spectra) measured at NBS to ± 0.0005 Å.

Torsional and Ring Strain Forces in Molecules—Studies have recently been made to determine the structures of a series of small, ring-shaped molecules: silacyclopentane; 2,5-dihydrothiophene; 2,3-dihydrothiophene; silacyclopentane; and cyclopentene oxide. These molecules are of particular interest to theoretical chemists because the precise shape of the ring is determined by a delicate balance between ring strain forces, which tend to make the ring planar, and torsional forces between hydrogen atoms attached to adjacent carbon atoms. The torsional forces act to pucker the ring into a nonplanar conformation. These two types of forces are also important for a number of large biologically active molecules. Before such large molecules can be under-

stood, however, we must be able to understand small molecules, which are easier to study. By studying certain low-frequency vibrations of these molecules by far infrared or by microwave techniques, the details of the forces controlling the ring structure in these molecules can be determined.

Plane Grating Spectrograph—A new plane grating spectrograph combining speed with high resolution has been constructed. It is being used to photograph spectra of atoms and molecules with resolving powers (greater than 5×10^5) not previously available in grating instruments at NBS. The general optical design is a modification of the two-mirror Czerny-Turner arrangement with a focal length of 3.34 m. The line definition was improved by increasing the focal length of the collimating mirror and positioning the slit very close to the photographic plate.

Transition Probability Measurements in the Neutral Argon Spectrum—Numerous recent determinations of transition probabilities of neutral argon are indicative of the importance of this noble gas in high-temperature technology. Nevertheless, serious discrepancies between the various experimental and theoretical results have remained. Therefore, two independent measurements and an extensive analysis of all other work on the argon spectrum have been performed to clarify this situation. The two experiments were, first, lifetime determinations of the prominent $5p$ states with a delayed coincidence apparatus, and, second, emission measurements of 80 infrared lines with a wall-stabilized arc. A number of the transitions measured with the arc originate from the $5p$ states so that they could be linked up with the lifetime results as well as some other arc data obtained at NBS to determine the internal consistency of all measured values. With this combined effort a reliable set of transition probabilities for the more prominent argon lines has been established.

Photodetachment Studies—Photodetachment studies on the ion (H_2O) OH^- have shown that this ion may play an important role in controlling electron densities in the D region of the earth's upper atmosphere and lower ionosphere. In this work, photodetachment has been observed to take place only in the ultraviolet and deep violet region of the spectrum: most visible radiation will not detach the most weakly bound electron from this ion. The observed spectrum indicates that the $\text{H}_2\text{O}-\text{OH}^-$ bond is relatively weak (<1 eV). Thus the relatively large photodetachment cross section ($<10^{-17}$ cm²) at short wavelengths may be accompanied by a large photodissociation cross section in the infrared region.

Photodetachment studies of S^- have provided for the first time an absolute cross section 2×10^{-17} cm², and a more precise value of the electron affinity, 2.095 ± 0.015 eV. This work also indicates that the previous application of threshold behavior theory is probably less exact than had been generally believed.

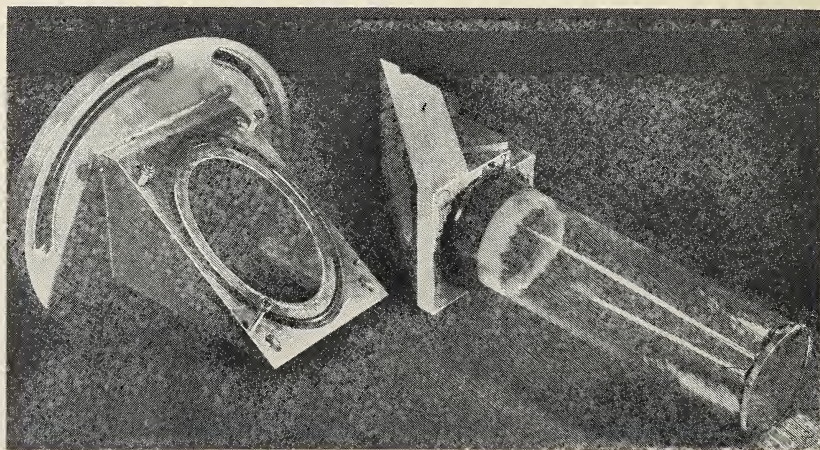
Compilation of Volume II of Critically Evaluated Data in Atomic Transition Probabilities (Elements Na through Ca)—The critical evaluation of atomic transition probability data for the second 10 elements of the periodic system (Na through Ca) has been completed and the tables have been published as part of the NSRDS-NBS series. Numerical "best" values for approximately 5,000 transitions, both allowed and forbidden, have been tabulated and for each value an estimated uncertainty has been given. The bulk of the data is estimated to be accurate within 50 percent. The best accuracy is 3 percent for the famous sodium D- lines. The compilation contains many original data which were obtained by numerical applications of the coulomb approximation and by utilizing the systematic trends of oscillator strengths within isoelectronic sequences.

Continuous Emission from a Hydrogen Plasma—The continuous emission from a hydrogen arc plasma has been extensively investigated theoretically as well as experimentally. The continuum studies are of considerable interest primarily because the hydrogen continuum has great potential to become one of the principal spectral radiation standards in the near ultraviolet and vacuum ultraviolet where no established radiation standard exists as yet. Computer calculations of the continuous emission have been performed over an extended range of temperature encountered in arc operations. The contributions from the hydrogen negative ion (H^-) and the hydrogen molecular ion (H_2^+) have been included, using recently calculated absorption coefficients, and the contributions of the extended line wings of the hydrogen lines have been considered for the first time. Intensity measurements with a pure hydrogen arc carried out at 13,000 K show that the relative spectral continuum distribution in the observed range from 2,800 to 6,000 Å follows the calculations within a few percent. However, the measured continuum is stronger by about 15 percent than the calculated one on an absolute scale. Some of the likely causes of this disagreement, like small deviations from thermodynamic equilibrium in the arc, are currently being investigated.

Microwave Spectra of Free Radicals—The microwave spectra of BrO and SF_2 have been observed for the first time. Transitions from the $^2\Pi_{3/2}$ state in BrO were detected, but not from the $^2\Pi_{1/2}$ state. The observed data for this molecule led to a rotational constant, hyperfine parameters, and nuclear quadrupole constants. Observation of the spectrum of SF_2 was of particular interest, since the chemical literature contains meager and conflicting reports of attempts to synthesize the molecule. The observed data for this molecule led to rotational constants, a dipole moment, and a prediction (with the help of a complete centrifugal distortion analysis) of the molecular force constants and infrared spectrum.

Analysis of Optical and Electron Scattering Data—A procedure has been developed to improve the analysis of optical- and inelastic-electron-scattering data. A least-squares computer program has been used to check the mutual consistency of models for the complex frequency-dependent dielectric constant (for example, the Drude-Lorentz model) and of different types of experimental data (for example, reflectance and inelastic electron scattering cross sections) for a number of elements (Al, Cu, Be, Ge, Sb, Bi, Na, and K) within the accuracy of measurement. The model parameters from satisfactory fits can be used for the identification of structure for comparison with theory, and to derive optical constants. In deriving optical constants from ultraviolet reflectance measurements, for example, the present approach obviates the necessity for arbitrary extrapolations of normal-incidence data, and better precision can be obtained than in the conventional analyses of data obtained at multiple angles of incidence.

Laser Electron Paramagnetic Resonance—A new technique involving an HCN laser in EPR was developed and given the name LEPR (Laser Electron Paramagnetic Resonance). This technique involves focusing the beam of the HCN laser on a small hole in the mirror of a Fabry-Perot interferometer centered in a uniform external magnetic field. The radiation is monitored by a Golay cell through a small hole in the opposite side of the interferometer. The output of the cell is connected to a 13.5 Hz phase detector referenced to the modulation of the magnetic field. A few torr of oxygen gas is placed in the Fabry-Perot, and the magnetic field is swept. Any absorption of energy by the oxygen gas is detected by the Golay cell. Using this technique, five lines in oxygen were observed. Considerable improve-



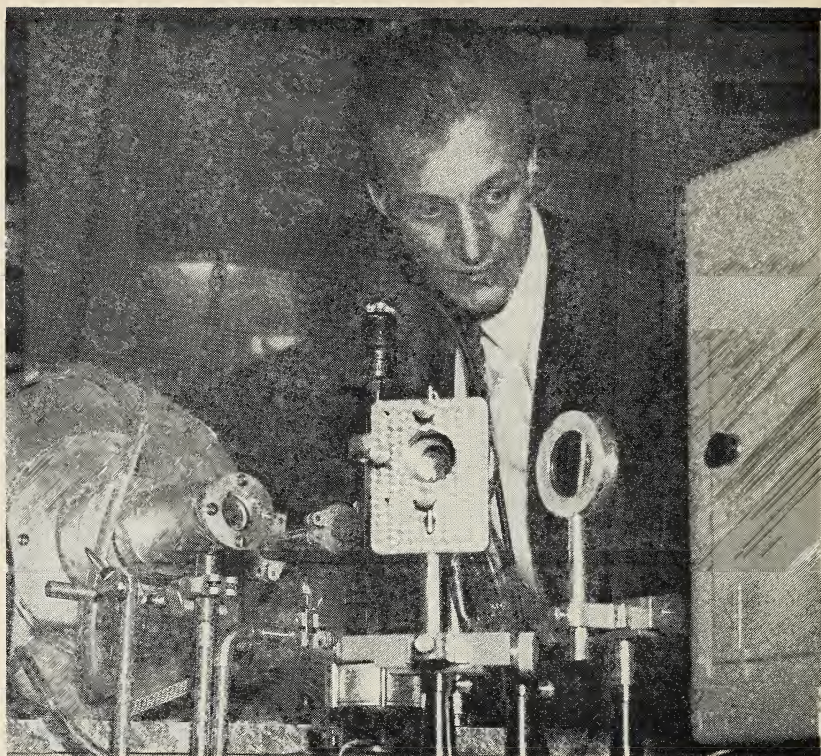
Sample cell used in laser EPR studies of oxygen (12 lines observed) and NO_2 (over 200 lines observed).

ment was achieved by making the oxygen absorption cell part of the laser and employing a Brewster window between the HCN and oxygen to determine the polarization of the radiation. With the improved sensitivity, a total of 12 lines has been observed and identified, and further improvements are planned. The spectrum of NO_2 is also being taken; thus far, over 200 lines have been observed.

Electron Density Measurements—The abnormal negative glow plasma in helium has been adapted for a thorough investigation of the microwave cavity technique used to measure the electron density in plasmas. This negative glow plasma, in combination with a low frequency microwave cavity, was used for experimental and theoretical investigations of the influence of longitudinal modes on the microwave measurements. A double perturbation technique allows for evaluation of the microwave field within the plasma and an extension of the cavity measurements beyond the limits of the simple perturbation method. Data indicate that use of the microwave technique, as interpreted by the simple perturbation theory, is more limited than commonly thought, due to the presence of excited longitudinal modes not accounted for by the theory. The double perturbation technique has in some situations extended the limits for the measurable electron density by about 1–2 orders of magnitude.

Heat Pipe Oven—A new, well-defined metal vapor device called the heat pipe oven has been developed for spectroscopic measurement. Based on a heat conductive element first developed at the Los Alamos Scientific Laboratories, the oven continuously generates homogeneous vapors of well-defined temperature, pressure, and optical path length. All three parameters can be measured directly with high accuracy. The vapor is confined by inert gas boundaries which remove the window problem and allow a direct pressure measurement without relying on vapor pressure curves. As such, the heat pipe oven is suitable for use in connection with vacuum UV spectrometers. It has already been used successfully in a number of different spectroscopic measurements (line broadening, transition probabilities, cross sections, etc.) and has been applied to the generation of metal vapor plasmas.

Line Broadening Theory—A new theoretical treatment of spectral line shapes emitted by plasmas has been developed. This approach, called the unified theory, provides the first line-shape theory capable of describing a line profile from the line center to the quasi-static wings. Calculations of the Lyman α line of hydrogen have been made and compared with experimental results and other theories. The calculations provide the first accurate description of the transition from Markovian to non-Markovian electron broadening. This approach should provide a strong foundation on which further investigations of line shape phenomena can be based.



A modified heat-pipe oven (left) is here being used in spectroscopic investigations.

Electron Impact Cross-Section Data Bank—A data bank containing evaluated experimental cross sections for low-energy electron impact on atoms and small molecules is now active. Data are gathered on a regular basis from the current scientific literature. Graphical display on microfilm can be prepared from the cathode ray tube used as a computer output device. This system will allow fast response to requests for the latest evaluated cross-section data and makes possible the updating of hard copy on a regular basis.

Dissociative Processes of Simple Molecules—An apparatus has been constructed to measure the angular and energy distributions of energetic ions produced by electron impact on molecules. Ion counting is used so that the processes can be observed near threshold where cross sections are small. Initial work on the energy and angular distribution of protons from H_2 has yielded a direct measurement of the momentum transferred to the molecule center of mass in the ionization process. This is the first such measurement and will provide theorists interested in the ionization process near threshold with some valuable checks on their theories. This work has also shown that the angular distribu-

tion of protons near threshold is not a pure cosine-squared distribution, but contains an isotropic part which may be due to autoionizing states of H_2 . After subtracting out the isotropic part, the resulting angular distribution seems to fit a more complicated function as predicted in a recent calculation.

Dissociation Energy of Li_2 from Laser-Excited Fluorescence—A new value for the dissociation energy (bond strength) of the Li_2 molecule has been established. This was accomplished by analyzing the fluorescence excited in the $^6\text{Li}_2$, $^6\text{Li}^7\text{Li}$ and $^7\text{Li}_2$ molecules by various lines of the argon ion laser. The fluorescent light was dispersed using a $\frac{3}{4}$ -meter spectrometer. A Birge-Sponer extrapolation of the $G(v)$ values gave $D_0^0 = 1.026 \pm 0.006$ eV. This value, combined with the dissociation limit of the upper state previously determined by Loomis and Nusbaum, proves that there is a potential maximum of about 0.12 eV above the asymptote of the potential curve for the Li_2 B $^1\Pi_u$ state. Experimental determination of the magnitude of the potential barrier for this fundamental molecule should serve as a useful check on various theoretical calculations of the interactions between atoms at intermediate distances.

Solid State Properties

Thermal Expansion of Platinum—The linear thermal expansion of two specimens of platinum has been measured with a high-precision optical comparator between 1000 and 1900 K. These data are believed to be accurate to 30 ppm for this material in the high-temperature range and will be very useful since platinum is widely used as a reference material for calibrating dilatometers and x-ray diffraction equipment.

Photoelectron Emission from Tungsten in the Vacuum Ultraviolet—Studies of the effects of adsorbed gases on the photoelectron emission from tungsten have shown the electron yield to be very sensitive to even partial monolayer coverages. Over a range of photon energies from 7.7 to 21.2 eV, each of the gases H_2 , N_2 , O_2 , CO , CO_2 , NH_3 , and H_2O produced specific yield changes which were dependent upon the species and quantity of gas adsorbed. The behavior of the electron yield during adsorption can be qualitatively correlated with work function changes, and also offers evidence for the appearance of additional electron emission produced by photoionization of the adsorbed gas.

Baseband Pulse Delay Lines—A miniature 50- Ω superconducting coaxial line (80 feet long, Nb inner conductor 0.01" O.D., Pb outer conductor 0.034" I.D., PTFE dielectric) has been analyzed in terms of the complete Bardeen-Cooper-Schrieffer (BCS) theory of superconductivity. The results have been compared with those based on the

two-fluid superconductivity model. The surface impedance of both superconductors, the line attenuation, and the picosecond time domain step responses have been calculated for a temperature of 4.24 K. Preliminary results indicate that with appropriate parameter values, the more tractable two-fluid model is adequate for engineering design considerations up to and including microwave frequencies. For higher frequencies, i.e., energies approaching or beyond the BCS energy gap, the complete BCS theory is required.

Low-Temperature Thermal Expansion—The low-temperature thermal expansion of over 30 aluminum, nickel, copper, and iron-base alloys was measured from liquid hydrogen temperature (20 K) to room temperature. Both the thermal contraction from room temperature and the thermal expansion coefficient were tabulated as a function of temperature. Comparison of similar alloys and alloy conditions led to the general conclusions that: (a) relatively large changes in composition are required to produce significant changes in thermal expansion, (b) thermal treatment or condition has little effect except when it produces a basic structure change, and (c) the thermal expansion coefficient at room temperature is a good indicator of the total length change that occurs as alloys are cooled.

Improved Superconductive Tunnel Junctions—A new method of fabricating tunnel junctions between superconductive niobium and lead films has been developed. The method consists of condensing the products of a glow discharge onto the surface of a cold (80 K) niobium film, followed by a thermal cycle and deposition of the lead film. This method gives a very high yield ($\sim 60\%$) of tunnel junctions with very low leakage current. After coating with a plastic layer to protect the film, these junctions become very rugged. They may be stored at ambient temperature, exposed to the atmosphere for many months, and subjected for many hundreds of cycles to a low temperature without detectable deterioration. They represent a significant step towards the fabrication of permanent instruments employing the Josephson effect.

Development of Super-Purity Aluminum—Design and construction of cryogenic aluminum magnets has been hindered in the past by the lack of large amounts of high-purity aluminum. Two years ago the highest purity available (99.9999) had residual resistance ratios of only 2,000 to 4,000. Now, through cooperation between NBS and industry, aluminum of the highest known purity, having residual resistance ratios of up to 45,000, has been produced. It is expected to be extremely valuable in physical and mechanical property measurements, as well as for potential use in magnets and other commercial applications.

Thermodynamic and Transport Properties

Determination of the Second Virial Coefficients and Intermolecular Potential for Several Important Gases—Methods developed for the optimum determination of virial coefficients and intermolecular potential functions from experimental data have been applied to the literature for the 14 important gases (Ar, Xe, Kr, He, H₂, N₂, Air, Ne, O₂, D₂, H₂O, D₂O, CH₄, CO₂). Tables of second virial coefficients and their first and second derivatives as well as intermolecular potential parameters, and third virials have been produced. These second virial coefficients and their derivatives are sufficient for the calculation of the real-gas corrections to the ideal-gas equation of state in many technical applications. The intermolecular potential functions can be used as input data in a variety of microscopic theories for macroscopic quantities. These tables represent a considerable improvement over those generally available previously.

Precise Experimental Determination of the Vapor Pressure of CO₂ to Within Several Hundredths of a Degree of the Critical Point—The vapor pressure of CO₂ has been measured with a temperature precision of 0.001° C and a pressure precision of 0.001 bar to within 0.001° C of the critical point. The data have been fitted to a nonanalytic equation consistent with the scaling-law approach. The usual analytic vapor pressure equation is based on the Clausius-Clapeyron equation, and contains the assumption that the liquid specific volume can be neglected with respect to the vapor specific volume, an assumption clearly in error at the critical point where both volumes are equal; thus, its fit exhibits systematic deviations near the critical point. The new equation produces a fit with a standard error an order of magnitude less than that of the analytic equation, and no systematic trend in its deviations.

Bose-Einstein Condensation—The condensation of an ideal Bose-Einstein system was studied using an approach in which the Bose transition acquires a very close resemblance to the second-order transitions about the liquid-vapor and magnetic critical points. The system exhibits an appropriate macroscopic order parameter related to the conjugate field through an ordered sequence of terms in reduced thermodynamic variables. In the vicinity of the transition, the equation of state takes on the homogeneous functional form predicted by the scaling hypothesis and exhibits a power-law behavior with non-classical exponents. The various ordered correlation functions (thermal expectation value for the various ordered products of Boson field operators) were determined and used to demonstrate the existence of a "critical eigenvector" conjectured by Green as a universal feature of critical points. An extension of certain properties of this simple

model has also proven useful in understanding the hydrodynamics of a superfluid condensate.

Determination of the Sensitivity of Thermodynamic and Transport Properties to the Intermolecular Potential Function—The simultaneous fit of equilibrium and transport properties has been assumed to be a particularly sensitive method for the determination of potential functions, as has the isotopic thermal diffusion ratio. Both have been examined for their inherent sensitivity to the potential function. Results reinforce those obtained for the individual properties and show that no new information can be obtained, in principle, from the requirement of a simultaneous fit to pairs of properties. A method for the testing of a functional form of an intermolecular potential function by the simultaneous fit pairs of properties was developed.

The isotopic thermal diffusion factor has never been properly studied because of the large uncertainties present in such data. It has now been confirmed that the isotopic thermal diffusion factor can be a very sensitive probe of the potential function provided measurements are made at temperatures outside an insensitive temperature range. Unfortunately much existing data were taken precisely at temperatures inside this insensitive range. The experimenter can now avoid such design errors in the future by choosing temperatures consistent with our results. The insensitive temperature range, $0.6 < T/T_B < 1.2$ where T_B is the Boyle temperature, was found to be essentially that found for other transport properties previously studied.

Analysis of PVT Data Obtained With Burnett Apparatus—In 1936, Burnett described a simplified technique for the measurement of the isothermal compressibility factor of a gas. The parameters necessary for this method are pressure and temperature, eliminating the troublesome volume measurement; however, a nonlinear statistical problem must be solved to evaluate the compressibility factor as a function of pressure. Many different data-reduction schemes have been developed for the evaluation of the virial coefficients and the compressibility factor, each differing in its approach to uncertainties involved. A meeting on the Burnett data-reducing problem was held at NBS, focusing on particular problem areas, and resulting in close cooperation among several laboratories. A round-robin problem was designed and circulated, and the comparison of the various data analyses is being used to define the areas of disagreement. This approach, established as a result of the meeting, will permit increased use of an experimentally simple method.

Development of a Method for Optimizing the Coefficients Determined from Measured Data Fitted to a Power Series for an Experiment of a Given Precision—In calculating the coefficients of a power series fitted to experimental data, several factors besides experimental precision affect the standard error of these coefficients. Using the prin-

ciples of statistical analysis, the effects of factors such as interval size, extrapolation, number, relative weight, and distribution of experimental points were studied by high-speed computer experiments. On this basis it was determined how to adjust these various factors to minimize the standard error for a given experimental precision. For the particular case of gas PVT data, a density expansion is possible and approximate theoretical knowledge of the first five coefficients is available. This allowed for the formulation of experimental conditions (other than improved precision) leading to a decreased standard error in one or more of the first four coefficients.

Critical Phenomena in Binary Liquid Mixtures—To study critical phenomena in binary liquid mixtures the system 3-methylpentane-nitroethane was selected. The refractive indexes of the liquid components of this system are such as to minimize multiple scattering in studying critical opalescence. Experiments were carried out to determine the intensity of scattered light, the curve of coexisting concentrations and the behavior of the surface tension as a function of temperature.

Scaling-Law Analysis—A systematic review of the experimental evidence for the validity of the scaling hypothesis has been completed using the available data in the critical region for a number of systems. An extension of the previous work on fluids, and a careful analysis of several ferromagnets, have shown the scaling ideas to be consistent. A closed-form equation of state based upon the scaling laws has been suggested to relate the appropriate thermodynamic quantities (for fluids—chemical potential, density, and temperature, for ferromagnets—internal magnetic field, net magnetization and temperature). Agreement between the proposed form and the experimental data shows that the equation of state is satisfactory within the entire critical region.

Light Scattering—The frequency spectrum of light scattered by temperature, pressure and concentration fluctuations in a binary liquid mixture was obtained using the linearized hydrodynamic equations. The theory indicates that light-scattering measurements provide an attractive means of measuring binary diffusion coefficients, as no gradients are imposed on the system.

Molecular Motion in Solids at Low Temperatures—Recent heat-capacity measurements on deuteromethane crystals have demonstrated that rapid transitions are taking place between the equivalent or nearly equivalent orientations of the molecules at temperatures well below 1 K. In addition, conversion between the different nuclear-spin states of the molecule accompanies the reorientation process. Similar behavior is well known in solid H_2 and D_2 , but it has long been thought that such processes were “frozen out” at such low temperatures in all other solids. The present results at 0.3 K show that this is not necessarily the case in simple molecular crystals.

High-Temperature Heat Capacity Standard—Over 20 years ago, NBS proposed synthetic sapphire (pure polycrystalline α - Al_2O_3) as a highly suitable heat-capacity standard for intercomparing calorimeters in different laboratories from below room temperature to near its melting point (above 2300 K). The heat capacity measurements subsequently made by NBS from 15 to 1173 K have been the standard reference since that time. A new sample of this substance has recently been prepared as a Standard Reference Material, and precise calorimetric measurements from 273 to above 2000 K were performed on several specimens. The results, using apparatus of improved precision, match the earlier values over the entire range of overlap, as well as joining the previous measurements below 273 K.

In addition to providing a new, carefully characterized sample, this recent work inferentially has revalidated the older samples still in use.

Technology of Liquid Helium—A publication on the "Technology of Liquid Helium" was edited and partially written by members of the NBS staff. This monograph brings together articles by 16 noted authorities in cryogenic technology. Individual chapters cover a full spectrum of subjects including Resources, Production and Conservation, Properties, Techniques for Liquefaction and Refrigeration, Storage and Handling, Safety, Cryoelectronics, and Applications for cryopumping, superconductivity, bubble chambers, and missile and space systems.

Thermal Conductivity of Gaseous and Liquid Hydrogen—The thermal conductivity of normal and parahydrogen has been measured at temperatures from 17 to 200 K and at pressures to 150 atm (15MN/m²). These measurements have filled a large gap in the knowledge of this property; they are the first comprehensive measurements on the compressed gas and liquid at temperatures below 78 K. The behavior of the thermal conductivity of hydrogen appears to be similar to that of other fluids except that the temperature dependence of the conductivity of the liquid at constant density is much larger than that of most other fluids except helium. These measurements complete a ten-year, NASA-supported program on the physical properties of parahydrogen at low temperatures.

Helium Heat Transfer—A 3-year program has been initiated to supply needed information on helium heat transfer. The technology of superconducting materials and cryogenically cooled high purity substances has reached a point where their use as electrical-current conductors is being planned into practical systems. But lack of reliable information on helium heat transfer is a serious obstacle to the development of industrial devices. This new NBS program is funded by the Atomic Energy Commission and monitored by a committee with representatives from all of the major AEC laboratories, NASA, DOD, and NBS. It will include the generation of property values for con-

ductivity and viscosity of Helium I. Additionally, studies will be carried out analytically and experimentally to determine the heat-transfer coefficients for supercritical Helium I and the heat transport through Helium II as well as the interface between Helium II and solid materials (Kapitza conductance).

Thermodynamic Properties of Argon—The thermodynamic properties of argon have been calculated from an equation of state which was fitted to experimental P - ρ - T data from the world literature. These calculations are one result of a study which was conducted over a period of several years and included exhaustive analysis of existing experimental data by both statistical and thermodynamic theory. The



He^3 - He^4 dilution refrigerator produces temperatures below 0.1 K. Refrigeration is produced in a chamber inside a coil (arrow) used to measure the temperature. Suspended from the chamber are two devices to be tested—a Josephson-junction thermometer on the disc and a heat exchanger to the left. In use the refrigerator is surrounded by a vacuum space and several thermal shields.

results of this effort are described in an NBS publication containing tabular values of density, internal energy, enthalpy, and entropy. Diagrams of the specific heats, compressibility factor, and thermodynamic properties are included.

Saturated Liquid Densities of Oxygen, Nitrogen, Argon, and Parahydrogen—Standardized density data were prepared for the saturated liquids of four commercially important gases—oxygen, nitrogen, argon, and parahydrogen. Users such as the Compressed Gas Association, the Department of the Air Force, and NASA require consistent and accurate values for the physical properties of these gases in order to achieve consistency in cost determinations. The State of California has used extracted tables in their code (law) for cryogenic measuring devices. The Compressed Gas Association is reissuing their pamphlet P-6 using these values as the data base.

He³-He⁴ Dilution Refrigeration—A refrigerator operating on the He³-He⁴ dilution principle has been designed and constructed utilizing a previously prepared tabulation of the thermodynamic properties of He³-He⁴ solutions. This refrigerator has achieved a continuous temperature of 0.020 K and a lowest temperature of 0.013 K. It was recently installed in a shielded room to minimize electromagnetic effects on sensitive instrumentation. Plans call for the experimental evaluation of transport and thermodynamic properties of He³-He⁴ solutions, determination of the physical properties of solids, and investigation of devices such as the Josephson junction thermometer.

MECHANICAL PROPERTIES

Classical Theory of Sound Propagation—An analysis of the equation customarily assumed to describe the propagation of acoustic waves in a fluid continuum considering viscosity, compressibility, and heat conduction has been carried out, calculating the transient as well as the steady-state response predicted by these equations. A bounded solution requires that an inequality involving the bulk and shear viscosity, the specific heats at constant volume and constant pressure, and the thermal conductivity be satisfied. This inequality appears to be obeyed for all fluids except the molten metals, including mercury, and possibly helium II. This failure probably indicates that some of the assumptions made in deriving the initial equation are inadequate when thermal effects are more prominent than viscous effects. The common assumption that the velocity of sound in an inviscid fluid varies continuously with increasing frequency from adiabatic to isothermal values was shown to be invalid except for one very special case.

Absolute Viscosity of Water—The rheology section completed an extended program in which measurements of the viscosity of a liquid

using two independent absolute techniques were compared. Ordinarily, measurements of viscosity yield only the viscosity of various liquids and reference is H_2O at 20 °C and 1 atmosphere. These can quite easily be made to 0.1 percent, but absolute measurements on any liquid to better than about 1.0 percent are difficult and are seldom undertaken. Moreover, most previous measurements have utilized flow through a capillary and involved certain end effects which may cause undetected systematic error. The NBS measurements involved two quite different types of flow, one inside an oscillating sphere, the second through a channel whose dimensions could be established very accurately. The results of these two measurements differ by 0.4 percent. By chance they bracket the value of H_2O previously accepted as the most reliable. Hence, the results of this program do not change previously accepted values for viscosity, but do for the first time provide a basis for assigning an accuracy (of ± 0.2 percent) to *all* such measurements.

APPLIED MATHEMATICS

The Institute for Basic Standards conducts a program in applied mathematics and statistics to meet varied needs in the development of new measurement techniques and in the evaluation of the results of measurement. The level of the mathematics involved makes it essential to conduct fundamental mathematics research on a fairly broad scale.

Computer-Controlled Scanning Microscope and Image Processor—A system has been designed and constructed for biological image-processing using a scanning microscope and several remote time-sharing computers. The scanning microscope, which is under computer control, measures the light flux passing through a specimen on the stage of a microscope. This measurement is made to one part in 256 for each point in square array of 256 by 256 points which is superimposed on the image appearing in the microscope. At present, the image thus quantized is recorded on magnetic tape for processing by a remote computer.

The remote computers (a PDP-10 and an ANFSQ32) are used to analyze the structure of the biological objects recorded on the tape. The morphological structure of such biological objects as nerve cells and blood cells is obtained by having the computer decompose the image into its component parts and then attempt to reconstruct the object according to known rules governing the prescribed shape of the whole object. This type of articular analysis is done in the programming language "LISP." This language provides facilities for the manipulation of partial orderings (tree structures) which represent the results of the image analysis.

Through the use of the interactive feature of the time-sharing computers, it is possible for a biologist to develop reasonably elaborate

criteria for the recognition and analysis of the objects portrayed in the microscope image because as he develops these criteria he can see their consequences immediately and refine or correct them at once. Currently, biologists are using this system to develop criteria for the analysis of the cytoarchitecture and morphology of central-nervous-system tissue, and for the study of autoradiographs of white blood cells as part of a study of cell kinetics.

Numerical Analysis—A method for computing absolute error bounds in terms of various matrix norms for the output of any matrix inversion program was refined and incorporated into an existing computer program. Error analysis for the stationary-phase method of approximation was completed. An explicit expression for the error term was found for the general case. Useful applications were made in the approximation by asymptotic expansions of Bessel and other special functions of mathematical physics. The method of Chester, Friedman, and Ursell for approximating definite integrals with nearly coincident saddle-points was extended.

FORTTRAN Version of OMNITAB—The Bureau's highly user-oriented, general-purpose computer program, OMNITAB, has been rewritten in ASA FORTRAN in order to make it as nearly machine-independent as possible. The program is being used successfully by scientists in numerous university, industrial, and government laboratories on at least five different makes of computers. OMNITAB is used easily, quickly, and effectively by nonprogrammers for the calculation of tables of functions, for solutions of nonlinear equations, for curve fitting, and for statistical and numerical analysis of data. The program reads data in free-field format and executes instructions written in the form of simple English sentences. It is particularly well-suited for exploratory non-routine data analysis.

Evaluation of Linear Least Squares Computer Programs—Two linear least squares test problems, both fifth-degree polynomials, have been run on more than twenty different computer programs in order to assess their numerical accuracy. Among the programs tested were representatives from various widely used statistical packages. Some of these were highly inaccurate in comparison to programs (such as NBS's ORTHO) that have been available for many years. A number of programs yielded results (coefficients) that were completely erroneous, containing not even one correct significant digit. The tests were run on several different computers, in double as well as single precision. It was found that those programs using orthogonal Householder transformations or Gram-Schmidt orthonormalization were much more accurate than those using elimination algorithms. Programs using orthogonal polynomials (suitable only for polynomial fits) also proved to be superior to those using elimination algorithms.

Selected Papers on Statistical Concepts and Procedures—The Bu-

reau has published Volume I, Statistical Concepts and Procedures, of the twelve-volume NBS Special Publication 300, Precision Measurement and Calibration. This 441-page volume collects, for easy reference, reprints of 40 selected articles on topics of special importance to metrologists. Each of six sections of the volume is preceded by a foreword pointing up the purpose for which the papers are included. Section titles are: (1) The measurement process, precision, systematic error, and accuracy; (2) Design of experiments in calibration; (3) Interlaboratory test; (4) Functional relationships; (5) Statistical treatment of measurement data; (6) Miscellaneous topics. A list of references and an extensive subject index are provided.

Finding Shortest Paths—In analyses of transport or communications networks, it is frequently assumed that a traveller will select (or a unit of traffic will be routed along) a path which is “shortest” with respect to time, cost, or some other pertinent feature. Because a single applied study may require solving a great many shortest-path problems, perhaps involving large networks, it is important to classify and experiment with various solution methods so as to know which techniques may be best suited to particular situations. Following preparation of a computationally oriented survey giving flow-charts and descriptions of over 20 variants, the Bureau conducted comparative tests of the more promising methods on a variety of real and “generated” networks.

Scheduling Algorithms—Proper scheduling in a transportation system increases the public service derivable from a given investment in fleet and facilities. Workers on mathematical scheduling methods have tended to ignore the important interactions between patronage and schedules. The Bureau has developed operational algorithms for near-linear (“acyclic”) networks which account for this interaction. Though focused on intercity rail movement, the technique should be valuable for other modes and for urban transport as well. In addition, Bureau-supported work has yielded the first rigorous treatment of jointly optimal scheduling for “express” and “local” service.

TECHNICAL ASSISTANCE TO OTHERS

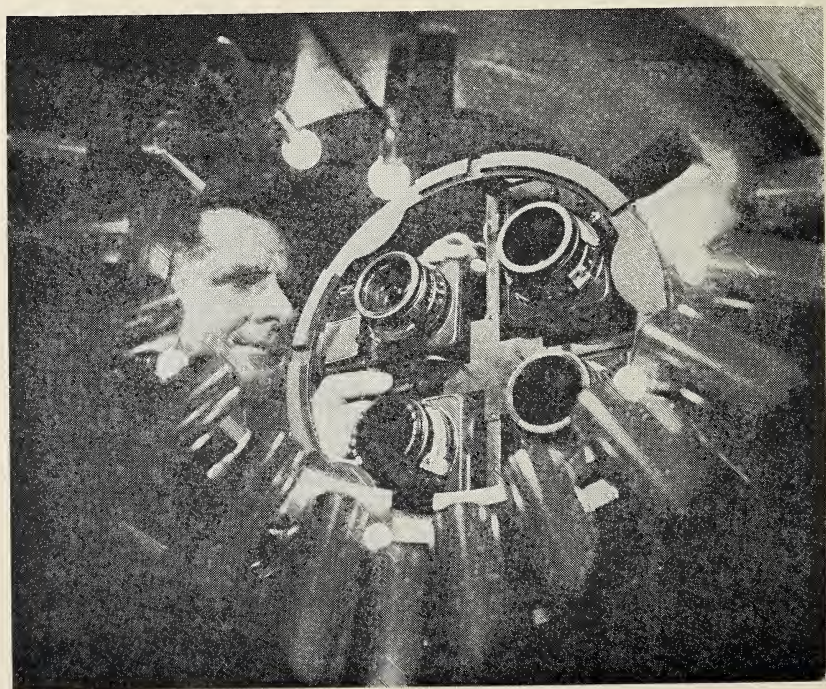
Advisory and Consulting Services

Location of Cargo-Consolidation Centers—The development of a system of inland consolidation centers for marine cargo has been suggested as a means of improving the efficiency of U.S. international trade activities. As part of a project conducted jointly with the Technical Analysis Division for the Federal Maritime Administration, the Operations Research Section developed a mathematical model to assist in the location and spacing of such centers, and to estimate some of the

potential benefits. The model was implemented in a computer program, and exercised in a number of scenarios employing available empirical data.

Precision Measurements for Mapping—NBS is assisting the U.S.A. Topographic Command in improving the application of precise microdensitometry and photographic image-structure evaluation in the production of maps. Starting with aerial photographs, cartographers employ several intermediate photographic and photomechanical stages in the preparation of the plates used to print maps. The assistance program involves training, consulting, preparation of physical standards of measurement, and assistance in perfecting measuring techniques. The improved ability to measure and quantitatively specify the pertinent image-structure characteristics of the materials at each stage should improve quality and effect economies.

Apollo IX Cameras Calibrated—The cameras used to photograph the moon on the Apollo IX mission were calibrated by NBS. Four cameras, each utilizing a different spectral region, were mounted in the main hatch window. Conventional black-and-white film, infrared film, and false-color film were used. The cameras were tested with films identical to those used on the flight. NBS measured the lens character-



Cameras used by Apollo 9 astronauts to photograph the moon were calibrated at NBS.

istics including back focal lengths, effective focal lengths, calibrated focal lengths for photogrammetric use, radial and tangential distortion, resolving-power throughout the field, T-stop settings, lens-transmittance factor, relative distribution of illumination, and lens-modulation transfer function. In addition, the shutter speed, shutter efficiency, and the wedge angles of the spectral filters were measured. Measurements were made with and without filters in place and the lenses were evaluated by photographic, photoelectric, and visual methods. The cameras provided photographs for use in detailed mapping of the surface of the moon.

Electroluminescent Formation Lights—Studies regarding the feasibility of installing strip-type electroluminescent (EL) formation lights on high-performance (mach 2) aircraft have been completed. Tests indicate that the EL lamps possess vibrational characteristics superior to those of their incandescent-lamp counterparts, while their linear geometry conveys better aircraft orientation to formation wingmen. These studies have resulted in the specification of EL-type formation lights for new aircraft (F-14 and F-15) while preparations are underway for retrofitting existing aircraft such as the F-4.

Field Tests of Fog Detectors—The distance one can see signal lights is an important factor in the safety of operation and landing of aircraft and in the navigation of ships. As part of the photometry program sponsored by the Department of the Navy, Federal Aviation Agency, Air Force, and Coast Guard, the Visual Landing Aids Laboratory of the Photometry Section, Meteorology Division, has completed field testing of several types of back-scatter fog detectors at Arcata Airport, Arcata, California—reputedly the foggiest airport in the United States. The tests included operational suitability, reliability, and correlation of fog-density measurements (obtained by the fog detectors) with direct measurements of atmospheric transmittance.

Recommendations on Archival Microfilm—Following several years of research on the causes and prevention of blemishes on microfilm, the results of a large-scale inspection of government microfilms were published, and final recommendations for the prevention of such blemishes were presented to the National Microfilm Association and the Archivist of the United States. Although there had been practically no loss of information, the potential threat to archival records was considered serious. The blemishes studied take several forms but are most often reddish spots about 0.1 mm in diameter. The blemishes were caused by minute amounts of gases generated during long-term degradation of the cardboard cartons in which the films were stored. Recommendations covered raw film, exposing and processing conditions, storage conditions, and inspection. For archival purposes, the importance of specifying storage and handling conditions as well as the characteristics of the films was emphasized.

Physical Properties of Glass-Fiber-Reinforced Plastic Rods—Studies were undertaken for the U.S. Coast Guard to develop and evaluate test methods for obtaining mechanical and electrical property data on glass-fiber-reinforced polymer rods. These rods are used for insulator guy systems needed for communications and navigational aids towers. The properties studied were tensile strength, creep strength, fatigue resistance, effect of ultraviolet radiation, water absorption, dielectric strength, and high-voltage flashover resistance. From the results of these investigations, a prototype insulator guy was specified which will be useful as a laboratory reference for evaluating improvements or variations in future insulator guy systems for Loran C towers and similar applications.

Hearing Aids Circular Revised—NBS Circular 534, "Hearing Aids" (1953) has been completely revised and brought up to date. This publication is directed toward the consumer and discusses sound and hearing, and the general properties, selection, and care of hearing aids. It also lists the principal hearing centers in the U.S., at which assistance to prospective users of hearing aids is available.

Radio Standards Information Center—A Radio Standards and Measurement Information Center was established to assist the NBS technical staff and provide scientists and engineers with information on the use of RF standards and calibrations provided by NBS. The Center will also provide information on the development, use, and evaluation of accurate measurement systems. Outputs include specialized bibliographies, survey articles, state-of-the-art reviews, and assistance in answering technical questions to the electronics industry, universities, private research agencies and standards laboratories, and other Government agencies working in the electromagnetic frequency range from 30 kHz through the millimeter wave region. Specific subjects include attenuation, power, noise, field strength, voltage, current, impedance, dielectric constant, pulse quantities, horn gain, and antenna patterns.

Radiation Protection Suit—An electromagnetic radiation protection garment was evaluated for use at frequencies below 100 MHz. This garment was designed to protect personnel who are exposed to high-level electromagnetic fields. Measurement techniques were developed to evaluate the shielding effectiveness of the suit in the presence of both high- and low-level fields. The low-level measurement results were encouraging, but high-level tests, using specially developed probes for measuring high-level E and H fields (approximately 100 mW/cm²), showed that the suit suffered from arcing and other nonlinear effects and hence was unsafe for human use.

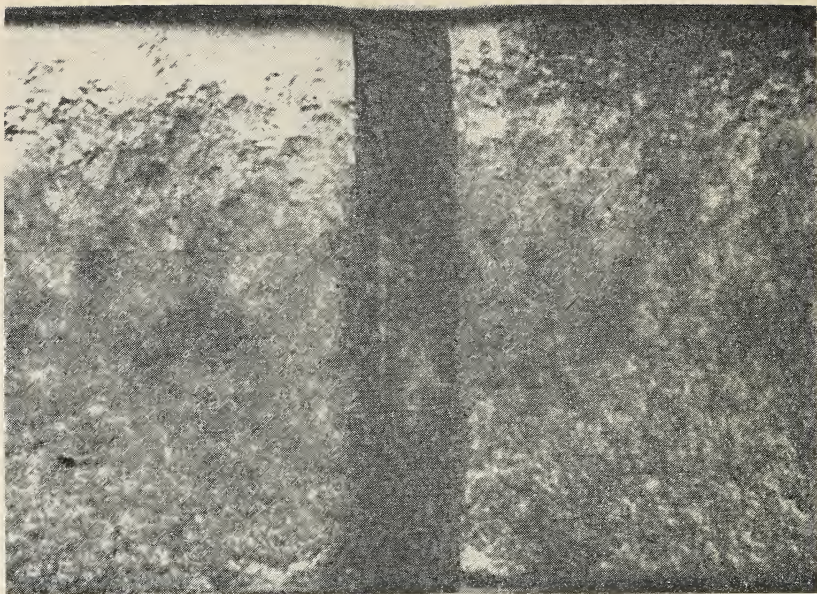
Biomedical Electronic Metrology—Work in biomedical electronic metrology has been initiated on a program basis. An extensive study was made of measurement needs, which led to the identification of

appropriate tasks and level of effort. Current work includes design of environmental test chambers for studying radiation effects upon animals and development of methods for measuring and calibrating radiation intensity from ultrasonic diagnostic and therapeutic instruments. Also, with the support of the HEW Bureau of Radiological Health, development was initiated on a new near-zone energy-density measurement device for determining the level of stray radiation from microwave ovens.

Apollo 11 Lunar Range Experiment—NBS has cooperated with scientists from the University of Maryland, Goddard Space Flight Center, Princeton University, Wesleyan University, and other institutions in preparing the optical retroreflector array that was carried to the moon on Apollo 11. The round-trip light travel time from the earth to the reflectors and back will be measured using short laser pulses. The accuracy of resulting distance measurements is expected to be 15 cm or better. One application of the range data will be to determine the lunar orbit, librations, and radius to much higher accuracy than they are now known. Improved information will also be obtained on the wobble of the earth about its rotation axis, the rotation of the earth, and the difference in longitude between widely separated observing stations. Such geophysical information should help in understanding the interaction between the core and the mantle of the earth, and should provide a direct test of whether the large-scale crustal movements predicted by recent theories of ocean-floor spreading and continental drift are taking place at present.

The Cryogenic Data Center—The NBS Cryogenic Data Center has a data bank of over 30,000 carefully indexed references to the properties of materials at low temperatures. During the past year the Center's professional staff noted over 8,300 articles of interest to the field of cryogenics in its review of an estimated 340,000 titles, abstracts, or full copy of the published and report literature. The articles noted were listed in the 52 issues of the "Current Awareness Service" that is now sent to 585 paid subscribers. The "Current Awareness Service" completed its fifth year of publication in April 1969, and "Superconducting Devices" (a quarterly survey prepared in cooperation with Stanford Research Institute and the Office of Naval Research) completed its first year of publication. There are now 115 paid subscriptions. All pertinent documents were entered into the automated information storage and retrieval system which is now in machine-readable form and machine searchable. A new method of segregating the references stored on magnetic tape was developed. It enabled the initiation of a Selective Dissemination of Information (SDI) program which identifies new publications in a particular field of interest.

Characteristics of Slush Hydrogen—In cooperation with NASA, flow characteristics of liquid-solid mixtures of hydrogen (slush hydro-



A magnified view of slush hydrogen being pumped through a 1-cm orifice. Slush hydrogen offers distinct advantages over liquid hydrogen as a rocket propellant.

gen) in transfer lines have been measured. Losses through a restriction were also determined with slush and found to be essentially the same as with liquid hydrogen. A system was constructed in which the solid content of slush could be increased by drawing off liquid and retaining solids with a screen. Using this technique, solid contents greater than 50 percent were attained in a dewar of freshly-made slush, while solid contents in excess of 60 percent can be realized by aging the mixture. Evaluation of stirring devices was made in the system to determine criteria for mixing the high-solid-content slush to a homogeneous, flowable mixture. Production of gelled slush hydrogen was also accomplished and an analytical expression was derived relating the quantity of gellant required to gel slush hydrogen to the quantity required to gel liquid hydrogen. Information on the characteristics and behavior of slush hydrogen is being sought for studies of applications to future space missions, and for the design of ground handling equipment.

Conferences and Symposia

Precision and Accuracy in Measurement and Calibration—A West Coast location was selected for a 3-day seminar on statistical techniques that had been presented four times in Washington. The Bureau reported experience in the development and use of statistical experi-

ment designs especially tailored to the needs of calibration laboratories for making economical intercomparisons among sets of standards. Presentations by NBS statisticians were complemented by reports from Bureau calibration laboratories that are implementing the new computer-aided methods for data analysis, preparation of calibration reports, and surveillance of the measurement ("production") process. The seminar was held at the West Coast University in Orange, California, and was sponsored by the National Conference of Standards Laboratories. Participants were 37 metrologists from industrial and government laboratories.

Photometry Seminar—Thirty-five registrants from photometric laboratories of private industry and government agencies participated in a four-day seminar on photometry given by the Photometry Section of the Metrology Division May 12 through 16, 1969. The seminar included lectures given by staff members on photometric quantities and terminology, photometry and the eye, photometric standards and methods, and projection photometry. The lectures were supplemented by laboratory visits and demonstrations and with individual conferences with staff members. A similar seminar was held earlier for 14 representatives of Navy standardizing laboratories.

Symposium on Thermal Expansion of Solids—Jointly sponsored by the Westinghouse Astronuclear Laboratory and NBS, this first symposium emphasized the complete spectrum of techniques used in measuring the thermal expansion property. The program consisted of 12 invited and 24 contributed papers, including a historical review of the theory of thermal expansion, a description of recent measurements at low temperatures, a review of x-ray and neutron diffraction methods, and a description of measurements at high temperatures with neutron diffraction. More than 100 scientists attended the three-day symposium, September 18–20, 1968.

Length, Angle, and Geometry Measurement Seminar—Thirty-two dimensional metrologists from industry and government standards laboratories and from the George Washington University Graduate School participated in a 5-day seminar. Lectures by NBS staff, laboratory sessions, and discussion periods covered: gage blocks; optical and mechanical measurement of length, angle, diameter, and flatness; gear metrology; and statistical programs for determination of the accuracy of measurement.

Fundamentals for Gas-Laser Length Measurement Seminar—Metrologists, physicists, and mechanical engineers from university, industry and Government participated in a 2-day seminar. Forty-one persons attended lectures by NBS staff covering fundamental interferometry, gas lasers, holography, NBS laser instrumentation, and practical pointers on use of lasers.

Symposium on High Pressure—An international symposium on "Ac-

curate Characterization of the High Pressure Environment" was held at the NBS October 14–18, 1968 and attended by 140 participants from the United States and abroad. Thirty-eight papers were presented, and four panel sessions were held covering current research at high pressures in static systems and in shock wave experiments. In panel sessions, five fixed points below 100,000 bars were proposed, and values for these points recommended. The equation of state of sodium chloride was recommended as a standard for the x-ray determination of pressure. The measurement of temperature in high-pressure apparatus and the measurements of phase transformations as a function of temperature and pressure using static and dynamic techniques were presented. Dynamic methods included studies of phase transformations ranging from 20,000 bars up to several million bars. Recent developments in static high pressure were also presented.

Short Courses in Noise and Power Measurements—By special request, two brief measurement courses were presented to specially selected U.S. Air Force personnel. One 4-day course covering RF power measurements was presented in February 1969, and a 5-day course covering both high-frequency and microwave measurements in noise was presented in April 1969. Both courses were designed to satisfy the specific needs of the Air Force and included discussions of NBS measurement techniques as well as anticipated Air Force measurement problems.

Seminars on Frequency and Time Stability—One hundred and twenty-two registrants from private industry and government agencies attended two seminars on Frequency and Time Stability given by NBS on February 18–20 and June 3–6, 1969. Four foreign countries were represented. The seminars developed a language that allows clear characterization of the instabilities in frequency standards. Measurement methods were described for obtaining data on the stability of precision signal sources such as cesium beams, hydrogen masers, rubidium gas cells, and quartz crystal oscillators. Flicker noise and other commonly encountered noises were discussed.

NATIONAL STANDARD REFERENCE DATA SYSTEM

The production of scientific information has undergone explosive growth during the past decade. Furthermore, the development of sophisticated instruments has meant that a greater part of this information is in the form of quantitative numerical data. These numerical data are essential to the advancement of scientific understanding, the planning of new research, and the utilization of research results in industry. However, the great mass of data now being produced has led to a serious problem in locating the data needed for a particular purpose and ensuring that it is the best available. The National Stand-

ard Reference Data System aims to solve this problem by collecting data from the world scientific literature, subjecting it to critical evaluation by experts, and condensing it into an accessible and easily usable form.

The National Standard Reference Data System (NSRDS) was established in 1963 under a directive from the Federal Council for Science and Technology and the President's Office of Science and Technology. The primary functions of NSRDS are: (1) to sponsor projects for the compilation and critical evaluation of numerical data in technical areas where the need for such data is clear; (2) to coordinate data compilation activities throughout the Government; (3) to establish standards of quality for products of the system; and (4) to develop techniques for handling and disseminating scientific data. The scope of the program is currently limited to well-defined properties of well-characterized physical systems. These boundaries are not perfectly sharp; however, the emphasis is on intrinsic properties which can be measured in a reproducible way and which are clearly defined in terms of universally accepted physical theory.

The core of NSRDS is made up of a group of data centers, each of which is responsible for a clearly defined technical area. There are currently about 25 of these centers. Roughly half are located in the technical divisions of the National Bureau of Standards, and the remainder are supported on a contract basis in universities, Government laboratories and industrial laboratories in all parts of the country. Each data center is responsible for the various phases of compilation of data within its technical scope. These consist of (1) locating the pertinent publications in the scientific literature; (2) indexing and abstracting the publications; (3) extracting and compiling the numerical data; and (4) subjecting the data to a critical evaluation in order to obtain a set of recommended values. The last phase is the most critical one and requires the intellectual input from experts who are experienced in the particular field. Every effort is made to utilize the services of outstanding research leaders for this critical evaluation phase.

The tangible products of NSRDS consist of tables of critically evaluated data, accompanied by a detailed discussion of the criteria for evaluation and a critique of the measurement techniques by which the raw data were obtained. Ideally, such a publication provides the best set of numbers for a scientist or engineer to use. In addition, it points out gaps in our existing knowledge and deficiencies in the measurement techniques, and thus helps to stimulate and guide future research. This feedback mechanism, by which the level of measurement and interpretation of scientific data can be upgraded, is regarded as one of the most important functions of NSRDS. It has, of course, always been one of the principal goals of the National Bureau of Standards.

The activities of NSRDS were given a specific congressional mandate through legislation enacted by the 90th Congress in July, 1968. The Standard Reference Data Act (PL 90-396) contains the following provisions: (1) a declaration that it is the policy of the Congress to make critically evaluated reference data readily available to scientists, engineers, and the general public; (2) a directive to the Secretary of Commerce to provide or arrange for the collection, compilation, critical evaluation, publication, and dissemination of standard reference data; (3) a directive to the Secretary of Commerce to prescribe standard criteria and procedures for the preparation and publication of standard reference data, as may be necessary; (4) authority for the Secretary, or a person or agency designated by him, to sell standard reference data and to allow the proceeds to be used by the Bureau; and (5) authority for the Secretary to obtain copyright, on behalf of the United States as author or proprietor, for standard reference data prepared or made available under the Act. This Act provides the authority for more varied channels of distribution of standard reference data. Plans are now being developed to utilize this authority.

Current Activities

The activities of NSRDS are classified under eight program areas; six of these represent technical areas in which data compilation projects are active; the remaining two cover more general functions carried out in the Office of Standard Reference Data. A summary of recent accomplishments and current activities in each program area is given below.

Nuclear Data

An NAS-NRC advisory panel meeting on nuclear data was held in November, 1968. The general conclusion of the meeting was that OSRD should work toward sharing the responsibility for financial support in the area of nuclear data, which previously has been almost entirely in the hands of the Atomic Energy Commission. The first step to implement this recommendation was taken in an agreement between OSRD and the AEC to provide joint support for the Berkeley Particle Data Center, which for several years has been compiling and publishing elementary particle data. Proposals for other projects on nuclear data are being studied, and it is hoped that some of these can be funded during the next year.

The Proceedings of the Neutron Cross Section and Technology Conference were published in two volumes with D. T. Goldman, Editor, as NBS Special Publication #209 (1968). The Program Manager also chaired a meeting of an ISO Working Group, in May, which was concerned with additions to the International Nuclear Energy Glossary of Terms.

Atomic and Molecular Data

This program has emphasized the production of reference data compilations in categories designated as highly important to the technical community by its advisory panels. Considerable emphasis during the past year was given to the publication of infrared spectral data based on the criteria established by the Board of Managers of the Coblenz Society. The first 1,000 research quality spectra have been issued and plans are underway for the preparation and issuance of the second group of 1,000 spectra. The success of this project has encouraged the initiation of similar activities in the fields of Nuclear Magnetic Resonance, Raman Spectroscopy, and Mass Spectrometry. An advisory group meeting to examine criteria for the publication of microwave spectra is also being planned. In addition to these activities, an ad hoc advisory panel met on May 27, 1969, to provide guidance for the compilation of interatomic distances in gas-phase molecules.

The following reports have been published:

1. "Theory of the Ionization of Atoms by Electron Impact," by M. R. H. Rudge, reprinted from *Reviews of Modern Physics*, Vol. **40**, No. 3, July 1968.
2. NSRDS-NBS-26, "Ionization Potentials, Appearance Potentials, and Heats of Formation of Gaseous Positive Ions," by J. L. Franklin and J. G. Dillard of Rice University, H. M. Rosenstock, J. T. Terron, K. Draxl of NBS, and F. H. Field of Esso Research and Engineering Company, 1969.
3. ORNL-AMPIC-11 and 12, "Bibliography of Atomic and Molecular Processes for January-December 1968," compiled by Atomic and Molecular Processes Information Center, Oak Ridge National Laboratory, under the direction of Dr. C. F. Barnett, 1969.
4. NSRDS-NBS-23, "Partial Grotrian Diagrams of Astrophysical Interest," by Dr. Charlotte E. Moore and Paul W. Merrill, 1968.
5. Special Publication 306-1, 2, and 3, "Bibliography on the Analyses of Optical Atomic Spectra, Sections 1, 2, and 3," by Dr. Charlotte E. Moore.
6. NBS Monograph 70, Volumes III, IV, and V, "Microwave Spectral Tables," by Paul F. Wacker, Marian S. Cord, Donald G. Burkhard, Jean D. Petersen, and Raymond F. Kukol, Matthew S. Lojko and Rudolph H. Haas, 1968, 1969.
7. NBS Technical Note 474, "Critically Evaluated Transition Probabilities for Ba I and II," by B. M. Miles and W. L. Wiese, 1969.

Solid State Data

The activities in the solid state program are reflected largely through the continuing efforts of the data centers established in this area. The assembly of bibliography and the preparation of compilations on

crystal data, alloy data, superconductor properties, and diffusion data are in progress. Two of the contributing efforts are being carried out in India. They are "Phase Transformation in Solids," under the direction of C. N. R. Rao, and "Crystal Defects in Alkali Halides," under the direction of S. C. Jain.

The following reports have been issued:

1. NBS Technical Note 464, "The NBS Alloy Data Center: Function, Bibliographic System, Related Data Centers, and Reference Books," by Gesina C. Carter with L. H. Bennett, J. R. Cuthill, and D. J. Kahan, 1968.
2. NBS Technical Note 482, "Superconductive Materials and Some of Their Properties," by B. W. Roberts, General Electric Research and Development Center, 1969.

Thermodynamic and Transport Properties

During the past year, projects initiated earlier were carried on under Office of Standard Reference Data sponsorship in the following areas:

Chemical Thermodynamic Data on Inorganic Compounds

Thermodynamic Data on Organic Compounds

Properties of Molten Salts

Thermodynamic Properties of Polar Gases: Ammonia

Low Temperature Specific Heats

Thermodynamic Properties of Liquid Metals and Liquid Oxides

Systems at High Pressure

Vapor-Liquid Equilibrium in Binary Mixtures of Non-Electrolytes at Low Pressures

Binary Metal and Metalloid Constitution Diagrams

Thermal Conductivity of Selected Materials, Including the Elements

Viscosity and Thermal Conductivity of Mixtures in the Gaseous and Liquid States

Transport Properties of Fluids

Two new projects were initiated on the evaluation of data near the critical points of binary liquid mixtures and on the thermodynamic properties of nitrogen. The first of these complements a program on the liquid-vapor critical region being carried out under other sponsorship. Together the programs constitute a thoroughgoing evaluation of the data in a region which is difficult experimentally but of great theoretical and practical interest. The project on nitrogen, in addition to its own intrinsic importance, is coordinated with an international program under the auspices of the International Union of Pure and Applied Chemistry.

Additional continuing projects under other sponsorship are also being carried out in the following fields:

Properties of Electrolyte Solutions

Thermodynamic Properties of Metals and Alloys

Thermodynamic Functions of Cryogenic Fluids

Thermal Conductivity of Metals and Alloys at Low Temperatures

The *Annual Index of Worldwide Published Thermochemical Studies* continues to be published yearly under the auspices of the International Union of Pure and Applied Chemistry, with substantial support from the Office of Standard Reference Data.

Several projects have resulted in major published reports during the year. These reports are:

Molten Salts, Vol. 1, Electrical Conductance, Density, and Viscosity Data, G. Janz, F. W. Dampier, G. R. Lakshminarayanan, P. K. Lorenz, and R. P. T. Tomkins, NSRDS-NBS-15, 1968

Theoretical Mean Activity Coefficients of Strong Electrolytes in Aqueous Solutions from 0 to 100 °C, Walter J. Hamer, NSRDS-NBS-24, 1968.

Thermodynamic Properties of Argon From the Triple Point to 300 K at Pressures to 1000 Atmospheres, A. L. Gosman, R. D. McCarty, and J. G. Hust, NSRDS-NBS-27, 1969.

Selected Values of Chemical Thermodynamic Properties, Tables for Elements 35 Through 53 in the Standard Order of Arrangement, D. D. Wagman, W. H. Evans, V. B. Parker, I. Halow, S. M. Bailey, and R. H. Schumm, NBS Technical Note 270-4, 1969.

Ionization Potentials, Appearance Potentials, and Heats of Formation of Gaseous Positive Ions, J. L. Franklin, J. G. Dillard, H. M. Rosenstock, J. T. Herron, K. Draxl, and F. H. Field, NSRDS-NBS-26, 1969.

The last named was sponsored under the program in Atomic and Molecular Properties but is of importance in the area of Thermodynamic and Transport Properties as well. Reports have been received in several other areas which should result in publications in the coming year.

Colloid and Surface Properties

There are six current projects in the field of colloid and surface properties. These cover the following fields:

Surface Tensions of Molten Salts

Critical Micelle Concentrations of Aqueous Surfactant Systems

Surface Tensions of Substances Liquid in the Vicinity of Room Temperature and Below

Light Scattering by Liquids

Electrical Properties of Interfaces

Physical Data Pertaining to Phase Transition Kinetics

A report on the surface tensions of molten salts is in press and two others are in the final stages of preparation.

Chemical Kinetics

A meeting of the Chemical Kinetics Advisory Group under the chairmanship of Dr. S. W. Benson was held on May 19, 1969. This group affirmed the view that the principal aims of the program should be directed toward compilations of chemical kinetic data and monographs for reactions in the gas phase. They also recommended the preparation of bibliographies for the assistance of those authors carrying out critical evaluations on subjects such as solution kinetics. The importance of varied approaches embracing data sheets, short reviews, and complete critical evaluations was endorsed.

The program has been responsive to this advice. The two data centers have prepared bibliographies for use by authors preparing critical evaluations. Data sheets covering the radiation chemical behavior of six compounds are in preparation. A compendium of rate constants embracing approximately 1,500 reactions has been compiled. The establishment of new data centers is under consideration.

The following reports have been issued :

1. NSRDS-NBS-20, "Gas Phase Reaction Kinetics of Neutral Oxygen Species," by Harold S. Johnston, University of California, 1968.
2. NBS Technical Note 484, "A Review of Rate Constants of Selected Reactions of Interest in Re-Entry Flow Fields in the Atmosphere," by M. H. Bortner, General Electric Company, 1969.
3. NBS Report 9884, "A Compendium of Evaluated and Estimated Rate Coefficients," by David Garvin, 1968.
4. "The Biradical Mechanism in Small-Ring Compound Reactions," by H. E. O'Neal and S. W. Benson, reprinted from the Journal of Physical Chemistry, 72, 1866 (1968).

Data Systems Design

The major efforts of the Data Systems Design Group have been divided among two coequal activities:

- (a) design of and programming support for computer-assisted typesetting and composition of data tables and associated textual and bibliographic material, and
- (b) development of a comprehensive package of general-purpose programs for data file manipulation.

Work has been completed on a series of general-purpose computer programs for automatic typesetting of computer-generated text and tables. One of these (TYPSET) handles program listings and formatted computer printout. Another called KWIND is specialized to typesetting KWIC (keyword in context) indexes. Both programs have provisions for converting the normal upper-case computer output to a more readable format with upper- and lower-case characters, and even Greek characters where appropriate.

In the area of data file manipulation a series of programs have been written to reformat and edit data files in three categories:

- (a) fixed-field files of fixed-length records,
- (b) free-field files of structured but variable-length records, and
- (c) free-field unstructured files of variable-length records which have been suitably flagged.

For files which satisfy the above characteristics the programs permit exceptionally easy file manipulation.

The programs have been documented in a series of Technical Notes issued through the Superintendent of Documents.

A distribution system for general-purpose computer programs on magnetic tape has been started as a pilot program for a larger effort on NSRDS data tapes. The machinery for distribution of magnetic tapes via the Clearinghouse for Federal Scientific and Technical Information is proving quite successful, judging from the reception by the technical press of the release of the first magnetic tape in the series.

Information Services

Despite the limited financial resources of the Office of Standard Reference Data, which have imposed severe restrictions on Information Services, this operation is continuing to progress and to make effective contributions to the NSRDS program. Some of the contributions are:

Data File—The Data File has established a very comprehensive collection of worldwide compilations of data in the physical sciences. Its holdings of reference data compilations are as extensive as will be found anywhere in the world. An annotated accession list of the holdings is in preparation.

Compilation and Editorial Services—This unit is responsible for the monthly *NSRDS News*. This newsletter has a worldwide circulation of over 4000 subscribers. This unit also acts as the editorial intermediary between the Office of Standard Reference Data, associated data centers and projects producing data compilations on the one hand and the government publication mechanism on the other. In the present reporting period, 20 publications have been produced and 17 are in process.

Inquiry Services—This unit provides coordinated replies to inquiries received by the Office of Standard Reference Data from the Data File, from the Compilation and Editorial unit of the Office, and from other sources in the NBS and NSRDS associated centers. If the query is within the capability and scope of the program, a reply is forwarded. Sometimes inquiries are referred elsewhere or reference sources furnished, or data compilations or other publications are provided. In the present reporting period, 558 inquiries were received, compared to 400 in the previous year.

Analysis and User Relations—In order to develop an effective information system to provide broad coverage of properties data in the physical sciences with a flexibility capable of meeting the needs of a large variety of specialists, this unit has been examining the users, actual and potential, of standard reference data. Toward this end, it conducted two very preliminary studies. The first queried by mail 212 persons who received NSRDS publications as to the use and usefulness of the publications received. Ninety-eight returns were received (47%). Of these, 86 indicated they found the NSRDS publications useful; 4 indicated they did not find them useful; one did not reply to that question; and 7 did not remember the publication.

The second effort was a pilot survey in the form of an interview/questionnaire aimed at providing information on technical reference data use patterns of NBS scientists. This pilot study revealed that 42, or 84 percent, of a representative sample of 50 NBS scientists use physical properties data. These scientists, on the average, spend approximately one hour of their working day in seeking or discussing their data requirements. One of the results of this pilot study is a revised interview/questionnaire suitable for survey of similar information by other organizations.

INSTITUTE FOR MATERIALS RESEARCH

Emphasis in the Institute for Materials Research (IMR) is on measurement methodology for well-characterized materials. This emphasis supports the unique mission to develop standards of measurement for the commercial, industrial, and educational communities of the Nation and to provide research and consulting services to other Government agencies. About 25 percent of IMR's work is for other Government agencies.

Work falls into the following broad areas:

1. Preparation and characterization of materials
2. Standard Reference Materials
3. Data on the properties of materials
4. Technical assistance to others.

PREPARATION AND CHARACTERIZATION OF MATERIALS

This area includes such activities as the synthesis of new materials, purification of materials, crystal growth, and the analysis of materials with respect to chemical composition and structure. Outputs of these activities are sample materials to be used for specific research purposes, and the development of new techniques and procedures for preparing or characterizing materials.

Preparation

Synthesis

Synthesis of Organoboron Compounds—A number of new organoboron compounds have been synthesized for the first time. Two of these, ethynyldichloro- and ethynyldifluoroborane, are the first reported members of the hitherto unknown family of organoboron halides of the acetylenic series. The syntheses were carried out using both photochemical and conventional synthetic techniques. The compounds appear to be significantly more stable than most boron-substituted acetylenes. Since they are thus amenable to detailed physical investigation, they are of considerable interest for detailed studies of structure and bonding in technologically important organoboron

derivatives. Preliminary microwave spectroscopic studies have been carried out in cooperation with members of the Institute for Basic Standards. Some novel organo-substituted compounds containing boron-boron bonds have also been prepared and are of particular interest as new derivatives containing preformed aggregates of boron atoms. The new compounds are being investigated both from the viewpoint of their fundamental chemistry and as possible precursors to useful boron-containing materials such as the borides.

Organometallic Complexes of Biological Interest—Interactions between transition metal ions in binuclear complexes have been observed in a large number of compounds. Research in this area has been extended, in a cooperative study with chemists at the State University of New York at Plattsburgh, to include complexes of biologically significant ligands such as the purine bases. Dimeric adenine complexes of copper(II) have been synthesized in both neutral and anionic forms, and the magnetic behavior of such complexes has been studied by electron paramagnetic resonance spectroscopy. These compounds are the first reported examples of purine-base complexes with unusual magnetic properties attributable to exchange-coupled metal-ion pairs. The complexes are of interest as model compounds for a number of biological systems, particularly in view of recent suggestions that the occurrence of metal ion pairs may be significant in biological processes involving enzymes and proteins with trace metal ions.

Reactive Intermediates in Chemical Synthesis—In the past several years increasing recognition of the importance of synthetic chemical processes derived from highly energetic conditions has resulted in an array of novel and useful chemical structures. To a large extent these advances have derived from the development of the characterization and descriptive chemistry of *reactive intermediates*—small molecules or molecular fragments produced thermally, by photo excitation, or in plasmas or flames—which determine the chemical pathways available outside the energy zone.

In previous studies, complex product mixtures resulting from interactions of low-density fluoride plasmas with ceramic or organic polymer materials were rationalized in terms of a few key reactive intermediates. Such an approach has been extended to examination of materials such as organometallics and their halides upon subjection to comparatively lower energy irradiation with visible and ultraviolet light. Primary and secondary reactions which occur at rates sufficiently different to permit identification by electron spin resonance (ESR) or mass spectrometry have been demonstrated. Solid solutions of methyltrichlorosilane and trichlorosilane show non-additive behavior at liquid nitrogen temperatures, where the methyl free radicals associated with photo-cleavage of methyltrichlorosilane by itself disappear in the presence of trichlorosilane to form a new, complex radical product. The

potential of such "reagent" intermediates for specific free radical syntheses is further illustrated by the photolysis of phosphorus trichloride at liquid nitrogen temperatures. The primary photo-cleavage reaction produces a novel radical, PCl_2 , along with mobile, reactive chlorine atoms. The latter produce secondary reactions with unreacted PCl_3 molecules to generate still another new radical, PCl_4 . Dilution experiments with the inert gas Xe indicate the secondary process can be inhibited in the solid PCl_3 matrix.

¹⁵N-Labeled Amino Sugars—Naturally occurring sugars that possess a chemical structure in which one or more oxygen atoms is replaced by a nitrogen atom are often found as components of substances, such as antibiotics, which have important biological functions. IMR scientists have now synthesized the first amino sugars having a ¹⁵N-content greater than 99 percent to study the nuclear magnetic properties of the isotope-labeled compounds and the potential of ¹⁵N magnetic resonance parameters for structural analysis.

Periodic Acid, a Novel Oxidant for Organic Compounds—Controlled oxidation of organic compounds is an important step in the synthesis of pharmaceuticals and other substances of biomedical significance. Periodic acid, a reagent well known for its ability to cleave 1,2-diols, has now been found also to be a useful oxidant of polycyclic, aromatic hydrocarbons. A unique, two-fold character of response to periodic acid by such hydrocarbons has been observed: production of (1) coupling-reaction products through a radical intermediate, or (2) quinones by a two-equivalent oxidation mechanism that does not involve a radical intermediate. A most interesting example is the oxidation of the hydrocarbon azulene with periodic acid, which gives a polymeric product that is highly paramagnetic and retains this free-radical property for some time at 485 °C.

Crystallization and Morphology of 66-Nylon—Current investigations are primarily aimed at determining the nature of the crystallization habit exhibited by 66-nylon when it is crystallized from dilute as well as concentrated solutions of the polymer in glycerol. The objective of this study is, among other things, the elucidation of the manner in which the chain molecules of this polyamide fold during crystallization. The polymer has been observed to crystallize in a variety of forms depending on the condition of crystallization. Among the habits observed on crystallizing from dilute solutions are simple parallelepiped lamellae as well as multi-layered lamellae crystals whose constituent chain-folded layers are apparently twisted. Concentrated solutions have been observed to yield on cooling complex petal-shaped aggregates as well as negatively birefringent spherulites. The relationship between these and other forms of crystallization exhibited by 66-nylon is being studied.



66-Nylon is being studied to determine the manner in which the chain molecules fold during crystallization. Shown here is a scanning electron micrograph of (X520) spherulites and other aggregates (top left) of 66-Nylon grown from a concentrated solution of the polymer in glycerol.

Polymerization at High Pressure—High pressures (10–20 kbars) have been utilized to produce new fluorinated copolymers of tetrafluoroethylene with the olefins 3,3,3-trifluoropropene; 3,3,3,2-tetrafluoropropene; 2-trifluoromethyl-3,3,3-trifluoropropene; and 3,3,4,4,5,5,5-heptafluoropentene-1. At autogeneous pressures only liquids and oils are produced, instead of the strong high molecular weight copolymers produced at high pressures. The comonomers all have some retardation effect on the polymerization of tetrafluoroethylene. The objective of the research which is sponsored by AQDP is the expansion of our basic comprehension of the molecular and structural factors that determine the mechanisms of polymerization, the nature of the transition state, and the molecular size.

The composition of the copolymers produced at various pressures from 3,3,4,4,5,5,5-heptafluoropentene and tetrafluoroethylene is a function of the composition of the monomer charged. The pressure does not affect the composition appreciably, although it drastically enhances the molecular size. Reactivity ratios found for these systems work against incorporation of the tetrafluoroethylene in the

copolymer. The rate of these copolymerizations becomes much more rapid at high (10:1) ratios of tetrafluoroethylene to the other monomer.

Separation of Polymer Molecules by Flow—The basic idea behind separation by flow is easily explained. An isolated polymer molecule flowing through a thin capillary and undergoing Brownian motion will have an average velocity greater than that of the solvent. This is because the center of the particle (assumed to be a rigid sphere) cannot get any closer to the walls of the capillary than its radius. It therefore samples only those solvent velocities away from the walls; since the solvent velocity is larger, the farther the distance from the wall, larger molecules will have larger average velocities than smaller molecules. This "Separation by Flow" mechanism has been advanced as the fundamental basis for the phenomenon of Gel Permeation Chromatography. The quantitative understanding of this basic phenomenon thus now allows the systematic design of instruments to separate molecules or particles of different size.

Chelating Agents as Potential Adhesives for Dental Restorations—One of the unsolved problems in restorative dentistry is the lack of adhesion between tooth tissues and permanent restorations under conditions existing in the oral cavity. Micro-leakage between cavity and restoration can result in secondary caries. In the present work, supported by the Dental Division of the United States Army Medical Research and Development Command, potential adhesive liners that may bond to tooth structure and plastic restorative materials have been studied. These tailor-made molecules were selected on the basis of theoretical considerations taking into account the chemical composition of the tooth surface and the compatibility of these compounds with cold curing acrylic restoratives. The newly synthesized compounds contained groups that react with calcium to form chelates and other groups that could copolymerize with dental resins.

Dentinal and enamel bovine tooth surfaces were treated with these compounds and the adhesion to a cold curing acrylic resin was then determined. The teeth were stored up to 30 days at 37 °C in solutions of different acidities. The tensile strength of the bond between tooth, liner, and restorative material was taken as a measure of the bonding efficiency of a compound. Treatment of the dentinal and enamel surfaces with most chelating agents produced only insignificant increases in bond strength or gave results that were not always reproducible. However, applications of dilute aqueous solutions of many heavy metal salts changed the surface of the enamel within a few seconds so that the restorative resin adhered even without the use of an intermediate liner after storage in water for 24 hours. This behavior can be explained by the formation of secondary covalent bonds, causing weakening of the bond of the resin's ester group on

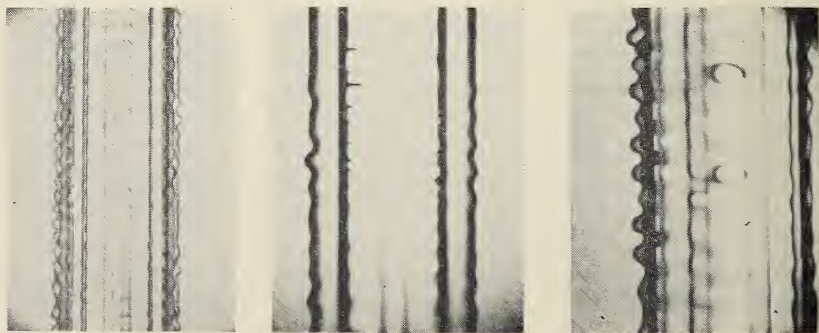
prolonged exposure to water. The results with similarly treated dentin surface showed less adhesion, but were promising enough to justify continuation of this line of investigation. It is hoped that the use of potential adhesives in combination with treatment of the surfaces with metal salt will lead to improved bonding at the tooth surface-acrylic interphase.

Electrodeless Electrolysis—Both solid materials and fused salts have been electrolyzed without being in contact with any solid conductor. The current was carried to the materials via glow discharge in a gas at a pressure of about 0.1 torr. Under these conditions some ceramics became electrically conducting, and dendrites of nickel, cobalt, copper, silver, tin, and magnesium were deposited from fused salts. The experiments show that metallic electrodes are not essential for electrolysis to occur.

Vapor Growth of Al_2O_3 Bicrystals—IMR has, with the support of the Advanced Research Projects Agency, developed a technique to grow aluminum oxide mono- and bicrystals by a vapor transport process. Monocrystals are being grown by this method with the goal of higher purity in order to improve the measurements of many physical and chemical properties. Bicrystals are being grown in order to measure properties of the interface.

Basically, the process consists of reacting AlCl_3 with oxygen under carefully controlled conditions of pressure and temperature with the resultant deposition of Al_2O_3 in single crystal form in a seed. By the use of double seed crystals, bicrystals containing symmetrical tilt boundaries have been grown in sizes suitable for research specimens. The purity of the crystals is quite high, of the order of 40 ppm by weight, or less, total cation impurities.

Morphology of Ice Cylinders—Recent experimental and theoretical results have shown that the initial morphology of growing crystals can be predicted. Experiments on the growth of single-crystal ice cylinders in slightly supercooled water have been carried out. The initially



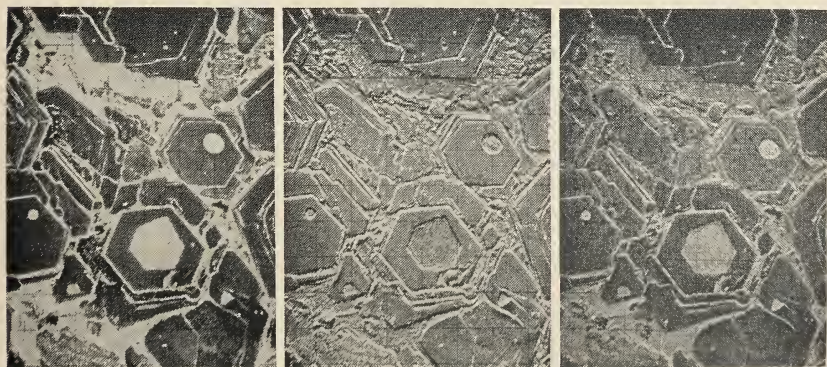
Experiment has confirmed theoretical predictions of the initial morphology of growing crystals. Shown here are perturbations on the surface of ice cylinders at supercooled temperatures -0.20 , -0.09 , and -0.08 °C, left to right.

smooth cylinder develops approximately sinusoidal perturbations parallel to the axis of the cylinder. The wavelengths, λ , of these perturbations have been measured as a function of the bath undercooling, ΔT . Based on a morphological stability analysis, theory predicts that of all possible wavelengths, the wavelength of maximum amplification will appear on the growing cylinder. Excellent agreement has been obtained between the theoretical prediction and the experimental results. The sinusoidal perturbations are the precursors of dendritic growth. Thus, the initial stages of the development of dendrites can be successfully treated by morphological stability theory.

Melt Growth of Cuprous Oxide Crystals—As a part of a crystal growth project sponsored by the U.S. Atomic Energy Commission, large single crystals of Cu_2O have been grown by a float zone process in controlled oxygen pressures. Typically, crystals of 1.5 cm diameter and up to several centimeters long have been grown, with total cation impurities of less than 40 ppm.

New Technique for Target Current Scanning in Electron-Probe Microanalysis—In the electron-probe microanalyzer, a beam of electrons focused on the surface of a specimen produces x rays, secondary electrons, and a current from specimen to ground, known as target current. The target current signal, which is affected by the back scattering of electrons, varies both with composition and topographic variations of the specimen surface.

In IMR laboratories, a concept has been developed for handling the target current signal in a way that permits separating and emphasizing the topographic aspects of a specimen independent of composition. This may render unnecessary in many cases the installation of secondary electron detectors and associated electronics. An amplifier

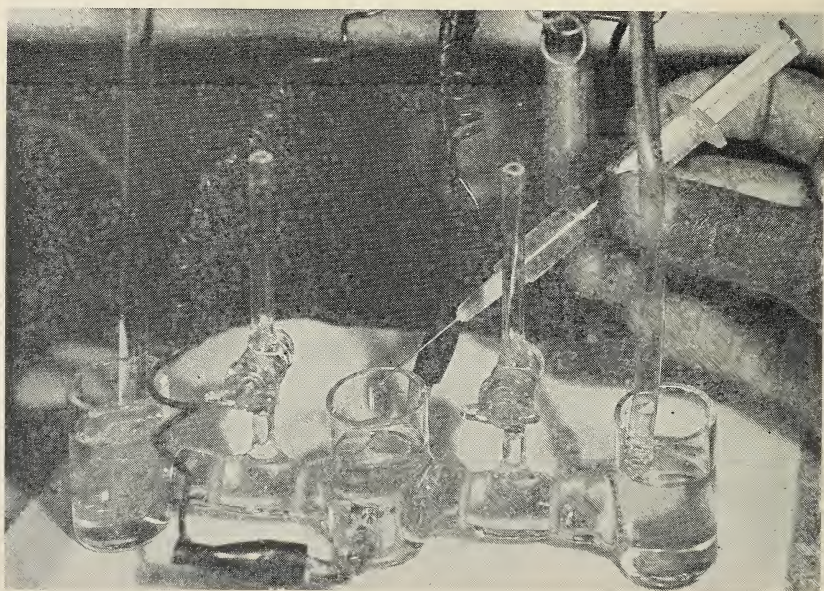


Target current scans of titanium boride contaminated with iron. The iron shows as the bright phase in the upper image. Upper image: linear target current, showing atomic number differences. Middle image: derivative image, showing topography. Lower image: combination of both signals.

has been constructed in which the target current signal and its first derivative are amplified and recombined in any desirable proportion. Application of this concept in the examination of a titanium boride specimen makes evident both the surface topography and the distribution of iron impurity.

Multi-Element Trace Analysis by Isotope-Dilution Spark-Source Mass Spectrometry—Highly sensitive methods have been developed for trace analysis of high-purity materials using the spark source mass spectrometer with isotope dilution techniques. In a typical analysis, for example, the determination of trace elements in zinc, a solution of the zinc is spiked with isotopically enriched solutions of the elements to be determined, the elements are electroplated onto high-purity gold wires, and the wires are sparked in the mass spectrometer. By measuring the mass spectra and applying appropriate calculations, the concentrations of the trace elements in the original zinc sample are determined.

The method permits the simultaneous determination of groups of six or more elements and provides determinations at concentrations of parts per billion, e.g., 20 ppb tin in zinc. The method was developed especially to provide analyses of NBS standard reference materials and has been applied to analyses of zinc, platinum, and gold. Further work indicates general applicability to the analysis of other materials such as steel and glass.



Cell for electroplating impurity elements from isotopically modified solutions onto gold electrodes prior to mass spectrometric trace analysis.

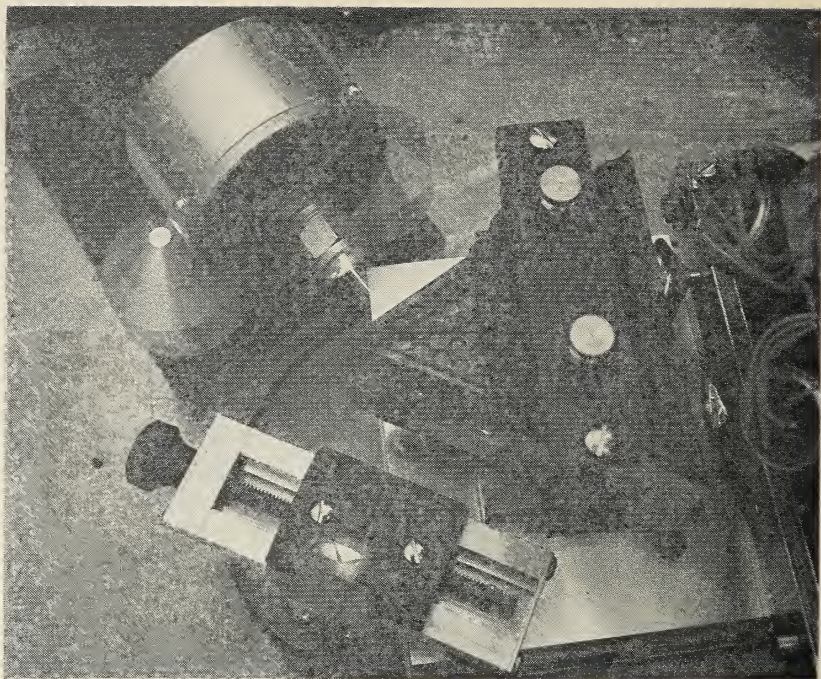
Improved Trace-Element Analysis—A new approach has been developed that offers at least a hundred-fold improvement in the selectivity and sensitivity of many existing spectrophotometric and spectrofluorometric procedures. It uniquely combines the inherent selectivity of solvent extraction and the statistical advantages afforded through utilization of three-component complexes. Improved sensitivity is obtained by performing exchange reactions on ion-pairs directly in the organic extract. In the proposed method conditions are readily obtained whereby a weakly chromogenic or fluorogenic cation is replaced instantaneously by a highly colored or fluorescent dye. Unreacted dye is readily extracted into water, leaving only the dye complex in the organic phase.

Recent studies involving nanogram amounts of gold and uranium have clearly demonstrated the improved selectivity and sensitivity of the extraction-exchange method. At least one other advantage is also apparent. The method shows promise of being a simple way of obtaining many new and potentially useful complexes which, heretofore, were impossible to prepare by conventional techniques.

Multi-Element Analysis by Molecular Absorption Spectrophotometry—In the metals industry great emphasis has always been placed on the speed of analysis, with accuracy being of somewhat secondary importance. The increasingly stringent demands of modern day technology, however, are creating new needs for more accurate as well as more rapid methods of analysis.

Absorption spectrophotometry, while always regarded as a highly accurate technique, has until now been limited almost exclusively to single-element analysis. Recent studies, however, have shown that the careful selection of chromogenic reagent in combination with computer evaluation of data can yield systems and procedures that are equally accurate for the determination of as many as five or six elements simultaneously. The primary prerequisites, in order of their importance, are degree of spectral overlap, reproducible formation, and stability of the metallo-organic complexes. These parameters have been investigated and closely evaluated in the recent development of a multi-element spectrophotometric procedure for the simultaneous determination of cobalt, nickel, and copper. Accuracy is about 1 percent at the part-per-million level and less than ten minutes are required for both analysis and computation.

Interferometer Control for Mössbauer Spectrometer—A Mössbauer spectrometer uses some type of device for producing a Doppler motion that provides the necessary gamma-ray energy shift. Electromechanical transducers are often used, but it is important that the motion be measured accurately. An electromechanical system has been modified for control by an interferometer. The device, which uses a laser light source, compares an interference fringe count with standard fre-



To provide the accurate constant acceleration needed for use with a Mössbauer spectrometer, this device compares an interference fringe count with a standard frequency, differences being used as feedback to control the motion. Constant acceleration is achieved by incrementing the standard frequency with time.

quency. Any difference is detected, and a correction signal is generated as feedback to the electronic system. Constant acceleration is achieved by incrementing the standard frequency linearly with time. This arrangement provides the extreme accuracy and precision of motion which enables the Mössbauer spectrometer to produce spectra of unprecedented reliability.

New Clean-Room Facility—A clean-room facility of advanced type has been designed and installed and is providing improved handling capability in ultratrace analysis. The facility consists of two clean rooms separated by a perforated wall partition, together with rooms to prepare personnel for entry into the clean areas. Horizontal laminar air flow, at an average rate of 100 lineal feet per minute, is provided for the clean areas. An absolute filter bank removes essentially all airborne particles greater than $0.5\ \mu\text{m}$ in diameter. Each cubic foot of air in the room adjacent to the filter bank contains less than 100 such particles, while a particle-level less than 1,000 is maintained in the second room. The rooms are currently used for microscopic examinations and for the assembly and disassembly of apparatus when extreme cleanliness is a requisite. Two laminar-flow fume hoods permit mild chemical reactions to be performed under ultra-clean conditions.

Improved Trace-Chromium Determination—A method of improved sensitivity and accuracy has been developed for determination of as little as one ten-billionth of a gram of chromium. The coulometric method consists of titration of chromium with electro-generated ferrous ion in a specially designed electrolysis cell of small volume to permit increased sensitivity. An improved end-point detection technique was also devised to facilitate the analysis. The new method provides sensitivity and accuracy of at least an order of magnitude better than previously available in the low concentration range. Microgram quantities of chromium can now be determined with an accuracy better than one part in a thousand while nanogram amounts can be determined to within a few percent of the amount present. From that point the accuracy drops off rapidly down to a lower limit of about a tenth of a nanogram (one ten-billionth of a gram). The method should be widely applicable to trace chromium determinations ranging from biological and clinical materials to metallurgical products. At NBS, it has been used for determination of chromium in ruby laser using only milligram amounts of the crystal in the determination.

Polarographic Analysis of Air Pollutants—Polarographic methods have been developed for the simultaneous determination of very low levels of iron, copper, cadmium, and lead present as air pollutants. Samples are collected on conventional filters which are ashed or from which the contaminant may be extracted by leaching in favorable cases. An ammonium oxalate supporting electrolyte is used, and the metal is determined by fast scan polarography. For extremely low levels of contaminants, preelectrolysis into a hanging drop electrode followed by anodic stripping voltammetry is used. The submicrogram detection limits make possible a number of application areas. The methods have been applied to a wide range of air-pollution problems including determination of the metallic contaminants of suburban and semirural residential areas, and to collaborative studies with the National Air Pollution Control Administration. They have also been applied to monitoring ambient lead in a laboratory involved in glass-making operations, and even to the determination of metallic constituents in relatively clean laboratory air to identify sources of blanks in trace analysis.

Analysis of Thin Metallic Films—Polarographic methods have been developed in IMR laboratories which have become extremely important for the microchemical analysis of thin metallic films such as are used in electronic circuitry. Such films must be accurately analyzed, yet there is not sufficient material to be accurately weighed, let alone enough for analysis by conventional methods. The polarographic method consists in dissolving the film and quantitatively determining each constituent by electroanalytical measurement with a dropping mercury electrode. Methods applicable to films composed of anti-

mony-bismuth, copper-nickel-chromium-aluminum, nickel-chromium, lead-selenium, and lead-tin-tellurium, respectively, have been developed to date, with total film amounting to only a few tenths of a milligram in each case.

Ion-Selective Electrodes for Microanalysis—Ion-selective electrodes are becoming increasingly useful in chemical analysis, but the physical dimensions of these sensors have limited their applicability to situations in which relatively large amounts of sample are available. IMR research on miniaturization of these sensors has resulted in two important developments. A commercial silver sulfide ion-selective electrode has been modified for use with small samples by drilling 5- to 10- μ l depressions directly into the polycrystalline silver-sulfide membrane of the inverted electrode. Such an arrangement permits measurements on 5 μ l of sample down to the lowest detection limits possible with this electrode.

The first successful truly miniature ion-selective electrode has resulted from redesign of the solid-state fluoride electrode. The construction resembles the conventional electrode except that the lanthanum fluoride membrane consists of a small conical-shaped crystal sealed in a plastic tube drawn down to a fine point. The sample volume requirement is less than 2 μ l (1/25 of a drop) and the needle-like profile of the electrode makes it adaptable to in situ, in vivo, and on-line continuous analysis. Further miniaturization in this manner is feasible and should provide electrodes of even greater versatility.

Aqueous Boric Acid-Borate-Mannitol Equilibria—Despite widespread use and extensive research on polyalcohols, especially mannitol, as addition agents in boric acid titrimetry, the stoichiometry and equilibria involved have not been clearly established. In view of the importance of this procedure in the determination of boron, IMR chemists have reinvestigated this system. Proton nuclear magnetic resonance spectra of boric-acid-borate-mannitol solutions in deuterium oxide and electrometric pH measurements of aqueous solutions were consistent with the formation of both 1:1 and 1:2 borate-mannitol complexes. The successive formation constants of the complexes were found to be only slightly different ($pK = -2.79$; $pK_2 = -2.19$). Failure to recognize this fact has led previous investigators to misinterpret data and to erroneous conclusions. Reevaluation in light of the present results brings several earlier discordant findings into agreement with the IMR conclusions and establishes a consistent model for this important analytical system.

Boron isotope analyses—A surface emission mass spectrometric technique for the precise measurement of boron isotope ratios has been developed. A single-filament tantalum ribbon source was used, and $^{11}\text{B}/^{10}\text{B}$ ratios were determined by measuring the relative abundances of $\text{Na}_2^{10}\text{BO}_2^+$ and $\text{Na}_2^{11}\text{BO}_2^+$ ions, at masses 88 and 89, respectively.

The effects of various parameters such as sample-mounting procedure, filament material, sample size, total sample composition, and sodium concentration were studied and alternative procedures were evaluated.

This precise technique was used to measure the absolute isotopic abundance ratios of two boron isotopic standards; SRM 951, $^{11}\text{B}/^{10}\text{B}=4.0362\pm0.00137$; and SRM 952, $^{11}\text{B}/^{10}\text{B}=0.053199\pm0.000032$. These standards and the new mass spectrometric measurement technique will be of significant value for the precise characterization of the boron used in nuclear reactors. They should also be useful for the characterization of isotopic variations in natural boron.

Atomic Weight of Rubidium—The atomic weight of rubidium has been redetermined by solid-sample thermal emission mass spectrometry. The absolute isotopic ratio was obtained by calibrating the instruments used with samples of known $^{87}\text{Rb}/^{85}\text{Rb}$ ratio, prepared from solutions of the separated isotopes. The resulting atomic weight of 85.46776 ± 0.00026 is not significantly different from the old value of 85.47 but shows a significant increase in precision.

Solvent Extraction With Hydrogen Bis (2-Ethylhexyl) Phosphate—Solvent extraction is one of the most useful techniques for separating one constituent of a mixture from interfering substances prior to analysis. Alkyl esters of phosphoric acid have been shown to be excellent extractants for metallic elements. One of these esters, that of 2-ethylhexanol, has been used extensively for rare-earth separations. A comprehensive study of the extraction of all the metallic elements into this reagent as a function of mineral-acid concentration has been completed. As a direct result of these studies, three new applications for this reagent in radiochemical separations have already been developed, and several other separations schemes, both for individual elements and for groups of elements, are under investigation. The separations are rapid, quantitative, and, with suitable choices of the back extractant, the mineral acid, and the concentrations, substantially interference-free.

Nondestructive Determination of Impurities in Ruby Laser Crystals—The effect of trace elements on the performance of solid-state lasers is as yet incompletely understood. In order to make possible a later correlation of laser optical properties with trace-element content, nondestructive neutron activation analysis methods have been applied to determination of over 10 trace elements in ruby at the ppm and ppb levels, with precisions of 2 to 5 percent. The samples were irradiated in the NBS reactor and the trace elements determined by gamma spectroscopy using a lithium-drifted germanium gamma-ray detector of large volume and high resolution. Because of the highly refractory nature of the matrix, other trace-analysis techniques proved to be of limited applicability. These new procedures provide a definitive characterization of ruby crystals and pave the way to an elucidation of the relationship between composition and optical behavior.

New Applications of the Cockcroft-Walton Neutron Generator—A comprehensive study of the utility of 3-MeV neutrons, from the $(^2\text{H}^2\text{H},n)^3\text{He}$ reactions, for compositional analysis of macro constituents, has been made. This expands considerably the analytical application of the small neutron generator, previously limited to 14-MeV neutron activation analysis. The study included experimental determination of the analytical sensitivity for more than 70 elements along with the gamma-ray spectra from these elements. In addition to their direct analytical application, these results may be used for the prediction of interferences from the buildup of the 3-MeV neutron flux encountered in 14-MeV neutron activation analysis from deuterium embedded in the tritium target. Thermalized 3-MeV neutrons from a small neutron generator were used successfully in the analysis of the metallic components of several oil-soluble organometallic Standard Reference Materials.

Determination of Oxygen in Sodium Metal—The corrosive nature of sodium metal used as a coolant in breeder reactors is markedly affected by oxygen content. The determination of oxygen in sodium by conventional methods is complicated by the possibility of surface contamination of the metal after sampling, yielding a result that is not representative of the bulk sample. In addition, the various reagents and equipment used may contribute to the analytical blank, and this blank may not be susceptible to accurate evaluation.

Photonuclear activation analysis, in which no blank correction need be made, has now been applied to this determination. The reaction utilized was $^{16}\text{O}(\gamma,n)^{15}\text{O}$. To remove surface contamination, the sample was etched after irradiation but before chemical separation and counting. A procedure to separate the radioactive oxygen from interfering substances in $3\frac{1}{2}$ min was developed, and accurate determinations were achieved in spite of the very short half-life of the product nucleus (2.1 min). Sensitivities of a few ppm and precisions of 10 to 15 percent were obtained.

Analysis of Surface Structure by Mössbauer Spectroscopy—Because the appropriate (Mössbauer) nuclear energy level of iron-57 decays with a high probability of emitting conversion electrons, Mössbauer-effect spectroscopy provides an interesting probe with which to examine chemical structure of surfaces of iron bearing materials. A proportional detector has been designed to detect these ~ 8 keV conversion electrons. The design has been optimized to provide the maximum signal to background ratio.

Gas-Water Chromatography—The use of aqueous solutions as the liquid phase in gas-liquid chromatography has been developed into a practical, rapid method of analysis. Techniques have been developed to compensate for the tendency of the solutions to volatilize during the analysis, and thus change composition. As a result, the vast body

of information on equilibria in water solutions can be applied to the design of chromatographic columns. Effective separation of the deuterated ethylenes C_2D_4 , $C_2H_2D_2$ from C_2H_4 has been achieved. The technique also can be used to determine gas-water partition coefficients and equilibrium constants for reactions occurring in aqueous solutions.

Mechanism of Steric Exclusion Chromatography—Steric exclusion chromatography, also called gel filtration or gel permeation chromatography, has in recent years become a powerful tool for the separation and analysis of large molecular species such as synthetic high-polymers, biopolymers, viruses, etc. Although widely applied, the precise mechanism of the separation process is not fully understood. This is partly due to the difficulties in measuring the pore size distribution of the gels which so far have been used with the method. By replacing the gels with glasses of precisely controlled and measurable pore size, the separation process was studied under controlled conditions. The results challenge the validity of the two presently most popular separation theories. The use of porous glass as separation medium is of further value to test present theories of the molecular conformation of macromolecules in solution.

Long range Atomic Ordering in Platinum-group Alloys—In many alloys containing two or more metals, the atoms of each element tend to occupy special positions over large regions within the crystalline lattice. This behavior can usually be produced by an appropriate heat-treatment at relatively low temperatures. The alloy is then described as possessing long-range atomic ordering. The extent of long-range atomic ordering can be measured by studying the relative intensities of x-ray beams diffracted by the crystal. A comprehensive study of the degree of long-range atomic ordering in a variety of binary alloys containing metals of the platinum family has revealed that the location of a given element in the periodic table can be related to its tendency toward atomic ordering.

The degree of long-range atomic ordering in an alloy often has a very significant effect on the mechanical properties of the alloy; particularly its strength and hardness. By intentionally varying the degree of atomic ordering, one can obtain desirable properties in dental gold alloys for example.

In a recent cooperative study with other laboratories it was found that changes in the degree of atomic ordering also produce significant changes in the temperature at which certain metal compounds (A15 phases) lose their electrical resistance completely and become superconductors. This discovery may have important implications since there is presently an intensive effort in many countries to develop alloys which would permit the economic utilization of superconductors to transmit electrical power. A basic understanding of the factors which control atomic ordering may permit the attainment of this goal.

It may also help to improve the properties of alloys now being used for dental and medical applications.

*Low Angle X-Ray Diffraction Intensities in *n*-Paraffins*—Problems concerning the structure and thermal behavior of the interlamellar regions of crystalline chain-folded polymers and *n*-paraffins have been studied with x-ray diffraction techniques. The x-ray diffraction from the crystalline *n*-paraffins, $C_{36}H_{74}$, $C_{44}H_{90}$, and $C_{94}H_{190}$, was examined at small angles as a function of temperature. The Bragg maxima (d_{00}) that occur at low angles result from a lamellar repeat distance which depends on the molecular length. In general, the intensity of these maxima was found to increase with increasing temperature in an approximately reversible manner. All the samples experienced solid-solid phase transitions in the temperature range of observation. Several possible mechanisms consistent with the temperature dependence of the intensity have been analyzed.

Polywater—In a collaborative study with the University of Maryland, a different form of water has been verified and has been shown to have a unique molecular structure. The interpretation of the infrared and Raman spectra indicates that this material is a stable polymer consisting of ordinary water molecules. This new form of water has been named polywater.

Polywater has properties remarkably different from those of normal water. Although polywater maintains the same chemical composition, it has a thermal stability to temperatures of the order of 500 °C; has a very low vapor pressure; has a density about 40 percent higher than ordinary water, and begins to solidify at -50 °C or lower to a glass-like state.

Several molecular structures for polywater have been proposed. These structures are consistent with the infrared and Raman spectra as well as with the properties, bonding, and the stability of the material. The infrared spectrum is unique: the O—H stretching bands prominent in normal water are absent and two new strong bands appear. One of the proposed structures shows that the O—H—O units are arranged in a plane and form hexagonal rings.

Crystallography of Calcium Phosphates and Related Compounds—Phosphorus in general and calcium phosphates in particular are very important in the life of vertebrates. Among the calcium phosphates found in the human body, either as skeletal material or pathological deposits, are $Ca_5(PO_4)_3(F, OH)$, $Ca_5H_2(PO_4)_6 \cdot 5H_2O$, $\beta Ca_3(PO_4)_2$ and $CaHPO_4 \cdot 2H_2O$. $CaHPO_4$ and $\alpha Ca_3(PO_4)_2$ are other important and relevant structures. The structures of $\alpha Ca_3(PO_4)_2$ and $\beta Ca_3(PO_4)_2$ are unknown and may be extremely difficult to determine. The crystal structures of the related compounds $Ca_5(PO_4)_3SiO_4$ and $Ca_4Mg_5(PO_4)_6$ were determined. These structures apparently have close and revealing relationships to $Ca_5(PO_4)_3(F, OH)$ and $K_3Na(SO_4)_2$, in



Proposed structures for polywater are discussed by members of the NBS—University of Maryland team investigating this material.

the case of $\text{Ca}_5(\text{PO}_4)_2\text{SiO}_4$ and to $\text{K}_3\text{Na}(\text{SO}_4)_2$, $\alpha\text{Ca}_3(\text{PO}_4)_2$, and $\beta\text{Ca}_3(\text{PO}_4)_2$ in the case of $\text{Ca}_4\text{Mg}_5(\text{PO}_4)_6$.

Analysis of the Shapes of Polycyclic Carbohydrate Molecules—The shapes of organic molecules are important to an understanding of their reactions. Most carbohydrates occur in cyclic nonplanar forms, but the forces that determine which of several different possible shapes each favors are not well understood. In this work, the shapes of sugar molecules formed by joining together several rings composed of five or six atoms have been studied by nuclear magnetic resonance techniques with computer analysis of the data. Assignment of the total shape of the molecules was aided by newly developed mechanisms, by which an excited nucleus can transfer radiofrequency energy to other nearby nuclei. The results show that electrostatic forces have an important influence on molecular shape.

Determination of Crosslinked Organic Networks—Three-dimensional organic network polymers have been available for several decades, but there have been serious impediments to the determina-

tion of their molecular structures. The copolymers of styrene and divinylbenzene are intermediates in the preparation of the derivative ion-exchange materials. Previously, it has been impossible to measure the styrene/divinylbenzene ratio *after* these compounds have reacted together. This problem has been solved by studying the infrared spectra of the copolymers. Comparison was made to similar spectra taken of NBS Standard Reference Material No. 705, polystyrene. The study revealed the presence of aromatic hydrogen (out-of-plane bending) vibrations, and these features have been identified with the corresponding divinylbenzene cross-linking agent. Measurement of cross-linking in the copolymer is thus feasible, and high-speed computation methods are being developed in order to do this with high accuracy.

Reduced Crystallographic Cells—Methods to obtain a unique reduced cell, once the three shortest noncoplanar lattice translations are known, have been developed in this laboratory. This work was undertaken because in addition to their theoretical interest, reduced cells have two important applications in crystallography: (a) they make it possible to determine the Bravais lattice from an arbitrary primitive cell of the lattice, (b) they provide a method for the classification of crystalline substances.

(a) *Determination of the Bravais Lattice*—If any primitive reciprocal cell can be derived from the x-ray diffraction patterns, then the corresponding direct cell, which is also primitive, can be reduced. The Bravais lattice can be determined from a table which relates the 44 reduced cell types to the Bravais lattices. The use of the reduced cells saves much of the preliminary work normally needed in crystal-structure analysis. The application of the reduced-cell concept becomes especially useful when crystals are grown and studied under special conditions, such as high pressure and high or low temperature, when it is difficult or impossible to determine the lattice symmetry by conventional means. Reduced cells will be applied to high-pressure diffraction studies and to the determination of crystal symmetry with computer-controlled diffractometers.

(b) *Classification of Crystalline Substances*—The present determinative listing of crystalline substances is made on the basis of cell dimensions within each crystal system. The choice of a conventional cell dictated by lattice symmetry is possible in all crystal systems except triclinic and monoclinic. The use of the reduced cell, however, makes it possible to classify uniquely all crystals including those crystallizing in the triclinic and monoclinic systems. The use of reduced cells has been applied to triclinic crystals.

Single-Crystal X-Ray Diffraction at High Pressure—The first successful structure determination by single-crystal diffraction techniques of a high-pressure polymorph in its region of stability has been com-

pleted. The study was conducted on benzene, an extremely important chemical material. Benzene is a liquid under ordinary conditions but crystallizes at about 5 °C. at ordinary pressure. The structure of this crystalline form has been studied extensively at low temperatures by conventional x-ray and neutron diffraction techniques. The crystal is orthorhombic and has a surprisingly complex arrangement of benzene rings. Benzene can be crystallized at room temperature at about 0.6 kbar and it was found that the crystal structure is the same as that obtained at low temperatures. This form is denoted benzene-I. If the pressure is raised on benzene-I, it transforms above 12 kbar to a denser crystalline form known as benzene-II which is stable only under high pressures. This crystal was the object of the present studies.

Single crystals of benzene-II were grown in the beryllium high-pressure diamond anvil cell and studied by precession x-ray diffraction techniques. Considerable effort was expended in an unsuccessful attempt to find the structure of the first crystal of benzene-II. The lack of success forced the conclusion that the intensity data should be checked using a different crystal. Several new crystals of benzene-II were grown and studied and although all crystals showed similar reciprocal lattice networks, the first crystal appeared to have a higher symmetry than subsequent ones. It was concluded that the original crystal was highly twinned. Data from the other crystals showed benzene-II to be monoclinic and the structure was readily obtained using computer techniques developed for use on the original crystal. In the new structure the benzene rings are packed more tightly than in benzene-I and are oriented more nearly parallel. The structure obtained by the diffraction studies was shown to be that arrangement producing the lowest repulsive energy between the closely packed rings. The structure obtained could not have been deduced by any other available techniques—i.e., powder diffraction—because of the low crystal symmetry and the low scattering-power of the benzene molecule.

Accurate Cell Parameter Data from Small Single Crystals—An existing x-ray diffractometer has been extensively modified to facilitate making more accurate cell parameter measurements than can normally be made with small (0.1 mm) single crystals. Because the instrument is intended for use with “ordinary” crystals which generally have sufficient mosaic character to limit the attainable precision, very high accuracy is not expected. The real value of the modification is in preparing crystals for intensity measurements with the automatic diffractometer. In this way the need for using the latter instrument for time-consuming hand measurements in order to establish cell parameters and orientation can be eliminated. Specimen temperature control is planned which will facilitate measurements of anisotropic thermal expansion on a wide variety of crystals. Use of a diamond-anvil high-pressure cell for measurements of cell parameters as a function of pressure is also being considered.

X-Ray Powder Diffraction Data—A program for the calculation of x-ray powder patterns has been started at IMR, and is part of the project for extending and improving data for use in the identification of crystalline phases. Patterns are calculated when it is impractical to produce experimental data because of the nonavailability of a good sample or because of experimental difficulties. Using the published data from the general literature, the d -spacings and relative intensities are calculated.

The intensities are computed as relative peak heights and simulate closely those results which would be obtained experimentally with a diffractometer and Cu radiation. Calculations are only made from highly reliable structural data. The results are published in the NBS Monograph Series of Standard X-ray Diffraction Powder Patterns and are also included in the Powder Diffraction File of the American Society for Testing and Materials.

Electron Microscopy of Rocks—Geologists studying the mechanics of deformation of rocks from the earth or the moon have used optical microscopy to observe deformation textures in their materials. In collaboration with the Geology Department of U.C.L.A., the feasibility of using transmission electron microscopy for studying geological materials was explored. The experimental conditions simulating rock deformation involve testing under high confining pressures and elevated temperatures. With brittle materials such as quartz, the test specimens and also the natural samples often are fractured and recrystallized indicating severe deformation. In order to prepare samples for study by electron microscopy, special thinning techniques were needed. Such techniques have been developed and used for many ceramic materials at NBS. By using ion bombardment, thin foils suitable for transmission electron microscopy were prepared from petrographic thin sections. Dislocation arrangements associated with plastic deformation in quartz rocks were observed directly; and some of the results were compared with observations made by optical microscopy.

Effect of Impurities on the Crystal Structure of Tantalum Pentoxide—Oxide compositions containing tantalum and other cations of similar size have been investigated at high temperatures. The smallest cations have been found to stabilize low temperature forms of Ta_2O_5 whereas slightly larger cations were found to stabilize the high temperature polymorph. Crystal structure analysis of the high-temperature form has revealed a completely new structure type. The crystal structure of the low-temperature form was found to be dependent upon the amount of smaller cations added to Ta_2O_5 and upon the total stoichiometry. Both play an important role in determining the number and type of polyhedra necessary to make up a unit cell.

Lattice Defects in Al_2O_3 by Transmission Electron Microscopy—

At low temperatures aluminum oxide is generally regarded as a purely brittle material with plastic deformation due to the generation and movement of dislocations possible only at temperatures in excess of 900 °C. However, by using an argon ion-bombardment technique to thin specimens in a controlled manner for examination in the electron microscope, it has been shown that plastic deformation does occur under hardness indentations at room temperature and that mechanical polishing introduces a high density of dislocations within a surface layer less than one micron thick. Direct evidence for plastic deformation at temperatures less than 900 °C has not been reported previously. Although the large-range movement of dislocations does not appear possible at low temperatures, the presence of such high densities of dislocations in the surface regions of mechanically polished single and polycrystalline samples of aluminum oxide is expected to alter their mechanical, optical, and chemical properties and, therefore, must be taken into consideration.

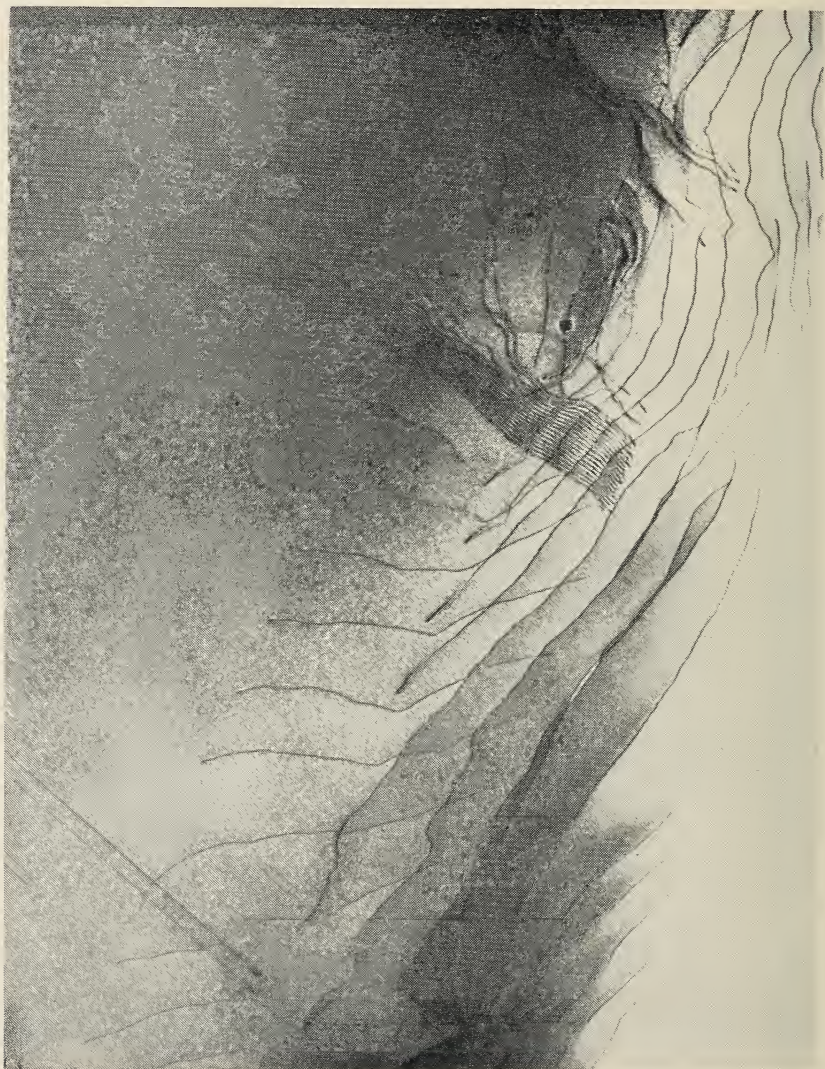
Because of their relatively high strength and chemical inertness even at high temperatures, ceramic materials such as aluminum oxide are important structural materials. Studies on the mechanical properties are, therefore, essential and a more complete understanding of these properties can be obtained by detailed investigations of lattice defects, such as dislocations, and their role in the deformation process. Since lattice defects also affect the optical properties, these studies are important to materials with laser applications.

*New Diffraction Theory for Imperfect Crystals—*A crystal which contains imperfections—such as dislocations, defects and impurities—diffracts x-rays to give fine structure detail in the diffracted beams which represents topographic images of the imperfections. The intensity of the diffracted beams also changes from the dynamical value to the kinematical value (known as the extinction effect), depending on the degree of crystal imperfection.

Until recently there has been no theory to explain the topographic images and the change in diffraction intensities (the extinction effect) in terms of the properties of existing imperfections. For example, the existing extinction theory requires the famous mosaic block model to represent crystal imperfections.

A new dynamical diffraction theory for imperfect crystals has been developed by IMR scientists. The theory has been derived from a quite general view of scattering. The ordinary dynamical theory, which is valid only for a perfect crystal, is merely a special case in this new dynamical theory of diffraction. Establishing such a theory is important, not only for determining the properties of crystal imperfections in a real crystal, but for crystal-structure determination with single crystals, where the extinction effect is the most difficult factor to estimate.

Characterization of a Hexagonal Alloy—A series of hexagonal silver-tin alloys has recently been characterized in terms of the defect dislocation structures and the stacking fault energy, using the technique of transmission electron microscopy. The electron microscope permits the direct observation of individual dislocations and stacking faults. Alloys having several different compositions were plastically



An example of the dislocation structures found on close-packed crystal planes in a silver-tin hexagonal alloy is seen in this transmission electron micrograph. Several examples of close-spaced dislocation multipoles are present, interacting with two distinct sequences of glide dislocations produced by plastic deformation. $M=30,000X$.

deformed to introduce dislocations, then examined. The studies concentrated on the variation in stacking faults energy with alloying, and on the importance of the several different dislocation glide systems that operate. Interactions between different groups of dislocations were studied and the results interpreted in terms of the known mechanical behavior.

Dislocations in Crystals—One way in which dislocations contribute to the mechanical properties of materials is as sources of residual or internal stress. A general theory of static sources of internal stress has been developed for finite anisotropic elastic bodies, including structures with complex topology. Using a projection formalism, the stresses and strains due to dislocations and “welded in” sources of internal stress have been calculated.

Another crystalline defect, a source of internal stress of somewhat higher complexity than the dislocation, is the disclination. A linear continuum theory of continuous distributions of static disclinations has been developed for the first time. This theory also relates to other developments in continuum mechanics.

STANDARD REFERENCE MATERIALS

From a beginning in 1906 with four standard samples of cast iron, the NBS Standard Reference Materials Program, which made the United States the first country with a national program of this kind, has grown to the point that almost 700 different SRM's are now produced or are nearing completion. These include 8 new categories available for the first time this year—labelled organic compounds, electrical and magnetic standards, ion activity standards, electron microprobe standards, certified analyzed liquids, high-purity metal standards, certified gas standards, and electronic and magnetic alloys—in addition to the 70 categories already available. These are the result of the continuing effort to keep the Standard Reference Materials program abreast of the growing and changing needs of the times.

This year has been one of continued growth and expansion of the SRM program. Maintaining a balance between providing renewal SRM's for industries which now rely heavily on SRM's, and responding to new national needs in areas such as health, air and water pollution, resources development and the like, has been difficult. That some balance has been achieved is indicated by the fact that during 1968, 66 new and 74 renewal SRM's were issued, and 54 SRM's were discontinued because the need for them had diminished or new SRM's were judged to better serve the overall interests of the Nation's science and technology. There were in stock at the end of 1968, 651 different items in 59 distinct categories totaling over 380,000 units. Work in

progress to produce 318 new and renewal SRM's was centered in 146 projects. Sales during this calendar year to 8,000 customers totalled just over \$1,000,000.

Examples which follow have been selected from the 44 new and 21 renewal SRM's completed and certified during fiscal year 1969 by 13 technical divisions of NBS.

High-Purity Metals—SRM 685—a new high-purity gold SRM was issued in wire form for spark-source spectrometry, in rod form for other methods of characterization and for reserach which requires a high-purity, homogenous standard material.

A high-purity gold vapor pressure SRM (SRM 745) also was issued for which the vapor pressure was accurately determined over the pressure range of 10^{-3} to 10^{-8} atmospheres and over the temperature range 1300–2100 °K.

The high-purity gold is issued with a Certificate of Analysis which gives "state of the art" information on the chemical composition, including values for coper, indium, iron, oxygen, and silver. Estimated limits of concentration are provided for 21 other elements which were detected by spark-source mass spectrometry. Extensive homogeneity testing was performed to establish that the gold was satisfactory within the precision of the analytical methods used. These included neutron activation, polarography, spectrophotometry, spark-source mass spectrometry, isotope dilution, and vacuum fusion in addition to optical emission spectrochemical analysis and residual resistivity ratio measurements.

The gold vapor-pressure SRM represents the first in a series of elements which will be certified for vapor pressure in the 10^{-3} to 10^{-8} atmosphere range. The series will cover the temperature range 600 to 3000° K.

In the field of high-temperature vapor pressure measurements there has been a need for standards. It is not uncommon for vapor-pressure measurements made by experienced investigators to differ by 30 percent, 50 percent, or even 100 percent. The gold vapor-pressure SRM was developed for use in the testing and calibration of vapor-pressure measurement apparatus and techniques, and for use in the detection of possible systematic errors. It also can be used for direct interlaboratory comparisons. The homogeneity of the gold was confirmed by extensive testing.

Biomedical Standards: SRM 912, 913, 914, 915—Four additional NBS Standard Reference Materials were certified as chemicals of known purity for use in calibration and standardization of procedures used in clinical laboratories. These supplement cholesterol, which was made available previously. The new SRM's are: urea, certified at 99.7 percent purity; uric acid, certified at 99.7 percent purity; creatinine, certified at 99.8 percent purity, and calcium carbonate, certified at 99.9

percent purity. The value of the purity of each of these SRM's has an estimated inaccuracy of 0.1 percent.

The homogeneity of each of the lots from which these standards were prepared was tested and proved satisfactory by chromatographic and spectrometric techniques. These methods also showed the materials to be free of measurable amounts of organic impurities, with the exception of a small amount (<0.07 percent) of biuret in the urea.

Spectrometric analyses of the ash obtained from each of the first three of these SRM's indicated only minor concentrations of the several metallic elements found. These results were substantiated by neutron activation analyses of the bulk materials.

The purities of the urea and creatinine were determined by phase-solubility analysis, and for the former, was also confirmed by differential-scanning calorimetry. The purity of the creatinine was further evaluated by setting limits on amounts of possible impurities that would be undetected by the chromatographic and spectrometric techniques employed. No organic impurities could be detected in the uric acid even after the application of techniques that should have concentrated the impurities, had they been present.

The calcium carbonate is a very high-purity material intended for use as a standard for all clinical determinations of calcium. Like the other four SRM's in the series it was prepared at the request of the College of American Pathologists and the American Association of Clinical Chemists. They placed a high priority on the certification and issuance of the calcium standard.

The rapid and accurate analysis of calcium in serum is an important clinical test. Hypercalcemia, when supported by other clinical evidence, is often indicative of hyperparathyroidism, and medical decisions involving immediate surgery are often made on the basis of the serum calcium value. The imperative need for accurate calcium determinations is obvious.

In clinical procedures, calcium is determined, usually as a calcium-fluorescent complex, by fluorometry, by flame emission, or by atomic absorption methods. The NBS calcium carbonate SRM will serve as the primary standard for the calibration of the instruments and methods used.

The material was examined for compliance with the specifications for reagent-grade calcium carbonate as given in *Reagent Chemicals*, 4th edition, published by the American Chemical Society. The material was found to meet or exceed the minimum requirements in every respect. Examination by thermal gravimetric analysis indicated the loss of a minute proportion of weight below 175°C (volatile matter), and the composition was stable above this temperature up to a temperature of 625°C , above which decomposition (evolution of CO_2) set in.

Replicate samples taken from a randomly selected region of the undried material were assayed by a coulometric acidimetric procedure. The results from nine independent determinations, based on expression of the assay as calcium carbonate, indicate a purity of 99.99+ percent with a standard deviation of 0.003 percent. Samples equilibrated at a relative humidity of 90 percent and assayed by this coulometric procedure showed a maximum moisture absorption of 0.02 percent as compared to samples that were dried for 6 hours at 210 °C. The moisture content on samples equilibrated at 75 percent relative humidity was found to be 0.01 percent. This water content was determined by the Karl Fischer method.

A semiquantitative survey for trace contaminants by emission spectroscopy indicated the presence of less than 0.001 percent of copper, iron, magnesium, manganese, and silicon in the material. By atomic absorption, magnesium was evaluated at 1.0, sodium at 0.4, and strontium 2.1 ppm; potassium was less than 0.4, lithium less than 0.05, and barium much less than 10 ppm. Neutron activation analysis indicated copper 0.9, manganese 0.6, and sodium 0.5 ppm. Copper was determined at 1 ppm by spectrophotometry.

Electronic and Magnetic Alloy Standards: SRM 1159, 1160—Two new nickel-base SRM's—widely used in the electronic and magnetic industries—were made available. One is a nominal 49 percent nickel, balance iron alloy, and the other is a nominal 80 percent nickel, 4 percent molybdenum, balance iron alloy.

These two new SRM's were issued meet a serious deficiency of standards for both producer and consumer of electronic and magnetic alloys. It is estimated that by 1975 the use, e.g., of permanent magnets will double from the present annual \$50 million market. Permanent magnets are in wide use in such applications as battery operated hand tools for home use, motorized garden and kitchen equipment, etc. These applications and others, where permanent-magnet motors are preferred for reasons of reliability and efficiency, are on a steeply rising growth curve.

Designed primarily for calibration in optical emission and x-ray spectrometric methods of analysis, the standards were issued in disk form 1¼ in. (3.1 cm) in diameter and ¾ in. (1.9 cm) thick. Lathe chips may easily be prepared from the disks in the user's laboratories, if desired, for checking chemical methods of analysis both for production control and for customer acceptance.

The material for the standards was prepared in a vacuum induction furnace and supplied to NBS in final form following a scheme of fabrication designed to produce material of the highest possible homogeneity.

Extensive homogeneity testing was performed in the Institute for Materials Research, and both materials were found to be satisfactory within the limits of precision of the analytical methods employed.

The standards were issued with a Provisional Certificate of Analysis certified for carbon, manganese, phosphorus, sulfur, silicon, copper, nickel, chromium, molybdenum, cobalt, and iron.

Ferrous Materials Standards: SRM 178, 1093, 1094, 340—A new chip-form carbon steel SRM was the first steel made by the basic oxygen furnace process to be certified. Two new steel SRM's were certified for oxygen and nitrogen and a new ferroalloy ferroniobium for niobium and other elements of interest.

The basic oxygen steel is a plain carbon grade. This was the first type to be certified by NBS as Standard Reference Materials. These first SRM's included steels produced by the acid Bessemer, the acid open-hearth, and the basic open-hearth furnace processes. A strong demand for these standards has existed for a period of more than 60 years, and has increased rapidly in recent years. The more stringent chemical-composition specifications in the steel industry and new, more rapid kinds of analytical equipment that rely on comparison techniques with standards of known composition are responsible for this demand. Furthermore, the demand is expected to grow with the continuing growth in use of both the basic oxygen furnace and the continuous-casting processes of steel manufacture. These relatively new commercial processes place urgent demands on the analytical laboratory for data within a very short elapsed time from sampling to the reporting of the final analysis.

The primary consideration in preparing steel Standard Reference Materials is the resultant chemical composition of the steel, not the method by which the steel is produced. This is fortunate, for in the United States, the acid open-hearth process is no longer used commercially, the use of the acid Bessemer process is rapidly diminishing, and the basic open-hearth is being superseded or supplemented by basic oxygen and electric furnaces. In the near future, steel for preparing renewal standards of the plain carbon steels may be available most readily from the basic oxygen furnace process.

The new SRM 178, chip form, carbon steel is intended for the same purposes for which the Acid Open Hearth Steel SRM 20f was used.

The role of oxygen and nitrogen as they affect the basic properties of metals—e.g., brittleness, ductility, etc.—is well established. A maraging steel can be produced which is dimensionally stable; oxygen present as inclusions affect this stability, especially in high pressure applications. It is therefore important that SRM's be available with known oxygen and nitrogen content to calibrate the various instruments or methods now used to determine these gases.

The two new SRM's certified for oxygen, SRM 1093 and SRM 1094, also have supplemental information given on the nitrogen content. A revised certificate of analysis was issued for SRM 1090, Oxygen in Ingot Iron, SRM 1091, Oxygen in Stainless Steels, and SRM

1092, Oxygen in Vacuum Melted Steel. For these three SRM's, oxygen values obtained by neutron activation and inert-gas fusion are included to support the values obtained by vacuum fusion and used on the previously issued certificate of analysis.

SRM 1093, with a value of 60 ± 5 ppm for oxygen (certified) and a nitrogen value of 4807 ± 10 ppm (not certified), is intended primarily for use as a standard for the neutron activation method for determining oxygen in steel. The oxygen was determined in a 2-inch-length of rod cleaned ultrasonically for 30 min in trichlorethylene, then dried in dry nitrogen. The rods were activated, together with standards of known oxygen concentration, in the NBS 14 MeV neutron generator. ^{16}N (7.2 s half-life) formed from the $^{16}\text{O}(n,p)^{16}\text{N}$ nuclear reaction was counted and the induced radioactivity compared to that of the irradiated standard.

The determination of oxygen in this particular steel by vacuum or inert-gas fusion is difficult because of the gettering action of the manganese which is of the order of 9 to 10 percent.

Although not certified at this time, nitrogen at 4807 ± 10 ppm was determined by a pressure bomb-distillation-indophenol-titrimetric method. Determination by vacuum fusion gave a value of 3540 ± 76 ppm of nitrogen. It is believed that the higher value is the more accurate.

The maraging steel, SRM 1094, is intended for the determination of oxygen. The low value of oxygen, 4 ± 0.4 ppm, makes determination by neutron activation difficult; present configuration of the NBS neutron generator precludes an accurate determination of oxygen at this level. Redesign of the equipment to increase the sensitivity of the technique at these low levels is now underway at NBS.

The vacuum-fusion oxygen value is the result of 60 determinations on 12 samples of the standard. One-gram samples were carefully cut from the rod material, cleaned, washed and dried. A furnace temperature of 1675°C and a vacuum of $<10^{-5}$ torr was maintained during the 4 min collection time from a high-purity nickel bath.

The nitrogen content, not certified at this time, was found to be 70.7 ± 3.2 and 61 ± 1 ppm for the pressure bomb-distillation-indophenol-photometric method and vacuum fusion technique, respectively.

Three SRM's—1090, 1091, and 1092—were certified for oxygen in March 1966. Since that time the inert-gas fusion and neutron activation methods have been studied and applied to the analysis of oxygen in these three SRM's. The results of this study are summarized on a revised certificate of analysis, together with the certified oxygen values, which remain essentially unchanged, but which are now supported by the additional analyses provided by the aforementioned methods.

Additional information on the nitrogen values of these three SRM's

is also provided, but the values were not certified. The nitrogen values were arrived at by four different analytical methods.

From the nitrogen results on the five SRM's, it was apparent that systematic biases exist in one or more or possibly all of the methods used for determining nitrogen. There was some evidence that nitrogen results by one or more of the methods are material-dependent. The complexity and magnitude of this effect is not now known. A comprehensive research effort is underway at NBS to resolve this question and to search out other possible systematic errors. When this research is completed these standards will be certified for their nitrogen contents.

At the request of the ferroalloy producers and Division F of ASTM Committee E-3, a ferroniobium, SRM 340, was prepared. The new SRM complements the list of other steel-making alloys that are available from NBS. Ferroniobium is an important inoculant in the steel production process, which is finding increasing use not only in stainless steel as a carbide-stabilizing element but also in carbon steels for purposes of hardenability. The ferroniobium standard certified as to its chemical composition is vitally important to the iron and steel industry since the ferroalloys are largely bought and sold on a contained basis for the alloy constituent. On the basis of analyses performed by NBS and by a number of cooperating laboratories, the new SRM is certified for niobium, tantalum, titanium, carbon, manganese, phosphorus, and silicon.

Computer Amplitude Reference Tape Standard: SRM 3200—An amplitude reference magnetic tape for use in the signal amplitude calibration of computer tape standard recording and reproducing systems was made available as a new SRM.

The characteristics of present-day magnetic computer tapes are the result of long-term research and development by the magnetic tape industry. Technological developments in related fields, and transducer-tape interplay, have stimulated continuing improvement of the tape media and its performance. This has often led to differences in tape characteristics and marginal operation when tapes are interchanged between systems. The only tapes previously available for quality assurance purposes have been industry-supplied standards.

At the request of industrial users of computer tape, Government procurement agencies, producers of magnetic computer tapes, and producers of tape-handling equipment, NBS has developed tape-measurement methods and will supply a "Secondary Standard Magnetic Tape-Computer Amplitude Reference," through the NBS Office of Standard Reference Materials.

This new material, consists of a 600-ft length of secondary reference tape, applicable test and calibration data, and a description of the equipment and procedures employed for measurement of the tape.

The secondary tapes themselves are 1/2-in wide unrecorded magnetic computer tapes wound on 8 1/2-in diameter precision reels.

Each tape will be calibrated in terms of 100 percent signal level, referred to the NBS Master Standard Magnetic Tape—Computer Amplitude Reference, which will be kept in repository at NBS. This signal-level calibration will be made at recording densities of 200, 556, 800, and 3200 flux reversals per inch, and calibration information in the form of signal output charts recorded near each edge and at the center will accompany each tape. A set of saturation curves relating the reproduce head output voltage (on the first read-after-write pass) to the write current at each bit density of both the NBS Master and each secondary tape will also be included. The write current will range from zero to at least 150 percent of the saturation current level. The measurement processes are being described in an NBS Special Publication.

Presently, the main criteria for the selection of amplitude reference tapes for calibration as secondaries are signal-amplitude uniformity and proximity of read-after-write output voltage to that of the Master. The instantaneous track-to-track variation in the average signal amplitude is limited to a maximum of 3 percent among the outputs from the NBS test tracks 2, 5, and 8. The location of these tracks is defined in ANSI Document X3.22-1967. The maximum variation in the longitudinal average signal output voltage along each test track is limited to 4 percent over the 600-ft length of tape. The absolute value of the average signal amplitude from each test track is within 10 percent of the NBS Master Standard Magnetic Tape-Computer Amplitude Reference. These criteria are applied for saturation current recording on degaussed (fully AC-erased) tape only.

Plutonium Assay Standard: SRM 944—The Office of Standard Reference Materials made available a new standard reference material, plutonium sulfate tetrahydrate, containing 47.50 percent of plutonium. This material was issued to provide a secondary standard for the assay of plutonium materials to supplement the presently issued plutonium metal standard. Each unit of the new SRM contains the equivalent of 0.5 g of plutonium.

Plutonium is a fissile material having impact on peaceful uses, particularly in power reactors, as well as military applications, of nuclear energy. Accountability problems are especially severe and will become even more so with the passage of time. The estimated inventory of plutonium by 1973 from domestic licensed facilities is 12,200 Kg.

Because of the stringent requirements for handling plutonium metal, the plutonium sulfate should prove to be particularly attractive for assay work requiring an accuracy of the order of 0.1 percent. The material is relatively insensitive to humidity at room-temperature over the range from 0 to 90 percent relative humidity, and provides a

solid from which appropriate-sized subsamples can be weighed easily for any particular use.

Electron Microprobe and Microchemical Standards: RM 480, 481, 482—The first three standards designated specifically for application to electron probe microanalysis were issued by the Office of Standard Reference Materials.

The first of these, a composite consisting of a tungsten-20 percent molybdenum alloy wire core embedded in a pure molybdenum rod onto which there had been electro-plated a layer of pure tungsten was prepared through the use of powder metallurgy. High-purity metal powders were used to produce a standard of the maximum homogeneity. Based upon the results of approximately 1,500 determinations of both tungsten and molybdenum by the electron microprobe, the material was found to be of high homogeneity of about the micrometer level of spatial resolution. A number of microprobe studies on this standard were made in IMR, including: (1) correction of the relative-intensity ratios to obtain concentrations in terms of input parameter uncertainties such as mass absorption coefficients and electron backscatter factors, and (2) effects of operating voltage on the microprobe absorption and atomic number correction.

Companion sets of six gold-silver and six gold-copper SRM's were certified for chemical composition and homogeneity. These sets are in wire form, 0.5 mm in diameter, and 5 cm. long, with colored coatings to identify the individual chemical composition.

The compositions of the alloy were chosen especially for direct calibration of the electron-probe microanalyzer for the gold-silver and gold-copper systems and for the testing of theories of correction calculations for electron-probe microanalysis. Because of the high homogeneity and well-determined composition, the standards will also be useful for other methods of microanalysis, including laser-probe and optical emission spectrometry, and spark-source mass spectrometry.

Standard reference materials for microanalytical methods have well-determined composition, and must be homogeneous on the microscopic level on which the analysis is performed. These sets of SRM's now offered by NBS is the result of a two-year program, including investigation of the compositions needed for the standards, their careful preparation, and their characterization. The materials were prepared from highest-purity gold, silver, and copper and the alloys were heat-treated at NBS to obtain maximum homogeneity.

Precautions were taken to minimize contamination. The alloy standards, in the form of wires, were heat-treated at NBS to improve microhomogeneity. The pure metal standards were examined by the residual resistance ratio technique and the total of active impurities in each was estimated to be about 0.001 percent. The final standards were

examined spectrographically for metallic impurities; no significant impurities were found at detection limits ranging from 0.0001 to 0.010 percent.

Variation in composition along the full length of each alloy wire was investigated by electron probe microanalysis, using a specially designed automatic data collection system for areas $25\text{ }\mu\text{m}$ in diameter at the two ends and at two evenly spaced intermediate positions. The observed differences in composition for the four positions, expressed as the range between the highest and lowest values for each alloy, were close to the repeatability of the method.

Homogeneity along the wires was also tested by measurement of the residual resistance ratio. These measurements indicated that the (macroscopic) variation of composition along all standard wires did not exceed 0.2 percent.

Variation in composition with the cross section of the wires at the four positions along the wire was also investigated by electron probe microanalysis. For each cross section, measurements were made along two diagonals at right angles. On each diagonal, determinations were made at 25 points, $1\text{ }\mu\text{m}$ or less in diameter, starting and ending at approximately $25\text{ }\mu\text{m}$ from the edge. For each alloy, the element which could be determined with the better precision was used in the evaluation. The variations was calculated in terms of the standard deviation for an individual determination for each traverse.

The homogeneity on a microscopic scale was further investigated by performing quantitative measurements in two-dimensional assays of 10×10 points on each of the four cross sections. The distance between adjacent points was $3.5\text{ }\mu\text{m}$. This was repeated for three of the cross sections so that 6 to 8 arrays were obtained on each alloy.

Isotopic Standards and Neutron-Density Monitor: SRM 951, SRM 952, SRM 953—Two new boron isotopic SRM's—a normal boron and a boron-10 standard—were prepared. The first is a boric acid of high purity and homogeneity for use in calibrating equipment and measuring the cross section of the boron (n,α)-Lithium [$\text{B}(n,\alpha)\text{Li}$] reaction. This cross section, particularly at thermal neutron energy (corresponding to a velocity of 2200 m/s) is one of the most important standard neutron cross sections used to calibrate equipment that measures neutron flux density.

The new boron isotopic standard was prepared from a lot of boric acid that is uniform in isotopic composition and that adjusts to a stoichiometric composition after about 30 minutes exposure to normal room humidity (approximately 35-percent relative humidity).

The boric acid has an acidimetric assay of 100.00 ± 0.02 weight percent and an absolute abundance ratio of $^{10}\text{B}/^{11}\text{B}=0.2473\pm 0.0002$.

The assay was performed by coulometric titration of samples varying in size from 0.2 to 1.0 g of boric acid, dissolved in 100 ml of a

preneutralized solution of 1 *M* in KCl and 0.75 *M* in mannitol. The inflection-point of the potentiometric curve obtained from measurements with a glass-calomel electrode system was taken as the end-point. The pH of the maximum inflection-point will vary from approximately 7.9 to 8.5 for the range of samples sizes given above. The titration was conducted in the absence of carbon dioxide. The indicated tolerance is at least as large as the 95-percent confidence level for a single determination of any sample in the lot of material.

The average essentially indicates a boron-hydrogen ion ratio of 1.0000, as separate examination shows the material contains less than 0.001 percent of free strong acid.

The abundance ratio was determined by single filament solid-sample mass spectrometry, using the ion Na_2BO_2^+ . Mixtures of known $^{10}\text{B}/^{11}\text{B}$ ratio (at a 1:4, 1:1, and 4:1 ratio) were prepared from high-purity separated isotope solutions and were used as comparison standards. A correction was made for the $^{16}\text{O}/^{17}\text{O}$ ($^{11}\text{B}/^{10}\text{B}$ ratio - 0.00079). The limits of error are based on 95-percent confidence limits for the means of the ratio measurements and on allowances for the known sources of possible systematic error.

The second SRM, a 95-percent boron-10 boric acid, was issued to supplement the normal boric acid and provides materials for the calibration of mass spectrometers at the 95-percent boron-10 level.

The boric acid has an absolute abundance ratio of $^{10}\text{B}/^{11}\text{B}$ of 18.80, and an acidimetric assay of 99.97 percent. The material is useful as a "spiking" material for the determination of boron by isotopic dilution, as well as for the calibration of mass spectrometers.

Many of the power reactors contain control systems involving boron, so the material should be particularly useful in the nuclear power field as well as in the determination of "trace element levels" in the ferrous metals and agricultural fields.

A description of the various methods used in the characterization of the normal and boron-10 enriched boric acid SRM's is published in full detail and available in NBS Miscellaneous Publication 260-17.

A new SRM, a cobalt in aluminum alloy, for the measurement and standardization of neutron densities was certified.

The accurate determination of thermal neutron densities is essential in irradiation tests in obtaining a basis for comparison of neutron densities among reactors, in applying data in the design of reactors, and in understanding the mechanisms of radiation damage. Cobalt alloys, particularly 0.1 wt percent cobalt in aluminum, have been widely used to measure thermal neutron densities in almost all irradiation experiments on materials. Of increasingly major importance, however, has been the need for a material of the highest possible homogeneity for which an accurate determination of the cobalt content is provided.

This SRM also will prove useful to the activation analyst for map-

ping relative variations in neutron densities along the length and width of irradiation containers, for determining the integrated neutron flux density (insofar as small lengths approximate a sphere), and for determining long-term variations of the neutron density at given irradiation positions. For most applications, these measurements will be made on a relative rather than an absolute basis.

The neutron-Density Monitor Wire was designed to meet these needs. It is a cobalt in aluminum wire 0.5 mm in diameter, which was prepared using specially selected high-purity starting materials. Based on results obtained at NBS by activation analysis and spectrophotometric analysis, the cobalt content is certified at 0.116 wt percent.

Extensive homogeneity testing of the wire was performed by a variety of methods at the NBS laboratories in Gaithersburg, Maryland, and Boulder, Colorado. The wire material selected for certification was found to be homogeneous for sample lengths of 1 mm or longer.

Acrylonitrile—Butadiene Rubber Standard: SRM 391—For many years the Office of Standard Reference Materials has made available to the rubber industry rubbers and other materials for rubber-compounding. The SRM's currently supplied are natural rubber (NR), two types of styrene-butadiene rubber (SBR), and butyl rubber (IIR). A new rubber is now added to this group, namely, acrylonitrile-butadiene rubber (NBR).

This rubber is intended for use in the standardization of testing among laboratories and as a reference rubber in quality control and testing for resistance to deterioration by oil, gasoline, and other hydrocarbon products.

The NBR used for the new SRM contains approximately 33 percent combined acrylonitrile. The rubber is wrapped in polyethylene film, and packaged in multiwall paper bags containing 25 kgs. The uniformity of the lot was established by tests on samples taken after each tenth package during manufacture.

The Mooney viscosity of this rubber was determined to be 49.0 ± 1.0 ML 1+4 (100 °C), using a modification of ASTM Designation D1646-68.

Compounds for evaluation were prepared in accordance with the formulation and mixing procedure described in ASTM Designation D15-66T for Standard Formula 1F using NBS Standard Reference Compounding Ingredients. The viscometer cure characteristics of each compound were determined at 150 °C according to ASTM Designation D1646-68. The minimum viscosity, incipient cure time, and cure index (the time required to increase from 5 to 35 points above the minimum) were measured.

The remainder of the compounds were vulcanized according to ASTM Designation D15-66T and measurements were made on these

vulcanizates for stress at 300-percent elongation, stress at failure, and elongation at failure (ASTM D412-68), strain under a stress of 400 pounds per square inch (2.75 meganewton per square meter) (ASTM D1456-61), and electrical resistivity [Ind. Eng. Chem. **44**, 159 (1952)].

This new acrylonitrile-butadiene rubber standard complements the other rubber and rubber-compounding SRM's available from the NBS Office of Standard Reference Materials.

Deuterated Hydrocarbons Standards: SRM 2175, SRM 2176—The Bureau announced the availability of two new deuterated hydrocarbon SRM's, ethane- d_6 and propane-1,1,1- d_3 . They are certified as chemical standards and are intended primarily to aid research workers in the analysis of deuterated or partially deuterated hydrocarbons.

In kinetic studies (pyrolysis, photolysis, radiolysis, etc.) in which deuterium labeling of reactants is used, partially deuterated molecules are formed as products. In order to specify the isotopic structure of these molecules, comparison mass spectrometric cracking patterns are determined in the same instrument in which the actual analysis of the product is made, since each commercial instrument shows a different behavior. It is for the determination of these comparative cracking patterns that these SRM's are intended.

The deuterated hydrocarbons were prepared by the photolysis of ketones and purified by a combination of gas chromatography, absorption, and distillation. Chemical purity was determined by gas chromatography and isotopic purity by mass spectrometry, and consideration of reactions entered into by the reagents.

The ethane- d_6 has a chemical purity of greater than 99.9 mole percent; the only detectable impurity being a trace of propane. Its isotopic purity is 99.8 ± 0.05 atom percent deuterium combined as 98.9 mole percent C_2D_6 and 1.1 mole percent C_2H_5D .

The propane-1,1,1- d_3 has a chemical purity of greater than 99.9 mole percent; the detectable impurities identified as approximately 0.03 mole percent ethane and 0.01 mole percent ethylene. Its isotopic purity is 99.4 mole percent $CD_3CH_2CH_3$ and 0.6 mole percent $CD_2HCH_2CH_3$.

Cyclohexane Dielectric Constant Standard SRM 1511—NBS issued Cyclohexane as the first of three liquids to be certified by the Office of Standard Reference Materials for dielectric constant standards. The dielectric constant of this material was measured at 20, 25, and 30°C and it is felt that calculated values can be used from 10 to 40°C without introducing appreciable errors. This SRM is useful for the determination of the geometric capacitance of two terminal dielectric constant cells and for checking the linearity of the geometric capacitance of the three-terminal, or absolute, cells.

The dielectric constant of most materials is commonly defined and measured as the ratio of the capacitance of a capacitor immersed in

the medium in question to that in vacuum. Because most test capacitors (cells) are nonideal, for accurate work they must be calibrated using air and one or more materials of known dielectric constant.

Reference Materials for Thermochemistry: SRM 724—Determinations of the heats of solution of a standard reference material, *tris* (hydroxymethyl)amino methane (TRIS) were carried out in a highly precise adiabatic solution calorimeter and used for preparation of a certificate of the heating value. This reference material (Standard Sample No. 724) is very much needed by scientific workers in the thermochemistry of solution processes, in order to detect and eliminate sources of systematic error. Such systematic errors have caused numerous discrepancies among measurements made in different laboratories. The precision of the recent measurements is somewhat better than 0.05 percent, and illustrates the quality of the measurements that can be made with this recently developed calorimeter. Both the heat of reaction with dilute HCl (ΔH negative) and with dilute NaOH (ΔH positive) have been determined, thus providing comparison reactions for both endothermic and exothermic reactions.

New Chemical Microstandard: SRM 1800—Problems encountered in the calibration of sensitive chemical analytical instrumentation have been resolved by a recent IMR discovery that individual ion-exchange beads can be used to hold extremely small and accurately known amounts of matter. Techniques have been developed for preparing beads containing from one nanogram (10^{-9} g) to less than a billion atoms. The preparative methods include the use of "clean" conditions for loading and isolating the beads. The first microstandard



Standard reference materials being prepared by the dispersal of chemically loaded ion-exchange beads on glass slides in a clean room.

has now been made available to the public. It consists of calcium ions loaded onto cation exchange beads in amounts ranging from 10^{-9} to 10^{-11} g. The material is designated Standard Reference Material No. 1800. Microstandards for a series of other elements are in preparation.

Oceanographic Conductivity Standards—The electrical conductivity of sea water provides the most precise and convenient measure of salinity available to the oceanographer. For the reliable identification of water masses and for the calculation of related properties such as density and sound velocity, measurements of conductivity are needed with a precision comparable with that of the best research measurements on electrolyte solutions. A series of sodium chloride solutions of certified conductivity has been prepared for use in calibrating oceanographic salinometers. The solutions have temperature coefficients of conductivity sufficiently close to those of sea water to have practical utility, and yet they are more readily prepared and characterized in terms of concentration and purity than either natural or synthetic sea water. With conductivities known to 0.01 percent, these reference standards are expected to find wide application in oceanographic work.

DATA ON THE PROPERTIES OF MATERIALS

This program stresses the precise measurement of materials properties and the development of new and improved measurement techniques. It embodies experimental and theoretical investigations of chemical and physical phenomena of importance to science and industry, and seeks to correlate chemical and physical properties of materials with their composition and structure.

Atomic and Molecular Data

Infrared and Ultraviolet Spectroscopic Studies of Reactive Molecules Isolated in Inert Solid Matrices—Using matrix-isolation techniques, the stretching fundamentals of SiCl_2 and SiCl_3 and all of the vibrational fundamentals of SiF_2 and SiF_3 have been observed. This permitted an estimate of the approximate structure of these species.

In ultraviolet studies of the reaction of photolytically produced H or D atoms with CO at cryogenic temperatures, the absorption counterpart of the hydrocarbon flame bands has been observed for the first time. The attribution of this band system to HCO has been confirmed. Both the CO-stretching and the HCO-bending vibrations have been found to be appreciably excited in this transition. The observation of the ground-state vibrational fundamentals of HCO in infrared spectroscopic studies made possible a detailed assignment of the hydrocarbon flame bands.

A furnace was developed for studies of the reaction of gases with high-temperature metal surfaces. Several of the first-series transition-metal (Cr, Mn, Fe, Co, Ni) dichlorides have been trapped in inert solid matrices in sufficient concentration for direct observation of their stretching fundamentals under conditions such that interactions with neighboring dichloride molecules are virtually completely eliminated. Two previously unreported electronic transitions have been observed between 500 and 440 nm for NiCl_2 , one of them with extensive vibrational structure.

Photolysis of matrix-trapped alkali metal atoms has provided a source of electrons in the matrix. These were used to interact with ground state C_2 species in an argon matrix to give C_2^- . A reassignment of the adsorptions previously ascribed to the Swann transition resulted. In a similar manner the species NO_2^- has been stabilized in sufficient concentration for the direct observation of one vibrational fundamental.

Magnetic Resonance of Copper in Rutile—The magnetic resonance spectrum of divalent copper ions in titanium dioxide (rutile) was examined in detail. Information was gained about both isotopes of copper and the interactions of the ion with the host crystal. The experiments determined the electronic g -factors, magnetic hyperfine coefficients, and the electric quadrupole coupling parameters. The magnitudes of the quantities were obtained experimentally and the absolute signs predicted theoretically from a model which gives a consistent picture of the ordering of the electronic states of the copper ion. The theory includes the effects of the orthorhombic crystal field and covalent sharing of the valence electrons of the copper.

Development of a Highly Stable Pulsed NMR Spectrometer—Scientists in IMR have recently developed an extremely stable pulsed NMR spectrometer which has made it possible to perform meaningful Carr-Purcell experiments. Such experiments are used to measure dynamic processes in molecular systems. The spectrometer is currently being used in a joint program by NBS and NIH scientists to study hindered rotation in cytosine derivatives. Cytosine is one of the molecular building blocks of DNA and RNA.

Determination of ^{13}C Relaxation Mechanisms—In a joint research program, IMR scientists and scientists at the Division of Computer Research and Technology and the National Institute for Arthritis and Metabolic Diseases (both at NIH) have for the first time made a quantitative determination of the contribution which various relaxation mechanisms (such as direct dipole-dipole, chemical shift anisotropy, spin-rotation, etc.), make to the total ^{13}C spin-lattice relaxation time. Such information has great importance for planning experiments on the \$200,000 super-conducting NMR spectrometer scheduled for delivery to NIH in the autumn of 1969. Also, such in-

formation is of importance in checking the fundamental theories of magnetic interactions in molecular systems.

Electronic Structure of P-F Bonds in Fluorophosphates—Using x-ray diffraction and NMR spectroscopy, IMR, in cooperation with University of Illinois scientists, has made it possible for the first time to determine the sign and the magnitude of the anisotropies of both of the chemical shift tensors (one for ^{19}F and one for ^{31}P) and the ^{31}P - ^{19}F spin-coupling tensor in the PFO_3^- anion. Such a determination requires a precise knowledge of the crystal structure of the fluorophosphate and a detailed line shape analysis of the ^{31}P and ^{19}F NMR line shapes for spectra obtained at -190°C . The knowledge of the sign and magnitude of the ^{31}P - ^{19}F spin coupling anisotropy has resulted in the first and only unequivocal experimental determination of the absolute sign of an NMR spin-coupling constant. This information is of great value both to synthetic chemists and to theoreticians.

Relaxation Process of Chromium Ion in Potassium Alum—The electron spin relaxation process of potassium chrome alum and potassium chrome aluminum alum was investigated by measuring the change in the static d-c magnetization as a function of the cw microwave power absorbed at electron resonance, and also by observing the transient recovery of the microwave resonance signal. At liquid helium temperatures the direct spin-lattice relaxation process is the dominant rate-determining process for magnetically dilute potassium chrome aluminum alum, but for the magnetically concentrated crystals the spin-lattice process can easily be obscured by the lattice-bath relaxation process. There is some evidence that the lattice-bath relaxation rate is determined by the thermal conductivity of the helium exchange medium. Within the experimental error the spin-lattice relaxation measurements at 14.5 GHz and those of other workers at 9 GHz are in agreement with the calculations made by Van Vleck in 1940. His theory predicts that for the direct process at high fields $\tau_1 TH^2$ is constant where T is the temperature, τ_1 is the direct spin-lattice relaxation time, and H the magnetic field. The experimental values of τ_1 agree with the calculated to about a factor of five and the exponential of H is approximately two. The theory also predicts that, to first order, τ_1 is isotropic and independent of the chromium ion concentration, and this also appears to be borne out experimentally.

Solid State Data

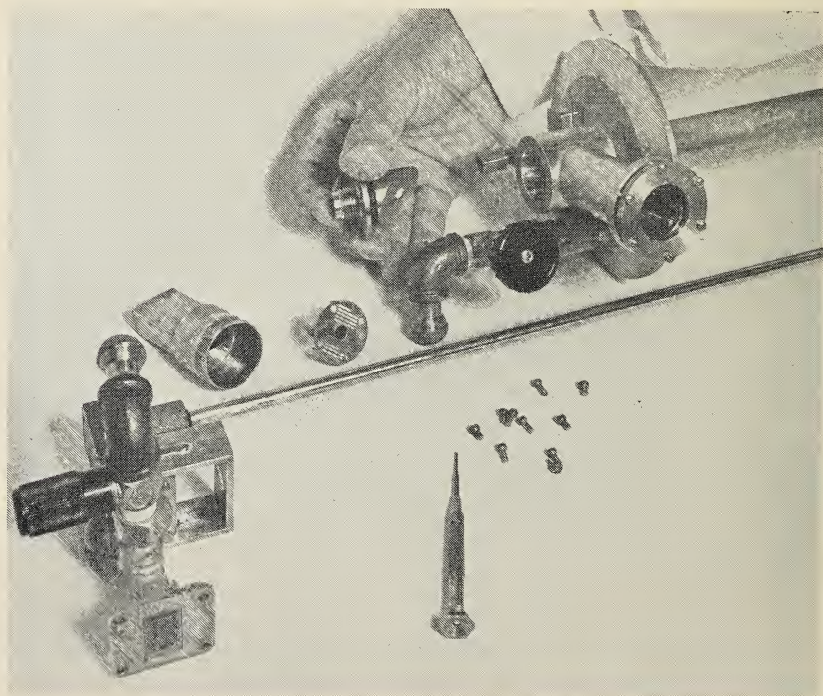
Versatile Probe for Optical-EPR Studies—A sample EPR probe, developed in IMR, can hold any sample material for experimental investigation under varying conditions of illumination, temperature (as low as 1.3 K), and microwave frequencies. The versatility of the new probe will aid scientists in improving the quality of lasers, in studying the photochromic properties of crystals, and in investigating the solid

state properties of crystals for prospective use in display panels and as holograms for storage purposes.

The probe is a pipelike device that can hold any crystalline or powdered material to be investigated. One of the probe's outstanding features is a rigid but adjustable coaxial coupling system (coaxial line) that physically and electrically connects the resonance "test" cavity at the lower end of the line to the microwave system at the upper end, with minimum energy losses and stray reflections. In addition to the coaxial line, the probe consists of a microwave source, a microwave transition coupling system, a light pipe, an iris assembly, and a resonance cavity.

The probe has been tested with considerable success in experiments on a double-doped ZnS: Cu, Ga crystal. NBS data on the photon absorption lines of the crystal at various magnetic field strengths and microwave frequencies showed good agreement with results obtained on the same crystal by other scientists.

Subsequently this probe was used to study photosensitive color centers in a single crystal of calcium oxide. An EPR width of 10^{-6} tesla was obtained, the narrowest line width ever observed in solid samples.



Optical-microwave probe showing the microwave transition coupling system, light pipe, iris assembly, and resonance cavity. The probe holds sample materials during EPR studies.

The increase in the production of color centers responding to excited light, and the color centers' decay after the removal of the excited light, could be traced by the EPR signal monitor.

Proposed Standard Reference Material for Mössbauer Spectroscopy—A new standard material for use in Mössbauer spectroscopy has been proposed as a result of nuclear magnetic resonance experiments recently performed by IMR scientists. The intermetallic compound, TiFe, possesses many advantages as a material with which to calibrate the applied magnetic field, H , in ^{57}Fe Mössbauer experiments. The Fe possesses a cubic symmetry, avoiding complications of quadrupole effects. The Knight shift has now been measured with good precision by nuclear magnetic resonance. A further advantage is that the Knight shift is nearly temperature-independent. Measurements of internal fields at the ^{57}Fe nucleus should be more precise using this new standard.

Luminescence in Semiconductors—The use of photoluminescence and photoconductivity in the elucidation of dynamical processes was applied to insulating strontium titanate and thallous bromide. Strontium titanate was found to exhibit a sharp emission line at 800 nm, with 31 satellite lines arising from phonon cooperation (vibrational sidebands). Excitation spectra show that the center responsible for the photoemission is not directly excited by resonant-energy transfer in which the excitation of electrons and holes is transferred to the exciting center by a radiationless transfer of energy. Then the excited center decays, emitting photons and phonons.

Another process, known as Auger recombination, has been observed in TlBr on excitons trapped at impurities. In this process the impurity exciton is deexcited by a spontaneous transfer of charge to the conduction band where it dissipates energy. The remaining energy at the center is dissipated by photon and phonon emission.

Temperature and Pressure Dependence of Dielectric Constant of Cadmium Fluoride—Although cadmium fluoride has a large insulator-like energy gap of 6.0 eV, doping transforms it into a well-conducting semiconductor. The static dielectric constant at room temperature and pressure was determined to be 8.33 and has a positive temperature dependence coefficient, thus agreeing with reported theories predicting a positive coefficient for compounds with a dielectric constant of less than 10. Pressure dependence along with the temperature dependence of the dielectric constant were combined with compressibility and thermal-expansion coefficient to complete the picture and give information on the temperature dependence of the polarizability, as well. A value of 0.80 at room temperature for the Szigeti charge was calculated.

Propagation of Acoustic Phonons in Heisenberg Paramagnets—The propagation of sound waves in ferromagnetic and anti-ferromag-

netic insulators has been studied within the framework of two models which describe the interaction between the spin system and the lattice. Expressions for the frequency shifts (phonon renormalizations) at high temperatures and near the transition temperature are obtained now in terms of time-dependent correlation functions. The frequency shifts for long-wavelength phonons are negative, increase rapidly in the vicinity of the transition temperature, and are less singular than the attenuation coefficients. The ratio of the frequency shift to the unperturbed phonon frequency is independent of the phonon frequency for long wavelengths. These results agree qualitatively with present experiments.

Development of Low-Threshold Laser Glasses—In a cooperative project with the Night Vision Laboratory, U.S. Army Material Command, experimental glasses doped with various rare-earth and transition metal atoms have been made in order to evaluate their fluorescent properties. Radiative and nonradiative energy transfer and decay processes have been investigated in silicate glasses codoped with Eu and Nd. Energy transfer from Eu to Nd was established from an examination of the excitation spectra and the increased Eu decay rates in the presence of Nd. Measurements of the concentration dependences of the Eu^{+3} and Nd^{+3} fluorescence lifetimes reveal the presence of four distinct processes arising from various ion-ion interactions: (1) self-quenching of the Nd^{+3} fluorescence, (2) self-quenching of the Eu^{+3} fluorescence, (3) nonradiative energy transfer from Eu^{+3} to Nd^{+3} , and (4) Eu quenching of the Nd^{+3} fluorescence. This last process decreases the radiative quantum efficiency of the $^4\text{F}_{3/2}$ state of Nd^{+3} , thus limiting the attractiveness of Eu sensitization for Nd laser action.

Polarizable Ion Models for the F Center in Ionic Crystals—The states of the F center have been considered on the basis of models which treat the movement of the nearest neighbors to the F center and the F electron in a self-consistent manner. The lattice is first described in terms of a classical ionic-crystal theory. The theory has been extended now to treat the nearest neighbor ions in a quantum mechanical manner. The one-defect electron (the F electron) is treated according to polarizable ion models. The absorption energy, the emission energy, the lifetime of the first excited state, the zero-phonon transition energies, and the Huang-Rhys factors are evaluated for two models which differ in the rigor used to compute the polarization of the nearest and next nearest neighbors. The model which contains the more rigorous evaluation of the polarization agrees best with the experimental results for CaO and MgO.

Thermodynamics and Transport Data

Chemical Thermodynamics Data Center—During the past year the Chemical Thermodynamics Data Center completed and published Part

4 of the Series, TN 270, "Selected Values of Chemical Thermodynamic Properties." This volume contains values for the room-temperature properties (heats and Gibbs energies of formation, entropy, enthalpy, and heat capacity) for compounds of 19 elements. This is part of the continuing NBS program on the maintenance of tables of self-consistent data in thermodynamics, and has now covered approximately one-half of the elements in the Periodic Table.

The Data Center has also completed a comprehensive Bibliography and Substance-Property Index of the published literature for 1968 with respect to thermodynamic measurements on inorganic compounds. It contains over 8,000 index entries obtained from nearly 2,800 published articles and will appear in the 12th Edition of the Bulletin of Thermodynamics and Thermochemistry, sponsored by the Commission on Thermodynamics of the International Union of Pure and Applied Chemistry.

Ionization and Appearance Potential Compilation—A comprehensive compilation has been prepared and published, covering ionization potentials, appearance potentials, and heats of formation of gaseous positive ions. These quantities are based on measurements employing techniques of mass spectrometry, photoionization, photoelectron spectroscopy, ultraviolet spectroscopy and, in some instances, on semi-empirical molecular orbital theory. The compilation covers the period 1955–1966, including information on more than 1,500 ionic species and giving more than 700 references to work in this field. This information is of fundamental interest in all areas of gaseous ionization phenomena.

Dissociation Energies of Fluorine and Hydrogen Fluoride—Determination of the dissociation energy of the F_2 molecule has been a difficult experimental problem. Many determinations of this quantity have been attempted, representing a number of different experimental techniques, and resulting in a wide range of proposed values. In a new approach to this problem, thresholds unambiguously assigned to dissociative ionization and ion-pair formation were measured with a photoionization mass spectrometer for the related molecules F_2 and HF. Although the resulting dissociation energies for both molecules are significantly smaller than previously favored values, these new data are supported by their consistency with the accepted heat of formation of HF. These measurements will affect the calculation of binding energies of fluorine-containing molecules. The new results have already stimulated a reinvestigation of the emission spectrum of the HF molecule.

Critical Phenomena in Binary Liquid Mixtures *—To study critical phenomena in binary liquid mixtures, the system 3-methylpentane-

* In cooperation with the Institute for Basic Standards.

nitroethane was selected at NBS. The refractive index of the liquid components of this system is such as to minimize multiple scattering in studying critical opalescence. Experiments were carried out to determine the intensity of scattered light, the curve of coexisting concentrations, and the behavior of the surface tension as a function of temperature. Stimulated by the work at NBS, several research groups elsewhere have initiated experimental studies to investigate other properties for the same system.

Deuterium Isotope Effect on the Ionization of Weak Acids—It has long been known that acids weak in water are still weaker when dissolved in deuterium oxide. The difference in strength, expressed as ΔpK (where K is the ionization constant), varies with the acidic strength (pK) in water in a manner thought my many to be linear. The straightline variation has been difficult to confirm, because of a lack of precise data for the pK of a moderately strong acid such as phosphoric acid or sulfuric acid. Such data are notoriously difficult to obtain. The ionization constant of phosphoric acid, however, was carefully measured at NBS 18 years ago and has now been determined, using identical techniques, in deuterium oxide over a temperature range of 5 to 50 °C. The values of ΔpK thus obtained thoroughly confirmed the linear variation, but a critical examination of the best data for a variety of acids revealed that only inorganic acids appear to fall uniformly on a straight line at low values of pK . Many moderately strong organic acids, on the contrary, show considerable deviations. It is concluded that the deuterium isotope effect is not as simple as has often been assumed.

pH Standard for Clinical Use—Changes in the acid-base balance of blood and other biologic fluids are a useful indicator of pathological conditions in the human body. For the most part, the region of critical interest lies between pH 6 and pH 8. Accurate pH measurement and control has been hampered by the lack of buffer materials capable of fixing pH in this physiologically important range of acidities. A new buffer substance called “*bis-tris*” (the chemical name is 2,2-*bis*[hydroxymethyl]-2,2′2′′-nitrilotriethanol) has recently been found suitable for pH measurement and control between pH 5.5 and 7.5. Furthermore, solutions of this substance are compatible with most biologic fluids. To make this buffer material most useful to clinical chemists, the acidic dissociation of “*bis-tris*” has been accurately determined from 0 to 50° C by careful electromotive-force measurements. In addition, the pH values of several concentrations of “*bis-tris*” buffers were determined. As a result of this work, it is now possible to control pH accurately in biomedical studies performed in the mildly acidic range and to standardize pH equipment at a pH near 6.5.

Standards for Ion-Selective Electrodes—The rapid increase in the types of ion-selective electrodes made available during the past two

years has brought a widespread expansion in the applications of these new sensors. New uses have stimulated the demand for standards of ion activity, the property most directly related to the response of these electrodes. A numerical scale of individual ion activities, consistent with the conventional scale on which the measurement of pH is based, has now been proposed by NBS, and the certification of activity standards for sodium and chloride ions is under way. The performance of electrodes for the determination of calcium and fluoride was evaluated by a procedure that has been adopted for the examination of each new type of sensor. The response is first compared with that predicted by theoretical equation as concentration and temperature are changed. The consistency of the response with the conventional scale of activity is evaluated, and the magnitude of liquid-junction errors in the measurement of ion activity is estimated. Priorities are established through consultation with the instrument manufacturers and users of the electrodes.

Melting Point of Aluminum Oxide—A task force on secondary temperature standards, chaired by an NBS staff member and sponsored by the Commission on High Temperatures and Refractories, International Union of Pure and Applied Chemistry, has completed a cooperative determination of the melting point of aluminum oxide (Al_2O_3). In all, nine scientific groups representing seven countries participated in the joint experimental effort. All work was performed utilizing a common supply of high-purity Al_2O_3 . Experimental techniques varied, depending upon the individual investigator. The value for the alumina point, as recommended by the task force, is 2053° with a standard deviation of $\pm 4^\circ \text{C}$ (ITS 1968).

In addition to actively supporting the task force, NBS is now developing an Al_2O_3 standard reference material. The high-priority standard sample, in 10 g lots, will have a certified melting point with an overall uncertainty of less than $\pm 5^\circ \text{C}$. The SRM will be particularly useful in the fields of high-temperature chemistry and refractories for in situ temperature-calibration purposes.

Heat Capacity Adjustments for Natural Rubber Confirmed—Recent precise measurements have confirmed NBS proposals for adjustments required in previously determined values of the heat capacity of natural rubber (*cis*-1,4-polyisoprene) near room temperature. Accurate knowledge of this thermophysical property is essential to efficient commercial manufacturing and processing operations in the automobile-tire and other rubber industries. The original values were determined in pioneering work at NBS in 1935. These values satisfied technological needs for several decades. However, refined industrial operations resulting from technological progress eventually suggested that these original values were not sufficiently accurate for thermophysical calculations required in the more advanced technological

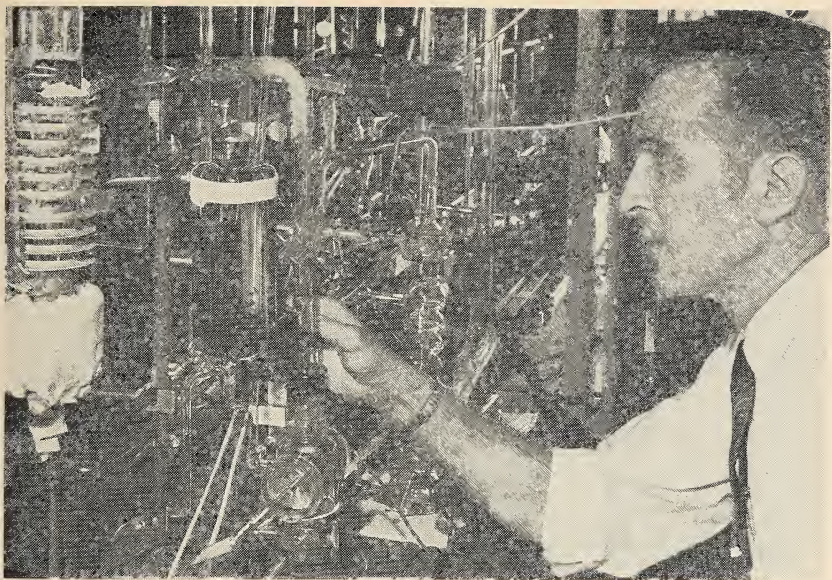
processes. On the basis of new knowledge gained from basic research on polymer crystallization during the intervening decades, NBS scientists concluded that the original measurements had been adversely affected by undetected crystallization processes which were unknown at the time those measurements were made. By taking this new knowledge into account, predictions were made for adjustments which needed to be applied to the 1935 values in order to obtain values sufficiently accurate to satisfy today's technological requirements. New heat-capacity measurements have now been made on synthetic (rather than natural) *cis*-1,4-polyisoprene in an adiabatic calorimeter which covers the temperature range from 2 to 360 K with a precision of better than 0.1 percent of the values. The synthetic polymer is the result of industrial research advances in the past decade and is considered to be technologically equivalent to the natural product, over which it has valuable economic advantages. These new measurements confirm the proposed adjustments, and the basis for the thermophysical calculations required in the rubber industry is again re-established on the basis of firm measurements.

Diffusion in Alloys—Kinetic equations have been developed describing diffusion in a random multicomponent alloy. It has been found that simple relations between the atom and vacancy diffusion parameters can be calculated if the atoms and vacancies in the crystal are assumed to be randomly distributed and suitable averages are taken. These relations, which should apply well to many nondilute alloys, predict a forbidden region in the plot of diffusion coefficient ratios versus composition, and indicate a possible kinetic source of negative diffusion coefficients.

Chemical Kinetics

Theory of Chemical Kinetics—Resonant scattering theory, originally developed for atom-electron interactions, has been applied by IMR scientists to the description of chemically interesting collisions. The result is a complete quantum mechanical description of the interaction between molecules in decomposition and combination reactions. Taken together with recent advances in the calculation of potential energy surfaces, this theory provides the basis for exact calculation of rates of chemical reactions.

High-Pressure Photoionization Mass Spectrometry—A unique photoionization mass spectrometer has been constructed by IMR scientists for investigation of ion-molecule reactions occurring at elevated pressures in the vapor phase. This instrument has been successfully used to derive accurate rate constants and cross sections for the reaction of thermal organic and inorganic ions in the pressure range 0–10 torr. Using selectively labeled deuterium-containing analogs, the stereospecificity of many of these processes has also been determined.



The kinetics and mechanisms of chemical reactions initiated by high energy radiation being studied with a rare-gas resonance lamp.

Photoionization in the Far Ultraviolet—In recent years considerable information has been accumulated concerning the kinetics and mechanisms of chemical reactions initiated by high-energy radiation, especially in the lower-molecular-weight hydrocarbons. Earlier work has demonstrated the feasibility of using photolysis techniques with high-energy photon sources as a means of studying the unimolecular decomposition of parent ions and superexcited molecules as well as the subsequent reactions of the fragment ions or radicals produced by such dissociations. Numerous investigations of this type have been carried out using krypton ($1236 \text{ \AA}$, 10.0 eV) and argon resonance lamps ($1067\text{--}1048 \text{ \AA}$, $11.6\text{--}11.8 \text{ eV}$). In order to extend such studies to still higher energies, self-contained helium ($584 \text{ \AA}$, 21.2 eV) and neon resonance lamps ($744\text{--}736 \text{ \AA}$, $16.7\text{--}16.8 \text{ eV}$) have now been designed, constructed and used successfully in such experiments.

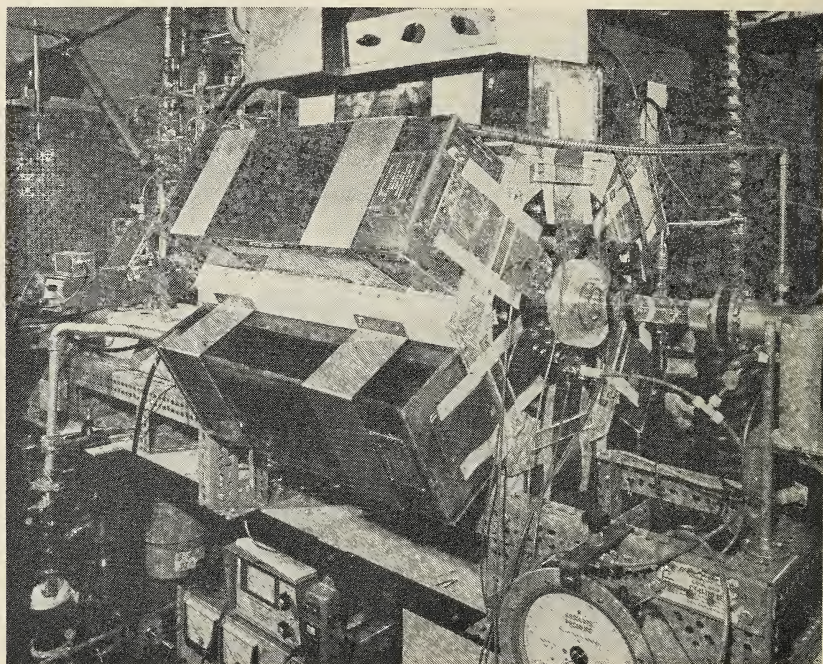
These helium and neon resonance lamps which have a high spectral purity, a high intensity, and a long lifetime have recently been used in several photoionization studies. The results have been compared with the results from the gamma-ray radiolysis of the same hydrocarbons. These same resonance lamps have been used to derive accurate extinction coefficients and ionization quantum yields for a large number of organic and inorganic compounds.

Decomposition of Hydrocarbons—Hydrocarbon molecules decompose at high temperatures by splitting into two free radicals by scission

of carbon-carbon bond. A systematic study of the rates of these decomposition reactions has been made using the single pulse shock tube technique. It shows that the rates and mechanisms are predictably dependent upon variations in molecular structure. As a result, a large number of decomposition rates may be predicted from a limited set of measurements. For example, from 9 measured decomposition rates 27 other rates have been predicted. Where cross-checks are available, the agreement is good.

Vacuum Ultraviolet Flash Photolysis—The vacuum ultraviolet flash photolysis of carbon suboxide has been shown to produce carbon atoms in addition to carbon monoxide. Using the method of kinetic spectroscopy, rate constants for the reaction of carbon atoms with hydrogen, oxygen, nitrogen, nitric oxide, and methane were measured for the first time. Rate constants for the interaction of methylene radicals (produced from the vacuum ultraviolet flash photolysis of ketene and diazomethane) with hydrogen, nitrogen, and methane were also determined by this technique.

Chemical Kinetics Mass Spectrometry—Reactions of atomic oxygen and of molecular oxygen in the lowest-lying excited state are of great importance in air pollution, upper atmosphere chemistry, and combustion. Rate constants for atomic oxygen reactions with about 25

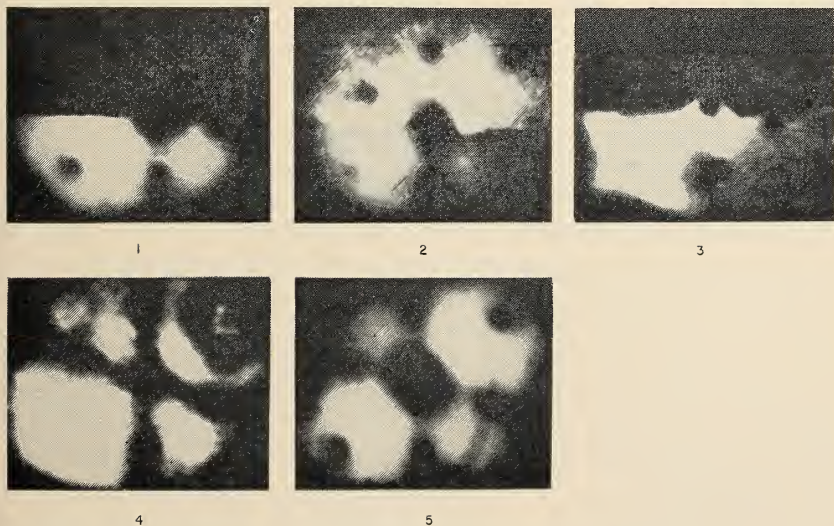


Vacuum ultraviolet flash photolysis apparatus used for the study of the chemical reactivity of free radicals.

alkanes and bromo- and chloralkanes have been measured at temperatures from 300 to 600 K. The first measurements have been made on the rates of reaction of excited molecular oxygen with organic compounds and the importance of these reactions in air pollution is under study.

Surface Data

New Technique for Surface Catalysis Studies—A novel method for observation of surface interactions has been developed. Two chemisorbed gases are completely separated, but contiguous—the zone of interaction initially is a region only a few angstroms wide. On heating, diffusion processes can occur to widen the zone. If interdiffusion takes place with increasing temperature, the process is visually observable. This new technique appears to have interesting applications in studies of surface catalysis. The instrument used is the NBS-developed field emission microscope-molecular beam combination. A field-emitter tip, whose axis is perpendicular to the gas beam, is covered with a monolayer of gas on that half of the tip exposed to the



Field emission patterns of two separated, contiguous, chemisorbed gases (carbon monoxide and oxygen) on tungsten :

1. Tungsten tip half-shadowed with oxygen (oxygen-covered portion not visible because of high work-function).
2. Carbon monoxide condensed over oxygen on tungsten (upper portion, oxygen; lower portion, carbon monoxide).
3. Pattern after heating tip to 350 K.
4. Pattern after heating tip to 700 K for 5 minutes.
5. Field emission pattern of clean tungsten.

beam. A second gas is similarly condensed as a second layer. Both depositions are done at 4.2 K. The temperature of the tip is raised to 50 K to separate the gases by surface migration, for at this temperature the second layer, but not the first, migrates. There results a monolayer over the entire tip, half of the tip covered with the first gas, the other half covered with the second gas.

Chemisorption on Single-Crystal Tungsten Surfaces—Several new experimental methods have been developed to study the chemisorption of gases on single-crystal tungsten surfaces. One study of hydrogen desorption from a (100) tungsten disk involved using a focused lamp to heat the suspended crystal uniformly without introducing temperature inhomogeneities. Two distinct binding states having desorption energies of about 25 kcal. mol⁻¹ and 32 kcal. mol⁻¹ were found. They are populated at room temperature in a two-to-one ratio with full coverage. The dipole moment per adsorbed atom was found to be 0.15 Debye, independent of binding state. Coadsorption of hydrogen and deuterium resulted in isotopic mixing in both states.

Oxygen chemisorption on a (100) tungsten was followed using 0⁺ ion desorption stimulated by electron impact. The β_1 state from which 0⁺ originates occupies about $\frac{1}{20}$ of a monolayer at full oxygen coverage. The ion-energy distribution curves at surface temperatures between 300 and 1200 K provided data leading to a characteristic vibrational frequency of 1270 cm⁻¹ for the β_1 state. This and other results strongly imply that the β_1 state is molecularly absorbed.

Ellipsometry—The ellipsometer is an optical instrument that measures the properties of surfaces and thin films on the surface by the changes in state of polarization of light reflected from the surfaces. Due to its sensitivity to very thin films, for example, the ability to measure thickness to a few Angstrom units, it has been applied to studies of polymer absorption, corrosion of metal surfaces, etc.

The errors that occur in measurements by the ellipsometer have been analyzed and means to eliminate them derived. The errors considered are an imperfect compensator in the ellipsometer, birefringence in a cell containing the surface, tilting of the surface, and roughness of the surface. A new Fortran computer program has been written to perform the involved calculation required to analyze the ellipsometer measurements.

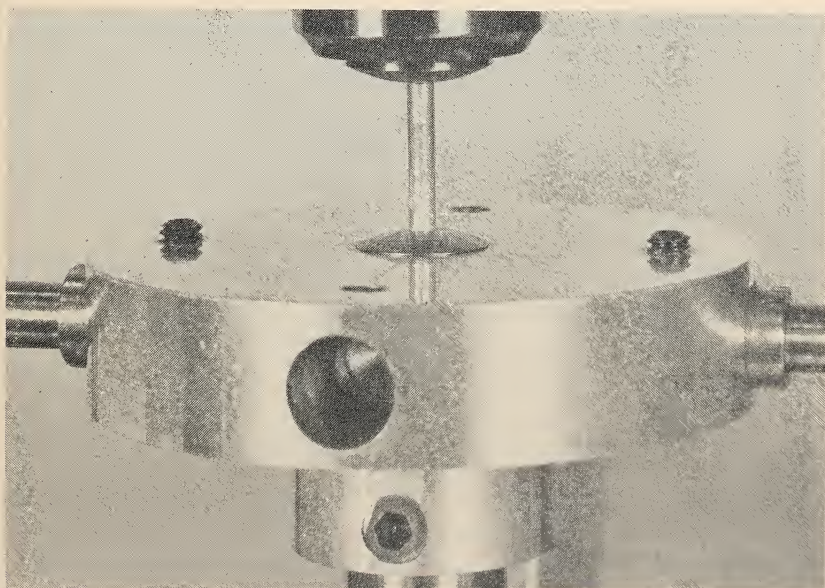
Mechanical Properties

Bulk Modules of Solid Linear Polymers—A bundle-of-chains model has been used to predict the bulk modulus and Gruneisen parameter of linear polymer crystals such as polyethylene and the n-paraffins. The theory uses nearest-neighbor pair potentials to calculate the second and third derivatives of the lattice potential energy with respect to volume. This model is expected to yield useful predictions of the

intermolecular thermal energy and, via the Debye equation of state, the thermal expansion coefficients for linear crystalline polymers. The predicted Gruneisen parameters were found to be considerably larger than for simpler molecular systems and were found to be in excellent agreement with results for ultrasonic and specific heat measurements on polyethylene.

Mechanical Properties of Dental Materials—A torsion pendulum system has been developed for determination of elastic properties and internal friction of dental materials. The specimen serves as the torsion bar in the system. From the frequency of the pendulum, the shear modulus of the specimen material can be determined and from the damping the internal friction can be measured. A value of 3×10^6 psi (2×10^9 N/m²) obtained for the shear modulus of dental amalgam is in agreement with values obtained by ultrasonic methods. The torsion apparatus is applicable to many restorative materials and to natural tooth structure. Studies of the effects of temperature variations on the mechanical properties of dental materials and tooth structure will be made with this apparatus.

Microscopic Studies of Elastic Strains in Materials—Stress-strain analysis in local regions of polycrystalline iron-silicon transformer sheet has been carried out using the divergent beam (kossel) x-ray diffraction method. This nondestructive measurement technique permits microscopic strain determinations near grain boundaries and



Part of the torsion pendulum system for determining elastic properties of dental materials.

other disturbed regions in the alloy and provides mapping the strain distribution. Previous mechanical and thermal treatments of the material determine the nature of the observed strain distribution together with the magnetic characteristics of the transformer sheet.

High Cyclic Bending of Steels—In repeated high cyclic bending, with constant load amplitude, the size and the shape of the plastic zone preceding the propagating crack is controlled by local structural conditions near the tip rather than by stress intensity. No significant correlations have been found between the experimentally determined sizes of plastic zone and the theoretically predicted values of Liu and Rice. The plastic zone sizes ahead of the propagating crack cannot be simply expressed as proportional to the rate of fatigue crack propagation, though a simple relationship exists between the rate and the stress-intensity factor. The relationship given by Paris, $\frac{dl}{dN} = Q \Delta K^n$, describes the rate of crack propagation only in a limited range of relative crack-length, $x < 0.5$. The extent of this range depends on the structure and on the level of applied cyclic stress. Beyond this range, the Paris equation cannot be applied and the crack propagation cannot be related to the stress-intensity factor.

Alloy Softening BCC Transition Metals—The experimental and theoretical basis for the unusual phenomenon of “alloy softening” has been clarified in three published papers. Alloy softening is the weakening of very pure metal deformed below a temperature of about 0.20 times the melting point upon addition of dilute concentrations of solute. Softening in interstitial alloys is attributed to elastic interaction between interstitials and screw dislocations leading to cross-slip, whereas softening in substitutional alloys is attributed primarily to electronic interaction, i.e., modification of the d -band density leading to reduction of the Peierls-Nabarro energy barrier. The relationship between alloy softening and the ductile-to-brittle behavior of bcc transition metals is being explored in high-purity interstitial alloys of iron.

Microplasticity in Metals—Microplasticity studies at room temperature on tensile specimens of 4340 steel in the conditions as normalized and as quenched and tempered to a tensile strength of 273,000 psi, and on a specimen of annealed Invar have revealed (1) that the practice, commonly employed, of prestraining a specimen before conducting microplasticity tests introduces appreciable instability of the specimen, and (2) the microplastic behavior of the prestrained specimen, especially at very small strains ranging from 1×10^{-7} to 1×10^{-4} , is not representative of that of the specimen in its initial heat-treated condition.

High-Temperature Creep—An NBS-designed-and-constructed ma-

chine has been installed and is being used to facilitate the study of the mechanisms underlying creep. Controlled through an analog computer, it provides a constant resolved shear-stress loading to single crystals compensating for slip-plane rotation and reduced cross section. It also has the capability of producing rapid temperature and stress changes and can operate in either high vacuum or inert atmosphere.

Ultrasonic Measurements in Molten Glass—It has been difficult, if not impossible, to perform high-temperature shear ultrasonic relaxation spectroscopy in molten glasses because of the high absorption of shear waves in the liquid. These structural investigations have been aided by improved ultrasonic detection using a phase-locking technique. A technique using signal-averaging and synchronized discriminating amplification common to sonar and some light-scattering experiments was modified for the purpose. This resulted in an increased range of absorption per wavelength of more than twice that of previously reported equipment. One inexpensive lock-in amplifier and a few general laboratory instruments can be easily incorporated into all ultrasonic systems with a video output, and will enhance signals of any R. F. frequency by approximately 25 dB, and increase the accuracy of amplitude measurements to about 0.1 dB. This added range enabled the study of shear ultrasonic relaxation spectra in molten glasses, and opened up a new research area: the ultrasonic detection of molecular clusters in immiscible oxide glasses.

Optical, Electrical, and Magnetic Properties

Vacuum Ultraviolet Excited Fluorescence—The fluorescence of chlorine produced from the vacuum ultraviolet photolysis of phosgene has been observed. From the dependence of the fluorescence upon the wavelength of the exciting radiation, it has been concluded that literature values for the electronic energies of two of the upper excited states of chlorine are too high by as much as one electron volt. It should be noted in this instance that a well-designed photochemical experiment has yielded results which are of primary concern to the molecular spectroscopist. It is an excellent example of how different disciplines interact in an active research area such as photochemistry.

Organic-Dye Lasers—IMR physical chemists have recently instituted a study of the properties of organic-dye lasers. The main emphasis of the program is on studying the mechanism of the laser process and the role of molecular triplet states in reducing the efficiency of the system. Theoretical calculations indicate that the addition of quenching agents specific for triplet states should greatly increase the efficiency. Experiments are in progress to test the validity of these calculations. A giant flash-photolysis system has been used to pump rhodamine 6G, and outputs in excess of 1J and pulse-lengths of 30 to 40 μ s have been obtained.

Optical and EPR Study of Orange Ruby—Orange discoloration has been a source of degradation in performance sometimes encountered in ruby lasers. A number of doped sapphire crystals have been grown and studied in this laboratory. Dopants include Cr (i.e., ruby) and other single-transition elements, and combinations including Cr as the second dopant. Optical absorption spectra taken before and after x-ray coloration show that the discoloration spectra produced are similar to those reported in laser rods, but only when Cr is present. Double-doping with Cr plus Mg produces the orange color without irradiation, and irradiation produces only a slight additional increase. Valence considerations suggest that the phenomena involve either a change of Cr valency or color centers or both. EPR spectra are being taken on a number of these samples in an effort to observe and identify the defects responsible.

Thermo-Optic and Piezo-Optic Properties of Laser Materials—The changes in refractive index caused by temperature and hydrostatic pressure have been determined for five neodymium-doped glasses and single-crystal ruby by an optical interference method. It is interesting to note that for ruby there is a decrease in refractive index for both the ordinary and extraordinary ray with applied hydrostatic pressure. Furthermore, there is an increase in refractive index with rise in temperature. These changes are not what might be expected on the basis of density considerations. Using the method of uniaxial loading in conjunction with the application of hydrostatic pressure, the two piezo-optic and two elasto-optic coefficients of each of the five glasses have been determined. The eight piezo-optic and eight elasto-optic coefficients of single-crystal ruby have also been determined.

These data are of immediate interest in laser technology because the temperature-induced changes in refractive index create a distortion of the wavefront of light generated in a laser solid. Since the refractive index is dependent on temperature, there is a direct change caused by the temperature gradient from the center to the edge of the rod. In addition, the temperature gradient creates internal stresses which produce further changes in index arising from the stress-optic effect. There are virtually no data in the literature on the temperature and stress coefficients of the refractive indices of important laser materials.

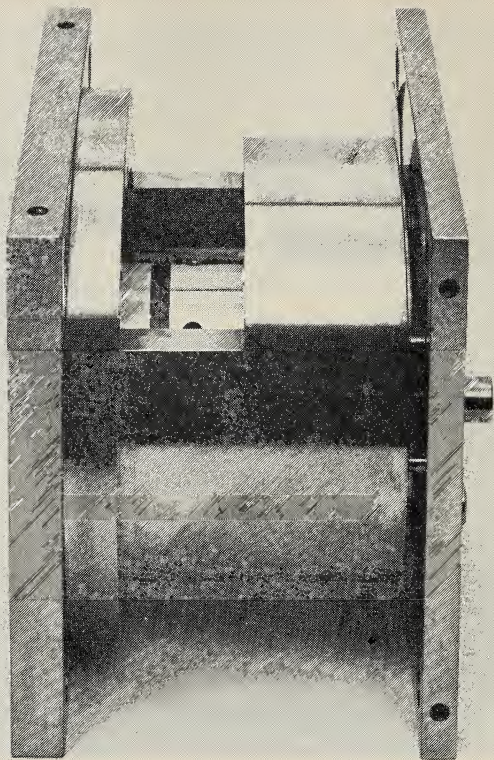
Plasmaron Structure in the Soft X-Ray Spectrum of Al—As part of a continuing study of the density of states in metals, the $L_{2,3}$ soft x-ray emission spectrum of Al has been carefully scanned for a low-energy edge due to a plasmon interacting resonantly with a hole (called a plasmaron), predicted by recent theoretical studies of the interacting electron gas. A very weak structure near the predicted location, but just within the noise level, was seen. An upper limit to the magnitude of such structures was established and it was suggested that the light alkali metals should provide a better test of the theory.

Refractive Index of Cuprous Chloride—The refractive index of single-crystal cuprous chloride (CuCl) was measured at room temperature in the wavelength range $0.42\text{ }\mu\text{m}$ to $22\text{ }\mu\text{m}$. CuCl is a potentially important laser-modulator crystal by virtue of its large electro-optic coefficient. Because it is cubic it lacks the undesirable feature of natural birefringence present in all commercial modulator materials. In addition, CuCl is transparent to beyond $20\text{ }\mu\text{m}$, making it useful for modulating the intensity of the CO_2 laser at $10.6\text{ }\mu\text{m}$. The measurements were performed in the wavelength range $0.42\text{ }\mu\text{m}$ to $1.2\text{ }\mu\text{m}$ with a commercial V-block refractometer. The range of wavelengths was extended to $22\text{ }\mu\text{m}$ by measuring the wavelength-dependence of interference fringes in thin polished plates. Materials were supplied by the Air Force Cambridge Research Laboratory and the Massachusetts Institute of Technology.

Infrared Absorption in Perovskites—The optical absorption spectrum of SrTiO_3 has been examined over the wavelength range of $2.5\text{ }\mu\text{m}$ to $25\text{ }\mu\text{m}$. Nine broad absorption bands were observed beyond $3\text{ }\mu\text{m}$, and a set of sharp lines at $2.86\text{ }\mu\text{m}$. The sharp lines are interpreted as due to OH impurities in the crystal, while the broad bands are believed to be due to multiphonon creation processes, except for one which is a single-phonon process. Selection rules for simultaneous creation of either two or three phonons were obtained, in dipole approximation, at selected critical points in the Brillouin zone. The observed bands were assigned, using these rules together with the experimental phonon-dispersion curves of Cowley and other published values for the higher-energy branches obtained at specific points in the zone by other techniques. Some of the phonon energies determined in this way have not been seen by neutron diffraction techniques. The results are typical of what is observed in other perovskite-type materials such as LiNbO_3 , KTaO_3 , and BaTiO_3 .

Structural and Spectroscopic Properties of Nickel(II) Pyrazole Complexes—X-ray diffraction and visible spectroscopic studies have been carried out on single crystals of some pyrazole complexes of nickel(II). The electronic-energy levels in two halogen-substituted complexes were shown to differ markedly from those in the closely related pyridine complexes. Detailed crystal-structure data showed that the unusual energy-level systems in these complexes were related to internal hydrogen bonding between the coordinated pyrazole molecules and the coordinated halogen atoms. The spectral assignments were based on a reduced effective field of the halogen atom at the site of the nickel(II) ion. These assignments were corroborated by the observed bond distances and pyrazole ring orientations.

Thin-Film Dielectric Measurements—Developments in the commercial manufacture of thin polymer films have generated considerable interest in the use of these films as electrical insulators. This interest



Three-terminal fluid displacement cell used to measure dielectric properties and average thickness of their films.

has resulted in a joint program between IMR scientists and a research scientist from FMC Corporation. A three-terminal fluid-displacement cell was designed and built and is being used to successfully measure the dielectric properties of commercial films as thin as $0.025\text{ }\mu\text{m}$. Not only does the two-fluid method used with this new cell permit dielectric constant measurements on a $25\text{-}\mu\text{m}$ film with an accuracy of better than 1 percent, but it also provides a unique way of measuring the average thickness of these films with an accuracy of the order of 1 percent. This program has resulted in significant advances in measurement capability that will be used by industry in the evaluation of insulating films and by NBS for the development of thin-film reference standards of dielectric constant and thickness.

Dipole Moments of Molecules in the Vapor Phase—An unconventional method for precise determinations of dipole moments of symmetric top molecules in the vapor phase has been used to obtain data on reactive gases with small dipole moments. The method is based on experimental measurements of the shape of the nonresonant Debye-

type microwave absorption in pure gases and gas mixtures. The measurements are made by varying the pressure at a single temperature where the gas is chemically and thermodynamically stable. The errors due to temperature variation are eliminated, and the sensitivity of the method permits determination of dipole moments too small to be detected by conventional methods.

Low-Field Tri-axial Helmholtz Coils—A circular, triaxial, fourth-order, air-core, Helmholtz-coil system was designed and constructed with the aid of the Navy Bureau of Weapons. The coil system was designed so as to generate a magnetic field homogeneous to 1 part in 10^4 over a volume of nearly 100 cm^3 . This coil system will be used for the calibration of magnetic flux density measuring equipment, and in a study of the feasibility of using electron paramagnetic resonance as a primary standard of magnetic flux density in the range of 0.1 to 30 millitesla.

Iron Nuclear Magnetic Resonance in Paramagnetic TiFe and TiFe-TiCo Alloys—For the first time in any paramagnetic material, the nuclear magnetic resonance of ^{57}Fe has been detected. The discovery was made by a team of IMR scientists including an NBS industrial research associate from the U.S. Steel Corporation. The team used the combined facilities of U.S. Steel laboratories for sample preparation and NBS laboratories for the resonance work. Each of the two samples used, enriched in the stable isotope ^{57}Fe , is valued at \$6,000.

Although the nuclear magnetic resonance is observed with relative ease in ferromagnetic materials due to a signal enhancement related to the magnetic domains, this observation demonstrates that it is also possible to observe the ^{57}Fe nuclear magnetic resonance in paramagnetic materials as well. This resonance is being used to study the electronic properties of TiFe and TiFe-TiCo alloys. These alloys are structurally related to TiNi, which has an unusual mechanical memory.

Precision High-Pressure Measurements Via N.Q.R. Spectroscopy—Pressure measurements up to 2 kbar have been made with a precision of ± 0.5 bar. This has been done using a servo-controlled nuclear quadrupole resonance spectrometer and a carefully purified sample of KClO_3 . The main source of error at the present time arises from the small temperature fluctuations ($\pm 0.005^\circ\text{ C}$) of the high pressure vessel.

Reactivity and Corrosion

Anisotropic Oxidation of Pyrolytic Graphite—Pyrolysis of a hydrocarbon at low pressure and high temperature can be used to deposit a form of polycrystalline graphite with preferred orientation. As a result of the orientation, with graphite basal plane preferentially

parallel to the surface of the substrate, anisotropy of physical and chemical properties is evident. IMR scientists have measured the ratio of air oxidation of graphite for rates parallel to and perpendicular to the preferred basal plane direction. From 800 to 1800 K, the ratio increases from about one to seven, with the faster oxidation parallel to the preferred basal plane direction. The offered explanation suggests that the same two-stage mechanism is involved in both directions: chemisorption followed by decomposition of surface oxides. The surface concentration of available sites is sufficiently different, however, that in one case the chemisorption is rate-limiting and in the other decomposition is limiting. This interpretation leads to activation energies in agreement with literature values for both steps of the mechanism.

Catalytic Decomposition of Hydrazine—Hydrazine and some of its derivatives are of interest as rocket fuels. However, hydrazine decomposes catalytically when stored in some metal containers, such as maraging steel. Investigations by IMR scientists show that the catalytic decomposition can be reduced to an insignificant rate by certain coatings electro-deposited on the steel. Among the best for the purpose are cadmium, silver, electrodeless nickel, and tin-lead alloy.

Stress-Corrosion Cracking of Glass—Glass is usually regarded as an inert material, being highly resistant to chemical corrosion. As a result it finds use as liners in water heaters, in industrial plants where chemical resistance is important and in the laboratory. Despite this well-known resistance, glass is susceptible to stress-corrosion cracking by water. The effect is so severe that, all other things being equal, the strength of glass is reduced by about 80 percent in the presence of water or water vapor.

Stress-corrosion in glass, as in other materials, results from the slow growth of surface cracks due to stress and environment. In glass the phenomena is believed to be due to a chemical reaction between the glass structure and the water molecules. The rate of reaction can be monitored by measuring the rate of crack growth as a function of temperature, applied stress, and environment, and the data obtained can be used to describe the process in fundamental terms used in reaction-rate theory. During the past few years the Institute for Materials Research has been conducting such experiments using fracture-mechanics techniques. Conclusions from such experiments are as follows:

1. Crack-velocity data quantitatively satisfies the Charles-Hillig theory for stress-corrosion of glass.
2. Glasses of different chemical behavior exhibit varying degrees of resistance to stress-corrosion cracking.
3. In water, the rate of reaction of hydroxyl radicals with the glass appears to control the rate of crack-growth.

4. In gaseous nitrogen containing water vapor, there are three distinct mechanisms of crack-growth. At low-stress intensity levels the rate of crack-growth is reaction-rate limited. At intermediate levels, the rate of crack-growth is limited by the rate of water transport to the crack tip. At the highest stress-intensity levels, crack-growth is independent of environment.

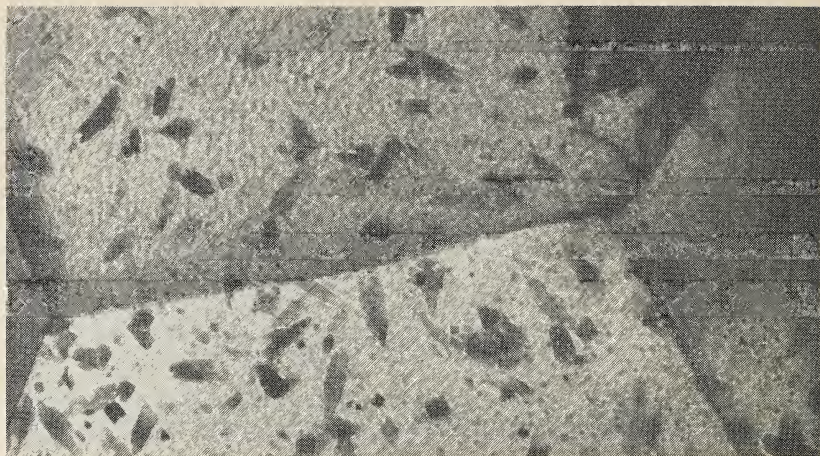
Stress-Corrosion Cracking of Copper Alloys—In a joint effort at the IMR laboratories, a research associate from the Research Institute for Advanced Studies (a division of Martin Marietta) and an IMR scientist have provided data that supports, in part, a stress-corrosion cracking theory. The theory proposes that the stress-corrosion cracking of alpha-phase copper alloys in tarnishing ammoniacal solutions proceeds discontinuously by the repeated formation and rupture of a brittle tarnish film. An important prediction of this "brittle-film" mechanism is that the cracking rate is determined by the rate of growth of the tarnish film. Earlier work on the alpha-brasses established that the cracking rate increases with increasing zinc content of the solid, with increasing temperature, and with the application of anodic potentials, but qualitative data was not available.

The ellipsometric data from the present study demonstrated quantitatively that the rate of tarnish growth increases significantly with zinc content, as does cracking rate. For example, the time taken for the film to attain a thickness of 900 Å was found to be 44 min for pure copper, 16 min for Cu-5 Zn, 317 min for Cu-10 Zn, 1 min for Cu-20 Zn, and ~ 1 is for Cu-30 Zn. The rate of tarnish growth determined quantitatively was also found to increase with increasing temperature and with applied anodic potentials, as does cracking rate.

Pure copper, which is not susceptible to stress-corrosion cracking in ammoniacal solutions, was found to have a low (less than alpha-brasses) film-growth rate. Further work is needed to explain why copper does not undergo stress-corrosion cracking in this solution.

Initiation of Pitting of Iron—Studies sponsored by the Office of Saline Water have shown that before the protective (passive) films on iron break down in the presence of chloride ions, subtle changes occur in the optical properties of the films. These changes occur before the ordinary indications of breakdown (e.g., an increase in current via galvanometric measurements) signal the onset of pitting. The changes in film properties, triggered by chloride ion, have been detected by ellipsometric spectrometry—a new technique which enables the measurement of optical spectra of very thin films on metal surfaces while they are immersed in solution. These studies are important in elucidating the mechanism of the pitting of metals, which is responsible for many corrosion failures.

Corrosion of Titanium Alloys—Studies of the initial stages of corrosion in a series of titanium alloys are in progress, using electron



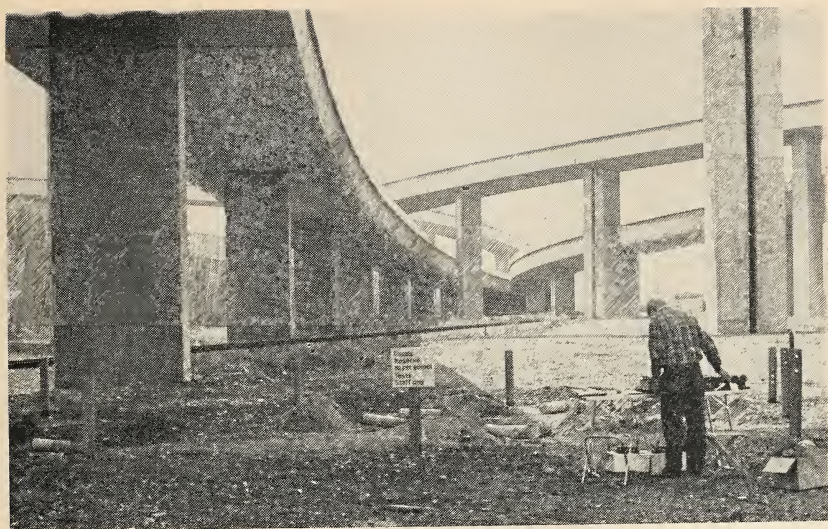
An electron transmission micrograph taken from a thin specimen of a titanium-palladium after corrosion in a salt-water solution. A thin film of titanium oxide containing small oxide crystallites is seen superimposed over the microstructure and grain boundaries in the alloy specimen.

microscopy and diffraction techniques. At the same time, quantitative chemical analysis of microscopic areas is proceeding using an electron probe micro-analyzer. The influence of high temperatures and pressures, using a salt-water corroding solution, are being investigated. Extremely thin corrosion films can be studied either in place on the alloy specimens or after removal to determine the physical and chemical nature of the films. The relative resistance to corrosion of the various alloys is interpreted in terms of the microstructure and chemical composition of the specimens. The results of these studies should guide the choice of the most suitable materials for applications to salt-water purification plants.

Microbiological Corrosion of Iron—A marine strain of *Desulfovibria* has been isolated which produces a mixture of schreibersite $(\text{Ni, Fe})_3\text{P}$, and iron sulfide when grown in a sea water medium containing ferrous ions and organic material. Schreibersite and iron sulfide (trolite) are found to be closely associated in certain meteorites and tectites.

The anaerobic corrosion of iron in the same medium without added ferrous ions was associated with removal of a protective iron sulfide film formed during growth of the organisms.

Corrosion of Steel Piles in Underground and Seawater Environments—The most extensive corrosion test programs ever undertaken are under way to determine the best means of protecting steel piles from corrosion. Measurements of corrosion rates are being made periodically on steel pile specimens exposed underground and in



Corrosion rates of steel pilings exposed underground and in seawater are being studied to determine optimum means of corrosion protection. Polarization techniques are used to measure instantaneous corrosion rates of the specimens.

seawater. Polarization techniques developed in the laboratory at NBS are being applied to monitor the instantaneous-corrosion rates of the steel pile specimens.

The study is planned to extend over a 15-year period in the seawater site and 25 years in the underground site. It is expected that the results will show a correlation between the amount of corrosion on the steel test piles, as determined by polarization measurements, and the actual corrosion on the specimens which will be measured when the test piles are extracted from the test sites after specific exposures.

In the seawater site, over 100 carbon-steel and low-alloy steel piles (35 ft long, 45 lb/ft) were installed 19 ft into the Atlantic Ocean bottom off the coast of Dam Neck, Virginia. Many types of protective methods are included in the investigation, ranging over coating systems (coal-tar epoxy, galvanized, aluminum- and zinc-flame spray, zinc-rich paints, etc.,) and cathodic protection by zinc and aluminum sacrificial anodes.

In the underground sites, 21 steel piles (35 ft long, 74 lb/ft) were driven to bedrock in connection with the construction of a Trans-canadian Highway Interchange in Montreal, Quebec, Canada.

Cooperating with NBS in these investigations are the American Iron and Steel Institute, Province of Quebec, the U.S. Army Coastal Engineering Research Center, the U.S. Navy Civil Engineering Laboratory, Amphibious Forces, and the Fleet Antiwar Aircraft Training Center.

Engineering Materials

Impact Properties of 431 Stainless Steel—The inclusion of refrigeration in the heat-treatment of 431 stainless steel has been a common practice. Data obtained in an investigation of the effects of refrigeration and strain on the notch toughness of 431 indicate that refrigeration should be avoided in the heat-treatment of 431 parts subjected to stress concentration or impact loading.

Fatigue Cracking in Chromium Diffusion-Coated Ti-Bearing Steel—Chromium diffusion coatings on Ti-bearing steel frequently lead to columnar growth beneath the Cr-rich layer. The grains are preferentially oriented. The depth of the columnar zone does not depend on the chromizing time. A high rate of cooling prevents columnar growth. No grain-boundary diffusion or carbide formation at the grain boundaries was observed. The Cr-rich layer is structurally homogeneous and consists of α -solid solution. The hardness of the coating does not vary with the chromizing time.

Dimensional Stability of Gage-Block Steels—The causes of dimensional instability in a 52100-type steel hardened to $R_c 65$ are being investigated and several important facts are indicated. Precision electrical resistance measurements, microhardness measurements, and carbide determinations indicate that the measured parameters change with long aging-time, and that these changes can be correlated with length changes. The results tentatively indicate that three structural processes are occurring: dissolution of carbides, diffusion of carbon to lattice imperfections, and re-precipitation of carbides at new sites.

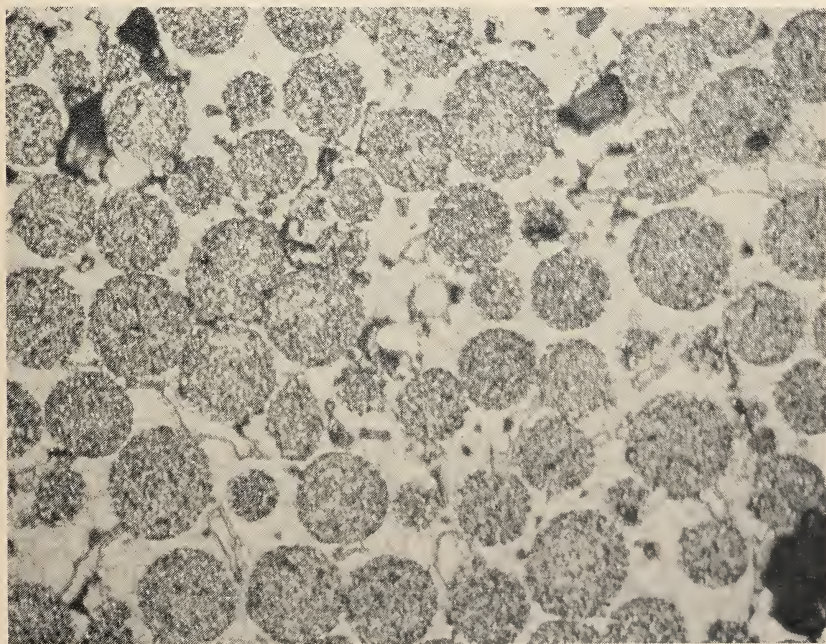
Properties of Electrodeposited Copper—A comprehensive investigation of the properties of electrodeposited copper, in progress for five years, has been completed. During the past year the large amount of data has been compiled into numerous tables and graphs and the complete work prepared for publication.

The project was sponsored jointly by the American Electroplaters' Society, the International Copper Research Association, the Copper Development Association, and the National Bureau of Standards. Investigations covered the types of plating baths used in industry: acid sulfate, cyanide, pyrophosphate, fluoborate, and amine baths. A number of properties were measured on each type of deposit obtained under about 150 different sets of operating conditions, the latter comprising variations in bath composition, temperature, and current-density. Properties measured and correlated included: tensile and yield strengths, modulus of elasticity, elongation, internal stress, fatigue limits, hardness, density, electrical resistivity, thermal expansivity, structure, and chemical composition. Effects of cold-working and annealing, and effects of cryogenic and elevated temperatures on some of the above properties, were also determined.

Development of a Radiopaque Composite Dental Restorative Material—Radiopaque esthetic direct filling materials are needed to facilitate dental radiographic diagnosis. An x-ray-opaque composite restorative material was developed having adequate esthetic properties for use in anterior teeth. The resin binder is a ternary eutectic dimethacrylate formulation. The powder consists of a BaF_2 -containing glass and fused silica both treated with a silane coupling agent and containing peroxide initiators.

One hundred and ten restorations were placed using this newly developed material. The restorations will be observed over a prolonged period. They had sufficient radiopacity and yet were esthetically pleasing.

Spherical Alloy for Dental Amalgam—A significant improvement in the physical properties of silver-tin-mercury alloys used in restorative dentistry has resulted as a culmination of research conducted at NBS. In this study, irregularly shaped particles formerly used to prepare dental amalgam alloys have been replaced by spherical alloy particles with a consequent improvement in strength and durability. In addition, the alloys can be packed into the tooth cavity with less pressure, resulting in greater comfort and convenience for both the dentist and his patient. The new alloy has recently been marketed both in this country and abroad.



Photomicrograph of dental amalgam alloy prepared with spherical alloy particles.

Gallium Alloys for Restorative Dentistry—A gallium-tin-palladium alloy has been developed which may be substituted in place of the mercury-tin-silver alloys now used to replace tooth structures lost as a result of caries. The new alloy is 50 percent stronger, several times more resistant to flow under an applied load, and has a thermal expansion coefficient closer to that of human teeth than the present dental amalgam alloys. In addition, this alloy wets tooth structure and thereby produces a more effective seal against fluid infiltration at the margins between the filling and the tooth. The alloy requires extensive biological and clinical testing before it can be made available for extensive use.

Creep of Pure-Gum Vulcanizates of Natural Rubber—The creep of vulcanizates of natural rubber cured with varying amounts of dicumyl peroxide was compared with that of vulcanizates cured by typical conventional sulfur-accelerator systems. The measurements involved indentation of a flat rubber disk as a function of time t and temperature T . The product of shear compliance J and T as a function of $\log t$ was represented by a family of curves with T as the parameter. For the vulcanizates cured with the sulfur-accelerator system, previous work has shown that the individual curves could be shifted along the abscissa to yield a single continuous curve with a slope which increased from a negligible value to a maximum and then decreased, continuing through a region of minimum slope extending over about 7 decades of time before ending in a region of increasing slope. The value of the creep was 1.5–2.0 percent per decade in the region of minimum slope. When the same procedures were applied to the vulcanizates cured with dicumyl peroxide, the individual curves of JT against $\log t$ at each temperature did not yield a single continuous curve when shifted. In a limited region just above the glass transition temperature, the creep was appreciable, but neither a constant-activation-energy shift nor a Williams-Landel-Ferry shift was satisfactory. Above this region it was clearly impossible to obtain a single curve, since the compliance-temperature product at a given time increased with increasing temperature while the corresponding creep of the dicumyl peroxide vulcanizates was generally too small ($<0.5\%$ per decade) to be measured between 5 s and 600 s, except when the compound contained less than 2.5 parts of effective dicumyl peroxide. Outside of these regions the shear modulus was found to increase linearly with temperature. The investigation included temperatures as high as 100 °C and effective dicumyl peroxide concentrations as high as 25 parts per hundred of rubber.

TECHNICAL ASSISTANCE TO OTHERS

IMR gives technical assistance and advice on materials to other Government agencies, and to science and industry. Arranging con-

ferences and seminars to facilitate dissemination of new data useful in solving materials problems is also an important service provided.

Advisory and Consulting Services

Life of Postage-Stamp Printing Plates Increased—Although prior recommendations of NBS have resulted in improved life of postage-stamp plates used by the Bureau of Engraving and Printing, a breakthrough in this area has been made. By judicious shot-peening of selected portions of the back of the plates, fatigue-resisting compressive stresses have been introduced and machining irregularities removed. The results on lifetime has been to increase service-life by more than an order of magnitude. One test plate now in service has registered 1,700,000 impressions and is still serviceable. This contrasts with untreated plates which may fail at less than 100,000 impressions.

Carbon Dioxide Standards—IMR chemists have developed a highly accurate gravimetric technique to prepare three standards of carbon dioxide in air for the Environmental Science Services Administration. The compositions were ascertained to within one part in ten thousand, and the mixtures will serve ESSA as primary standards for the continuing investigation of atmospheric composition.

Calibration Standards for Salinometers—The conductivities of 20 samples of sea water of varying salinities were measured for the Department of the Navy. These samples will be used for the calibration of oceanographic salinometers. The same techniques were also used to measure the conductivities of two different lots of Standard Sea Water with an accuracy of 50 ppm. Standard Sea Water is a convenient and widely accepted reference for oceanographic measurements but is available at only one value of salinity.

Conferences and Symposia

Symposium on the Structural Properties of Hydroxyapatite and Related Compounds—Sponsored jointly with the Office of Naval Research, in cooperation with the American Dental Association and the Georgia Institute of Technology, this symposium reviewed critically the information available on apatites. The properties of these compounds have importance in diverse areas such as tooth, bone and pathological mineralizations, minerals, fluorescent coatings, water purification, and oceanography. Twenty papers were presented and 125 visitors participated in the symposium. The papers are to be published as a text.

Symposium on the Thermodynamic Properties of Bulk Polymers—Through its Polymers Division, the Institute for Materials Research sponsored this symposium at the NBS Gaithersburg Laboratories on

March 10–12, 1969. The symposium assembled over a hundred polymer scientists from industrial, university, institutional and governmental laboratories to focus discussion on the specific aspects of polymer science indicated by the symposium title. Thirty papers were discussed on subjects including: crystalline, vitreous, and elastomeric physical states; crystallization, melting, glass transformation, and other physical transitions and relaxations; elasticity of elastomers; special methods for determining thermodynamic properties; state properties and their derivatives for common and special situations; and the relationship between thermodynamic and molecular properties.

Symposium on Ion-Selective Electrodes—This symposium, held at NBS on January 30–31, 1969, was attended by approximately 450 physical and biomedical scientists. The present state of the art and future capabilities of these electrochemical sensors were evaluated. A broad spectrum of topics ranging from principles, characteristics and basic chemical studies to applications in biomedicine and industrial analysis and control was reviewed. The published proceedings of this symposium, consisting of the complete texts of the invited review papers plus selected segments of the panel discussions, is available from the Government Printing Office as NBS Special Publication 314.

Modern Trends in Activation Analysis—The Third International Conference on Modern Trends in Activation Analysis, sponsored jointly by the National Bureau of Standards, the U.S. Atomic Energy Commission, the International Atomic Energy Agency, and EURISOT, European Communities Commission, was held October 7–11, 1968. The Conference was attended by over 400 scientists, including 100 from countries other than the United States. Each day a plenary lecture, given by a recognized world authority, outlined the state-of-the-art for the various phases of activation analysis. This lecture was followed by simultaneous sessions with contributed papers. A panel discussion at the end of each day's sessions provided a synopsis of the major points of discussion in each session, allowing attendees to hear the highlights of sessions they were unable to attend. More than 150 technical papers were presented. The published Proceedings of the Conference are now available.

Conference on Fundamental Aspects of Dislocation Theory—Held at NBS under the auspices of IMR on April 21–25, 1969, this conference was devoted to a thorough appraisal of dislocations in crystals. One hundred theorists from 18 different countries attended to center on the questions: "How far have we come?" and "Where do we go from here?" Seventy papers were presented, ranging over all aspects of the subject including its applications to mechanical properties of materials, lattice mechanics of defected crystals, biophysical applications to proteins, and liquid crystals. The published proceedings are expected to be available in the spring of 1970.

Symposium on "Nuclear Standards for Chemistry and Technology"—This Symposium was held September 8–9, 1968, sponsored jointly by the American Chemical Society's Division of Nuclear Chemistry and Technology and the ACS Committee on Standardization Relations. The Symposium was organized to bring workers in the diverse areas of safety, health, quality assurance, and specific engineering standards together to discuss present programs and future needs in the relatively new and rapidly growing field of nuclear technology. The published proceedings of the Symposium are available as NBS Special Publication 310.

"Symposium on an International Standard Reference Materials Program"—This Symposium was held May 26–28, 1969, at the National Bureau of Standards, under the joint sponsorship of NBS and the International Committee on Weights and Measures (CIPM). Thirty-three representatives from 15 countries and 4 international agencies attended. The goal of this first International Standard Reference Materials Symposium was to explore the possibilities and problems of an international SRM Program. A summary of the Symposium discussions is being published in *Metrologia*.

INSTITUTE FOR APPLIED TECHNOLOGY

The Institute for Applied Technology (IAT) attempts to meet the Nation's need for measurements and standards related to the artifacts of our society. Thus, the IAT programs are concerned with technological or "engineering" measurements and standards which deal with products, commodities, devices, processes or systems. These measurements and standards are the language of the market place for coupling user requirements with the performance capabilities of products, processes or devices, and thus bring order and quantification to man's use of his technical skills.

The Institute provides a technical base for the development of engineering and product standards, and measurement methodology. Its programs are oriented to industry, to the States and regions of the country, and to all levels of Government. They bridge the interface between science and its applications, and by coupling science and technology with the daily activities of commerce, industry, and Government, they stimulate economic progress.

The Institute is responsible for several mandatory legislative assignments. Among them are the Flammable Fabrics Act as amended in 1967, the Fire Research and Safety Act of 1968, and the Fair Packaging and Labeling Act.

IAT activities fall into two major categories: Technological Measurements and Standards, and Technical Innovation. A substantial part of the work done in the IAT program is technical assistance to other parts of the Bureau, other Government agencies, and to industry. Work done in motor vehicle safety research and technical analysis is virtually all done in support of the mission of other agencies of Government. In other fields, a lesser but still substantial volume of IAT work is done at the request of others.

TECHNOLOGICAL MEASUREMENTS AND STANDARDS

This category covers the Institute effort for the broader extension of the national measurement system into the engineering and technological fields. It is a program to apply principles of good measurement, rather well defined in the science area, to the determination of performance or other significant characteristics of products systems and

devices important either in Commerce or to Government needs. In essence, the objective is to find ways by which standards or a national and sometimes international consensus of measurement methodology can be developed for items, systems, and devices which are not readily evaluated in terms of existing measurement methods.

The performance concept introduces man into the measurements and standards process, and the whole idea centers on the belief that products, processes, services, and systems can be described and their performance can be measured in terms of user's requirements without regard to their physical characteristics, design, or method of their creation. Key to this method is the identification of measureable performance criteria for meeting the user's requirements, thereby coupling subjectively derived needs with objective technical measurement methods.

As the complexity and technical sophistication of articles of commerce increase, it becomes increasingly difficult to define those characteristics of a product which best measure its performance. The virtue of the performance concept in measurement and standard development is that it stresses the end to be attained, and leaves the means to obtain the desired end wide open.

Today many complex products are actually complete "systems," and the only feasible way to evaluate them is in terms of their performance against criteria established to meet the user's requirements. The concept encourages innovation when producers can meet the performance required by whatever design of product will do the job.

The idea of performance criteria also has ramifications beyond those concerned with articles of commerce. Our national welfare depends critically on making informed decisions related to complex social and economic policies and programs. To do this, one needs to define criteria of performance for these policies and programs and be able to measure or predict possible benefits and costs of alternative actions. Through the techniques of system analysis and operations research, technology has provided a means for helping such decision-making.

Institute activities in technological measurements and standards range from the development of performance criteria and test methods for individual items to the study and analysis of complex systems such as a multi-State transportation network.

During the past year several organizational changes were made to concentrate the Institute efforts in the technology area. The Center for Computer Sciences and the Clearinghouse for Scientific and Technical Information were separated from IAT. In addition, a product testing laboratory developed for the General Services Administration (GSA) was transferred to GSA. Added to the Institute were a

Measurement Engineering group and an Instrument Shops group. The Institute inaugurated an Office of Fire Research and Safety with responsibility for planning the programs anticipated when funds become available under the terms of the Fire Research and Safety Act of 1968.

Program elements include :

Technological Measurements and Standards

Building Technology

Electronic Technology

Systems Analysis

Motor Vehicle Safety

Engineering Materials

Industrial and Consumer Products

Technological Innovation and Diffusion

Invention and Innovation

Building Technology

The spiraling of construction costs combined with the tremendous backlog of housing units required to be produced in the next 30 years have accelerated the move to the industrialization of the building process. This change, from an industry heavily dependent on hand craftsmen techniques, to one of mass-production is generating pressures for up-dated standards, improved techniques for determining user requirements, and advanced capability for evaluating the performance of complex components. In order to meet this demand, the NBS programs for building technology have been broadened to cover more than the traditional materials research, which alone is no longer adequate to provide answers for the new questions raised by industrialization of the building process. All of these questions involve a consideration of cost to some extent. Therefore, this broadened scope must include studies of building economics. The program of economic studies has been initiated as a multiagency project, designed to develop a computerized construction cost control system that relates unit building prices, operation, and maintenance costs. This information can be used to project estimates of both building and operating costs from preliminary planning, through construction, to ultimate building use.

The scope of research in building technology in the past year was expanded by the creation of a "Sensory Environment Branch" with three sections, namely; Environmental Engineering, Psychophysics, and Building Transport Systems Section. The environmental engineering program will continue the work which has been going on with regard to the materials and equipment which create the favorable interior environment of buildings. The psychophysics program will study techniques for evaluation and measurement of the responses of building occupants to the visual, aural, thermal, and spatial char-

acteristics of the interior environment. The transport systems program will gradually be enlarged from plumbing activity to include electrical and communications systems, and food, water and fuel distribution, and movement of people. A "Mobile Acoustical Laboratory" has been acquired. This laboratory can be used at the site of existing structures.

The application of advances in building technology depends on dissemination of new information as it is developed. NBS has hosted or sponsored conferences, seminars, and symposia held throughout the year to this end; such as: Man and His Shelter—Roofing Technology—Durability of Insulating Glass—Wind Loads. . . . It is anticipated that there will be larger numbers of such conferences in the future.

A new program in urban technology has been initiated to develop an information framework of user needs for housing, especially for low-income housing. The questions to be examined include what information is germane, how it may be manipulated, where it falls in relation to other information, when it is applicable, and to whom it pertains. Criteria for user needs are derived from all sources in dividing the fields of sociology, physiology, and psychology. Where criteria cannot be developed from these sources, the program will develop substitute evaluative techniques and a basis for judgment decisions.

The building technology program continued the traditional laboratory and field support provided to Federal agencies, State, and local Government groups concerned with building construction and technology. Technical consultation and advice was made available to industry, and the staff continued to work closely with the many private standards-making groups throughout the country.

Outdoor Exposure of Building Materials—The Building Research Division completed the establishment of six outdoor sites for testing of building materials. These sites were selected to provide diverse weather conditions and are located at the following facilities:

NBS, Gaithersburg, Maryland
Fort Holabird, Baltimore, Maryland
U.S. Naval Station, Roosevelt Roads, Puerto Rico
Nellis Air Force Base, Las Vegas, Nevada
Fort Lewis, Tacoma, Washington
Fort Greely, Fairbanks, Alaska

Permanent racks for holding specimens are now in place and materials are being exposed at all sites.

Many studies are underway to evaluate the effects of these various climates on materials. Some of the current studies are concerned with (1) weathering characteristics of aluminized steel and roofing, (2) the spalling resistance of porcelain enamels, (3) the color stability of plastics, and (4) the durability of paints.

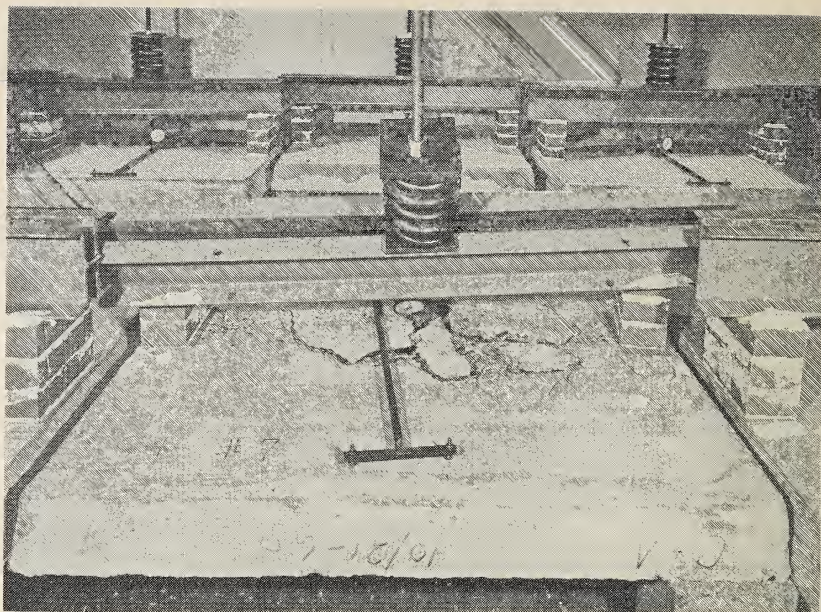
A program will be implemented to obtain or measure the exposure parameters at the sites. Static sampling devices from the Public Health Service, designated as "Effects Packages", have been placed at the NBS (Gaithersburg) and Ft. Holabird (Baltimore) sites. These devices indicate air quality and in conjunction with weather data will help establish the relative severity of exposure conditions at each site.

Wind Effects on Buildings—The effects of wind on buildings is the subject of a study jointly sponsored by the Environmental Science Services Administration and NBS. This problem has assumed great importance with the recent trend toward lighter more slender buildings and larger window panels. U.S. Weather Bureau records indicate the average yearly damage to structures by wind in the continental United States is approximately \$600 million with two years on record for which the damage exceeded one billion dollars. The study will ultimately result in improved design techniques for structures exposed to wind. Most building codes make some provision for wind loads but are largely based upon results of wind-tunnel studies in which prototype conditions were incorrectly modeled. A number of pressure sensors have been installed in the Building Research Building at the NBS site to measure both steady and fluctuating pressures over the exterior walls. Simultaneous recordings of the oncoming wind are obtained by means of fast-response anemometers on an array of six towers placed upwind of the building. Once the techniques associated with instrumentation, data acquisition, and data reduction are perfected, the study will focus on taller buildings where the response of the structural frame to aerodynamic forces is of primary concern.

Strength of Inserts Embedded in Reinforced Concrete Slabs—As the cost of construction continues to increase, more and more designers are looking for methods to optimize floor space utilization. One method commonly used is to suspend from the ceiling equipment which might otherwise be occupying premium floor space. An increasing number of devices suitable for suspending such loads are being used in industrial, institutional and commercial buildings.

One such device being used with reinforced concrete slab construction is an anchor commonly called a concrete insert. These concrete inserts are made to receive either an ordinary threaded rod or the head of a machine bolt. They are simply fastened to the formwork prior to placing the concrete. This simplicity offers advantages over other devices such as embedded anchor bolts which must penetrate the normally reusable formwork.

As far as is known, no systematic study of the factors which affect the load-carrying capacity of inserts in reinforced concrete slabs has been published. It is known that some manufacturers have investigated certain variables, but the scope and the results of the tests are not known. Ordinarily, the manufacturer's catalogs are the only source of data regarding the load-carrying capacity of most of these inserts.



To save floor space, equipment is often suspended from inserts embedded in concrete ceilings. A systematic study was made of the factors affecting the strength of these inserts. Here, an insert has been tested to failure in a sustained load test.

Because of the limited availability of concrete insert data the Building Research Division, in cooperation with the Post Office Department, conducted a comprehensive study of the variables influencing the ultimate load carrying capacity of some commonly used inserts.

In the test, the effect of cyclic fatigue on the pull-out strength of the inserts was being investigated.

In a report recently issued, design criteria for the inserts were recommended. These criteria take into consideration 10 variables commonly encountered in construction.

Study of Occupancy Loads in Postal Facilities—The Post Office Department is faced with the problem of moving an ever increasing volume of mail efficiently and rapidly from pickup to delivery. To cope with the problem, efforts are being directed toward mechanizing the mail handling and processing operations. This equipment accounts for a large portion of the occupancy load that is imposed on Post Office structures. It is expected that the loads on these facilities will increase as more and more automatic mail processing equipment is developed and installed in the buildings. Present data is inadequate for design of post office structures for the expected loadings over the useful life of the building. Consequently, the present design procedure must, of necessity, include exceedingly conservative values for loadings.

The Building Research Division has undertaken a project to study the existing loads in a sample of postal mail handling facilities across the nation. The main objective is to investigate the magnitudes and distributions of actual occupancy loads imposed on the structures for the purpose of making engineering recommendations for values of loads to be used in the design of new facilities.

The work has been in progress for approximately one year and was carried out in two phases. The initial phase of the work was to develop background information on mail handling operations, process machinery, and facility layouts, and to formulate the most suitable measurement techniques and evaluative methods for conducting the study. At the same time, load sensing devices and data acquisition instrumentation for field measurements were designed and developed in the laboratory.

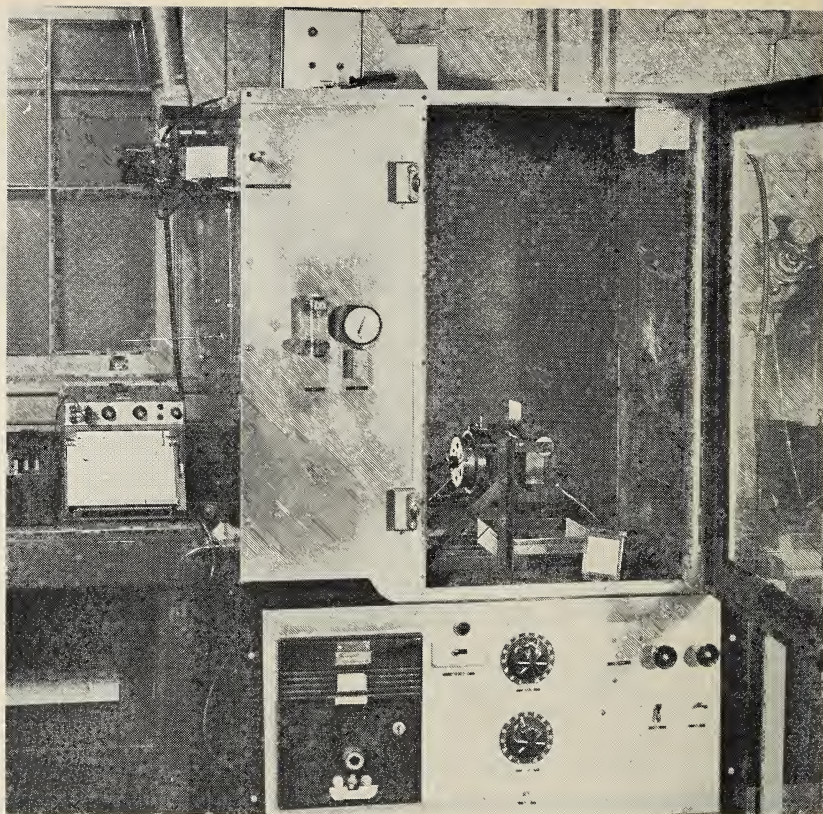
The next phase of the work was concerned with the field survey of actual loads in specific structures selected as a representative sample of Post Office mail handling facilities across the nation. Computer programs are being developed for automatic data processing and evaluation. Three facilities, located at Greensboro, N.C., Chicago, Ill., and Buffalo, N.Y., have been surveyed thus far with plans to survey at least six more over the next year.

*Measuring the Smoke Generating Characteristics of Materials—*The smoke density chamber, designed and developed at the Bureau, was put into commercial production following comparative testing and preparation of a formal proposal of method. The illustration shows the first commercially produced model. There are nine chambers in existence and 16 under delivery to testing laboratories and private corporations.

The smoke density chamber method measures the smoke generating characteristics of solid materials by monitoring the attenuation of a light beam by the suspended particulate matter generated from materials under both pyrolytic decomposition (smoldering) and flaming combustion within a closed chamber. The resulting measurement is expressed in terms of maximum specific optical density and the time to generate a specified fraction of the maximum specific optical density.

Developmental testing with the smoke density chamber resulted in several design and technique improvements. These included: (1) a multihole burner for better flame anchoring and uniform distribution during flaming exposures, (2) techniques for supporting thermoplastic materials, and (3) a procedure for evaluating the influence of delamination, cracking, peeling or other separations on the smoke-generating characteristics of laminated or faced composite materials.

*National Conference of States on Building Codes and Standards—*In fiscal year 1969, continued staff support was given to a number of States in organizing a National Conference of States on Building



The smoke-generating characteristics of materials are studied in this smoke density chamber, which measures the attenuation of light resulting from the smoke generated by smoldering or flaming materials.

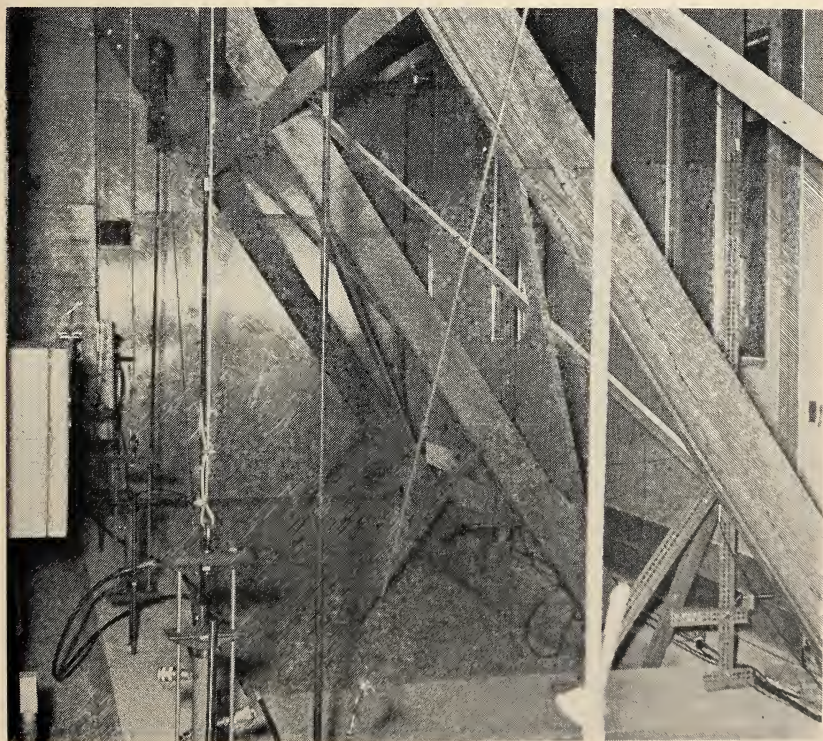
Codes and Standards. Modeled after the National Conference on Weights and Measures, the objective is to obtain more uniform building standards among the States. Since a majority of the States do not have research facilities, the National Bureau of Standards building technology program findings will serve in support of the States in the National Conference.

The second annual meeting of this group was held in April 1969, attended by representatives from 36 states, the District of Columbia and Puerto Rico. Revised procedures and by-laws were adopted, standing committees established, and the Conference unanimously approved the goal of achieving a greater degree of intrastate and interstate uniformity within and among the states in matters of building code requirements and standards.

Floor-Surface Resistance to Movements of Wheeled Vehicles—The Public Health Service has been faced with an increasing demand for

use of different floor coverings, such as carpeting, in hospital and other health care facilities. The National Bureau of Standards developed a testing procedure to measure starting and rolling friction of wheeled vehicles on various floor surfaces. A hospital bed was selected as the test vehicle to be used in the study as it is the most prevalent piece of portable equipment in the hospital. Instrumentation was devised to measure the forces required to initiate motion as well as to measure the forces required to maintain motion of the bed. Forces were measured with the bed empty and with an applied live load of 300 pounds. Nine different floor coverings were tested while the test bed was equipped with casters designated by the manufacturer as "hard" and "soft." Results show that soft coverings may offer almost five times as much resistance to movement as does resilient floor covering.

Performance Tests of a Prefabricated Building—The structural and thermal performance of a prototype prefabricated building system was determined for the U.S. Navy. The building system is being developed for use as an advanced-base, relocatable structure. The building was made of modular sandwich panels. Floor, wall and roof panels



Part of the equipment used in structural tests of a prefabricated building, one wall of which is seen at the far right.

consisted of interior and exterior aluminum skins bonded to a kraft paper-honeycomb core. The test building was prefabricated in Florida, trucked to NBS, and erected for test in the large Building Research Division environmental test chamber.

Structural tests to simulate wind loads and snow loads were made. Air leakage at the windows and doors and at the joints of the prefabricated elements was measured with a small inside pressure and revealed by smoke tests. Heat loss and condensation observations were also made.

As a result of the performance tests a number of design changes were recommended to improve the thermal and structural performance in the further development of this building system.

Field Studies of Air-Cleaning Systems in Post Offices—In cooperation with the Post Office Department, the Bureau's Building Research Division made measurements of the diurnal and seasonal variations of the airborne particulates in postal work spaces under operating conditions. The purpose of this work was to determine the performance of different types of filters and air-cleaning systems in use to help prescribe the most economical and effective ways for cleaning air. Airborne dirt is not only important for health and housekeeping reasons, but it is also a possible cause of failure in mail-handling and processing machines. The field measurements involved application of techniques used for air pollution studies as well as several new techniques developed specifically for building spaces and ventilating systems.

Electronic Technology

The electronics industry is one of the fastest growing and most heavily research-oriented in this country. In the context of R&D funds spent by the industry, NBS activities are relatively small, but the Bureau's influence is broad. Devices are being developed faster than adequate measurement methods for the control of the fabrication processes and for the characterization of the finished devices. The Bureau's contribution to the fields lies in this critical area. Its program emphasizes the development of improved basic measurement methods of significance to advances in the entire technology.

Since the Government purchases more than half of the industry output, the Bureau's work is of interest to many Federal agencies. The Bureau has undertaken joint programs on methods of measurement for semiconductor materials, process control, electronic devices and transducers. The objectives of these programs are enhancement of the performance, interchangeability, and reliability of discrete semiconductor devices, integrated circuits, and transducers and improvement of their use through better methods of measurement specifying materials and devices and in control of device fabrication processes. The

goal is to provide a set of measurement methods which have been carefully evaluated for technical adequacy, which are acceptable to both users and suppliers, and which can provide a common basis for the purchase specifications of government agencies. At the end of the fiscal year investigations were underway in a large number of measurement areas in addition to activities related to technical and information services.

Inhomogeneities—Variations in the resistivity of a silicon wafer can cause significant variations in device characteristics. In large scale integrated circuits and large area power devices these variations result in nonuniform current distributions which may be unnoticed until they cause device failure. Although it is possible to measure resistivity variations by means of a point-to-point measurement with conventional four-probe or spreading resistance techniques, a scanning technique is more suitable in many applications because it does not require the placement of probes on the region of interest. A study of photovoltaic method for measuring resistivity variations shows that this technique gives qualitative resistivity profiles on circular silicon wafers. Recent efforts have been directed toward the establishment of an analytical expression to relate the photovoltage measured on the diameter of a circular wafer to the resistivity gradient. The studies of various surface preparations have also led to the selection of a technique which reliably and consistently reduces the adverse effect of surface recombination in *p*-type silicon wafers.

Deep-Lying Trapping Levels in Indium Antimonide—Semiconducting compounds of the III-V type, such as indium antimonide, contain naturally occurring trapping centers located near the middle of the forbidden energy gap. These centers control the electrical properties of high-purity specimens from which most of the shallow doping impurities have been removed. Although observed in all reported specimens of high-purity indium antimonide, the origin of these centers remained uncertain. From results of NBS experiments in which electrical resistivity, Hall coefficient, and majority and minority carrier lifetimes in high-purity indium antimonide specimens were measured before and after the addition of lithium, the donor-like nature of the center was confirmed; a simple lattice vacancy, probably an indium vacancy, appears to be responsible for the trapping center. The trapping centers are apparently frozen in at a concentration of approximately 10^{14} cm^{-3} during crystal growth. Growth of crystals at lower temperatures, as, for example, by vapor deposition, would be expected to result in a lower concentration of vacancies.

Wire-bond evaluation—A dominant cause of device failures in both radiation and nonradiation environments is failure of the wire bond used in connecting the semiconductor chip to the external leads. Most wire bonds are of good quality and do not fail. The problem is the

early identification of those which will eventually fail in use. Survey and evaluation of methods used for determining strength of wire bond systems in semiconductor devices have been undertaken. A comprehensive study of existing procedures and techniques is nearing completion and a critical review of the present status of wire bond testing is being prepared. Systematic studies of factors which influence wire bond quality have been initiated in order to identify those parameters for which tests must be standardized. An improved method of grasping wire has been developed for use in pull tests which are commonly employed to determine the strength of the wire bond.

Radiation Damage in Silicon Nuclear Detectors—Silicon nuclear radiation detectors operating on earth-orbiting satellites and deep space probes can be destroyed by high fluxes of low-energy protons and electrons which exist in the trapped-radiation belts or which arise from transient solar events. In cooperation with the NASA-Goddard Space Flight Center, the Bureau's Electronic Technology Division has been studying radiation damage effects in silicon surface-barrier transmission detectors so that the useful lifetime of such satellite-borne detectors can be predicted. Effects of damage by protons with energies between 50 keV and 5.0 MeV have been studied; damage from electrons with energies between 100 keV and 2.0 MeV is currently being investigated. The study has shown that general guidelines for reducing the effects of damage by low-energy charged particles include (1) the operation of detectors at the highest fields possible, and (2) a reduction of the density of defects produced by radiation in the high field or barrier region of the detector. With respect to the latter point, the useful lifetime of silicon transmission detectors operating in the presence of low-energy protons was found to be extended several orders of magnitude when the detector was mounted so that the rear, ohmic contact was irradiated rather than the front, surface-barrier contact.

Thermal Management—Power dissipation in a semiconductor device is limited by the maximum permissible temperature of the semiconductor junction. To assist users in determining junction temperature under actual operating conditions, semiconductor manufacturers specify the thermal resistance and in some cases the transient thermal response of the device. These are determined indirectly by means of electrical measurements, or calculated from heat transfer considerations. In either case, accuracy and reproducibility are poor, and adequate correlation between calculated and measured values and between values obtained by different methods of measurement is lacking. Much of this difficulty results from the fact that the current distribution is usually not uniform over the area of the junction, causing localized variations in junction temperature and further discrepancy between the predicted junction temperature and the actual value at

the highest, and therefore most critical, local temperature. The seriousness of this problem has been repeatedly emphasized by suppliers and users of semiconductor devices, individually and through the Electronic Industries Association.

In response to these needs, a critical review of thermal resistance and thermal response measurements in diodes and transistors has been undertaken. The validity and reproducibility of existing methods of determining thermal resistance is being studied in the laboratory. Thermographic phosphors are being used to determine the relationship between the junction temperature predicted by the methods and the actual junction temperature in the device. A study to determine the feasibility of using temperature-sensitive phosphors to measure the temperature distribution on the surface of a transistor chip has been completed and equipment assembled to facilitate the use of such phosphors for this purpose. Better understanding of the thermal performance of semiconductor devices will permit better prediction of their power-handling capabilities under both continuous and pulse operation and will enhance efforts to detect anomalies by screening for abnormal heat dissipation.

Characterization of Germanium for Nuclear Radiation Detectors—A comprehensive program for measurement of the characterization of materials and quality of gamma-ray detectors fabricated from test specimens of germanium submitted for analysis is being carried out in cooperation with the Atomic Energy Commission.

Presently 68 germanium crystals contributed by various domestic and foreign suppliers, and U.S. and Canadian national laboratories and universities are being evaluated. The correlation of three methods for determining oxygen in germanium—infrared absorption, lithium precipitation and lithium mobility—is being studied. Lithium ion drift-mobility in germanium has been measured between 24 and 61 °C, the temperature range normally encountered in the fabrication of lithium-drifted germanium gamma-ray detectors. The values measured were higher than those previously obtained by extrapolation from data obtained above 150 °C. Based on these studies, a method for evaluating germanium crystals by determining the mobility of lithium has been developed for the American Society for Testing and Materials Committee F-1.

Piezoelectric Response of Polymers—Piezoelectric materials are used in a wide range of applications to generate and to measure motion, pressure, and flow. In many cases their applicability or their efficiency is limited because presently used piezoelectric materials are hard, stiff, dense, and brittle. Polymers provide a choice of mechanical properties ranging from those of light, soft, foam rubber to some approaching those of crystalline materials. However, the piezoelectric response of available polymers, when it exists, is too small for practical applica-

tions. NBS investigation shows that proper treatment with heat and intense electric fields could develop a piezoelectric response in some inactive polymers and significantly increase the response in polymers having slight activity. The results of the investigation suggest that improved methods of treatment could develop sufficient piezoelectric response in polymers for technological and industrial applications.

Systems Analysis

In any program for promoting the application of technology to complex problems of industry or Government, the use of systems analysis or operations research is almost a mandatory requirement. The NBS Technical Analysis Division (TAD) seeks to develop, test, and disseminate systems analysis techniques which are applicable to public sector problems in terms of program planning, resource allocation, and program execution.

The IAT systems analysis group is the largest such organization within the civilian agencies of the Government. It serves other agencies in the solution of their specific systems analysis problems, and helps these agencies to develop their own capacity to tackle complex systems problems. It also conducts research on the cost-benefit analyses for Government programs.

During FY 1969 the IAT systems analysis group started a modest research effort in standard definitions for the concepts and elements used in the economic evaluation of Government programs, particularly in the area of applied science projects. Methods were proposed for measuring the economic value of Government services to individual citizens in contrast to services for business or public agencies.

Post Office Department Mechanization Projects—A systems engineering project is currently being conducted to develop specifications for mail-handling systems. These specifications may be used by the Post Office Department Bureau of Research and Engineering as a guide in developing more efficient and effective hardware.

A parallel study identifies and quantifies the relationships between physical characteristics of letter-sized mail and mechanical mail processing. This study will define the limits of mechanical processing.

A research program at the Human Factors Laboratory of the Bureau of Research and Engineering is directed and staffed by NBS personnel from TAD's Behavioral Sciences Group. Research projects include identification of the man-machine aspects of postal mechanization. Recent studies have included evaluation of closed circuit television as a means of viewing the addresses on letter-sized mail; examination of the relationship of the number of digits in a numeric code to the processing time for a piece of mail; and a comparison of machine pacing and self-pacing in the operation of two configurations on numeric keyboards.

Maritime Consolidation Centers—In response to the U.S. Maritime Administration, the Bureau investigated the advantages of strategically locating centers where goods for export and import could be consolidated and loaded in containers. Such centers would be established at intermediate points between importers or exporters of commodities. Cargo would accrue until a full container could be shipped.

The Division analysts have developed a model for evaluating the costs and benefits which the shipping industry would derive from the establishment of such centers. It was found that the specific location of points where goods could accumulate and be containerized would be of definite advantage to the shipping industry and the U.S. Merchant Marine.

Progress in Transportation Simulation Studies—The Northeast Corridor Transportation Project sponsored by the Department of Transportation has advanced to an operational stage in which the complex of models has been used to evaluate a set of alternative transportation systems for the Northeast Corridor. A variety of new transportation technologies—high-speed rail, tracked air cushion vehicles, vertical takeoff aircraft and the like—have been examined using the models. The result of the simulation tests are undergoing review and will be used to make suggestions as to choosing among possible Federal government policies with regard to the development and implementation of intercity transportation facilities.

The Department of Housing and Urban Development asked the NBS Technical Analysis group to develop the necessary concepts for adaptation of the Northeast Corridor Transportation models for use as urban transportation analysis tools. Some of these concepts were directly applicable to the intraurban transportation scene. Three sub-models were developed for experimental purposes.

Coast Guard Operations—In response to a Coast Guard request a data base was constructed for analyzing the search and rescue operations of Coast Guard units throughout the country. A computer program was developed to edit raw data tapes provided by the Coast Guard, reformat records of operations, sort reported cases by district and operating facility and print summary statistics. The Coast Guard is using the outputs for each of several years to evaluate hypotheses concerning the activities of its shore stations.

A second project underway is the construction and exercising of a simulation model for a research and rescue force integration analysis.

Urban Studies Projects—The Technical Analysis Division embarked on a cooperative project with the International City Managers' Association, Fels Institute of the University of Pennsylvania and the Department of Housing and Urban Development to determine the applicability of systems analysis and operations research techniques to the resolutions of urban problems.

Numerous cities throughout the country have begun to apply systems techniques to the resolution of varying types of urban problems. Staff members have recently been involved in an effort to evaluate the state-of-the-art. A forthcoming publication will cite the cities using the systems methods and the problems to which they have been applied. The publication will serve as an important source document for government agencies, city staffs, consulting firms and universities active in the area of urban problems.

During FY 1969, the Division acted as "technical coach" to the city staff of East Lansing, Michigan. A model was constructed which enabled the city staff to determine the optimal number and location of fire stations necessary to protect the city now and in the future.

In cooperation with the Montgomery County School Planning Board, Maryland, a model was built to determine an optimal school districting plan. The model was applied to the elementary schools in the Takoma Park area. An operational computer code of a transportation algorithm was modified to meet the districting criteria of the Planning Board. This methodology reduces the time required to establish these districts.

Motor Vehicle Safety

During fiscal year 1969 the Office of Vehicle Systems Research in the National Bureau of Standards continued its research activities in tire systems, occupant restraint systems, braking systems, and vehicle structures. This work is sponsored by the National Highway Safety Board of the Department of Transportation in order to provide the technical basis for motor vehicle safety performance standards.

Tire Systems—A major activity of the tire research program was concerned with developing a uniform quality grading system for motor vehicle tires to enable consumers to make an informed choice in the selection of tires. The tasks involve establishing (1) standard methods of test for the several properties required to evaluate tire quality; (2) quality levels for each property; and (3) a means of conveying the information to the consumer. For this system a method for rating the relative tread wear has been developed. It requires the inclusion of various pavements surfaces, prescribed amounts of cornering, braking, and acceleration. The tests are designed so that effects of wheel position, vehicle, and weather can be estimated and removed from consideration in the evaluation scheme.

In progress is work to establish wet traction standards for tires, standards for resistance to impact damage, and methods for nondestructive examination of tires.

Occupant-Restraint Systems—Included in the second series of sled tests at the Holloman Air Force Base were experiments involving human volunteer subjects riding on automotive production seats and

restraint-simulating crash levels up to 14 g. The kinematic trajectory of these subjects has been plotted from the high speed motion picture coverage. Computer analysis of the data from these tests is producing data on velocity and movement of various body members, seat belt and shoulder harness loading, leg, and muscular reaction, etc.

New laboratory procedures are being developed for static and dynamic sled testing of restraint systems. A test method has been developed for determining the degree of resistance to lateral intrusion of various vehicle door systems.

Braking Systems—Road test procedures and instrumentation are being developed to measure the safety performance of a braking system and its components. To establish correlation between road tests and laboratory tests, research programs have included the utilization of a special purpose double-ended inertia dynamometer, a friction materials test machine, friction assessment screening test machine (FAST), and a "scale" machine from England. One of the objectives of this effort is to develop laboratory methods for safety performance standards for replacement brake linings.

Recommendations were made to the National Highway Safety Bureau for upgrading the brake fluid regulations with respect to actual performance requirements and packaging and labeling of fluids. Un-



Instrumented test vehicle used for evaluation of braking systems under normal and various types of failure conditions.

der development has been a humidity exposure test procedure to establish percent water pickup by the fluid after one to two years service. Testing facilities for automotive flexible brake hoses are nearing completion.

Engineering Materials

IAT provides advisory, consultative, and laboratory investigation services relating to selected engineering materials. The laboratory work is mainly for the development of test procedures which can be used by other testing laboratories. The staff responsible for this program works with national and international groups to establish standards for these materials. Services also include establishing a technical base for other Government agencies to set purchase specifications.

Magnetic Tapes for Satellite Recorders—The early failure of NASA flight recorders is one of the most important causes of satellite failures. New satellite designs calling for performance for several years, rather than a few months, magnify the problem. NBS has continued its study of the changes of physical and chemical characteristics of magnetic tapes during their use. The interactions of tape and head surfaces are of special concern. Tapes are operated over dummy heads of known composition, with controlled parameters of atmosphere, temperature, tape tension, and tape speed. Changes in such properties as stiffness, hardness, surface roughness, coefficient of friction, and wetting characteristics are measured. Detection and identification of material that deposits on heads is carried out by contact angle measurements, interference microscopy, and infrared spectroscopy.

Experimental tapes, expected to have improved stability, have been produced in this laboratory. These tapes are based on polyimide resins and have improved friction and hardness properties as well as high resistance to heat, chemicals, and radiation.

Textile Evaluation by the Apparel Industry—A cooperative program conducted with The Apparel Research Foundation resulted in a book, "Testing Programs for the Apparel Industry—Evaluation of Materials and Components," and a series of training programs based on the book. This work was based on a survey of the industry. The booklet describes three types of textile testing programs, testing equipment, its intended use, and approximate price. Test methods described include those of ASTM, AATCC, and the Federal Government. The training programs presented were fully subscribed and others are planned.

Plastics Technology—An example of effective consultative and advisory service is a problem undertaken for the U.S. Navy. The Navy experienced difficulty in specifying and obtained a very heavy intricate polypropylene part that would meet difficult performance require-

ments. This item is a major component of a new weapons system. NBS was able to obtain satisfactory parts, by means of a relatively new forming technique, that had both improved design and mechanical properties. At the same time the cost of these items was reduced from approximately \$450 each to about one dollar.

Collaborative Reference Program for Paper—Accurate paper testing procedures are of major concern to the pulp and paper industry. Because of the large volume of production, even small systematic errors in test values can be very costly. A collaborative reference program, administered by the Bureau at the request of the Technical Association of the Pulp and Paper Industry (TAPPI), provides a mechanism for participating laboratories periodically to check the level and uniformity of their testing in comparison with that of other laboratories. Test samples are distributed bimonthly to the participants and the results of the test determinations are returned to the Bureau for an analysis of the data. Reports, in which the laboratories are identified only by code number, are prepared bimonthly and include test results, averages, and standard deviations for individual laboratories and for the group as a whole. The first shipment of samples under the program was made in March 1969 to over 100 participating laboratories.

Thermal Stability of Modified Celluloses—Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) are useful tools for studying thermal stability, phase transitions and changes in structure of materials. These techniques have been used to obtain information on the effects of modifying cellulose through reaction with specific oxidizing agents to produce varying amounts of aldehyde, carboxyl and ketone. The nature of the carboxylic acid salt, especially calcium and aluminum salts, is of interest in relation to paper mill practice. Aluminum sulfate is widely used in papermaking, and calcium carbonate is sometimes used as a filler.

Cellulose normally shows a massive DTA endotherm at about 350 °C. The temperature of this endotherm usually is lowered by modifications made through oxidation, and by covering the carboxyls with aluminum. The temperature of this endotherm is raised if the carboxyls are converted to calcium salts. Thus, aluminum tends to unstabilize cellulose and calcium tends to stabilize it.

Aluminum sulfate that is incorporated into paper during the paper-making process is thought to contribute to instability because of hydrolysis, producing sulfuric acid. Both DTA and TGA data indicate that the effects of alum and of sulfuric acid are quite different. At the same time, it appears that cross-linking occurs in each case.

Flammable Fabrics—Various combinations of ignition, heat transfer, flame spread, and the liberation of toxic or irritant products of combustion represent the hazardous characteristics of flammable

fabrics and related materials. Research was initiated on the relationships between these fundamental characteristics and with hazards to life and property. In response to notices by the Department of Commerce that there may be need for new or amended flammability standards for wearing apparel, carpets, and rugs, several test methods are under development or evaluation as proposed standards. The notices were based in part on flammability test data obtained and analyzed by NBS on garments recovered by the Department of Health, Education, and Welfare from burn cases. NBS technical experts are cooperating with the Department of Health, Education, and Welfare in the design of better data gathering programs. NBS sponsored a two-day symposium on the measurement of flammability. The symposium was attended by over 500 representatives of industry, the academic world, and government agencies.

Engineering Standards

Standards Information—In fiscal year 1969, the Bureau, through its Office of Engineering Standards Services took a step toward improving communication among the nation's standards-writing groups by announcing the availability of information on engineering and related standards and specifications. During the past year, 1,178 standards were added to the collection, bringing the total to 19,000 from 353 U.S. trade, professional, and technical societies. A Key-Word-In-Context (KWIC) Index was compiled, which lists the standards alphabetically by key words in their titles and gives the number and sponsoring group for each standard. This Index will aid individuals in determining if there is a standard (or standards) in their areas of interest and in locating groups that could answer their questions on specific standards. Standards-writing groups will be able to use the Index before they initiate a standards project to determine if there is an existing standard covering the same subject. A need for this type of information is evidenced by the 900 inquiries concerning the KWIC Index which were received between January 1969 and the end of the fiscal year. About 5,600 inquiries regarding standards and standardization activities were received in fiscal year 1969.

Product Standards—One standard and two amendments were published, five other recommended standards were approved for publication, and 14 were circulated to industry for determination of their acceptability. Additionally, four proposed standards were circulated for comment and 29 standards were transmitted for committee approval. Reviews of 312 existing standards were in progress and recommendations for withdrawal, revision or revalidation were made on 29 standards.

Consumer Standards—Progress was made on several programs of interest to consumers in fiscal year 1969. A voluntary standard for salt

packages was approved for publication and four other consumer commodity packaging standards were initiated. These proposed standards cover toothpaste, instant nonfat milk, instant mashed potatoes, and green olives.

Another area of consumer interest concerns standards which establish size classifications, according to significant body measurements, for patterns and apparel. In this area, revisions of the "Women's" and "Boys'" standards were formally initiated; under development were a revision of the "Girls'" standard, and a new standard for male "Students," and a "How to Measure" standard.

Fair Packaging and Labeling—The National Bureau of Standards has continued to pursue the implementation of the responsibilities assigned to the Secretary of Commerce under the Fair Packaging and Labeling Act. With cooperation by producers, distributors, and weights and measures officials from the states, counties, and cities, voluntary decisions have been reached covering a significant portion of the packages subject to the statute. Evidence of industry action in this area can be seen in the tabulation below and also in the marketplace.

Percent Reduction in Package Sizes by Commodity

[Achieved by agreement as of July 1969]

Product	Percent reduction	Product	Percent reduction
Adhesive bandages-----	73	Paper towels-----	76
Dry breakfast cereal-----	52	Peanut butter-----	60
Cheese-----	36	Pickles-----	* 35
Cookies and crackers-----	23	Potato chips-----	* 33
Dry detergents-----	75	Refrigerated dough	
Instant coffee-----	20	products-----	* 24
Instant tea-----	67	Salad and cooking oils-----	53
Jellies and preserves-----	37	Soft drinks-----	* 33
Macaroni products-----	50	Syrups-----	* 20
Mayonnaise and salad		Tea bags-----	33
dressing-----	20	Toilet tissue (1 ply)-----	20
Paper napkin-----	28	Toilet tissue (2 ply)-----	36

* Estimated.

New State Standards—During fiscal year 1969, seven additional States received new State standards. The seven included Georgia, Hawaii, Kentucky, Missouri, North Carolina, Pennsylvania, and Wisconsin. To date, the Bureau, under this program, has provided new equipment to 30 States. Manufacture of additional sets of precise physical standards and instruments is continuing and the ultimate goal is to update all State weights and measures laboratories.

Basic laboratory training (two weeks) has been given to 23 State metrologists at various State laboratories.

Technical Training—The demand for weights and measures training continues to increase. This past year 31 training schools were held, an increase of five over the previous year. These schools were conducted throughout the country and at the NBS weights and measures laboratory. Training included basic laboratory techniques, advanced courses, and special purpose field work. In addition to techniques used in the laboratory, the courses cover items such as the effect of the Fair Packaging and Labeling Act on State and local enforcement policies of the weights and measures groups; field testing operations; and special training for foreign visitors, Federal agencies, and private industry.

National Conference on Weights and Measures—The 54th National Conference on Weights and Measures was held in Washington during June. Major revisions, based on the past year's experience, were made in the Model State Packaging and Labeling Regulation. These changes were made to clarify requirements of the States with respect to consumer package quantity labeling, and to assure the consistency of the Model regulation with Federal requirements under terms of the Fair Packaging and Labeling Act.

The Conference updated Handbook 44 "Specifications, Tolerances, and other Technical Requirements for Commercial Weighing and Measuring Devices."

Standards Engineering—The engineering staff of the Office of Weights and Measures identifies, analyzes, and solves technical problems in the measurement area of commerce. During the past year, they have studied and reported on such diverse subjects as wire and cordage measuring devices, a new method of calibrating rental car odometers and taximeters, and propane vapor meters. The vapor meter project resulted in the adoption of a new tentative code in NBS Handbook 44, the basic working reference used by weights and measures officials throughout the country. A study to help local weights and measures officials calibrate grain moisture meters has been initiated.

Two new publications concerned with important areas of Standards and Engineering were distributed during '69; NBS Handbook 105-1, Specifications and Tolerances for Field Standard Weights, the first in a series, and an unpublished report.

TECHNICAL SUPPORT ACTIVITIES

The Institute for Applied Technology (IAT)—Provides engineering and development assistance in applying measurement technology to specific measurement problems. These services are used by other research programs in the Bureau, and by other Federal agencies. Work of this nature involves electronic techniques or combinations of electronics with mechanical, thermal or optical methods. It also includes the production of instrument prototypes. Examples of the services follow.

Performance of AC-to-DC Voltage Transfer Standards—A recently completed standard is based on comparing the average of a rectified alternating voltage with a direct voltage, the principle inherently used by a majority of present-day AC voltmeters. With this standard, a comparison accuracy of 20 ppm (parts per million) was obtained at audio frequency, increasing to 200 ppm at 100 kHz. This work complements the earlier development of an AC-DC transfer standard based on comparing the peak value of the alternating voltage with a standard direct voltage.

Laser Emissions—In the field of length metrology, there is much interest in using particular laser emissions as intermediate length standards. Such use requires that the laser emission be calibrated against a primary standard wavelength, a process that is tedious and time-consuming when done manually. Work continued on a scanning Fabry-Perot interferometer for comparing two light emissions, the goal being to allow laser wavelength calibrations to be made quickly and automatically.

Comparing Circular Grooves—A precise gaging method for measuring and comparing circular grooves used to align the electrodes of precision capacitors. This instrument, which is not a jig or fixture, can also be used to measure precisely the diameter of circles as well as circular grooves. The importance of this instrument is reflected in the requirements of the laboratory to align circular vee grooves to an accuracy of ± 0.0001 of an inch or less.

TECHNOLOGICAL INNOVATION AND DIFFUSION

Technological innovation may be defined as that process whereby means are devised and applied for stimulating new technologies, channeling them in promising useful directions, and exploiting their use for purposes other than those for which they were originally developed. Technological diffusion, in the context of IAT programs, is largely restricted to disseminating the results of Government sponsored R&D.

Invention and Innovation

The Office of Invention and Innovation is the IAT activity which helps to develop an environment conducive to technological change. Its basic program has three aspects: providing a national basis for formulation of climate-setting Federal policies, offering assistance to inventors, and education.

In FY 1968, the Office assumed responsibility (as assigned by the Assistant Secretary of Commerce for Science and Technology) for the program, planning and budgeting activity of the Department of Commerce Technology Program. Department programs included in this responsibility are the Patent Office, Office of State Technical Services,

the NBS Clearinghouse for Federal Scientific and Technical Information and the Office itself.

Organization of American States—At the request of the Director of the National Bureau of Standards, the Office conferred with a Working Group of the Organization of American States to devise a strategy for the technological development of Latin America. The recommendations of the Working Group were considered at the 1969 Trinidad meeting of the OAS Cultural Council.

Invention Programs—Twenty-four states held invention expositions and inventors' seminars during fiscal 1969 and several other states are planning to join this effort. The programs are organized and conducted by state organizations and universities and are aimed at facilitating the licensing, development, and exploitation of inventions. NBS provides assistance in planning and undertaking the expositions and also provides experts for the seminar programs.

Invention Policy—The Office of Invention and Innovation, in cooperation with the National Inventors Council, embarked on a policy study to determine the role of the Department of Commerce in encouraging and providing assistance to independent inventors and inventor-entrepreneurs. The study will analyze the problems faced by inventors and inventor-entrepreneurs who seek to bring their inventions to fruition, describe present programs and mechanisms for aiding them, identify deficiencies in these methods of assistance, and explore alternative ways in which the Department of Commerce and other agencies could properly help to overcome these deficiencies.

Education for Innovation—A study of the role of the Department of Commerce in the education of prospective inventors, innovators and entrepreneurs is being conducted in cooperation with the National Inventors Council and the American Society for Engineering Education. It is a sequel to the 1965 National Conference on Creative Engineering Education, which was organized by the Commerce Department. The study will determine what has been done to promote creative education since the Conference and what needs and opportunities now exist with respect to the education of prospective inventors, innovators and entrepreneurs. The study will provide the basis for a report by the National Inventors Council to the Secretary of Commerce.

Innovation Studies—An analysis of policies to promote technological innovation is being performed in conjunction with the George Washington University Program of Policy Studies. This study stems from two earlier events—one, the report to the Secretary on "Technological Innovation: Its Environment and Management," and the other, an agreement by NBS with the Arms Control and Disarmament Agency to assist them in the formulation of policies that would ease the impact of a substantial cut-back in defense spending on high-technology enterprise and put to salutary use the resources thereby

released. Among the aims of the study are the development of a descriptive model of the processes by which technology is created, applied and diffused; an evaluation of specific federal programs and policies that could or do influence these processes; the development for comparative analysis of innovation profiles of selected defense and civilian industries; and the identification of key leverage points in the model where policies may be beneficially applied.

CENTER FOR RADIATION RESEARCH

The Center for Radiation Research (CRR) develops improved techniques and instruments for detecting and measuring ionizing radiation, obtains basic data on the interactions of radiation with matter, and investigates the structure of the various forms of matter. The center also maintains radiation sources and standards for providing national services essential to industrial, medical, and research applications. Radiation of concern to the Center is that with energies between approximately 5 keV and 300 MeV, produced by radionuclides, reactors, and particle accelerators.

Because of its unique facilities and competences, the Center not only has responsibility for carrying out the NBS mission in the area of radiation, but also for making its resources and capabilities available to other Government agencies, industry, and universities. This policy of facility sharing ensures efficient utilization of large multipurpose facilities, such as the reactor and linear accelerator, by making them available to a broad segment of the scientific community. A number of collaborative programs are now functioning successfully, in which university and Center scientists are jointly performing experiments of mutual interest. Approximately one-half of all the guest workers currently at NBS are working in the Center.

Although the programs of the CRR are complex and diverse, they can be grouped roughly as follows:

1. Radiation Measurement and Standards
2. Nuclear Physics Research
3. Radiation Theory
4. Structure of Materials
5. Facilities Operations
6. Technical Assistance to Others

The experimental research effort is supported by a broad theoretical effort aimed at interpretation and analysis of the results.

Radiation Measurement and Standards

Calorimetric Dosimetry by Means of Refractometry—An interferometer has been designed and constructed for measuring small changes in refractive index. In a transparent medium, the refractive index will

change due to a temperature rise resulting from radiation energy deposition. The interferometer, functioning as a refractometric calorimeter, is capable of measuring doses from 1 kilorad to 5 megarads at dose rates from 200 rad/s to 5 megarad/s. Measurements can be made, without the need for cables or heat-sensing probes, in any transparent medium for which the temperature-dependence of the index is known or can be determined. It is possible to make a spectrophotometric analysis of the irradiated sample simultaneously with the interferometric measurements.

Energy Deposition by Electrons in Aluminum—Energy-absorbed distributions at various depths in aluminum have been measured with silicon detectors for normally incident monoenergetic electrons with energies of 0.5, 0.75, and 1.0 MeV. Pulse-height distributions were recorded from silicon ($Z=14$) transmission detectors with thicknesses of about 100 and 200 μm at depth intervals of about 200 μm in aluminum ($Z=13$). From a knowledge of the response of the detectors, the pulse-height distributions were converted to energy-absorbed distributions. The data yield both (1) the probability per incident electron that a specific amount of energy will be deposited in a given layer and (2) the total energy deposited in a given layer of aluminum.

X- and Gamma-Ray Instrument and Source Calibrations—Fifty-six conventional roentgen meters with 200 condenser ionization chambers were calibrated in exposure units at various energies. Instruments capable of higher precision in exposure measurement are being submitted in greater numbers, with 20 such devices having been calibrated. In addition, the x- and gamma-ray facilities have been used for standard exposures to various kinds of thermoluminescence dosimeters.

Twenty-one small gamma-ray sources were calibrated in exposure units, including five cesium-137 sources. The capability for calibrations of small cesium-137 sources was only recently established.

A program has been drawn up for automating and computerizing the exposure-calibration facilities. Part of the equipment for automatic handling has been constructed in Bureau shops, and contracts have been awarded for additional items, including data-acquisition equipment.

Thermoluminescence Dosimetry—Work was completed on a comparison of the thermoluminescence (TL) sensitivity of LiF and $\text{CaF}_2:\text{Mn}$ for ^{60}Co gamma rays and 15-MeV electrons; no difference was found in the sensitivity of these materials exposed to the two types of radiation. The measurements of TL response of $\text{CaF}_2:\text{Mn}$ irradiated in a CaF_2 medium to electrons between 1 and 4 MeV were not reproducible, possibly because the response was influenced by a nonreproducible electron-charge distribution in the (insulating) CaF_2 medium. Work on this problem is continuing.

A search for a possible dependence of F -center formation in LiF on the gamma-ray exposure rate turned out negative for rates between about 1.5×10^3 and 5×10^6 R/h. This result is compatible with the dominant role given F -centers in some of the current models for the thermoluminescence process in LiF of TLD grade.

Dose Distributions Measured with Radiochromic Dyes—Thin foils of approximately 1 mg/cm^2 containing stabilized dye precursors have been used successfully to measure gamma-ray and charged-particle energy deposition in extended media. The production of dye by irradiation is measured spectrophotometrically in terms of increase in optical density. The response does not vary appreciably over wide ranges of radiation energy and intensity. Experimental measurements of energy deposition as a function of depth of penetration of high-energy electrons and protons in various substances have shown reasonably good agreement with theoretical Monte Carlo calculations. Energy deposition in inhomogeneous media irradiated with gamma rays has also been measured with high resolution, using the thin-dye systems placed across interfaces of different materials.

Monitoring of High-Energy Electron Beams—The development of nonintercepting beam monitors employing ferrite toroids as pulse transformers has been refined to permit measurement of high-energy pulsed beams with an accuracy of the order of 0.1 percent for pulsed current well below 1 mA. The absolute accuracy of our high-energy,



The color density of a radiochromic dye film is measured after electron irradiation. Density variations indicate changes in the dose distribution of the electron beam.

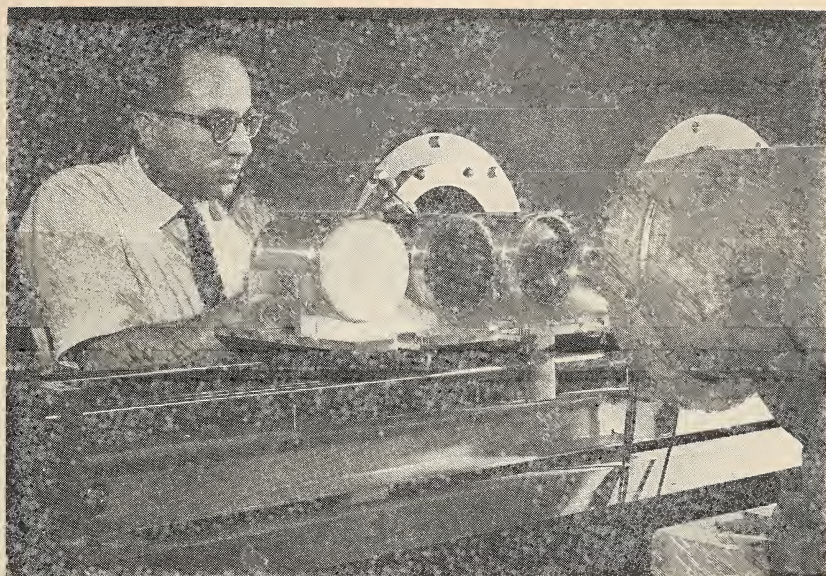
high-beam-power Faraday Cup is being studied by using the nonintercepting monitor to measure directly the difference between beam current and current collected by the Cup. These differences can be reproducibly measured to within a few hundredths of a percent of the total current. Differences of a few tenths of a percent due to secondary electrons produced in the vacuum window of the Cup and back-scattered electrons are observed and are being investigated.

Reactor Flux and Power Measurement—The neutron flux and power distribution in the NBS reactor were determined using new techniques developed at the Bureau as well as more standard methods. The usual thermal flux measurement technique of activating cobalt wires was supplemented by the activation of tungsten wires which were used to determine the epithermal flux by comparison to the cobalt measurements. The absolute value of the neutron flux was determined by activating a cobalt- ^{10}B glass bead. The relative power distribution was also determined independently by gamma-scanning each fuel element individually in a reproducible geometry. The power distribution determined in this way agreed with that from the flux measurements within a few percent, demonstrating the feasibility of the gamma-scanning technique.

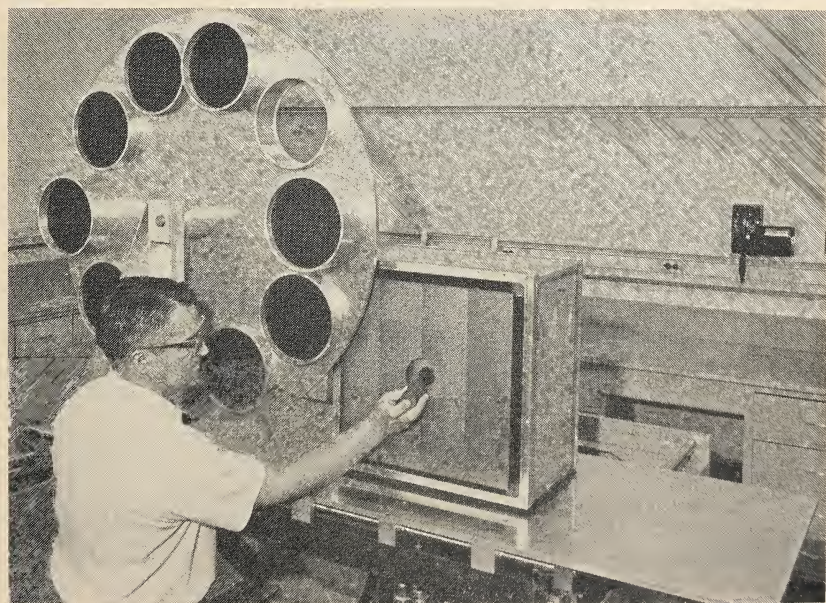
Neutron Cross Sections—The total neutron cross sections of hydrogen, aluminum, calcium, titanium, iron, and nickel were measured in the neutron energy range 0.5 to 15 MeV. The neutron time-of-flight method was employed, with the NBS electron linear accelerator used as the pulsed neutron source. The energy resolution varied from 0.2 percent to 0.9 percent; both the statistical precision and the normalization accuracy were better than 2 percent. The hydrogen cross section was measured in order to check a recent suggestion that this cross section may not be smooth, but may have some structure. Our measurements showed that the cross section was, indeed, smooth. The rest of the elements measured are important constituents of shielding or construction materials, and the measurements were made primarily for the purpose of providing data to be used in transport calculations.

Chemical Dosimetry—A study has been completed on the systematic errors which may be introduced in the spectrophotometric determination of ferric ion concentration by incorrect evaluation of a nonlinear calibration curve of the spectrophotometer. Several methods have been developed for using correctly a nonlinear relationship between absorbance and molarity, in calculation of absorbed dose.

Radioactivity Standards—A total of 341 radioactivity standards were sold in fiscal year 1969. Fifty-one source calibrations were made: 41 for other agencies and companies and 10 for NBS laboratories. Fifteen series of standards were produced totalling 1359 units, five of these series were prepared for use with Ge(Li) detectors. Two new stand-



Preparations of neutron time-of-flight experiment to measure neutron cross section of hydrogen (polyethylene sample at left-center). A compensating carbon sample is being adjusted.



Absorbed-dose measurement apparatus showing the absorber wheel and graphite calorimeter block. Remote-control operation changes the absorber thickness and moves the calorimeter and comparison standards (not shown) parallel and perpendicular to a beam of electrons or x-rays.

ards were developed: plutonium-238, which is the first NBS alpha-particle disintegration-rate standard; and thorium-228-thallium-208 gamma-ray standard, for which a new method of calibration was developed utilizing alpha-particle and gamma-ray counting methods. Work is almost complete on two new reference materials—nickel-63 solution standard and krypton-85 gamma-ray gas standard.

High-Energy Electron and Photon Absorbed-Dose Standards—A calorimeter intended to be the basis for the Bureau's absorbed-dose calibration service was constructed and all tests preliminary to actual use were completed. The instrument will be used initially to determine the dose deposited in carbon by electron beams with energies from 30 to 50 MeV.

Cobalt-60 Source Calibration—The exposure rate from a cobalt-60 source furnished by the National Research Council, Ottawa, Canada, was measured with the Bureau's standard cavity chamber. The measured rate agrees within 1 percent with the rate predicted from the power output of the source as determined by calorimetry.

High-Intensity Monoenergetic Photon Beams—Experimental studies are being carried out to develop high-intensity monoenergetic photon beams in the energy region from approximately 0.5 to 100 keV. These beams will be produced with electron accelerators by direct electron excitation of K x rays in different targets. It is expected that for direct current accelerators, the beam intensities will be approximately three orders of magnitude larger than the current methods in which K x rays are produced by photon rather than by electron excitation.

Nuclear Physics Research

High-Energy Electron Scattering—A broad program of high-energy electron scattering is conducted using the NBS electron linac. The experimental apparatus is capable of measuring the spectrum of scattered electrons with a resolution of 0.08 percent. Inelastic scattering from ^{12}C has been studied, concentrating on the excitation region 17–27 MeV. The low lying excited states of ^{16}O have been examined in an experiment made feasible by the unique high-resolution capability of the apparatus. In particular, it was possible to resolve the first two excited states near 6 MeV, separated by only 80 keV, and measure their form factors. This work is expected to contribute substantially to our understanding of the structure of this nucleus. Other experiments in progress are aimed at measuring the properties of the nuclei ^{13}C , ^{19}F , ^{46}Ti , ^{48}Ti , ^{50}Ti , ^{88}Sr , and ^{89}Y . Many of these experiments are collaborative efforts with research teams from a number of universities.

A study is in progress to measure the cross section for scattering of electrons from protons. The NBS apparatus is particularly well suited for this work in which the aim is to measure this very funda-

mental cross section with an accuracy substantially better than one percent.

Nuclear-Structure Studies by Electron and Photon Excitation—The program to investigate the systematics of nuclear energy levels excited by electrons and photons is continuing. Experiments being actively carried on include excitation of nuclear levels in isotopes of hafnium, iridium, gold, and erbium with the possible inclusion of tungsten and mercury being considered. Systematic variations in the energy structures of these nuclei are being studied and the results being compared with existing theoretical calculations.

Heavy-Ion Spectrometer Experiments—Successful and reliable operation of the heavy-ion spectrometer to measure charged particle spectra and angular distributions was achieved. Measurements were started on the cross section for the reaction, ${}^4\text{He}(\gamma, d)\text{D}$. An agreement was made with the University of Illinois to carry out a collaborative experiment to measure the cross sections for the photodisintegration of the mass-3 nuclei (${}^3\text{H}$ and ${}^3\text{He}$).

Nuclear Spectroscopy—The nuclear orientation of radioactive iridium-192 in a ferromagnetic matrix was studied in detail. The angular distribution of gamma rays and conversion electrons with respect to the nuclear spin direction was measured for eight electromagnetic transitions and the multipolarity mixtures for each of these transitions determined. A measurement of the beta ray angular and energy distributions determines the tensor rank of the matrix elements contributing to the beta transitions. A solid state beta detector and pre-amplifier operating at a temperature of 1.5 K and large germanium gamma ray detectors provided the necessary resolution and efficiency.

Inelastic Electron Scattering—Following a previous large-angle, moderate-energy experiment, the data are being extended to include higher incident energies (up to 3 MeV), forward scattering angles (less than 90°), and low atomic number targets. These parametric studies are being carried out to determine the effects of the atomic binding of the target electrons on the observed scattered electron spectra. Disturbing variations from the available theoretical calculations are still evident and the data are now in the process of being analyzed.

Inelastic Electron Scattering Cross Sections—Investigations of inelastic electron scattering for 100-, 200-, and 400-keV electrons have shown low and high energy tails in the energy spectra of inelastically scattered electrons. In addition, the angular distribution of these electrons over the region from 20 to 180 degrees has shown a sharp maximum in the region of 90 degrees.

Isospin Sum Rules for Photonuclear Reactions—The study of the electro-magnetic aspects of the three-nucleon system has continued. Three sum rules for the two isospin components in the ${}^3\text{H}$ and ${}^3\text{He}$

photodisintegration reactions have been derived. One of these rules relates the photon absorption cross sections to the charge radii of the nuclei. This rule has also been generalized to other nuclei and provides a valuable guide to the prediction of the strengths of photoabsorption cross sections of the two final isospin states.

The Photonuclear Data Center—Compilation and abstracting activities have continued along with the preparations for publication of the second supplement to Miscellaneous Publication 277, the Photonuclear Data Index. This supplement, scheduled for publication early in FY 70, will be an index to data published from January 1965 through the first half of 1969. Work was started on building up a computer-based library of photonuclear cross-section data. Over 150 cross-section curves have been digitized and entered in the data file.

Radiation Theory

High-Energy Physics—Assuming $SU(6)_w$ invariance of the meson-baryon amplitudes and a universality principle which connects these to the photoproduction amplitudes, it has been possible to describe the energy dependence of many forward photoproduction processes involving pseudoscalar and vector mesons. For example, results obtained for

the ratio $\frac{(\gamma p \rightarrow K + \Sigma^0)}{(\gamma p \rightarrow K + \Lambda)}$ disagree with the simple quark model but fit much better with the experimental results.

Encouraged by success in the photoproduction work, efforts were directed to the meson-baryon processes in an attempt to make some sense out of the apparent tremendous breakings of the symmetry there. It was found that the symmetry is broken in a rather regular, though still not understood, way which nevertheless allows quantitative predictions for forward and backward meson-baryon reaction cross sections.

Meson Effects—A model calculation of the photoproduction of π mesons from nucleons was completed, and numerical results which agree fairly well with existing experiments were obtained by fitting a single parameter. A calculation of the anomalous magnetic moment of the deuteron, allowing the admixture of nuclear resonances ($N^*_{3-3}(1236)$, $N^*_{1-1}(1470)$) in the wave function, was made. Results were such as to improve agreement of existing calculations with experiment. Finite energy sum rules were used to predict high energy behavior of meson-baryon scattering amplitudes, using low energy experimental data for these processes as input. New mathematical properties of the finite energy sum rules were explored and exploited.

Electron Transport Theory—In continuing study of electron transport using Monte Carlo techniques, calculations have been made of electron transmission and reflection, energy deposition, and brem-

ssstrahlung production in thick targets. Specific applications have included determination of the spatial distribution of energy deposition in the atmosphere by auroral electrons (2 to 20 keV), in the presence of the earth's magnetic field; calculation of the response of Si detectors to electrons with energies from 0.15 to 5.0 MeV; and calculation of the electron flux generated in water by electrons and bremsstrahlung beams with energies up to 60 MeV.

A code has been developed which will calculate the energy deposition by electrons in multilayer media. These calculations have applications in determining the dynamic response of solids by laser interferometric techniques. The production of characteristic K x rays and bremsstrahlung has also been calculated.

Quantum Electrodynamics—Higher order radiative corrections to the scattering of electrons from nuclei at high momentum transfers were studied with the aim of permitting more accurate analysis of data from the newer linear accelerators. In particular, radiative corrections to the scattering cross section in second Born approximations, and the radiative tails in the same approximation, are being calculated.

Time Reversal—The first direct demonstration that time reversal symmetry (T) is violated in K^0 meson decay has been given. Heretofore T violation has only been inferred indirectly via the known CP violation and the CPT theorem. However, CPT symmetry itself is being tested in K^0 decays. (The T test is more sensitive.) This work was published in a journal series and has received attention in the technical news magazines. It is relevant to the current search for an electric dipole moment of the neutron, in which the NBS high-flux reactor may play a role.

The mathematical techniques involved in K^0 decays have also been applied to a study of energy transfer between impurities in matter. Mechanisms of energy transfer are currently of biologic interest. Additional papers on charge transport in stressed matter and on photo-production of phonons have been prepared.

Structure of Materials

Dynamics and Interionic Forces in Phosphonium Salts—The vibrational and rotational dynamics of phosphonium halide crystals have been studied by Laser-Raman and NMR spectroscopy in cooperation with other groups at NBS and the University of Maryland. A comparison of the spectroscopic results with previous structural information suggests that the crystal potential hindering the rotation of the phosphonium ions is dominated by short-range repulsive forces. The results are in sharp contrast to those for the isostructural ammonium salts. It is hoped that continued neutron diffraction and inelastic scattering studies on these crystals, combined with theoretical cal-

culations, will yield generally consistent force models for both ammonium and phosphonium salts.

Phase Transitions in Metal Hydrides—The lattice vibrations and diffusion of hydrogen in several phases of vanadium hydride have been studied by quasielastic and inelastic neutron scattering in collaboration with scientists at Argonne National Laboratory. The $\alpha \longleftrightarrow \beta$ phase transition has been shown to be associated with a very large change in the hydrogen diffusion rate. In addition, an abnormally low activation energy for diffusion and a large concentration dependence for the diffusion is found in the α phase. These observations, coupled with significant differences in the vibration spectra for the α , β , and γ phases, suggest that the transitions are associated with a reordering of the hydrogens. The implications of these results with respect to the properties of other transition-metal hydrides are being studied.

Facilities Operations

Linear Accelerator—During fiscal year 1969 approximately 1700 hours of beam time were provided for experiments using the NBS linac. This was 63.4 percent of the scheduled beam time. Continued modifications to the accelerator and beam transport equipment have been accomplished to improve beam quality and reduce maintenance. The fraction of the scheduled time during which beam was delivered to experiments has increased 11 percent over the corresponding figure for FY 68. This increase was largely due to the fact that for about 20 percent of the year it was possible to operate the linac round-the-clock on a four day a week schedule. The beneficial impact of this round-the-clock operation was somewhat impaired by a series of injector problems, but was still very significant both to the accelerator performance and to experiments.

Experiments using the accelerator include programs in electron scattering, photoneutron production, production of heavy ions, measurement of neutron total absorption cross sections, studies in beam monitoring, tests of accelerator theory, programs in activation analysis, and production of radioactive sources for materials studies using Mössbauer sources and for studies of photonuclear reactions. Programs in electron and x-ray beam dosimetry, accelerator research, production and use of monoenergetic positron and photon beams, studies of transient chemical reactions, and studies of neutron capture gamma rays are in preparation.

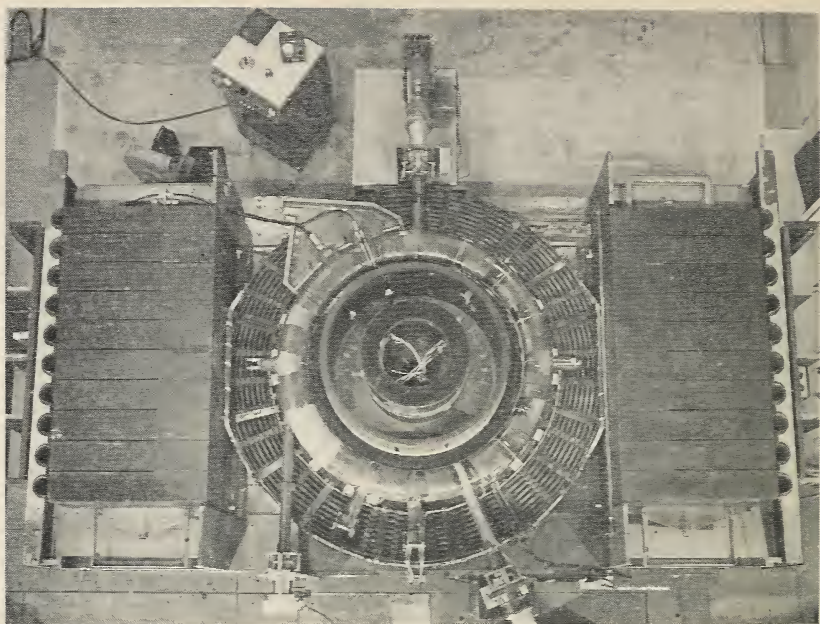
Use of the linac by organizations outside of CRR has increased appreciably during this period. For groups outside of NBS most of this use is in collaborative programs with NBS personnel. There are presently 25 Guest Workers representing 12 organizations outside NBS (mostly universities) involved in programs using the linac. Most

of these Guest Workers spend a relatively small fraction of their time at NBS, doing the analysis of their data at their home institutions. Experimental data-taking for two doctoral theses was completed this year, with preliminary data-taking started for five other doctoral candidates. Work using the NBS linac that was supported by six other government agencies was carried out during this period.

Accelerator Research—A problem of long-standing interest in the theory of linear electron accelerators is the ultimate beam current possible for extremely short, high-intensity beams. This question is directly related to the energy lost by such a beam due to microwave excitation of the accelerating structure. The loading by very short beam bursts might be expected to be large due to the broad frequency spectrum implied by short beam bursts; however, theoretical estimates of these effects differ considerably in the magnitude of the effect, and even in the dependence on electron energy. The existence of enhanced beam loading for short pulses is of particular importance for a new electron ring accelerator concept presently being studied in the USSR and at the University of California. An experiment has been performed to measure these effects using low beam currents under precisely controlled experimental conditions. The energy lost by a very short (few nanosecond) electron beam pulse in passing through an accelerating waveguide was measured. Energy shifts of a few parts in 10^5 were detectable. Enhanced beam loading, consistent with some theoretical predictions, was observed.

4-MeV Accelerator—Improvement and updating of the NBS 4-MeV Van de Graaff electron accelerator is continuing, with most modifications occurring in the peripheral experimental equipment to increase its flexibility and usefulness for the largest possible number of diverse experiments. Such improvements are typified by the addition of a new 180° scattering chamber designed to study electron-produced gamma and x rays at large scattering angles. The existing 18-in scattering chamber has also been outfitted with a new target-holding mechanism which allows the experimenter to mount a variety of different targets and to change the inclination of the target to the incident beam direction without cycling the vacuum system. In addition to the experimental programs of the Bureau, a number of outside users have and are utilizing this facility for numerous experimental projects.

180 MeV Synchrotron—The move of the NBS Synchrotron from the old NBS site to Gaithersburg was completed early in the fiscal year. Electron beam current is generally about twice what it was in Washington with better stability. The accelerator is used almost exclusively as a far ultraviolet light source by the Atomic Physics Division, IBS. With new experimental apparatus the light may be used at several points simultaneously, thus greatly increasing utilization of the accelerator.



Far ultraviolet light is transmitted through tangential port (left-foreground) of donut resting on magnet of NBS Synchrotron—seen here from above.

Isotope Separator—A large number of irradiations have been carried out with the isotope separator in the last year. Oxygen and neon isotopes were implanted into niobium foil for Pennsylvania State University for nuclear-excitation experiments; iron-57 was implanted into aluminum in connection with the development of a Mössbauer detector for the NBS Analytical Chemistry Division and the Atomic Energy Commission; aluminum-27 and chromium-52 were implanted into silicon crystals, and krypton-86, on a special target, for the Naval Research Laboratory; bismuth-209 was irradiated in the NBS linear electron accelerator and separated and examined in the low-level counting facility, with some indication of radioactivity down to mass 200. A technical note on praseodymium profiles and penetration in iron foils, in connection with nuclear orientation experiments, was presented at the International Isotope Separator Conference held in Brookhaven in September 1969.

Technical Assistance to Others

Shielding for Civil Defense—Changes in the methods used by the Office of Civil Defense to calculate shielding properties of structures against fallout, which were recommended by NBS, have been adopted by OCD. Recommendations on procedures for calculating protection from nuclear weapons are being generated by NBS.

Particle Accelerator Conference—The third Particle Accelerator Conference was held at the Shoreham Hotel, Washington, D.C. on March 5–7, 1969. Approximately 800 persons from industry, national laboratories, universities, and Government agencies attended. Although it was organized as a national conference, the proceedings were enriched by significant foreign participation. The purpose of the Conference was to promote and to facilitate communication among scientists and technologists engaged in the planning, design, and operation of all types of particle accelerators and related devices.

APPENDIXES

ORGANIZATION OF THE NATIONAL BUREAU OF STANDARDS ¹

The Bureau is headed by a Director who is appointed by the President with the advice and consent of the Senate. The Director is assisted in the overall management of the Bureau by a Deputy Director. An Associate Director for Administration is responsible for the planning and operation of facilities and of administrative management services in support of the Bureau's technical programs. An Associate Director for Information Programs is responsible for the dissemination and accessibility of scientific and technological information.

Technical program activities are conducted in three Institutes and two Centers. Each is headed by a Director who is responsible for the development and direction of research programs and central national services essential to the fulfillment of a broad segment of the Bureau's mission. These major organizational units are:

(1) The Institute for Basic Standards, which includes the Office of Measurement Services and 11 technical divisions (5 in Boulder, Colorado) each serving a classical subject matter area of science and engineering; also in IBS are 3 administrative divisions serving the technical divisions located at Boulder;

(2) The Institute for Materials Research, which consists of 6 divisions, organized primarily by technical field;

(3) The Institute for Applied Technology, which includes 10 divisions oriented to high technology industries;

(4) The Center for Radiation Research, which includes 4 divisions concerned with the theory and application of radiation; and

(5) The Center for Computer Sciences and Technology, which includes 5 divisions concerned with the selection, acquisition, and utilization of automatic data processing equipment.

¹ As of June 30, 1969.

DIRECTOR

ALLEN V. ASTIN*

DEPUTY DIRECTOR

L. M. KUSHNER

OFFICE OF THE DIRECTOR

Assistants to the Director

G. E. AUMAN

C. N. COATES

R. D. HUNTOON

A. G. MCNISH

Special Assistant for Program Planning

R. E. FERGUSON

Legal Advisor

A. J. FARRAR

Office of Academic Liaison

S. SILVERMAN

Senior Research Fellow

C. EISENHART

Staff Units Reporting to the Deputy Director

Office of Industrial Services-----G. S. GORDON

Office of Engineering Standards Liaison-----A. A. BATES

OFFICE OF ASSOCIATE DRECTOR FOR ADMINISTATION

R. S. WALLEIGH

Deputy Associate Director

P. H. SCHRADER

Patent Advisor

D. ROBBINS

Accounting Division-----J. P. MENZER

Administrative Services Division-----G. W. KNOX

Budget Division-----J. E. SKILLINGTON

*Dr. Astin retired on August 31, 1969.

Management and Organization Division_____	J. T. HALL
Personnel Division_____	G. R. PORTER
Plant Division_____	H. GRAHAM
Supply Division_____	N. H. TAYLOR

OFFICE OF THE ASSOCIATE DIRECTOR FOR INFORMATION PROGRAMS

E. L. BRADY

Assistant for Program Coordination and Evaluation
P. H. KRATZ, Acting

Office of Standard Reference Data_____	D. R. Lide, Jr.
Atomic and Molecular Data	Data Systems Design
Chemical Kinetics	Mechanical Properties
Colloid and Surface Chemistry	Nuclear Data
Information Services	Solid State Properties
Thermodynamics and Transport Data	

Clearinghouse for Federal Scientific and Technical

Information_____	H. E. Sauter
Document Distribution	Joint Publications Research
and Reproduction	Document Processing
Automated Systems and	
Services	
Administrative Operations	

Office of Technical Information and Publications_____	W. R. Tilley
Special Activities	Photographic Services
Editorial	Graphic Arts
Publications	Computer Assisted Printing

Library Division_____	E. L. Tate
Resources Development	Reader Services
Library Auxiliaries	

Office of Public Information_____	A. V. Gentilini
Office of International Relations_____	H. S. Peiser, Acting

INSTITUTE FOR BASIC STANDARDS

Director
E. AMBLER

Assistant Director
R. J. CORRUCINI

Deputy Director, Institute for Basic Standards/Boulder¹

B. W. BIRMINGHAM

Office of Measurement Services
J. M. CAMERON

Applied Mathematics-----	E. W. Cannon
Numerical Analysis	Statistical Engineering
Operations Research	Systems Dynamics
Electricity Division-----	C. H. Page
Resistance and Reactance	High Voltage
Electrochemistry	Absolute Electrical
Electrical Instruments	Measurements
Metrology Division-----	K. G. Kessler, Acting
Photometry	Length
Image Optics and Photography	Engineering Metrology
Colorimetry and Spectrophotometry	Mass and Volume
Mechanics Division-----	L. K. Irwin
Sound	Hydraulics
Vibration Measurements	Aerodynamics
Engineering Mechanics	Pressure Measurements
Rheology	Vacuum Measurements
Fluid Meters	Humidity Measurements
Heat Division-----	R. P. Hudson
Heat Measurements	Statistical Physics
Cryogenic Physics	Temperature
Equation of State	Radiation Thermometry
Atomic and Molecular Physics Division-----	K. G. Kessler
Spectroscopy	Molecular Spectroscopy
Infrared Spectroscopy	Electron Physics
Far Ultraviolet Physics	Atomic Physics
	Plasma Spectroscopy
Radio Standards Physics Division ¹ -----	H. S. Boyne
Solid State Electronics	Quantum Electronics
	Plasma Physics
Radio Standards Engineering Division ¹ -----	R. C. Sangster
HF Impedance Standards	Microwave Calibration Service
RF Transmission and Noise	Microwave Circuit Standards
RF Power and Voltage	Electromagnetic Field Standards
Time and Frequency Division ¹ -----	J. A. Barnes
Frequency-Time Dissemination	Frequency-Time Broadcast Services
Research	

¹ Located at Boulder, Colorado.

Atomic Frequency-Time Standards

Laboratory Astrophysics Division ¹ -----	L. M. Branscomb*
Cryogenics Division ¹ -----	D. B. Chelton, Acting
Cryogenic Technical Services	Properties of Cryogenic Fluids
Cryogenic Data Center	Cryogenic Systems
Cryogenic Properties of Solids	Cryogenic Metrology
Cryogenic Fluid Transport Processes	
Administrative Services Division ¹ -----	B. F. Betts
Supply	Office Services
Drafting Services	
Instrument Shops Division ¹ -----	W. A. Wilson
Instrument Shops 1 & 2	Welding-Sheet Metal
Glassblowing Shop	
Plant Division ¹ -----	E. A. Yuzwiak
Construction-Maintenance	Special Services
Custodial Services	

INSTITUTE FOR MATERIALS RESEARCH

Director
J. D. HOFFMAN

Assistant Director
E. HOROWITZ

Office of Standard Reference Materials-----	J. P. Cali, Acting
Analytical Chemistry Division-----	W. W. Meinke
Radiochemical Analysis	Microchemical Analysis
Spectrochemical Analysis	Analytical Mass Spectrometry
Electrochemical Analysis	Organic Chemistry
Analytical Coordination	Activation Analysis
Chemistry	
Separation and Purification	
Polymers Division-----	R. K. Eby, Acting
Polymer Dielectrics	Dental Research
Polymer Chemistry	Polymer Characterization
Polymer Crystal Physics	Polymer Interfaces
Molecular Properties	Thermophysical Properties
Metallurgy Division-----	E. Passaglia
Engineering Metallurgy	Corrosion
Alloy Physics	Metal Physics
Lattice Defects and Micro- structures	Electrolysis and Metal Deposition
Crystallization of Metals	
Inorganic Materials Division-----	J. B. Wachtman, Jr.
Inorganic Chemistry	Physical Properties
Inorganic Glass	Crystallography
High Temperature Chemistry	Solid State Physics
Physical Chemistry Division-----	J. R. McNesby, Acting
Thermochemistry	Mass Spectrometry
Surface Chemistry	Photo Chemistry
Elementary Processes	Radiation Chemistry

*Dr. Branscomb became director of the National Bureau of Standards on September 1, 1969.

¹ Located at Boulder, Colorado.

INSTITUTE FOR APPLIED TECHNOLOGY

Director

H. E. SORROWS, Acting

Deputy Director

M. W. JENSEN, Acting

Office of Engineering Standards Services----- D. R. Mackay
Product Standards Section Information Section
Mandatory Standards Section

Office of Weights and Measures----- T. M. Stabler, Acting

Office of Invention and Innovation----- D. V. DeSimone
Innovation Studies Program Invention Programs
Engineering Education Program

Office of Vehicle Systems Research----- P. J. Brown
Tire Systems Occupant Restraint Systems
Braking Systems

Product Evaluation Division----- S. B. Newman*
Plastics and Textiles Paper Evaluation
Fibrous Systems Fabric Flammability
Viscoelastic Materials

Building Research Division----- J. R. Wright
Structures Materials Durability and Analysis
Fire Research Codes and Standards
Scientific and Professional Liaison Building Systems
Sensory Environment Branch
Environmental Engineering Psycho-physics
Building Transport Systems

Electronic Technology Division----- M. G. Domsitz
Semiconductor Characterization Instrumentation Applications
Electron Devices Semiconductor Processing

Technical Analysis Division----- W. E. Cushen
Transportation and Highway Studies Economic Analysis
Systems Analysis and Human Development of New Methodology
Factors

Instrument Shops Division----- F. P. BROWN
Instrument Shops 1-5 Optical Shop
Glassblowing Tool Crib
Welding and Sheet Metal Electroplating Shop

Measurement Engineering Division----- G. F. MONTGOMERY
Electronic Instrumentation Electronic Optical Development

Microwave and Mechanical Instrumentation

CENTER FOR RADIATION RESEARCH

Director

C. O. MUEHLHAUSE

Deputy Director

R. S. CASWELL

Radiation Theory

Health Physics

*Now M. R. Meyerson.

Reactor Radiation Division-----	R. S. CARTER
Reactor Operations	Neutron Solid-State Physics
Engineering Services	Radiation Effects

Linac Radiation Division-----	J. E. LEISS
Linac Operations	Photonuclear Physics
Radiation Physics Instrumentation	Electronuclear Physics

Nuclear Radiation Division-----	H. H. LANDON
Nuclear Physics	Radioactivity

Nuclear Spectroscopy Section

Applied Radiation Division-----	J. W. MOTZ
X-Ray Physics	Dosimetry

CENTER FOR COMPUTER SCIENCES AND TECHNOLOGY

Director

H. R. J. GROSCH

Office of Information Processing Standards-----	J. O. HARRISON, JR.
Planning and Coordination	Software Standards
Hardware Standards	Applications and Data Standards

ADP Management Standards

Office of Computer Information-----	M. R. FOX
-------------------------------------	-----------

Computer Services Division-----	W. B. RAMSAY
Business Applications	Computer Operations
Scientific Applications	Systems Programming and Training

Systems Development Division-----	C. T. MEADOW
Programming Research	Management Systems
Information Science	Instructional Systems

Information Processing Technology Division-----	J. P. NIGRO
Measurement Automation	Computer Systems
	Performance Measurements

FIELD ESTABLISHMENTS

Institute for Basic Standards

Metrology Division Field Station:	
Visual Landing Aids Field Laboratory	Arcata, California
Time and Frequency Division Field Station:	
Standard Frequency Station WWV-WWVL-WWB	Fort Collins, Colorado
Standard Frequency Station WWVH	Maui, Hawaii
Laboratory Astrophysics Division Field Station:	
Poor Man's Relief Mine, Four-Mile Canyon	Boulder, Colorado

Institute for Applied Technology

Office of Weights and Measures Field Stations:	
Master Railway Track Depot	Clearing, Illinois

SUMMARY OF NBS STAFF AS OF JUNE 30, 1969

	Washington	Boulder	Total
Full-time permanent staff ¹ -----	2, 867	566	3, 433
Other staff ² -----	502	68	570
Total paid staff-----	3, 369	634	4, 003
Research associates and guest workers-----	135	15	150
Total NBS staff-----	3, 504	649	4, 153
Professional staff with academic degrees:			
Physicists-----	401	114	515
Chemists-----	263	6	269
Engineers-----	181	82	263
Mathematicians-----	47	4	51
Other-----	130	7	137
Total-----	1, 022	213	1, 235

¹ Includes Post Doctoral Research Fellows.

² Summer, Youth Opportunity Corps, Part-time, Intermittent and Temporary.

FINANCIAL DATA FOR FISCAL YEAR 1969

<i>Program and source of financing</i>	<i>Obligations incurred in thousands of dollars (rounded)</i>
Supported by NBS Appropriations	
Operating programs:	
Research and technical services-----	\$36, 100
Civilian industrial technology-----	39
Special foreign currency program-----	900
Construction and facilities programs:	
Plant and facilities-----	2, 441
Construction and facilities-----	2, 793
Total obligations, NBS appropriations-----	42, 273
Supported by other funds: ¹	
From other Federal agencies-----	24, 960
From other sources-----	4, 073
Total obligations, other funds-----	29, 033
Total program-----	71, 306

¹ Work supported by other funds consists of research and development programs for other Government agencies; consultative, advisory, and technical services, the performance of various tests and calibrations, and the manufacture and sale of standard reference materials for other Government agencies and the public.

RESEARCH ASSOCIATES AND GUEST WORKERS

Research Associates and Their Sponsors During Fiscal Year 1969

American Dental Association

Argentar, Mr. Harold
Bowen, Mrs. Joy C.
Bowen, Dr. Rafael L.
Brown, Dr. Walter E.
Brunetti, Dr. Anthony P.

Carlson, Mr. Elmer T.
Caul, Mr. Harold J.
Chandler, Mr. Harry H.
Gregory, Mr. Thomas M., Jr.
Kinsbury, Mrs. Pamela
Mabie, Mr. Curtis P., Jr.
Manuszewski, Mr. Richard C.
McDowell, Mr. Hershel
Moreno, Dr. Edgard C.
Paffenbarger, Dr. George C.
Palcic, Miss Julia M.
Patel, Mr. Praful
Rupp, Dr. Nelson Woodward
Wallace, Mrs. Betty M.
Waterstrat, Mr. Richard M.

American Electroplaters Society
Johnson, Mr. Christian E.

American Society for Testing and Materials
Bell, Mrs. Jacqueline Y.
de Groot, Mr. Johan H.
Evans, Mrs. Eloise H.
Grimes, Mr. John W., Jr.
McMurdie, Mr. Howard F.
Morris, Mrs. Marlene C.

American Society for Testing and Materials (Concrete and Concrete Reference Laboratory)
Anderson, Mr. Harry G., Jr.
Atkinson, Mr. George O., Jr.
Dise, Mr. John R.
Johnson, Mr. Marlin C.
Katz, Mrs. Anne K.
Liskey, Mr. John F.
McCarthy, Mr. Dennis D.
Spring, Mr. Curtis B.
Sturm, Mr. William F.
Wallace, Mr. Dennis R.

American Viscose Division (FMC Corporation)
Oneal, Mr. Glen, Jr.

Bell Aerosystems
Rogers, Ernest E.

Children's Hospital
Borasky, Dr. Rubin

Consumers Union of U.S., Inc.
Kanagy, Dr. Joseph R.

Control Data Corporation
Buckland, Mr. Stanley, F., Jr.

Corn Refiners Association, Inc.
Thomas, Mr. James H.
Vomhof, Dr. Daniel W.

Corning Glass Works
Justice, Mr. Benjamin

Dow Chemical Company
Hamilton, Mr. Robert M.

Eastman Kodak Company
Prsybylowicz, Dr. Edwin P.

Electrical Testing Laboratories, Inc.
Mohan, Mr. Kshitij

Factory Mutual Research Corporation
Kappraff, Mr. Ronald M.
Orloff, Mr. Lawrence
Rockett, Dr. John A.
Torrance, Dr. Kenneth E.

International Business Machines Corporation
Cleveland, Mr. Norman G.
Phillips, Dr. Sidney L.

Japan Electronic Industry Development Association
Yamadori, Mr. Yuji

Kennecott Copper Corporation
Harvey, W. William

Manufacturing Chemists Association
Clark, Mr. Joseph E.
Gill, Paul C.
Herndon, Mr. John L., III
Slater, James Alan

Martin Marietta Corporation (Research Institute for Advanced Studies)
Mengenbergl, Dr. Hans-Dieter

Nippon Electric Company (Japan)
Inawashiro, Mr. Tutomu

Owens-Corning Fiberglas Corporation
Fitch, Mr. William E.

Porcelain Enamel Institute
Burdick, Mr. Milton D.
Baker, Mrs. Margaret A.
Gugeler, Mr. Lauren A.

State of Maryland
Benjamin, Arthur

Underwriters Laboratories, Inc.
Castino, Mr. Guy T.

U.S. Navy Marine Engineering Laboratory
Arora, Dr. Om P.

U.S. Naval Ship Research & Development Center
Hammond, Barry L.

U.S. Steel Corporation
Martin, Mr. John F.
Swartz, John C.

ADVISORY COMMITTEES

STATUTORY VISITING COMMITTEE

The Statutory Visiting Committee was established by Act of Congress to advise the Secretary of Commerce and the Director of NBS. This committee is appointed by the Secretary of Commerce. It meets at the call of its chairman. Dates indicate expiration of appointment.

Dr. Robert L. Sproull, Vice President, University of Rochester (1971),
Chairman
Prof. Norman F. Ramsey, Dept. of Physics, Harvard University (1970)

Dr. E. R. Piore, Vice President & Chief Scientist, International, Business Machines Corporation (1972)

Dr. Elmer W. Engstrom, President, Radio Corporation of America (1973)

Dr. J. E. Goldman, Senior Vice President, Research & Development, Xerox Corporation (1974)

TECHNICAL ADVISORY PANELS

The panels advisory to the National Bureau of Standards were established in 1959 under the terms of a contract between the National Bureau of Standards and the National Academy of Sciences. The Advisory Panels are responsible to the National Academy of Sciences-National Academy of Engineering-National Research Council, Division of Physical Sciences. One panel is established for each of the three Institutes of the Bureau, and one panel for each of the major divisions within the Institutes. The advisory panels to the Institute for Applied Technology are appointed by the President of the National Academy of Engineering. All other panels are appointed by the President of the National Academy of Sciences. The divisional panels ordinarily have eight members although by special arrangement a few have more or less. Terms are usually three years.

Institute for Basic Standards

Advisory Panel to Institute for Basic Standards

Dr. William G. Amey, Leeds & Northrup Company
Dr. John A. Hornbeck, Sandia Corporation
Prof. John Todd, California Institute of Technology
Mr. Eric J. Schneider, Engis Equipment Company
Prof. S. R. Beitler, American Society of Mechanical Engineering
Dr. E. F. Hammel, University of California
Dr. Wade L. Fite, University of Pittsburgh
Prof. Sanborn C. Brown, Massachusetts Institute of Technology
Dr. Paul D. Coleman, University of Illinois
Mr. Nathan Cohn, Leeds and Northrup Company
Prof. Robert Novick, Columbia University
Dr. Clyde McKinley, Air Products and Chemicals, Inc.

Advisory Panel to Applied Mathematics Division

Prof. John Todd, Chairman California Institute of Technology
Prof. Francis J. Anscombe, Yale University
Prof. Philip J. Davis, Brown University
Prof. Charles R. DePrima, California Institute of Technology
Prof. H. O. Hartley, Texas A. & M. University
Prof. M. H. Martin, University of Maryland
Prof. Marvin L. Minsky, Massachusetts Institute of Technology
Prof. John Riordan, The Rockefeller University

Advisory Panel to Electricity Division

Dr. William G. Amey, Leeds & Northrup Company, Chairman
Prof. Raymond M. Fuoss, Yale University
Prof. H. N. Hayward, University of Illinois
Mr. William J. Johnson, Philadelphia Electric Company
Prof. George B. Hoadley, North Carolina State University
Dean R. B. Lindsay, Brown University
Mr. S. C. Richardson, General Electric Company
Mr. Douglas C. Strain, Electro Scientific Industries, Inc.

Advisory Panel to Metrology Division

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Prof. Isay A. Balinkin, University of Cincinnati
Mr. B. R. Buus, General Electric Company
Dr. Alsoaph H. Corwin, The Johns Hopkins University
Mr. J. K. Emery, The Van Keuren Company
Dr. Robert E. Hopkins, University of Rochester

Mr. Louis Polk, Dayton, Ohio
Dr. George J. Zissis, University of Michigan

Advisory Panel to Mechanics Division

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Prof. Lynn S. Beedle, Lehigh University
Dr. B. B. Dayton, The Bendix Corporation
Prof. Cyril M. Harris, Columbia University
Dr. Arthur T. Ippen, Massachusetts Institute of Technology
Dr. Harry F. Olson, RCA Laboratories
Prof. R. S. Rivlin, Lehigh University
Dr. M. E. Shank, Pratt & Whitney Aircraft

Advisory Panel to Heat Division

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Prof. G. B. Benedek, Massachusetts Institute of Technology
Prof. John E. Kilpatrick, Rice University
Dr. James E. Mercereau, California Institute of Technology
Dr. John P. McCullough, Mobil Oil Corporation
Dr. James W. Moyer, Northrop Corporation.
Prof. John G. Phillips, University of California
Prof. Howard Reiss, University of California

Advisory Panel to Atomic and Molecular Physics Division

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Prof. C. O. Alley, University of Maryland
Dr. James E. Drummond, Boeing Scientific Laboratories
Prof. Vernon W. Hughes, Yale University
Prof. Richard C. Lord, Massachusetts Institute of Technology
Prof. Robert Novick, Columbia University
Prof. W. E. Spicer, Stanford University
Prof. George Wallerstein, University of Washington

Advisory Panel to Radio Standards Physics Division

Prof. Sanborn C. Brown, Massachusetts Institute of Technology, Chairman
Dr. George Birnbaum, North American Aviation Science Center
Dr. Arnold L. Bloom, Spectra-Physics
Prof. A. Javan, Massachusetts Institute of Technology
Dr. Josua Menkes, Institute for Defense Analyses
Dr. Robert W. Terhune, Ford Motor Company

Advisory Panel to Radio Standards Engineering Division

Dr. Paul D. Coleman, University of Illinois, Chairman
Mr. Stuart L. Bailey, Atlantic Research Corporation
Mr. Ray Bailey, Newark Air Force Station
Prof. Herbert J. Carlin, Cornell University
Dr. Archie Gold, Army Ballistic Defense Agency
Mr. Theodore S. Saad, Sage Laboratories, Inc.
Mr. C. E. White, AVCO Corporation

Advisory Panel to Time and Frequency Division

Mr. Nathan Cohn, Leeds and Northrup Company, Chairman
Dr. L. S. Cutler, Hewlett-Packard Company
Dr. John M. Holt, Research Mathematics Group
Mr. Chesley Looney, National Aeronautics and Space Admin.
Dr. A. O. McCoubrey, Varian Associates
Dr. J. A. Pierce, Harvard University
Dr. Gernot M. R. Winkler, U.S. Naval Observatory

Advisory Panel to Laboratory Astrophysics Division

Prof. Robert Novick, Columbia University, Chairman
Dr. James E. Drummond, Boeing Scientific Research Laboratories
Dr. John W. Evans, Air Force Cambridge Research Laboratory
Prof. Ronald Geballe, University of Washington
Dr. Wayland C. Griffith, Lockheed Missiles & Space Company
Dr. George H. Herbig, University of California

Prof. Dudley Herschbach, Harvard University
Dr. John A. Hornbeck, Sandia Corporation
Prof. Robert P. Kraft, University of California

Advisory Panel to Cryogenics Division

Dr. Clyde McKinley, Chairman Allentown Laboratories
Dr. Frederick J. Edeskuty, Los Alamos Scientific Laboratory
Dr. Alan M. Lovelace, Air Force Materials Laboratory
Dr. William R. Lucas, National Aeronautics and Space Administration
Dr. John L. Mason, AiResearch Manufacturing Company
Dr. J. V. Sengers, University of Maryland

Advisory Panel to Institute for Materials Research

Dr. William P. Slichter, Bell Telephone Laboratories
Dr. William O. Baker, Bell Telephone Laboratories
Prof. John Bardeen, University of Illinois
Dr. Arthur Bueche, General Electric Research Laboratory
Prof. L. B. Rogers, Purdue University
Prof. Walter H. Stockmayer, Dartmouth College
Dr. J. H. Crawford, University of North Carolina
Prof. John E. Willard, University of Wisconsin

Advisory Panel to Analytical Chemistry Division

Prof. L. B. Rogers, Purdue University, Chairman
Prof. Walter J. Blaedel, University of Wisconsin
Dr. T. S. Burkhalter, Texas Instrument Company
Mr. M. D. Cooper, General Motors Corporation
Dr. John F. Flagg, American Cyanamid Company
Dr. Vincent P. Guinn, Gulf General Atomic, Inc.
Dr. N. B. Hannay, Bell Telephone Laboratories
Prof. Sidney Siggia, University of Massachusetts
Dr. Ralph E. Thiers, Bio Science Laboratories

Advisory Panel to Polymers Division

Prof. Walter H. Stockmayer, Dartmouth College, Chairman
Dr. Raymond F. Boyer, Dow Chemical Company
Dr. Frederick J. Fowkes, Lehigh University
Dr. Fred Leonard, Walter Reed Army Medical Center
Prof. Carl S. Marvel, University of Arizona
Dr. Fraser P. Price, University of Massachusetts
Prof. Duane F. Taylor, University of North Carolina
Prof. Bruno H. Zimm, University of California

Advisory Panel to Metallurgy Division

Dr. Walter A. Dean, Aluminum Company of America
Dr. G. J. Dienes, Brookhaven National Laboratory
Dr. Julius J. Harwood, Ford Motor Company
Dr. Richard A. Oriani, United States Steel Corporation
Dr. Robb M. Thomson, State University of New York
Prof. David Turnbull, Harvard University
Dr. H. G. F. Wilsdorf, University of Virginia

Advisory Panel to Inorganic Materials Division

Dr. J. H. Crawford, University of North Carolina, Chairman
Dr. George J. Bair, Corning Glass Works
Dr. Theodore L. Brown, University of Illinois
Dr. Harris M. Burte, Air Force Materials Laboratory
Prof. Clyde A. Hutchison, Jr., The University of Chicago
Dr. J. S. Kasper, The General Electric Company
Prof. John L. Margrave, Rice University
Prof. Alan M. Portis, University of California
Dr. Elias Snitzer, American Optical Company
Prof. J. H. Van Vleck, Harvard University

Advisory Panel to Physical Chemistry Division

Prof. John E. Willard, University of Wisconsin, Chairman

Professor Kyle D. Bayes, University of California
Dr. Hartwell F. Calcote, AeroChem Research Laboratories, Inc.
Dr. F. G. Ciapetta, W. R. Grace & Company
Dr. R. E. Fox, Westinghouse Electric Corporation
Prof. B. S. Rabinovitch, University of Washington
Prof. T. N. Rhodin, Cornell University
Dr. Bruno J. Zwolinski, Texas A. & M. University

Advisory Panel to Institute for Applied Technology

Mr. Kenneth C. Allen, Hobart Manufacturing Company
Dr. Robert A. Hechtman, McLean, Virginia
Dr. Leon Podolsky, Pittsfield, Massachusetts
Prof. Philip Morse, Massachusetts Institute of Technology
Dr. Jack Moshman, LEASCO Systems & Research Corporation
Dr. W. J. Harris, Jr., Battelle Memorial Institute
Dr. Carl H. Madden, U.S. Chamber of Commerce
Dean Joseph R. Passonneau, Skidmore, Owens & Merritt
Mr. Jacob Rabinow, Bethesda, Maryland
Mr. Paul Strassmann, National Dairy Products Corporation
Mr. Michael Witunski, McDonnell Douglas Corporation

Advisory Panel to Building Research Division

Dr. Robert A. Hechtman, McLean, Virginia, Chairman
Prof. Ray Clough, University of California
Mr. Edward N. Davis, Underwriters Laboratories, Inc.
Dr. J. Vincent Fitzgerald, Tile Council of America, Inc.
Dr. William H. Lindsay, Jr., Mechanical Contractors Association of Philadelphia, Inc.
Mr. Andrew R. Lolli, State Department of General Services
Mr. Raymond C. Reese, Toledo, Ohio
Dean Charles E. Schaffner, Polytechnic Institute of Brooklyn
Dr. J. A. Stavrolakis, American Standard
Mr. Herbert H. Swinburne, Nolan, Swinburne & Associates
Mr. Charles H. Topping, E. I. DuPont de Nemours & Company

Advisory Panel to Electronic Technology Division

Dr. Leon Podolsky, Pittsfield, Massachusetts, Chairman
Mr. J. A. Caffiaux, Electronic Industries Association
Mr. Ralph E. Clarridge, IBM Corporation
Mr. Ivan G. Easton, General Radio Company
Mr. H. J. Luer, Bell Telephone Laboratories
Mr. Peter R. Perino, Statham Instruments, Inc.
Dr. Robert Pritchard, Stanford University
Mr. Robert I. Scace, General Electric Company
Mr. Samuel H. Watson, Radio Corporation of America
Dr. Richard C. Webb, Colorado Instruments, Inc.

Advisory Panel to Technical Analysis Division

Prof. Philip Morse, Massachusetts Institute of Technology, Chairman
Prof. George B. Dantzig, Stanford University
Mr. Leslie C. Edie, The Port of New York Authority
Mr. Martin L. Ernst, Arthur D. Little, Inc.
Mr. David B. Hertz, McKinsey and Company, Inc.
Dr. William Linvill, Stanford University
Dr. Hugh J. Miser, Hartford, Connecticut
Prof. Thornton L. Page, Houston, Texas
Dr. George Pettee, Research Analysis Corporation
Prof. Gustave J. Rath, Northwestern University
Mr. Robert H. Shatz, United Aircraft

Advisory Panel to Center for Computer Sciences and Technology

Dr. Jack Moshman, LEASCO Systems & Research Corporation, Chairman
Mr. H. G. Asmus, American Federation of Information Processing Societies
Mr. T. E. Climis, IBM Corporation

Dr. William W. Eaton, Washington, D.C.
 Mr Elmer C. Kubie, Computer Company, Inc.
 Mr. J. Don Madden, Association for Computing Machinery
 Dr. Alan J. Rowe University of Southern California
 Mr. F. Gordon Smith, Business Supplies Corporation of America
 Dr. Willis H. Ware, The RAND Corporation

Advisory Panel to Center for Radiation Research

Dr. Marshall R. Cleland, Chairman, Radiation Dynamics, Inc.
 Dr. S. C. Abrahams, Bell Telephone Laboratories
 Dr. Peter T. Demos, Massachusetts Institute of Technology
 Dr. R. B. Leachman, Kansas State University
 Prof. E. W. Montroll, University of Rochester
 Dr. Charles J. Mullin, University of Notre Dame
 Dr. George F. Pieper, National Aeronautics and Space Admin.
 Dr. W. C. Roesch, Battelle-Northwest
 Prof. Erwin F. Schrader, Case Institute of Technology
 Prof. C. G. Shull, Massachusetts Institute of Technology

AWARDS AND HONORS

Recognition of the Bureau's contributions to science and technology often takes the form of awards and honors from Government, academic, professional, and industrial groups. The following list reflects such recognition bestowed on Bureau staff members during fiscal year 1969.

<i>Recipient</i>	<i>Award</i>
Astin, Allen V.	Elected Honorary Member, American Society of Heating, Refrigerating and Air Conditioning Engineers Elected Member, National Academy of Public Administration
Bates, Roger G.	American Chemical Society Award in Analytical Chemistry
Bennett, John	George Kimball Burgess Award, Washington Chapter of American Society for Metals
Branscomb, Lewis M.	Career Service Award, National Civil Service League
Brauer, Gerhard M.	Souder Award of the International Association for Dental Research
Foster, Bruce E.	Henry L. Kennedy Award of American Concrete Institute Frank E. Richart Award, American Society for Testing and Materials
Jacox, Marilyn E.	Recognition Award, Washington Academy of Sciences
Lloyd, Edward C.	Awarded Fellow Member Certificate by Washington Section, American Society of Mechanical Engineers
Lonnie, Mansfield (retired)	Awarded Plaque from Mail Order Association of America for work on standards of clothing sizes
Marton, Ladislaus L.	Elected Fellow, Institute of Electrical and Electronics Engineers Honorary Membership, Electron Probe Analysis Society of America
Meinke, W. Wayne	George von Hevesy Award, Journal of Radio-analytical Chemistry Special Award from American Nuclear Society Elected Honorary Member, Society Analytical Chemistry, Great Britain
Meyerson, Melvin R.	Burgess Memorial Award, Washington Chapter, American Society of Metals
Milligan, Dolphus E.	Recognition Award, Washington Academy of Sciences
Napolitano, Albert	Elected Fellow, American Ceramic Society

Paffenbarger, George C.

Parsons, Douglas E. (retired)

Taylor, John K.

Wildhack, William A.

Williams, Morgan L.

Burdick, Milton D.

Coriell, Sam R.

Currie, Lloyd A.

Dibeler, Vernon H.

Franklin, Alan D.

Hamer, Walter J.

Isbell, Horace S.

LaFleur, Philip D.

Meinke, W. Wayne

Pope, Chester I.

Scribner, Bourdon F.

Sorrows, Howard E.

Steiner, Bruce W.

Gold Medal, Pierre Fouchard Academy
Elected Honorary Fellow, International

College of Dentists

Distinguished Service Award, Underwriters'
Laboratories, Inc.

Awarded Honor Scroll, Washington Chap-
ter, American Institute of Chemists

Honorary Membership, Instrument Society
of America

District Meritorious Certificate Award by
American Welding Society

Elected Fellow, Washington Chapter of the
American Institute of Chemists

EUGENE C. CRITTENDEN AWARD

(National Bureau of Standards)

For superior performance by support personnel.

<i>Recipient</i>	<i>Area</i>
Hester, James	Electrical services
Hydro, John, Jr.	Glassblowing
Lantz, Herman L.	Floor care
Lederer, Grace S.	Procurement services
Mayers, Susan B.	Cleaning services
Michalak, John L.	Structural testing
Pittman, Arthur, Jr.	Payroll problems
Schneider, Lawrence	Precision instruments

SAMUEL WESLEY STRATTON AWARD

(National Bureau of Standards)

For outstanding scientific engineering achievements in support of the NBS mission.

David R. Lide, Jr.

EDWARD BENNETT ROSA AWARD

(National Bureau of Standards)

For outstanding achievement in the development of standards of practice, the standards by which industry judges its operations, its production processes, and the quality of its products.

W. Wayne Meinke

DEPARTMENT OF COMMERCE EXCEPTIONAL SERVICE AWARDS (Gold Medal)

Recipient

Costrell, Louis
Kostkowski, Henry J.
Kushner, Lawrence M.
Lide, David R., Jr.
Shuler, Kurt E.
Group Award:
Muehlhause, Carl O.
Landon, Harry H., Jr.
Carter, Robert S.

Technical Area

Radiation Physics Instrumentation
Radiation Thermometry
Research Management
Molecular Structure
Chemical Physics

Nuclear Radiation

DEPARTMENT OF COMMERCE MERITORIOUS SERVICE AWARDS (Silver Medal)

Recipient

Allen, David W.
Coates, Clarence N., Jr.
Cullen, William C.
Cuthill, John R.
DeVoe, James R.
Garfinkel, Samuel B.
Heinrich, Kurt F. J.
McCrackin, Frank L.
Marshak, Harvey
Martin, William C., Jr.
Oser, Hans J.
Schooley, James F.
Tilley, William R.
Walton, William W.
Weiss, Andrew W.
Wollin, Harold F.
Joint Award:
Shields, William R.
Murphy, Thomas J.

Technical Area

Atomic Frequency and Time Standards
Legislative Programs
Materials Durability
Alloy Physics
Radiochemical Analysis
Radioactivity Standards
X-ray Spectrometry
Ellipsometry
Cryogenic Physics
Atomic Spectroscopy
Systems Dynamics
Cryogenic Physics
Technical Information
Building Research
Plasma Spectroscopy
Weights and Measures

Analytical Mass Spectrometry

DEPARTMENT OF COMMERCE SUPERIOR SERVICE AWARDS (Bronze Medal)

Recipient

David, Richard M.
Garing, Herbert H.
Henley, Elizabeth L. M.
Ledford, Albert E., Jr.
Lunsford, Katherine S.
Massie, Minnie R.
Missimer, Joshua K.
Pearson, Cornelius H.
Peterson, Ruth L.
Roberts, Marion S.
Snyder, Wilbert F.
Williams, Earl S.

Technical Area

Radiation Chemistry
Instrument Shops
Administrative Issuance Systems
Molecular Energy Levels
Thermometer Calibration
Thermometer Calibration
Fibrous Systems
Thermophysical Properties
Spectroscopy
Employee Development and Relations
Radio Standards Engineering
Electrical Instruments

EDUCATION, TRAINING, AND UNIVERSITY LIAISON

A comprehensive employee development program, ranging from broad surveys to very detailed treatment of a new or specialized area of research, is available to all staff members and to members of other Government agencies and industrial personnel on a space-available basis. Courses and seminars are implemented primarily through the NBS Graduate School and special educational programs for subprofessional and non-professional employees, through non-Government educational and training facilities, and through interagency offerings. The programs range from adult basic education through postdoctoral research, and are offered at both the Boulder and Gaithersburg Laboratories. The primary objectives are to increase employee knowledge, skills, and efficiency in assigned duties in the categories more fully described below, and to prepare Bureau staff members to respond to the rapidly changing technology at all levels. Programs also include emphasis on maintaining liaison with the public, industry, commerce, and science.

In accord with Federal policy the Bureau is increasing its participation in projects aimed at stimulating community interest. Instruction by our scientists in the fields of their expertise, and facilities are being made available to the universities, public schools, professional societies, and the industrial and scientific community. In addition to numerous seminars offered through the Graduate School program and in cooperation with other Government and industrial agencies at the division and institute levels, there is the weekly National Bureau of Standards Scientific Colloquium at which current topics of broad interest to the Bureau are presented. These are open to the public and are attended by members of the university, industrial and Government communities, as well as by NBS personnel. Speakers are drawn from the outside community and from the NBS staff. Monthly colloquia under the joint sponsorship of the National Institutes of Health and the National Bureau of Standards are also offered, with each institution alternating as host.

NBS Graduate School

The NBS Graduate School curriculum includes graduate and undergraduate courses in the physical sciences, mathematics and specialized branches of engineering. A series of scientific colloquia and seminars designed to update and continue the education of the postdoctoral scientist are led by research leaders from the Bureau and from other research centers and universities. In addition, general staff development courses are offered, such as Supervision and Management, Reading Improvement, Technical Report Writing, and administrative and clerical conferences, workshops, and courses.

Two special programs, designed for technicians and subprofessional laboratory personnel, offer courses both in-house and in cooperation with the Montgomery Junior College leading to two NBS Technician Certificates and/or the A. A. degree at the Montgomery Junior College. Surveys periodically redetermine course offerings and keep the program in step with the changes and variations in educational requirements and the changing technology. Graduate degrees based partly on credit obtained for courses or thesis research carried on under the NBS Graduate School Program have been earned by 2 Bureau employees this year, bringing the total to 350 graduate degrees earned at 45 different universities since the establishment of the educational program in 1908.

The Graduate School at Boulder is associated with the University of Colorado in a Joint-Course program and Adjunct Professor Plan. Various graduate departments of the NBS Graduate School at Boulder and the University offer courses which mutually benefit the Government and the University.

Postdoctoral Research Associateships

The National Bureau of Standards, in cooperation with the National Research Council, National Academy of Sciences-National Academy of Engineering, offers a number of awards for postdoctoral research.

These awards provide young scientists of unusual ability and promise an opportunity for fundamental research in various branches of the physical, engineering, and mathematical sciences. Applications are evaluated by a Board of Selection appointed by the National Research Council. The NRC-NBS Postdoctoral Research Associateship program has been in existence since 1954. There have been 187 awards made during these years.

Postdoctoral Research Associates on Duty During Fiscal Year 1969

<i>Name</i>	<i>University</i>	<i>NBS Adviser</i>
Alderman, Donald W.	Cornell University	R. J. Mahler
Bielefeld, Michael J.	U. of Pennsylvania	J. J. Spijkerman
Borie, Edith F.	U. of North Carolina	L. C. Maximon
Cahill, Kevin E.	Harvard University	S. Meshkov
Davis, Douglas	U. of Florida	H. Okabe
DeLancey, George B.	U. of Pittsburgh	H. Oser
Denenstein, Arnold M.	U. of Pennsylvania	F. K. Harris
Duerst, Richard W.	U. of California (Berkeley)	G. F. Kokoszka
Ensign, Thomas C.	U. of Wyoming	T. Chang
Epstein, Gabriel L.	U. of California (Berkeley)	J. Reader
Fickett, Frederick R.	Oregon State University	R. Powell
Gibson, Benjamin F.	Stanford University	M. Danos
Golub, Stephen	Columbia University	B. Steiner
Greenhouse, Jeffrey A.	U. of California (Berkeley)	W. J. Lafferty
Handy, Larry B.	U. of Wisconsin	F. E. Brinckman
Hegstrom, Roger A.	Harvard University	J. H. Shirley
Hoegy, Walter R.	U. of Michigan	R. Mountain
Knoeck, John W.	Iowa State University	J. K. Taylor
Latanson, Ronald M.	Ohio State University	A. W. Ruff
Ledbetter, Hassell M.	U. of Illinois	R. P. Reed
Ott, William R.	U. of Pittsburgh	W. Wiese
Parke, William C.	George Washington University	R. W. Hayward
Pierce, Stephen J.	U. of Calif. (Santa Barbara)	M. Newman
Plummer, Earl W.	Cornell University	R. D. Young
Ponzini, Robert G.	Michigna State University	S. Meshkov
Raveche, Harold J.	U. of California (LaJolla)	R. Mountain
Searles, Stuart K.	U. of Alberta (Canada)	P. Ausloos
Shirk, James S.	U. of California (Berkeley)	A. M. Bass
Stokowski, Stanley E.	Stanford University	L. H. Grabner
Sullivan, Donald B.	Vanderbilt University	R. A. Kamper
Thornton, Donald D.	Syracuse University	B. M. Mangum
Weisman, Irwin I.	U. of California (Berkeley)	L. H. Bennett
Williams, H. T.	U. of Virginia	M. Danos
Zalewski, E. F.	U. of Chicago	R. A. Keller

Other Employee Development Programs

In addition to the programs for technicians described above, the National Bureau of Standards recognizes the need to develop and upgrade the non-professional staff, which plays an essential role in the support of the scientific staff. Therefore, courses in adult basic education are offered in cooperation with the Montgomery County Public Schools, designed to help the individual in his work. Also, foreign-born individuals who need improvement in oral and written English can enroll in classes being taught by experienced adult teachers who are aware of the problems facing the non-English speaking individual.

Non-Government Education

Non-Government education falls into three categories . . . full-time (3 to 12 months) graduate study and research assignments at universities and research centers; full-time (less than 3 months) attendance at institutes, seminars, short concentrated courses and workshops; and part-time, job-related academic courses at universities and in industry. In the last year 654 staff members at Washington and Boulder were trained through non-Government facilities, and 6 career employee were selected for full-time graduate study or research assignments at universities and research centers. Participants in approved full-time training programs receive full salary and expenses, including tuition, fees, travel, and per diem, as well as transportation of family and household effects. In addition, 648 staff members, mostly from technical divisions, attended job-related courses on a semester basis, and shorter concentrated courses at universities and in industry.

Interagency Training

Courses made available through the Interagency Training Programs are an additional effective means of improving program operations for NBS personnel. Courses are offered at Government facilities in Supervision, Management, Office Skills and Practices, and specialized programs. This pooling of agency resources offers broader employee development opportunities at a saving to the Government. During 1968, 230 National Bureau of Standards employees took advantage of the interagency offerings.

PUBLICATIONS*

PUBLICATIONS IN THE BUREAU'S SERIES

During the year NBS publications totaled 1058 published papers and documents.

Of the formal publications, 98 appeared in the *Journal of Research*, and 676 in the journals of professional and scientific societies. Also, 172 summary articles were presented in the Bureau's *Technical News Bulletin*.

In the non-periodical series, 112 documents were published: 8 in the Monograph series, 2 in the Handbook series, 28 in the Special Publication series, 6 in the Building Science series, 50 in the Technical Note series, 7 in the National Standard Reference Data Series, 1 in the Applied Mathematics series, 9 in the Federal Information Processing Standard series, and 1 in the Product Standards series.

Journal of Research. Reports National Bureau of Standards research and development in physics, mathematics, chemistry, and engineering. Comprehensive scientific papers give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Illustrated with photographs, drawings, and charts.

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, \$9.50; foreign, \$11.75. Single copy price varies. SD Catalog No. C13.22/sec.A:74.

Section B. Mathematical Sciences. Issued quarterly. Annual subscription: Domestic, \$5; foreign, \$6.25. Single copy, \$1.25. SD Catalog No. C13.22/sec.B:74.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, \$5; foreign, \$6.25. Single copy, \$1.25. S.D. Catalog No. C13.22/sec.C:74.

Journal of Research

Section A. Physics and Chemistry

J. Res. Nat. Bur. Stand. (U.S.), 72A (Phys. and Chem.), No. 4 (July–August 1968)

Mass spectrometric study of photoionization. X. Hydrogen chloride and methyl halides. Morris Krauss, James A. Walker, and Vernon H. Dibeler.

The fourth and fifth spectra of vanadium (V IV and V V). Laura Iglesias.

Variation of absorbance-curve shape with changes in pigment concentration. Gerald L. Howett.

Periodic acid, a novel oxidant of polycyclic aromatic hydrocarbons. Alexander J. Fatiadi.

*Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (foreign postage, one-fourth additional). The NBS non-periodical series are also available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151. Reprints from outside journals and the NBS *Journal of Research* may often be obtained directly from the authors.

A galvanic cell with a low emf-temperature coefficient. Gerald N. Roberts and Walter J. Hamer.

Synthesis of cerite. Jun Ito.

Tables of collision integrals for the $(m, 6)$ potential function for 10 values of m . Max Klein and Francis Smith.

Spectrum of relaxation times in GeO_2 glass. A. Napolitano and P. B. Macedo. Résumé of values of the Faraday. Walter J. Hamer.

J. Res. Nat. Bur. Stand. (U.S.), 72A (Phys. and Chem.), No. 5 (September-October 1968).

Studies in bomb calorimetry. A new determination of the energy of combustion of benzoic acid in terms of electrical units. K. L. Churney and G. T. Armstrong. Effect of low pressures on the room temperature transitions of polytetrafluoroethylene. G. M. Martin and R. K. Eby.

Electrostatic potentials and their spatial derivatives about point defects in ionic crystals. Herbert S. Bennett.

Electric fields produced by a charge density in ionic crystals. Herbert S. Bennett.

Mass spectrometric study of the photoionization of some fluorocarbons and trifluoromethyl halides. Clemente Juan Noutary.

Infrared matrix spectra of lithium fluoride. Stanley Abramowitz, Nicolo Asquista, and Ira W. Levin.

Electronic transition moment integrals for first ionization of CO and the A-X transition in CO^+ . Some limitations on the use of the r -centroid approximation. Paul H. Krupenie and William Benesch.

The configurations $3d^N4p$ in doubly ionized atoms of the iron group. C. Roth.

Electron impact excitation of hydrogen Lyman- α radiation. Robert L. Long, Jr., Donald M. Cox, and Stephen J. Smith.

J. Res. Nat. Bur. Stand. (U.S.), 72A (Phys. and Chem.), No. 6 (November-December 1968).

Interaction energy surfaces for $\text{Li}(2^3\text{S})$ and $\text{Li}(2^3\text{P})$ with H_2 M. Krauss.

Energy levels and classified lines in the first spectrum of technetium (Tc I). W. R. Bozman, C. H. Corliss, and J. L. Tech.

The first spectrum of tungsten (W I). Donald D. Laun and C. H. Corliss.

Theoretical interpretation of the even levels in the first spectrum of tungsten. Y. Shadmi and E. Caspi.

Stable carbon isotope ratio measurement with a gas density meter. S. P. Wasik and W. Tsang.

The single crystal spectrum of hexakis(imidazole)nickel(II) nitrate. Curt W. Reimann.

Crystalline alpha and beta forms of 3-O- α -D-glucopyranosyl-D-arabinopyranose. Horace S. Isbell, Harriet L. Frush, and J. D. Moyer.

Preparation and solubility of hydroxyapatite. E. C. Moreno, T. M. Gregory, and W. E. Brown.

High temperature dehydroxylation of apatitic phosphates. T. Negas and R. S. Roth.

J. Res. Nat. Bur. Stand. (U.S.), 73A (Phys. and Chem.), No. 1 (January-February 1969)

Specific heats of oxygen at coexistence. Robert D. Goodwin and Lloyd A. Weber. Specific heats C_p of fluid oxygen from the triple point to 300 K at pressures to 350 atmospheres. Robert D. Goodwin and Lloyd A. Weber.

Thermodynamic properties of fluid oxygen at temperatures to 250 K and pressures to 350 atmospheres on isochores at 1.3 to 3.0 times critical density. Robert D. Goodwin.

Two new standards for the pH scale. Bert R. Staples and Roger G. Bates.

Calculation of diffusion coefficients in ternary systems from diaphragm cell experiments. P. R. Patel, E. C. Moreno, and T. M. Gregory.

Calculated line strength for the transition array $(3d^3+3d^24s)-3d^24p$ in Ti II . H. Mendlowitz.

Morphological stability of a cylinder. Sam R. Coriell and Stephen C. Hardy. Distorted tetrahedra in strontium copper åkermanite. Jun Ito and H. Steffen Peiser.

Tritium-labeled compounds XII. Note on the synthesis of D-glucose-2-*t* and D-mannose-2-*t*. Horace S. Isbell, Harriet L. Frush, C. W. R. Wade, and A. J. Fatiadi.

A Survey of blemishes on processed microfilm. C. S. McCamy, S. R. Wiley, and J. A. Speckman.

J. Res. Nat. Bur. Stand. (U.S.), 73A (Phys. and Chem.), No. 2 (March–April 1969)

Color representation of electron microprobe area-scan images by a color separation process. Harvey Yakowitz and Kurt F. J. Heinrich.

Configurations $3d^n4p$ in singly ionized atoms of the iron group. C. Roth.

Configurations $3d^n4p+3d^{n-1}4s4p$ in Sc II, Ti II, and V II. C. Roth.

Effective interactions in the even configurations of the third spectra of the iron group. Y. Shadmi, E. Caspi, and J. Oreg.

Test of a kinetics scheme: Emission in $H(^2S) + NO(^2II)$. M. Krauss.

A nuclear magnetic resonance and relaxation study of dimethoxyborane. T. C. Farrar and T. Tsang.

The effects of low energy irradiation on organometallics. Organometal halides of group IVA. F. E. Brinckman, Gerald F. Kokoszka, and Norman K. Adams, Jr.

Measurements of gaseous diffusion coefficients by a gas chromatographic technique. S. P. Wasik and K. E. McCulloh.

Phase relations in the Ru- IrO_2 system in air. C. L. McDaniel and S. J. Schneider.

Heats of reaction of natural rubber with sulfur. Norman Bekkedahl and James J. Weeks.

A table of rotational constants of symmetric top molecules giving rise to microwave spectra. Matthew S. Lojko and Yardley Beers.

Energy levels, wave functions, dipole and quadrupole transitions of Fe^{+++} ions in sapphire. Jacques Lewiner and Paul H. E. Meijer.

J. Res. Nat. Bur. Stand. (U.S.) 73A (Phys. and Chem.), No. 3 (May–June 1969)

New even levels and classified lines in the first spectrum of tungsten (W I) C. H. Corliss.

The heat of combustion of beryllium in fluorine. K. L. Churney, and G. T. Armstrong.

Deuterium isotope effect on the dissociation of weak acids in water and deuterium oxide. R. A. Robinson, Maya Paabo, and Roger G. Bates.

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PATENTS

The following U.S. Patents have been granted to NBS inventors and assigned to the United States of America, as represented by the Secretary of the Department noted in parentheses:

- Ruehrwein, Robert A., and Hashman, Joseph S., No. 3,378,351, April 16, 1968. Complex Compounds of Molecular Oxygen (Army).
- Wall, Leo A., and Antonucci, Joseph M., No. 3,394,190, July 23, 1968. Synthesis of Perfluoroparacresol, Polyoxyperfluorobenzylene and Related Monomers and Polymers (Navy).
- Henig, Seymour, and Palasky, Ervin C., No. 3,397,392, August 13, 1968. Information Storage and Category Selector (Commerce).
- Thompson, Moody C., Jr., and Vetter, Maurice J., No. 3,400,330, September 3, 1968. Refractometer That Measures the Difference in Refractive Indices of a Gas at Two Frequencies (Commerce).
- Carlson, John G., No. 3,410,100, November 12, 1968. High Vacuum Baffle Using Cooled Chevron-Shaped Members (Commerce).
- Cruz, Jose E., No. 3,417,350, December 17, 1968. Variable Impedance Device with Relative Rotation Between Conductors (Commerce).
- Bender, Peter L., and Owens, James C., No. 3,424,431, January 28, 1969. A Distance Measuring Instrument Using a Pair of Modulated Light Waves (Commerce).
- Wall, Leo A., and Antonucci, Joseph M., No. 3,429,935, February 25, 1969. High Temperature Substitution Reactions of Hexafluorobenzene (Navy).
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- Thompson, Moody C., Jr., and Wood, Lockett E., No. 3,437,821, April 8, 1969. Radio—Optical Refractometer for Measuring Integrated Water-Vapor Refractivity (Commerce).

