NBS TECHNICAL NOTE **714**

Reactor Radiation Division:

Annual Progress Report for the Period Ending October 31, 1971

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[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.
[2] Part of the Center for Radiation Research.
[3] Located at Boulder, Colorado 80302.

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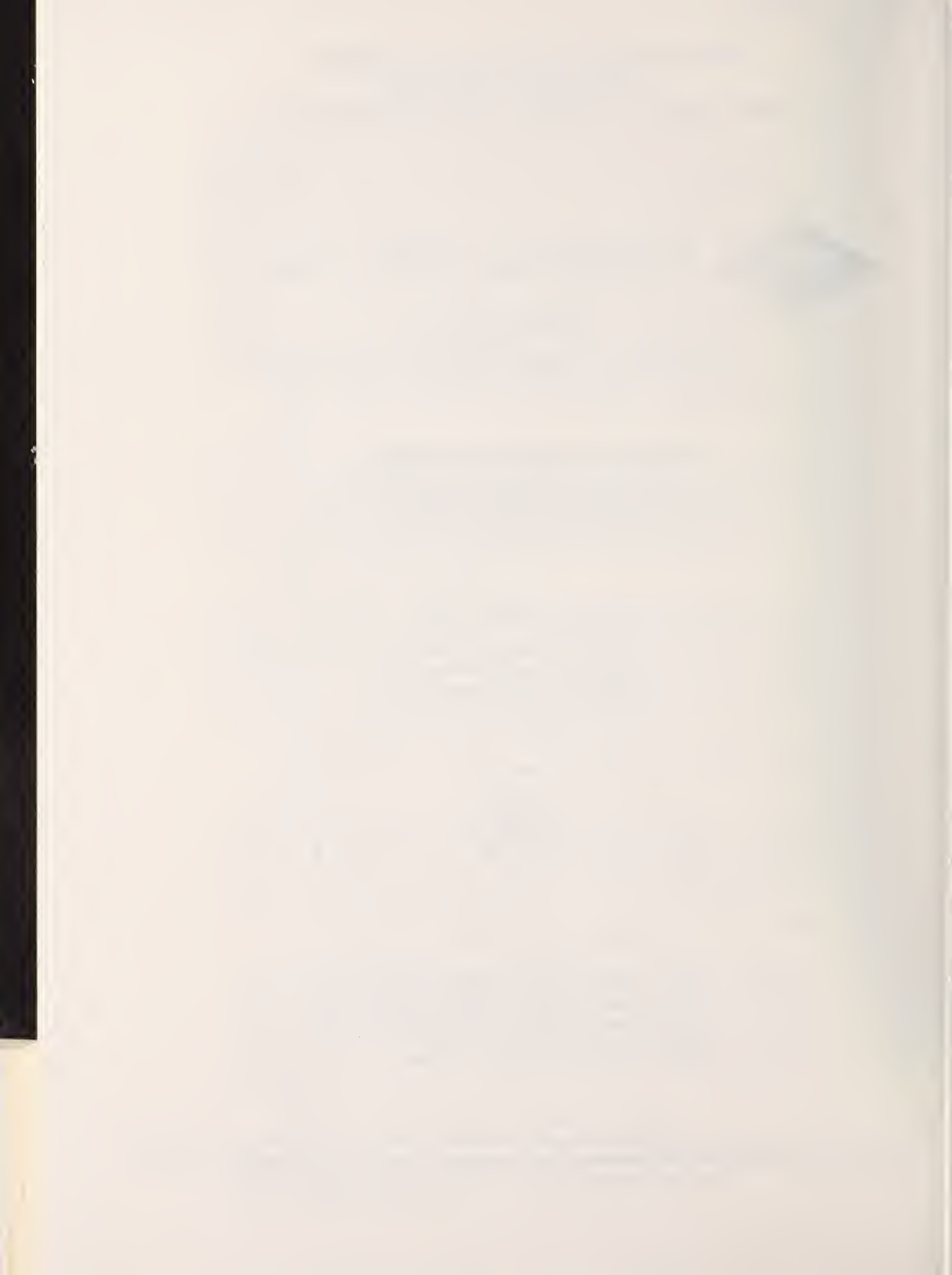
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Reactor Radiation Division:

Annual Progress Report
for the Period Ending October 31, 1971

Robert S. Carter, Editor

Reactor Radiation Division
Institute for Materials Research
National Bureau of Standards
Washington, D.C. 20234

FOREWORD

The National Bureau of Standards Reactor was built not only to serve the needs of the NBS but also those of the greater Washington Scientific Community and other government agencies. The Reactor Radiation Division was established to operate the reactor and to foster its scientific and technological use. Toward this end, the Division has a small nucleus of scientists experienced in the use of reactors for a wide range of scientific and technical problems. In addition to pursuing their own research and developing sophisticated experimental facilities, they actively seek out and encourage collaboration with other scientists, engaged in challenging programs, whose work can benefit from use of the reactor, but who as yet do not have the reactor experience necessary to take full advantage of the facilities available. The Division also provides irradiation services to a wide variety of users as well as engineering and other technical services.

The reactor operates at 10 Mw and is designed to provide a broad spectrum of facilities ranging from intense neutron beams to extensive irradiation facilities, making it one of the most versatile high flux Research Reactors in the country. Thus it is able to serve a large number of scientists and engineers in a broad range of activities both within and outside the NBS.

Use of the reactor by individuals and organizations outside the Reactor Radiation Division has continued at a high level during the past year. Thirty-three scientists from other parts of the Bureau, representing 20 man-years of effort, use the reactor regularly either directly or in cooperation with scientists in the Division. An additional 80 scientists, engineers, and technicians from other agencies, universities and industry spent 34 man-years of effort using the reactor during the past year.

The purpose of this report is to summarize the activities of the large number of users outside the Division as well as the work in the Division. The summaries are divided into two groups. One is the research activities and the other is the operation and service activities. The research activities are further broken down into three groups: the first includes all the work of the Division and most of the collaborative work; the second describes the activities of our formal interagency collaboration based on long-term arrange- ments where scientists from other agencies are actually stationed at the reactor; and the third catagory is the summary of activities of other NBS divisions which use the reactor directly rather than through collaboration with Reactor Radiation Division scientists.

R. S. Carter, Chief
Reactor Radiation Division

REACTOR RADIATION DIVISION

Annual Progress Report
for the period ending October 31, 1971

Edited by R. S. Carter

This report is the annual progress report of the Reactor Radiation Division for the period October 1, 1970 to October 1, 1971. It summarizes the activities of the large number of users outside the Division as well of the work in the Division.

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RESEARCH PROGRAMS

A. Division and Interdivision Collaborative Programs

Thermal Neutron Flux Measurements
V. W. Myers and M. Ganoczy

A bath consisting of manganese sulfate dissolved in water is being used at one of the NBSR beamports. The purpose of the experiment is to have a known thermal flux in which cobalt glass beads can be activated. A neutron beam of the order of a millimeter radius is directed into the bath, and the system is calibrated by the addition of a known ^{56}Mn activity into the bath. Cobalt glass beads activated in the known thermal flux can then be used for calibrating a counting apparatus. An unknown thermal flux can then be measured by activating an identical cobalt glass bead in the flux and then counting it in the calibrated counting system.

Intercomparison of Neutron Diffractometers
V. W. Myers

The Reactor Division is collaborating in a program sponsored by the International Union of Crystallography for comparison of neutron flux at various neutron diffraction instruments around the world. Gold foils are activated at the spectrometers and then sent to one of the following laboratories for counting: Australian Atomic Energy Commission: Institutt for Atomenergi, Kjeller, Norway; NBS. The NBS counting apparatus was calibrated by activating a gold foil in the thermal neutron density standard maintained at the Center for Radiation Research. The Kjeller and NBS counting facilities have been intercompared by means of a gold foil that was irradiated at Kjeller and counted at both of these facilities. The Kjeller and NBS results on the gold foil's d.p.s. differ only about 1 percent. This is satisfactory since the two counting facilities were calibrated independently of one another, and the inherent accuracy of each facility is of the order of 2 percent.

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Group Theoretical Analysis of Neutron Scattering in Crystals

R. C. Casella

and

S. F. Trevino
(Picatinny Arsenal)

A general program for employing projection operator techniques (first suggested by Elliott and Thorpe) to study the effects of crystal symmetry on the structure function for inelastic neutron scattering is being implemented. Certain logical difficulties in the original proposal have been overcome and simplifications introduced. The method has been applied to $NaNO_3$ (See K. W. Logan, S. F. Trevino, R. C. Casella, et al., Interagency Collaborative Programs, Sec. B) and a paper submitted for publication. Further work on Brillouin-zone-boundary and other general related problems is in progress and expected to continue.

Asymptotic Scattering Theorems

R. C. Casella

Work on the form of the elastic cross section $d\sigma/dt$, (t= momentum transfer) when total cross sections satisfy $\sigma(\bar{A}B) \neq \sigma(AB)$ as the square of the c.m. energy $s \to \infty$ has continued, based upon Casella's earlier discovery of an infinite number of zeros in the analytic continuation of the scattering amplitude in the variable $x = (lns)^2 (-t)$. [$\bar{A}$=anti-A]. The degree to which the zeros approach the physical axis in this variable determines the amount of dampening of possible oscillations in $d\sigma/dt$ caused by the zeros. A paper was submitted for publication in the "Ettore Majorana" series, edited by A. Zichichi. Work has begun in an attempt to understand the nature of inclusive reactions, $A + B \to C +$ anything, and the origin of the emperical result, max J(resonance) $\propto s$, in view of the unitarity relation, max J(total) $\propto \sqrt{s}$ lns. [J= angular momentum].

Properties of Crystal Lattices: The Derivative Lattices and Their Determination

A. Santoro

and

A. D. Mighell
(Inorganic Materials Division)

A method has been developed for driving the superlattices and sublattices from a general lattice, and a Table of Matrices $\underset{\sim}{S}$ for generating all unique superlattices associated with a lattice for $\underset{\sim}{S} = 2,3,4$ has been derived. The matrices for generating the unique sublattices can be obtained by taking the transpose of the inverse of the matrices $\underset{\sim}{S}$.

The Structure of $[Co(Imidazole)_6]\ (NO_3)_2$

A. D. Mighell and C. W. Reimann
(Inorganic Materials Division)

and

A. Santoro and E. Prince

As part of a continuing study of compounds containing metal-organic complex cations, the structure of $[Co(Imidazole)_6]\ (NO_3)_2$ has been determined by x-ray and neutron diffraction. This study was carried out in order to obtain accurate parameters of the imidazole molecule and to work out the details of hydrogen bonding in the packing of the cations. In addition, a comparison of the results of the x-ray and neutron diffraction determination is of interest in itself, as only a few compounds of this complexity have been studied with both techniques. Finally neutron diffraction is particularly suited for this complex, as the crystal shows no damage when exposed to neutrons.

In Table 1 the bond distances and angles for the imidazole ring found in the recent study are reported for the x-ray determination (column 4) and for the neutron determination (column 5). Corresponding quantities agree quite closely. In the table, the bond distances and angles obtained in the present study are compared with those found in molecular imidazole (column 2), in $Im_6Ni(NO_3)_2$ (column 3), $Im_6Cd(NO_3)_2$ (column 6) and $Im_6Cd(OH)$ $(NO_3) \cdot 4H_2O$ (column 7). The data in Table 1 shows that although the interaction between the rings and their environment is not the same in all the compounds compared, the geometry of the imidazole ring is remarkably constant.

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The positions of the hydrogen atoms were determined by neutron diffraction and the bond distances involving these atoms are given in Fig. 1. This unambiguous location of the hydrogen atoms completely clarifies the hydrogen bonding in the crystal. Fig. 2 shows a projection of the structure of $Im_6Co(NO_3)_2$. Note that the distances between the nitrogen atom N(2) and the oxygen atoms O(1), O(2), and O(3') are all consistent with hydrogen bonding between the hydrogen atom H(4) (attached to N(2)) and these oxygen atoms. However, the neutron study shows conclusively that the distance H(4)...O(2) is 2.44Å and the distance H(4)...O(3') is 2.79Å and the distance H(4)...O(1) is 1.99Å. As two of these H...O distances are greater than the 2.4Å limit for hydrogen bonding to oxygen (Hamilton and Ibers, Hydrogen Bonding in Solids, Benjamin Inc., New York, 1968), only the H(4)...O(1) interaction is a hydrogen bond.

The complex cation Im_6Co^{++} determined with x-ray and neutron diffraction methods is illustrated in Figs. 3 and 4, respectively. A comparison between the two figures shows that the shapes and the orientation of the thermal motion ellipsoids are very similar for the two determinations. In the imidazole rings the directions of the largest vibration tend to be perpendicular to the planes of the rings. This is particularly evident if one observes the thermal ellipsoids of the hydrogen atoms in Fig. 4.

TABLE 1. Unique bond distances and angles in $Im_6Co(NO_3)_2$ and in related compounds.

	Imidzaole* -150 C	$Im_6Ni(NO_3)_2$	$Im_6Co(NO_3)_2$ **	$Im_6Co(NO_3)_2$ ††	$Im_6Cd(NO_3)_2$ †††***	$Im_6Cd(OH)(NO_3)\cdot4H_2O$ †††
M^{++}-N(1)		2.129Å	2.1599Å	2.1729Å	2.361(3)Å	2.364(3)Å
N(1)-C(1)	1.326Å	1.316	1.324	1.313	1.316(4)	1.313(7)
C(1)-N(2)	1.349	1.329	1.334	1.339	1.325(5)	1.333(6)
N(2)-C(2)	1.369	1.352	1.357	1.358	1.357(6)	1.361(8)
C(2)-C(3)	1.358	1.366	1.354	1.358	1.340(6)	1.331(8)
C(3)-N(1)	1.378	1.372	1.378	1.368	1.366(5)	1.370(6)
C(3)-N(1)-C(1)	105.4°	105.4°	105.21°	105.39°	106.1(3)°	105.7(4)°
N(1)-C(1)-N(2)	111.3	111.6	111.27	111.50	110.6(3)	111.1(4)
C(1)-N(2)-C(2)	107.2	107.9	107.75	107.48	107.7(3)	106.9(4)
N(2)-C(2)-C(3)	106.3	106.1	106.43	105.92	106.6(4)	107.0(5)
C(2)-C(3)-N(1)	109.8	109.1	109.32	109.68	108.9(4)	109.3(5)
M^{++}-N(1)-C(3)			130.32	130.25	130.9(2)	127.6(3)
M^{++}-N(1)-C(1)			124.27	124.14	123.0(2)	126.8(3)
N(1)-M^{++}-N(1')		88.1	88.23	88.30		90.4(1)
[00.1] M^{++}-N(1) bond		56.1	56.00	55.95	56.3	54.5
N(3)-O(1)		1.239	1.235	1.228	1.231(2)	1.286(6)

* Martinez-Carrera, S. (1966). Acta Cryst. 20, 783-789.

** By x-ray diffraction, this work

*** The standard deviations on bond distances and angles were estimated from the errors in coordinates and in lattice parameters.

† Santoro et al. (1969). Acta Cryst. B25, 842.

†† By neutron diffraction, this work.

††† Mighell and Santoro, Acta Cryst., In Press.
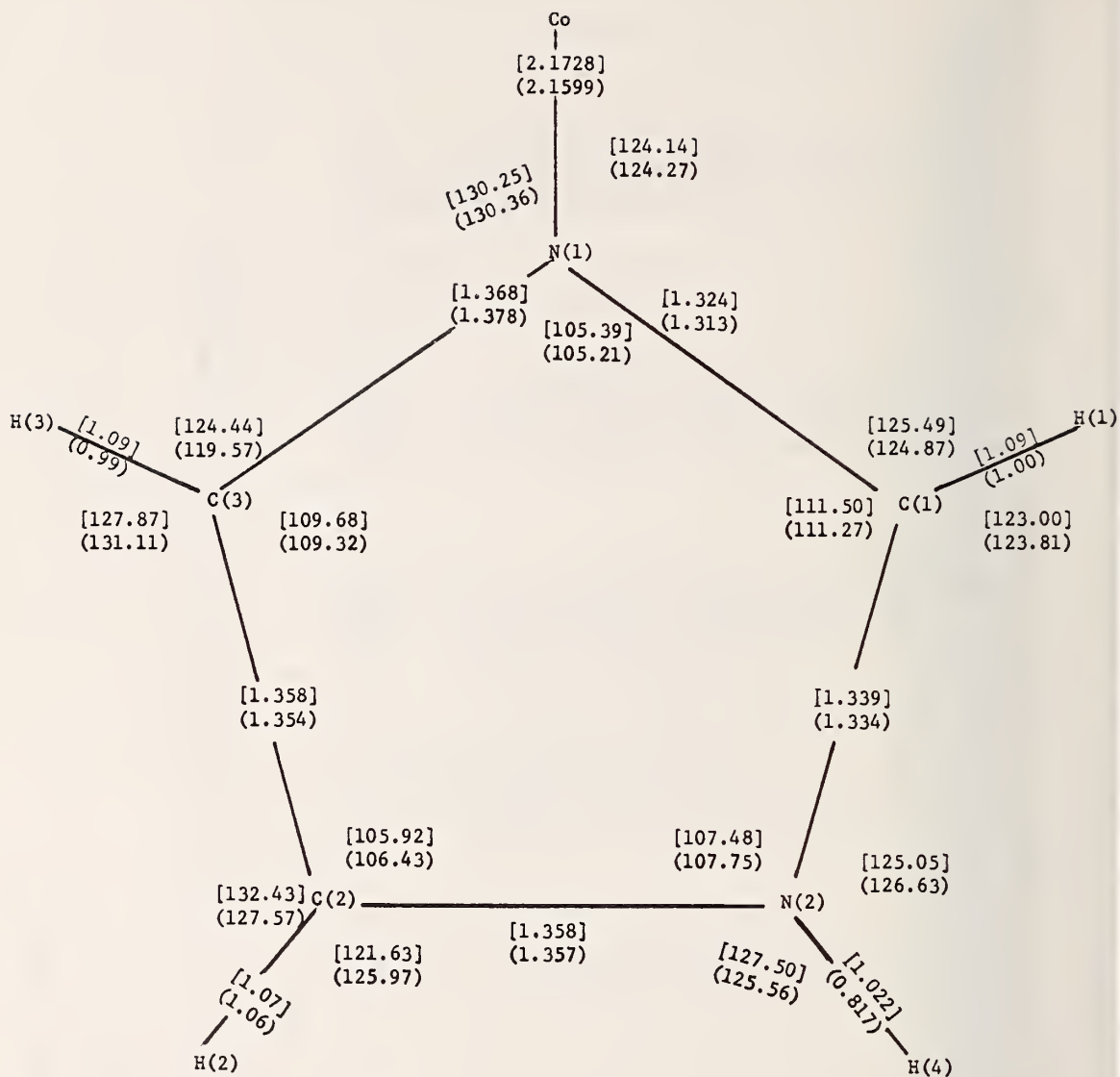
Fig. 1. Bond distances and angles for the coordinated imidazole ring in hexakis (imidazole) cobalt(II) nitrate. The angles and distances in brackets were determined by neutron diffraction and those in parenthesis by x-ray diffraction.

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Fig. 2.

Fig. 3.

Fig. 4.

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The Configuration of the Water Molecule in Cupric Fluoride Dihydrate
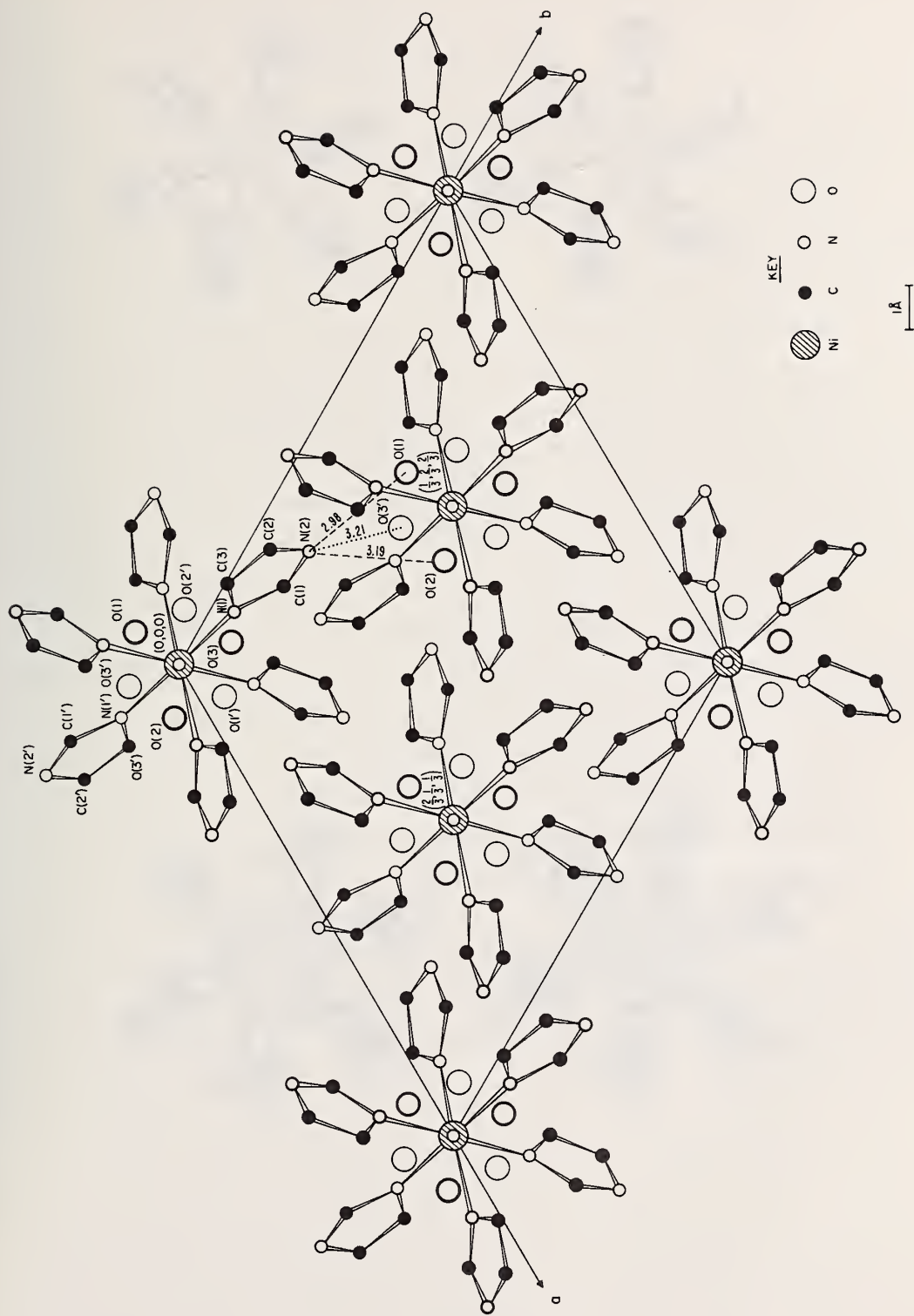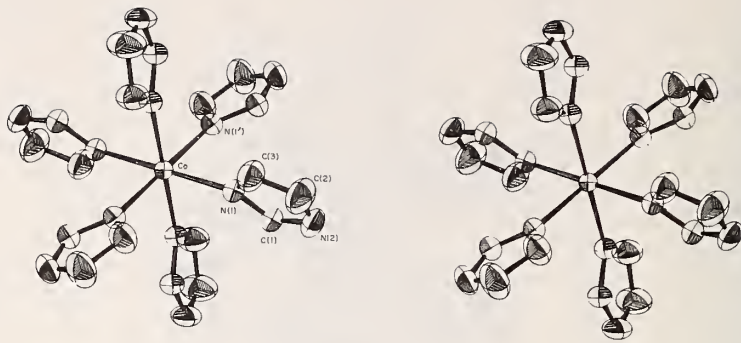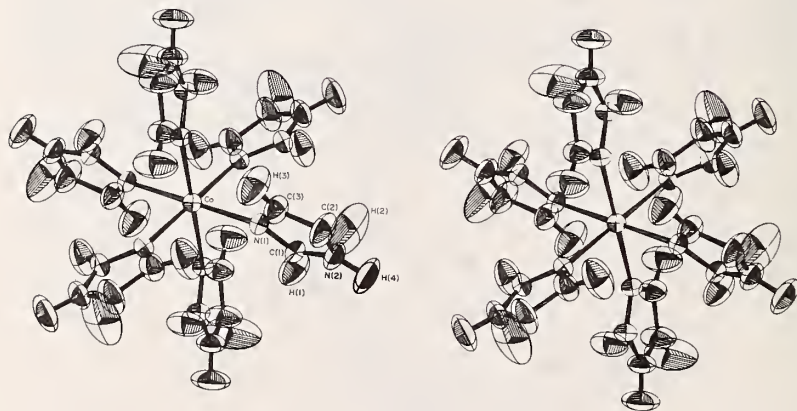
E. Prince

Cupric fluoride dihydrate, $CuF_2 \cdot 2H_2O$, was one of the first hydrated crystals to have its structure determined by single-crystal, neutron-diffraction techniques. In the original determination the H-O-H bond angle in the water molecule was found to be $115.5\pm4°$, in contrast to a value of 104.5 in a free water molecule in the vapor phase. As a much larger number of structure determinations became available, it appeared that this value of the bond angle was considerably out of line with those found in other, similar crystals. The original determination was done with 3 sets of 2-dimensional data taken on 3 different crystals mounted with 3 different axes vertical. Only 6 OkO reflections were common to the 3 sets, and several of these were known to be affected strongly by secondary extinction. In view of the uncertainties in the original determination and the fact that the structure appeared to be unusual, a redetermination of the structure was undertaken, using 3-dimensional data taken from 1 crystal.

$CuF_2 \cdot 2H_2O$ is monoclinic, with cell dimensions $a=6.7060Å$, $b=7.3958Å$, $c=3.3002Å$, $\beta=109.38°$ Z=2, space group C2/m. Of 168 reflections within the limits of the diffractometer there was observable intensity for 163. A least-squares refinement of these data led to a final weighted R index of .038 and the parameters shown in Table 1. Fig. 1 shows the 8-membered ring formed by 2 water molecules and the fluorine atoms to which they are hydrogen bonded, together with the copper atoms that coordinate to the water molecules. The H-O-H bond angles is $110.6\pm.2°$, which is much more in line with the values found in similar, hydrated crystals. Note, however, that this angle is still larger than the F-O-F angle $(96.5°1^0)$, which indicates that the presence of the hydrogen bond is not the principal cause of the opening of the bond angle from the free molecule value. The observed value is close to the tetrahedral angle of 109.5° which may be the preferred angle for a water molecule bond in this type of structure.

TABLE 1. Final atomic parameters for $CuF_2 \cdot 2H_2O$. Numbers in parentheses are the standard deviations of the last significant figure.

Atom	X	Y	Z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu	0	0	0	1.06(6)	.72(7)	1.00(6)	0	-.09(3)	0
F	-.2235(2)	0	.4619(3)	1.67(6)	1.33(8)	1.67(7)	0	-.26(4)	0
O	0	.2620(2)	0	2.03(7)	.84(6)	2.22(8)	0	-.46(6)	0
H	-.1065(3)	.3363(2)	-.1989(6)	2.71(10)	2.07(9)	2.85(8)	.14(4)	.14(5)	.19(5)
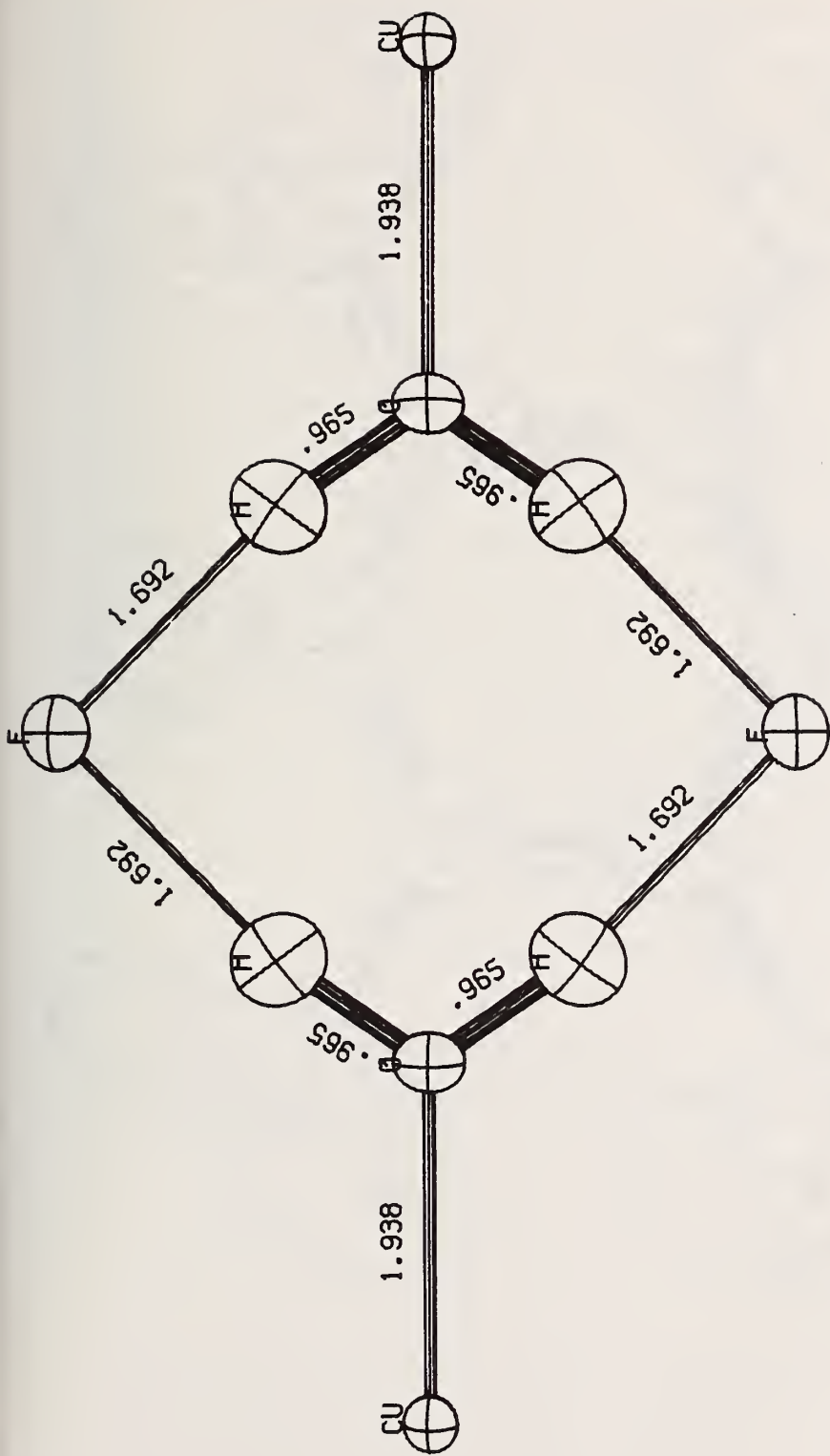
Fig. 1. The 8-membered ring formed in $CuF_2 \cdot 2H_2O$.

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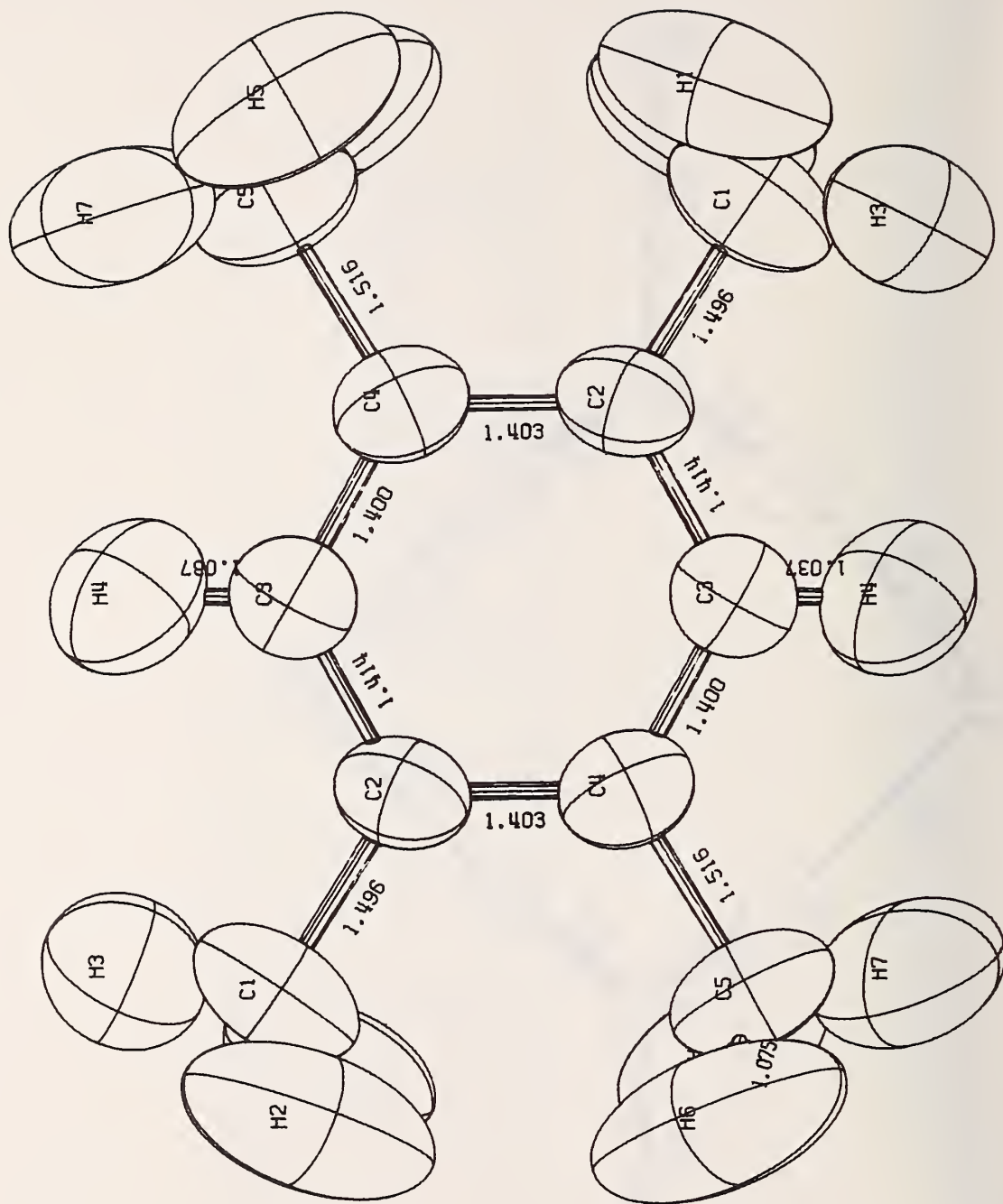
The Structure of Durene

E. Prince, L. W. Schroeder and J. J. Rush

The work on the crystal structure of durene (1,2,4,5-tetramethyl benzene) described in the 1970 progress report has continued during this year. The structure is monoclinic, space group $P2_1/a$, with $a=11.57\text{Å}$, $b=5.77\text{Å}$, $c=7.03\text{Å}$, $\beta=112.03°$. Of 870 independent reflections within range of the diffractometer 485 had intensities significantly above background. The data have been refined by least squares methods, using several different weighting schemes; the lowest value of the weighted R index achieved so far is $R_w = .066$.

At this level the general features of the structure are clear, but certain details are inconsistent, and the refinement cannot be considered completely satisfactory. The position parameters for the carbon atoms indicate a molecular structure (see Fig. 1) which is exactly planar within the precision of measurement, with C-C distances, both within the ring and between the ring and the methyl groups, very close to the expected values for this type of compound. There is, however, excessive scatter in the C-H distances. Also, although all of the thermal ellipsoids correspond qualitatively to what would be expected from a model which makes the principal thermal motion a libration of the entire molecule about its axis of maximum inertia, to which are added librations of the methyl groups around the C-C bond, there are some glaring inconsistencies. In particular, the hydrogen atom attached to the ring, H4, should not have a component of its greatest vibrational amplitude parallel to the C-H bond, and the tangential component of the motion of hydrogen H3 is less than the similar component of the carbon on which it is riding, which is obviously impossible.

A study of the refinement methods that can be applied to this data set is continuing, and an additional data set will be collected at low temperature.

The Structure of Anhydrous Monocalcium Phosphate

B. Dickens
(Polymers Division)

and

E. Prince

The Dental Research Section of the Polymers Division of NBS is conducting crystallographic studies of calcium phosphates and carbonates and related compounds. The knowledge gained is expected to provide insight into processes of mineralization in vivo. The complete characterization of many of these compounds includes knowlecge of the positions and thermal parameters of hydrogen atoms. 1212 reflections have been measured by neutron diffraction on a single crystal of $Ca(H_2PO_4)_2$ and the hydrogen positions have been found. Two of the five crystallographically different hydrogens in the crystal structure are nominally centered between their coordinated oxygen atoms. Such hydrogen bonds are chemically important and are of considerable theoretical interest. In the case of $Ca(H_2PO_4)_2$, however, abnormally great secondary extinction effects have mitigated further analysis because the limits of the usual correction formulas have been exceeded and the thermal parameters probably contain appreciable systematic error. More accurate ways of applying the extinction correction are being studied.

Neutron Diffraction Study of Two Dental Amalgam Alloys

R. M. Waterstrat
(Polymers Division)

Dental amalgams are silver-tin-mercury alloys which are widely used in restorative dentistry. The amalgam is prepared by mixing a silver-tin alloy powder (Ag_3Sn) with liquid mercury to form a metallic paste which can be shaped to conform to the tooth cavity. After the alloy hardens by a diffusion reaction, it is essentially a composite material consisting of partially reacted Ag_3Sn particles (γ phase) which are bound together in a matrix of finely crystalline Ag_2H_3 compounds (γ_1 phase) and Sn_7Hg compound (γ_2 phase).

Recent work by Dr. L. B. Johnson at the University of Virginia has demonstrated that the formation of the tin-mercury compound (γ_2 phase) can be prevented or at least suppressed

by the addition of about 10% gold to the silver-tin alloy powder (γ phase). This produces a dental amalgam which is probably more resistant to degradation since the γ_2 phase is the softest and most easily corroded of the three constituent phases. Dr. Johnson's findings, however, were based largely on x-ray diffraction data. X-rays reveal information mainly from a thin surface layer rather than from the entire volume of the sample. Furthermore, the x-ray data is subject to much uncertainty since crystals within the thin surface layer may be strongly oriented. A relatively small preferred orientation of the crystals would produce significant changes in the x-ray intensities and would make it very difficult to estimate the relative percentages of each phase in the alloy.

R. M. Waterstrat of the NBS Dental Research Section suggested to Dr. Johnson that neutron diffraction would be a more suitable experimental method than x-ray diffraction since the neutron beam is capable of penetrating a much larger volume of the sample[*]. Dr. Johnson subsequently furnished samples of his new dental amalgam prepared from the 10% Au alloy and also from a conventional alloy without the Au addition. These samples were examined by neutron diffraction at the NBS reactor in cooperation with Dr. S. Pickart, Dr. J. J. Rhyne and Dr. E. Prince of the reactor group.

A comparison of neutron diffraction patterns from the two amalgam samples has revealed that lines due to the γ_2 phase, which were present in the pattern from the conventional amalgam, were entirely absent in the pattern from Dr. Johnson's 10% Au amalgam. One may, therefore, conclude that the Au addition has either eliminated or greatly suppressed the formation of the γ_2 phase. In addition there was some evidence for a new Au-containing phase in Dr. Johnson's alloy since an unidentified line appeared in the pattern from this amalgam which did not appear in the pattern from the conventional amalgam. A comparison of the relative line intensities also suggested that Dr. Johnson's alloy contained a greater percentage of unreacted phase particles than the conventional alloy. These findings are all compatible with Dr. Johnson's suggestion that the new amalgam will prove to be more durable than the amalgams now being used in dentistry.

[*] The mass absorption coefficients for Ag and Hg irradiated by X-rays (CuK_α) are 223 and 223 while for neutron irradiation the corresponding values are 0.20 and 0.63, respectively. These values may be used to calculate that about 99% of incident x-rays are absorbed by a

0.02 mm surface layer of the amalgam whereas only about 1% of incident neutrons would be absorbed in a 5 mm layer. The cylindrical samples which were used in the neutron diffraction study were irradiated in a direction perpendicular to the cylinder axis and were about 6 mm in diameter.

Refinement of the Structure of Orthoclase

E. Prince

and

G. Donnay
(Dept. of Geological Sciences, McGill University
Montreal, PQ, Canada)

Orthoclase is a feldspar, a class of alumino-silicate minerals which is one of the major constituents of the rocks in the earth's crust. The basic structure is one of MO_4 tetrahedra linked together by sharing the oxygen atoms at the corners between neighboring tetrahedra. The distribution of aluminum among crystallographically nonequivalent tetrahedral sites is of great interest to geologists, because the degree of order or disorder can be correlated with the thermal history of the rocks in which the mineral is found. Also, there have recently been suggestions that a substantial amount of water could be bound in the supposedly anhydrous minerals of the earth's crust by means of small amounts of substitution of OH^- ions for $O^=$ ions in these crystals.

Aluminum and silicon are neighbors in the periodic table, so they cannot be distinguished by x-ray diffraction on the bases of scattering amplitude alone. Unfortunately the scattering amplitudes for neutrons for these elements are also very similar. Because of this, all attempts to determine the Si-Al distribution have made use of the fact that the Al^{3+} ion is significantly larger than the Si^{4+} ion, and therefore the ionic occupancies are directly related to the M-O distances, as determined from an accurate refinement of the crystal structure. The M-O distance in this type of structure varies approximately linearly with aluminum content from $1.61\overset{\circ}{A}$ for pure SiO_4 to $1.75\overset{\circ}{A}$ for pure AlO_4. A neutron diffraction refinement of the orthoclase structure was undertaken in order to determine the distances accurately, and to see if there was any indication of the presence of OH^- in the structure.

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There is evidence that the true structure is triclinic, with cell dimensions a=8.5610Å, b=12.965Å, c=7.2-77Å, α=90.048°, β=116.059°, γ=89.814°. However, a systematic comparison of reflections of the type hkℓ with reflections $\bar{h}\bar{k}\ell$ did not reveal any significant department from monoclinic symmetry. Therefore, in the refinement, the α and γ angles were assumed to be 90°, and the refinement refers to an "averaged" clinic structure. Of 764 reflections within the range of the diffractometer, 721 had observable intensities. A least squares refinement using these data gave a final weighted R index ($R_w = [\Sigma_w (F_o - F_c)^2 / \Sigma_w F_o^2]^{1/2}$) of .038 with the atomic parameters listed in Table 1.

Figure 1 shows the configurations of the two MO_4 tetrahedra. The mean MO distances indicate that site SI1 contains approximately 1/3 aluminum and 2/3 silicon.

TABLE 1. Final atomic parameters from the refinement of the structure of orthoclase. Numbers in parenthesis are the standard deviations of the last significant figure.

Atom	x	y	z	B_{11}^*	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
K	.2842(2)	0	.1388(4)	1.6(1)	2.5(1)	2.1(1)	0	.54(7)	0
SI1	.0096(2)	.1843(1)	.2247(2)	1.66(5)	2.26(5)	1.23(5)	-.24(4)	.72(4)	-.15(4)
SI2	.7085(2)	.1176(1)	.3446(2)	1.29(5)	.86(5)	1.00(5)	.00(4)	.51(4)	.02(3)
OA1	0	.1452(1)	0	2.04(6)	1.47(6)	1.82(6)	0	.91(5)	0
OA2	.6366(2)	0	.2853(2)	1.86(6)	.90(5)	1.47(5)	0	.30(4)	0
OB	.8255(2)	.1460(1)	.2279(2)	2.11(5)	2.37(5)	2.16(5)	-.23(4)	1.43(4)	.16(3)
OC	.0353(1)	.3119(1)	.2599(2)	1.60(4)	1.32(4)	1.68(4)	-.31(3)	.61(3)	-.36(3)
OD	.1819(1)	.1252(1)	.4082(1)	1.83(4)	1.53(4)	.93(4)	.13(3)	.21(3)	.11(3)

* units are $\mathring{A}^2$

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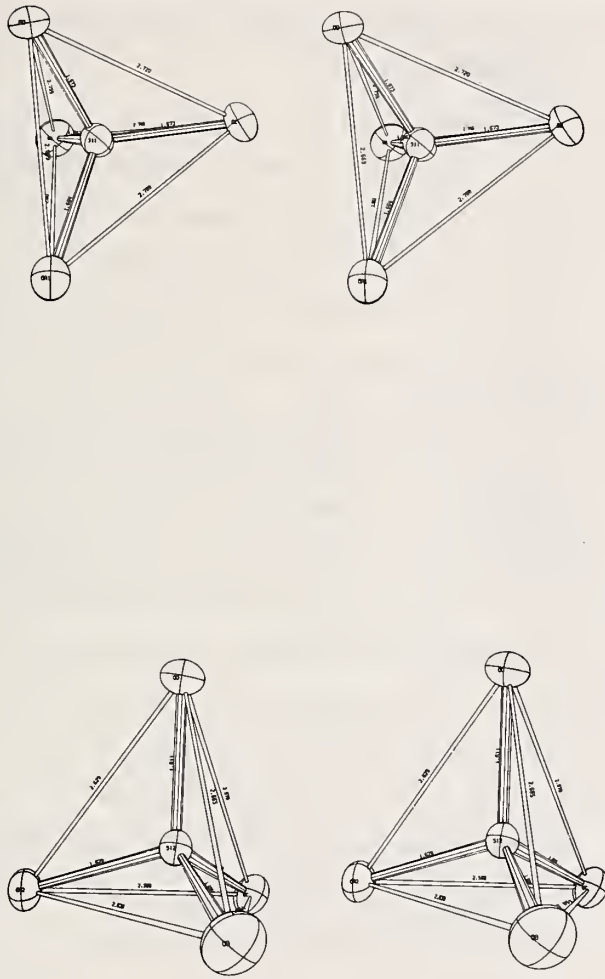
Fig. 1. Stereo pairs showing the configurations of the two M-O tetrahedra in orthoclase. The standard durations of M-O bond lengths are .002Å.

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Work has continued on a systematic study of the diffusion and vibrational dynamics of hydrogen bond in polymorphic transition-metal hydrides. The work completed or in progress on several hydride systems is summarized below:

1. Vanadium Hydrides- L. A. de Graaf and J. J. Rush

In collaboration with H. E. Flotow and J. M. Rowe of Argonne National Laboratory, we have analyzed in detail inelastic scattering spectra measured previously for the α (bcc) phase of $VH_{0.20}$ and the α and β (bct) phases of $VH_{0.57}$ over a range of neutron momentum transfer (Q) of $2.0-4.1\overset{\circ}{A}^{-1}$. Some of the measured spectra are shown in Fig. 1, with the separation into inelastic scattering (horizontal line), elastic scattering from vanadium and quasielastic scattering from the protons indicated. The extension of our measurements of quasielastic line broadening to larger momentum transfers than those available with cold ($4\overset{\circ}{A}$) neutrons by the use of an incident wavelength of $2.42\overset{\circ}{A}$ has proved to be essential in obtaining definite information about the jump mechanism of the hydrogens in the bcc lattice. The measured full widths at half maximum of the quasielastic peaks for $VH_{0.20}$ at 212°C have been compared with widths calculated for several models involving jumps between octahedral and tetrahedral interstitial sites. The experimental and theoretical results are shown in Fig. 2, along with the previous cold neutron data. None of the models tested predict the rapid increase of the line width at large Q, but from the behavior of the width curves for $VH_{0.57}$ at $Q^2 < 14\overset{\circ}{A}^{-2}$ we conclude that the diffusion in $\alpha-VH_{0.57}$ is dominated by jumps between first-neighbor tetrahedral sites. This is the first time to our knowledge that jump mechanisms have been established for a Group V-A metal-hydride.

A number of possible deficiencies of the jump diffusion model have been examined and we have tentatively concluded that the most important factor is the assumption of instantaneous jumps. Due to the very rapid diffusion in α VH_x the relaxation (residence) times derived from our results are about 0.5 and 1 ps (1 ps = 10^{-12} sec) for $VH_{0.20}$ and $VH_{0.57}$, respectively, whereas the jump time between sites is ~ 0.04 $M_{eff}^{1/2}$ ps (M_{eff} is the effective mass of the "dressed" proton in the lattice). Thus the jump time is not "instantaneous" but is approaching comparability with the residence time between jumps. This apparently explains the more rapid breakdown of the jump diffusion model for $VH_{0.20}$ (Fig. 2) and the differences in the measured quasi Debye Waller factors both between $VH_{0.20}$ and $VH_{0.57}$ and between those derived from the present measurements and the earlier cold neutron data. We are currently

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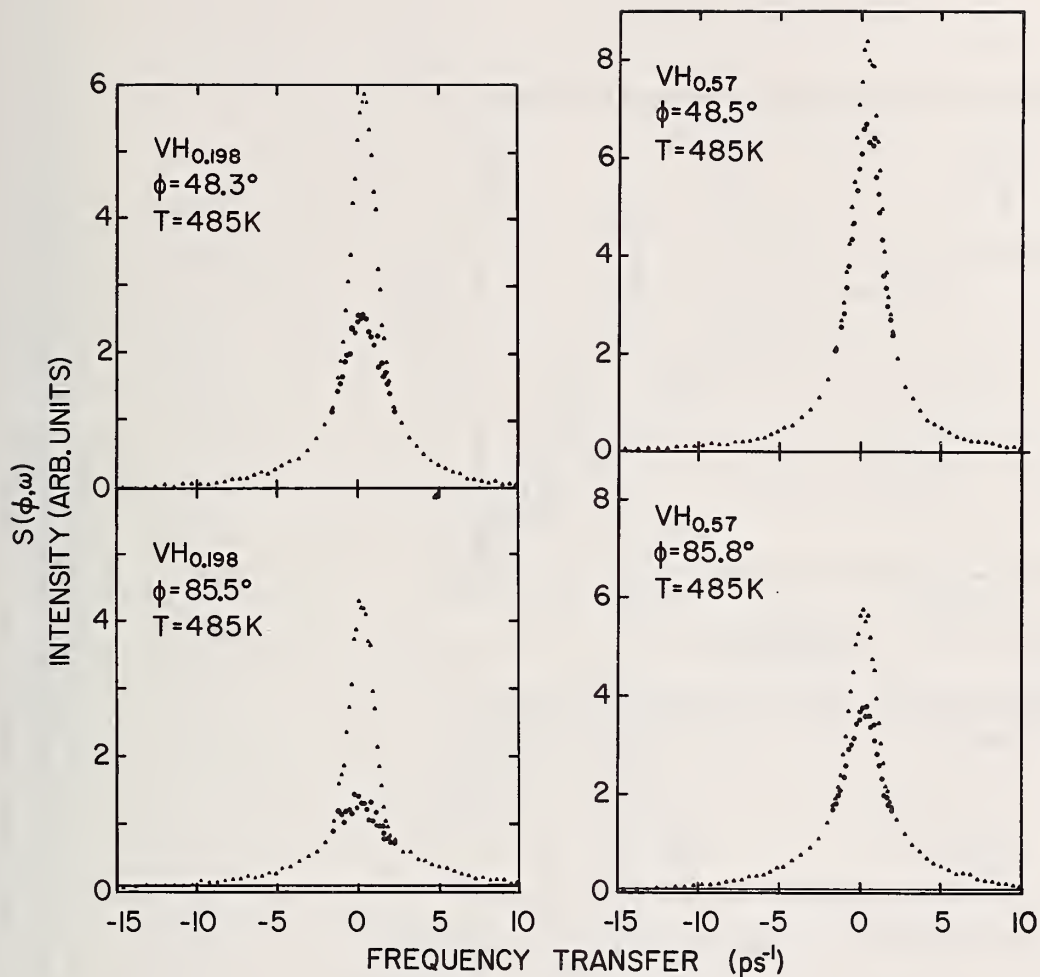
Fig. 1. The separation of the measured spectra $I(\phi,\omega)$ in inelastic, elastic, and quasielastic components. The data points are given by triangles, the points corrected for vanadium scattering by dots.
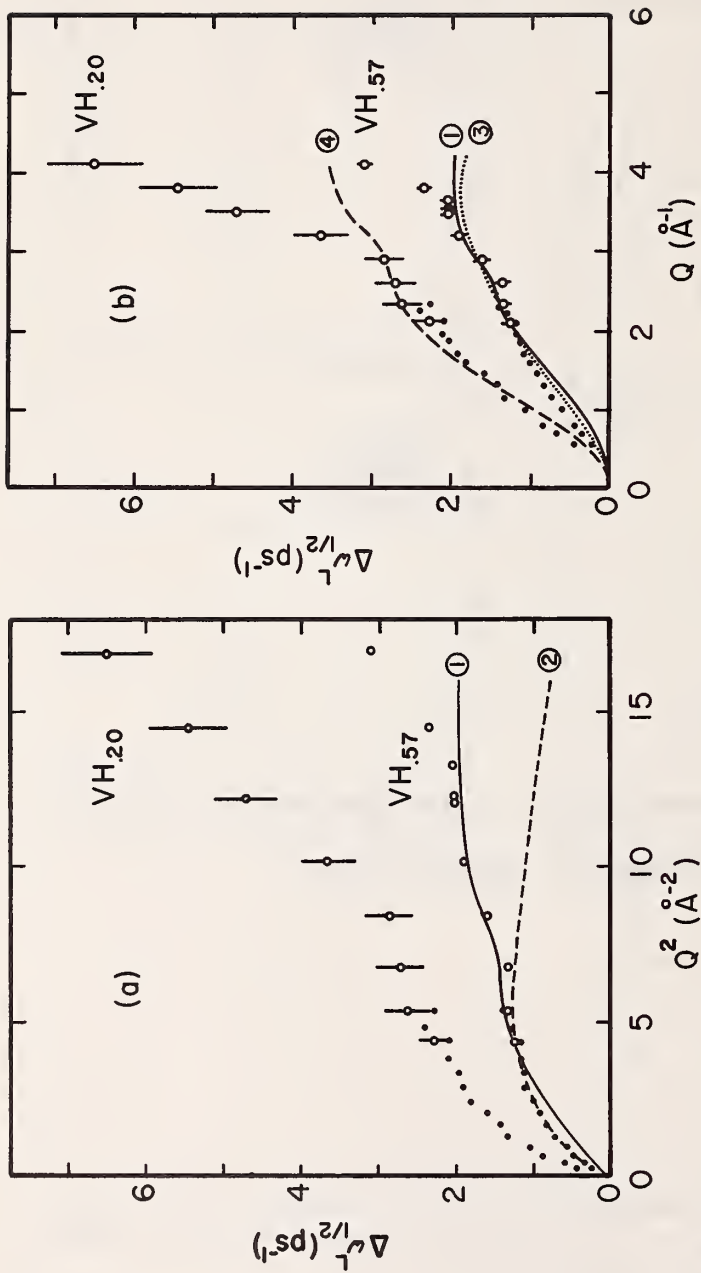
Fig. 2. The measured widths of the quasielastic peaks, $\Delta\omega^L_{1/2}$ compared with the predictions of several models (see text). The open circles with error bars are the results obtained in the present measurements, the filled circles are the results of the earlier cold-neutron experiments.[3] Curve 1: jumps between nearest-neighbor tetrahedral sites, τ =1.0 ps; 2: jumps between nearest-neighbor octahedral sites, τ=1.25 ps; 3: jumps between nearest and next-nearest neighbor tetrahedral sites, τ_1=τ_2=2.0 ps; 4: jumps between octahedral and tetrahedral sites, τ_{tt}=0.55 ps, τ_{to}=τ_{ot}=0.11 ps, τ_{ot}=0.01 ps.

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preparing a paper for publication which fully discusses these results and their implications.

2. Tantalum Hydrides - L. A. de Graaf, R. L. Livingston, and J. J. Rush

In collaboration with H. E. Flotow of Argonne National Laboratory we have extended our neutron scattering studies of the Group V-A metals to tantalum hydride. Spectra have been measured for α-TaH$_{0.15}$ at temperatures from 22 to 340°C and momentum transfers of 0.45 to 3.6Å^{-1}. These data are presently being analyzed. Preliminary examination of the quasielastic line widths seems to indicate a leveling off at high Q. In addition our preliminary results indicate a considerably higher activation energy for diffusion and lower diffusion rate for the hydride. Such results would be consistent with the ideas and theoretical predictions developed in the explanation of the VH$_x$ results described above. Work is continuing on the analysis of the TaH$_{0.15}$ data, including a comparison with various diffusion models and calculation of quasifrequency distributions for the hydrogen vibrations. Further experiments with other hydrogen concentrations are planned.

3. Palladium Hydrides - L. A. de Graaf and J. J. Rush

In collaboration with G. A. Ferguson of the Naval Research Laboratory, a large single crystal of palladium (f.c.c. structure) has been electrolytically loaded to a concentration of PdH$_{0.03}$. This crystal will be used for a detailed study of the diffusion of hydrogen in various crystal directions by quasielastic neutron scattering using the new four-rotor cold-neutron beam facility at the NBSR.

A smaller Pd single crystal has been loaded with deuterium to PdD$_{0.6}$ to text the quality (mosaic spread) of the resulting "crystal" for future lattice dynamics experiments. A neutron diffraction examination showed an effective mosaic spread about 2-3 times greater than is desired. Further tests will be tried with different loading conditions.

Work has continued on detailed studies of phase transitions involving orientational disorder of ionic groups or molecules in crystals. The precise nature of the rotational disorder in the high temperature (generally cubic) phases of hundreds of inorganic and organic crystals is not in general well understood - nor is the microscopic basis of the dramatic changes in physical and thermodynamic properties associated with such phase transitions. We have chosen several prototype materials (ionic and organic) and are attempting to obtain a more detailed understanding of the dynamical and structural nature of such phase transitions by combining results from a variety of techniques including neutron diffraction and inelastic scattering, infrared spectroscopy and Raman scattering.

1. Hydrosulfide Salts - L. A. de Graaf, J. J. Rush, R. C. Livingston and L. W. Schroeder

The structure, ionic reorientation and vibrational dynamics of sodium hydrosulfide in both its low-temperature trigonal phase and its high temperature fcc "rotator" phase have been studied by neutron diffraction and inelastic scattering neutron powder pattern techniques for the trigonal and cubic phases at 296 and 379K, respectively. The results are shown in Fig. 1. Several possible models describing the orientations of the SH^- ion in the trigonal phase were compared with the experimental structure factors. A model in which the SH^- ions are aligned along the trigonal axis clearly gave the best agreement between calculated and observed structure factors. This structure is illustrated in Fig. 2 along with the high temperature fcc structure. Thus the transition from the cubic to the trigonal phase apparently involves alignment of the SH^- ions along one of the cubic diagonals which then becomes shorter than the others, resulting in the less symmetric trigonal structure. The analysis of the room temperature diffraction data also yielded an S-H distance corrected for thermal motion of 1.35Å.

Several structural models were also considered for comparison with the cubic phase results. The isotropic "free rotor" model can be rejected in favor of a less randomly disordered model by comparison with the neutron inelastic scattering results, which clearly rule out quasifree or random rotation. The limited diffraction data in this phase, however, do not enable a distinction to be made between disordered models.

In a related experiment the dynamics of the SH^- ions in NaSH have been studied by neutron inelastic scattering using the hybrid time of flight spectrometer at the NBSR. The incident neutron wavelength was 2.42Å and for elastic scattering the time of flight

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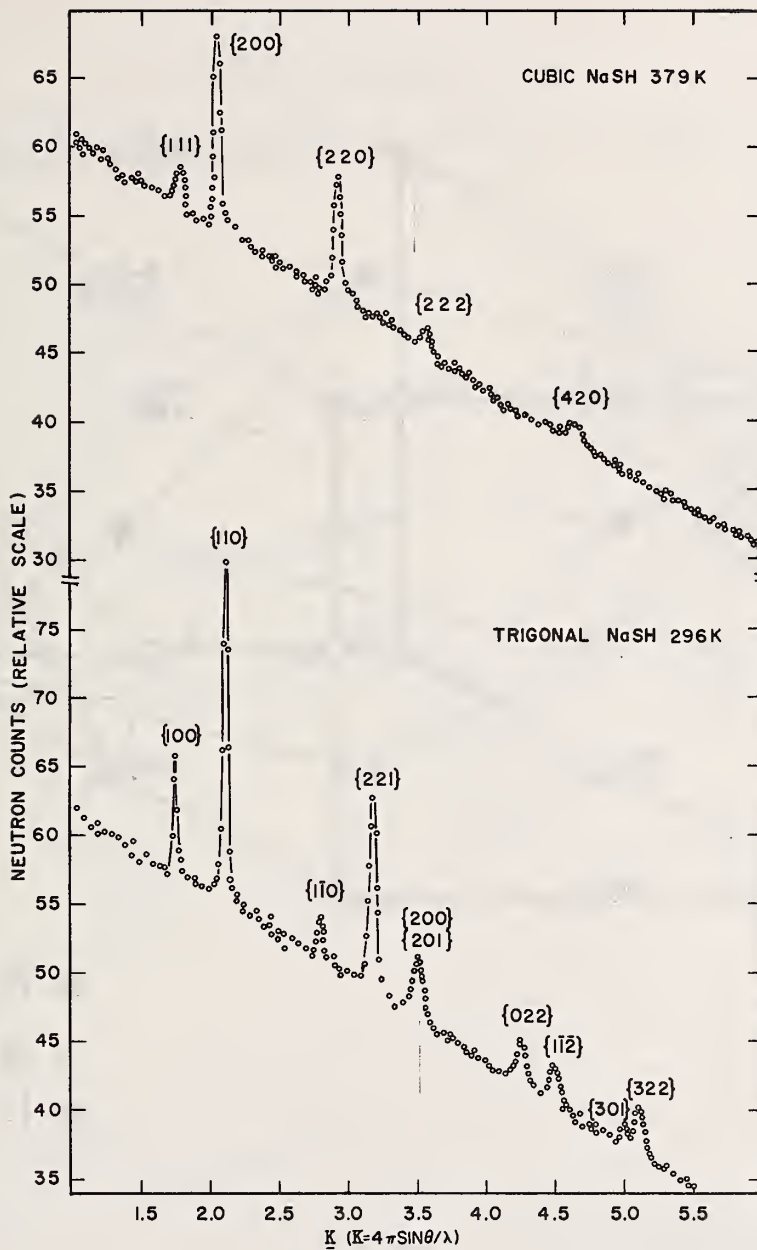
Fig. 1. Neutron diffraction patterns of powdered NaSH in cubic and trigonal phases.

Na
S
1/2H
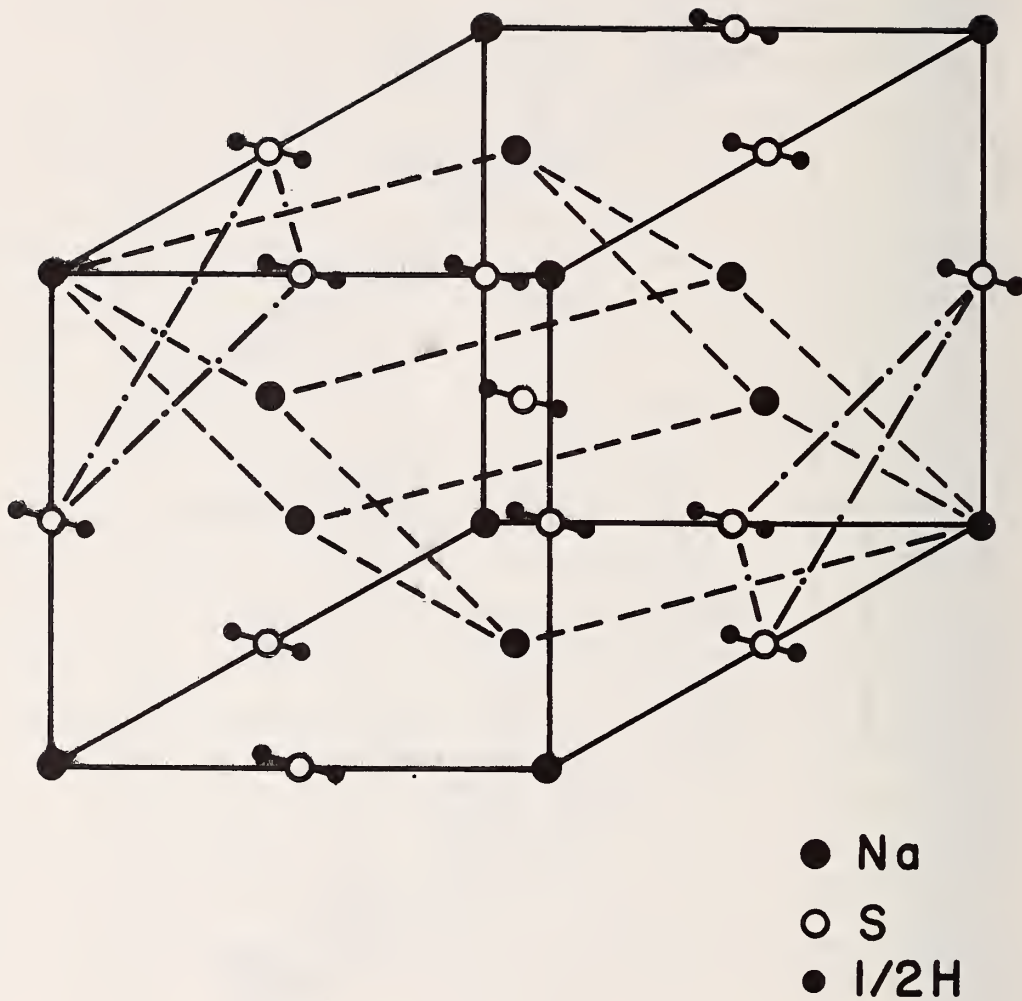
Fig. 2. Composite drawing of the NaSH crystal phases. The SH⁻ ions are shown aligned
along [111] of the cubic phase. The three vectors from the Na atom at the
corner to Na atoms in a (111) plane define the translations of the trigonal cell.
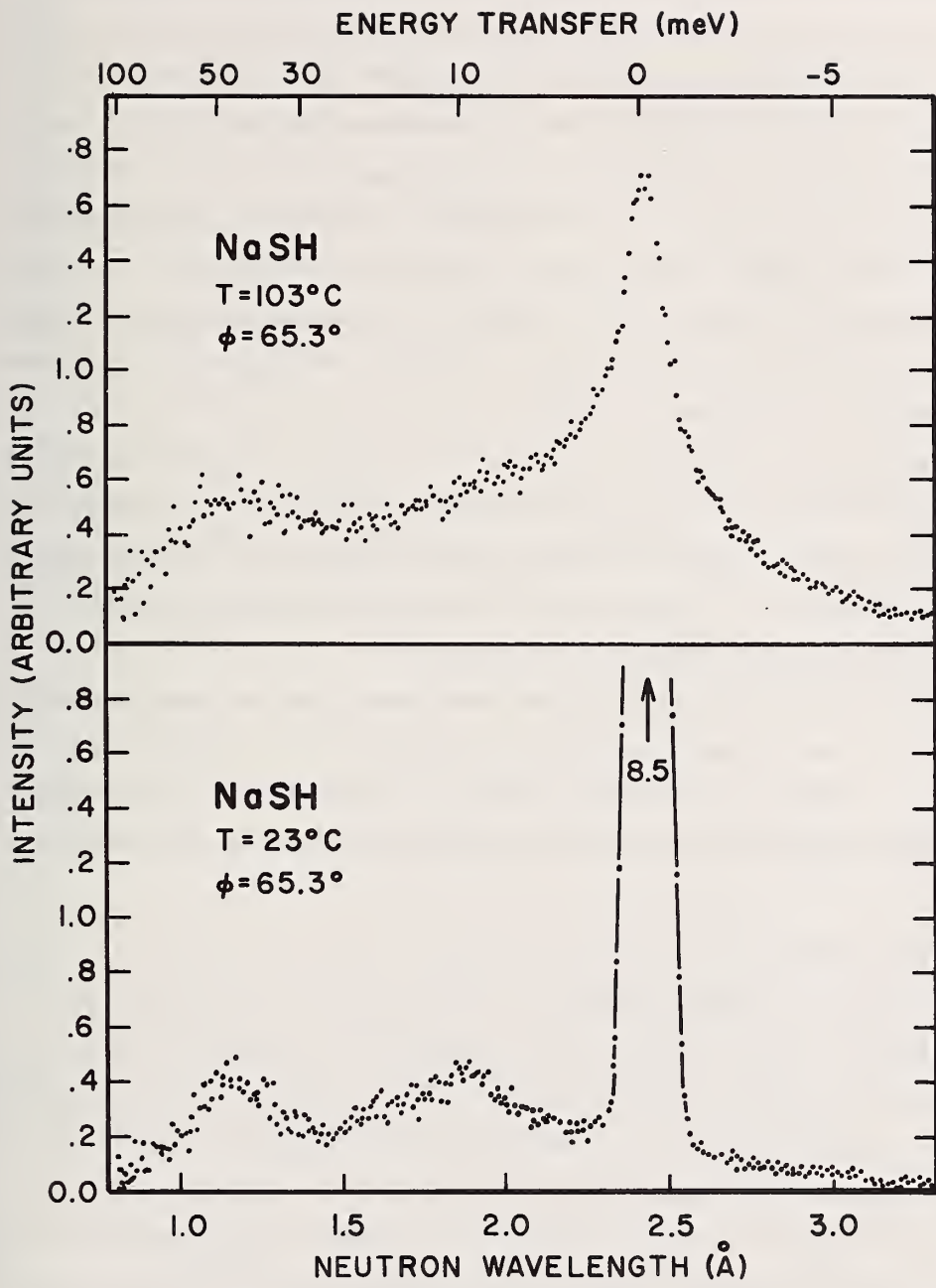
Fig. 3. Inelastic neutron spectra vs. neutron wavelength for NaSH in its cubic and
trigonal phases. Intensities are corrected for background, neutron chopper
transmission and detector efficiency.

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resolution was 3.8% and the momentum transfer (Q) range was 0.4 to $3.2\overset{\circ}{A}^{-1}$. Examples of measured inelastic scattering spectra above and below the phase transition ($T_t \sim 360K$) are shown in Fig. 3. The spectrum at 296K in the trigonal phase shows a very sharp elastic scattering peak at $2.42\overset{\circ}{A}$, as well as two bands of inelastically scattered neutrons, one peaked at ~ 50 meV (400 cm^{-1}) energy gain and the second around 10 meV. The two inelastic bands are assigned, respectively, to the SH$^-$ librational modes and to translational lattice modes. The spectrum measured in the fcc phase at 379K shows no great change in the peak of the librational band, but does exhibit considerable broadening of the elastic scattering line. This behavior can be directly attributed to the onset of rotational jumping of SH$^-$ ions in the lattice on a time scale of $\sim 10^{-12}$s.

The temperature and Q dependence of the observed broadening has been extracted from the data and is being compared in detail with theoretical widths calculated using various models in which the SH$^-$ ions are assumed to reorient with rapid jumps between a limited number of quasi-equilibrium positions. Relaxation times and the activation energy for the SH$^-$ reorientations should also be derived from our analysis.

Experiments are also currently in progress to synthesize CsSH, an interesting analogue to the sodium salt, so that neutron spectral measurements may be extended to a study of the phase transition in this system. It should be noted that these materials are extremely reactive and thus require considerable care in handling and in preparing neutron sample holders.

2. Potassium Cyanide - J. J. Rush and E. Prince

KCN is one of the broad group of polymorphic compounds M$^+$XY$^-$ (including the hydrosulfides discussed above) whose crystal phase immediately below the melting point has a CN$^-$ ions. Previous attempts to determine the nature of the disorder in the cubic phase of KCN by x-ray diffraction and by neutron powder diffraction have failed to provide much information about the orientation and dynamical behavior of the cyanide ions. In view of this we have, in collaboration with D. L. Price and J. M. Rowe of Argonne National Laboratory, performed neutron diffraction measurements on single crystals of KCN both at room temperature and at 180K, close to the phase transition at 167.5°K. Such single crystal measurements can obviously provide better intensity data on many more reflections than powder patterns and thus allow a more meaningful test of various models for the cyanide disorder.

In our diffraction measurements all reflections in an octant of reciprocal space were measured, using an incident neutron wavelength of 1.062Å. Sets of data at both 180K and 295K were collected with the crystals mounted in an orientable variable-temperature cryostat. Out of a possible 39 measurable reflections, the number actually observed was 24. The agreement in intensity within sets of equivalent reflections was excellent. The experimental structure factors were compared to models in which the CN^- ions were constrained to be oriented at random among sets of equivalent crystal directions, namely the (111), (110) and (110) directions of the fcc cell. Each model fit had as variable parameters (a) thermal parameters allowing isotropic translational motion of the K^+ ions and both isotropic translation and libration of the CN^- ions, (b) the CN distance, (c) an extinction correction, and (d) a scale factor.

The analysis of these results is almost complete. The tentative conclusions reached thus far on the basis of a careful examination of the diffraction results and preliminary force model calculations are as follows:

a. The non equality of reflections with equal values of $(\underline{h}^2 + \underline{k}^2 + \underline{l}^2)$ but different values of $\underline{h}$, $\underline{k}$, and $\underline{l}$ clearly rules out the possibility of freely rotating cyanide ions.

b. The model fits suggest that the (111) equilibrium orientations are less likely (contrary to previous predictions). However the [100] and [110] models give equivalent fits, so that no definite statments about equilibrium orientations are possible.

c. Very large translational mean-square displacements are predicted from all the fits. These displacements cannot be explained by any reasonable harmonic model for the lattice dynamics and may have important implications concerning the nature of the fcc phase in KCN.

A publication will be prepared shortly on these results and the effects described above are being investigated further. Lattice dynamics experiments planned on KCN which are discussed elsewhere provide a natural extension for our structure study.

3. Plastic Crystals - L. A. de Graaf, R. C. Livingston, and J. J. Rush

Neopentane, $C(CH_3)_4$, is one of a large group of organic molecules close to spherical in shape which undergo a crystal phase transition below the melting point from a less symmetric to a highly symmetric cubic structure. These transitions are analagous to those in the inorganic crystals described above and are generally associated with large entropy changes and a waxy or plastic character of the material in the high temperature phase.

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We have extended our earlier neutron scattering studies to an investigation of the rotational behavior in neopentane by the careful measurement by infrared spectroscopy and analysis of the line shape of a vibration-rotation band (at 925 cm^{-1}) in both solid phases and in the liquid. We experienced considerable difficulty in making these measurements due to the high vapor pressure of neopentane in the plastic (fcc) phase. A special variable temperature cell was designed, constructed and modified until the necessary conditions for the infrared measurements were achieved. In both the liquid and plastic crystal phases the observed infrared band is considerably broadened indicating a high degree of rotational mobility. The band shape narrows sharply in the low-temperature crystal phase. Measurements over a range of temperatures in the plastic phase are being continued. The band shapes are being analyzed by Fourier inversion to derive correlation functions and times for the rotational motions. When completed our results will be compared to those obtained by neutron scattering, so that the information derived by the optical and neutron methods may be correlated, and a better understanding of the intermolecular forces and rotational processes may be achieved.

Lattice Dynamics Studies

1. The Dynamics of Sodium and Potassium Bifluoride: Infrared, Raman and Neutron Studies - J. J. Rush and L. W. Schroeder

Analysis of the spectroscopic results described previously has been completed and the neutron incoherent scattering spectra obtained for $NaHF_2$ and KHF_2 have been used to calculate quasifrequency distributions $g'(\omega)$ for the vibrations of the HF_2^- ions. The $g'(\omega)$ derived for $NaHF_2$ using the one-phonon incoherent approximation is shown in Fig. 1 along with the infrared absorption band observed in the far IR. Our extensive Raman, infrared and neutron measurements combined with previous data, and comparisons made with recent spectroscopic and theoretical results on NaN_3 (which has the same $R\bar{3}m$ crystal structure as $NaHF_2$) have enabled us to provide a fairly detailed assignment of the internal and lattice modes for $NaHF_2$ and KHF_2. These assignments are listed in Table 1. A publication on these results has been prepared and submitted.

2. Dynamics of Potassium Cyanide - J. J. Rush

As an extension of our structural studies on the structure and phase transition of KCN we are currently measuring Laser-Raman spectra of a single crystal of KCN in all of its crystal phases. The information we obtain on the librational and translational lattice modes will be correlated with our structural study to seek a better understanding of the nature of the orientational disorder. The lattice modes measured at q=o will also provide useful information for our study of the lattice dynamics and interionic forces in KCN which we hope to begin at the NBSR in the near future in collaboration with scientists from Argonne National Laboratory.
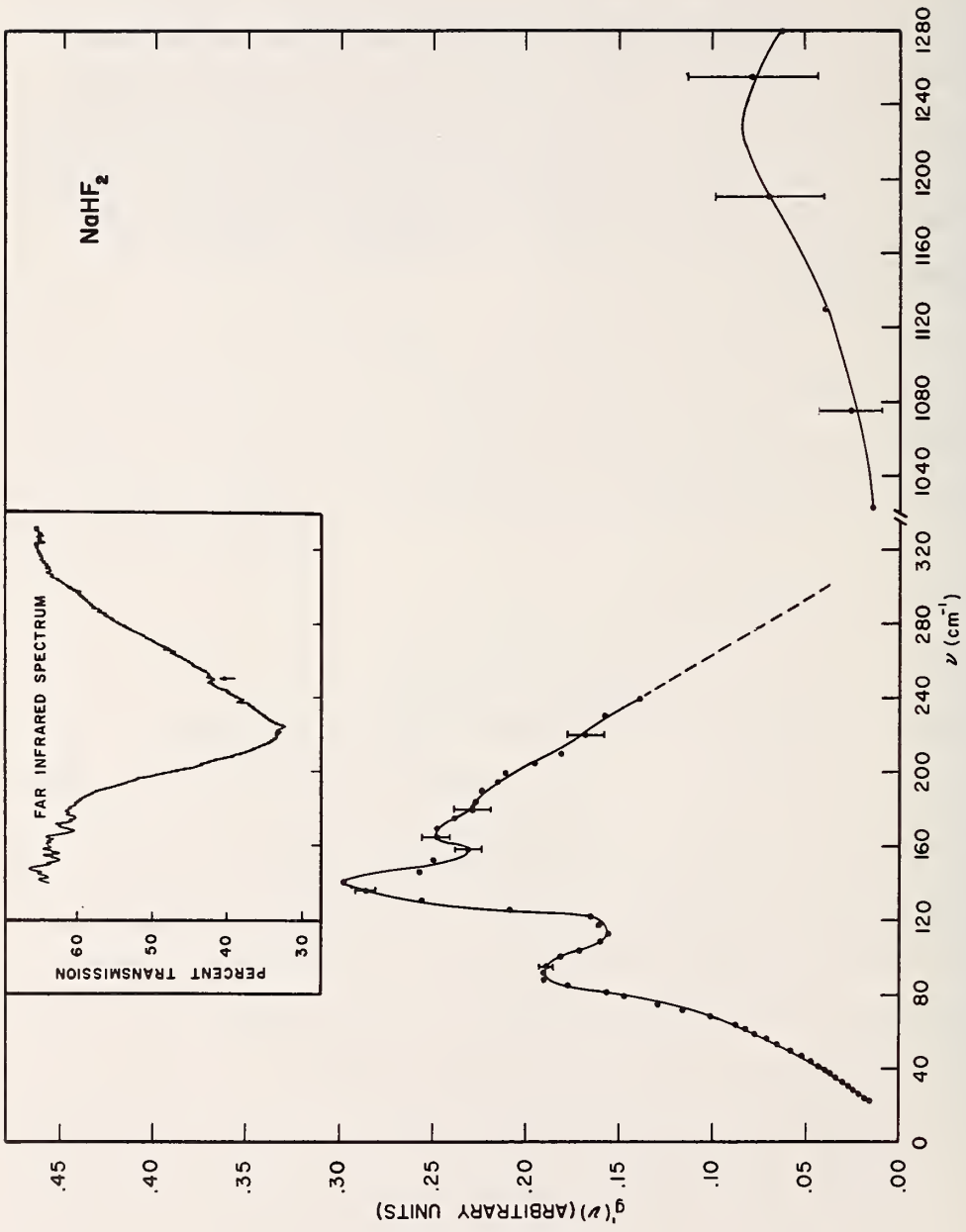
Fig. 1. "Effective" vibrational frequency distribution, $g'(\omega)$ for $NaHF_2$.

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TABLE 1. Assignments of internal and lattice modes for $NaHF_2$ and KHF_2.

	NaHF$_2$	Present Assignments (cm^{-1})		Previous Assignments (cm^{-1})	
Mode[a]	Active Species	H	D	H	D
ν_3	A_{2u} (IR)	≈1500	1070[b]	1577	1150
ν_2	E_u (IR)	1210, 1220[d]	893[b]	–	–
ν_1	A_{1g} (R)	630.5	630.5	–	–
L	E_g (R)	145	145	–	–
T	E_u(IR) , A_{2u}(IR)	228(250,sh) 180[d]	–	226	–
T'	A_{2u}(IR) E_u(IR)	90 ITA)[f], 142(LA)[d]	–	–	–

	KHF$_2$				
ν_3	E_u(IR)	≈1450[c]	1033[b]	1440	1045
ν_2	A_{2u}(IR)	1233	912[b]	·1234	891
	E_u(IR)		895.5[b]	1227	
ν_1	A_{1g}(IR)	596	–	595[i]	–
	B_{2g}(R)	603	–	604[i]	–
L	B_{1g}(R)				
	E_g(R)	143.5[e]	–	136[i,e]	–
T	E_g(R)	104[e]	–	100[i,e]	–
	A_{2u}(IR) , $2E_u$(IR)	–	–	146, 174, 130	–
T'	A_{2u}(IR) , E_u(IR)	–	–	–	–

[a]
ν_i = internal mode; L = Libration mode; T = Translatory (optical) mode; T' = acoustic mode; LA = longitudinal acoustic modes; TA = transverse acoustic modes; IR = infrared active; and R = Raman active.

[b] value for DF$_2^-$ "defect" concentration.

[c] Estimated from frequency for DF$_2^-$ using an isotope shift H/D = 1.40.

[d] From our neutron scattering spectrum.

[e] A rigorous assignment of these L and T modes would require polarization single crystal Raman spectra.

The "Rotator" Phase Transition in n-Alkanes

John D. Barnes
(Polymers Division)

Since the last reporting period a further series of inelastic scattering measurements were carried out on n-nonadecane. The instrument used was the crystal-monochromator, Fermi chopper facility located at the BT-5 beamport.

These experiments differed from the previous one in that considerably more refined sample preparation and containment procedures were used. Neutron spectra were measured over a range of momentum transfers (for elastic scattering) from 0.4Å^{-1} to 3.2Å^{-1}. A large number of Vanadium runs were made in an attempt to characterize the instrumental resolution more completely than in the previous experiments. n-Eicosane was selected as being worthy of attention because it is the next member of the n-alkane series above n-nonadecane and it does not exhibit a solid-solid phase transition below its melting temperature. Measurements on n-nonadecane at 77 K were made for the purpose of confirming some of the structure found in the weighted frequency distributions measured at higher temperatures. The scattering angles used in these experiments were dictated by a desire to avoid significant contributions from coherent Bragg scattering.

A model for the diffusive broadening observed in the quasileastic peaks measured on n-nonadecane in the Solid I ("rotator") phase has been developed. This model is based on a particle performing a random walk among a series of equally spaced sites on a circle. The self-correlation function $G_s(r,t)$ can be calculated in a closed form for this model. The Fourier transform of $G_s(r,t)$, namely the "Scattering Law", can likewise be calculated in closed form. The fitting of this model to experimental data is now being done.

Inelastic Neutron Scattering from Vanadium Above and Below the Superconducting Transition

Bernard Mozer
(Inorganic Materials Division)

Inelastic neutron scattering data has been obtained from vanadium at 5.6K, a temperature above the superconducting transition temperature, 5.21K, and at a temperature of 4.5K. Data were obtained using 1.5Å and 2.45Å neutrons at several scattering angles using the Fermi chopper time-of-flight system installed at the NBS reactor. The 1.5Å neutrons, obtained

from (111) planes of a copper monochromater, were used to study the whole excitation spectra of vanadium. The resolution over most of the spectra is determined by the spread in energy in the incoming beam from the crystal mosaic and collimating systems. The energy spread is about 2×10^{-3} e.v. The 2.45Å beam was used to study the lower energy part of the excitation spectra where the energy resolution in the incoming beam is 0.7×10^{-3} e.v. Preliminary results indicate that the excitation spectra of vanadium above and below the superconducting transition are very similar yet there are indications of differences. Additional work is in progress to elucidate these differences.

Neutron Diffraction Studies of Clustering in $Cu_x{}^{62}Ni_{1-x}$ Alloys

Bernard Mozer
(Inorganic Materials Division)

$Cu_x{}^{62}Ni_{1-x}$ alloys made with essentially nickel-62 isotope were prepared for neutron diffraction studies of clustering in these alloys. The alloys were made by Mr. D. Fickle, of the NBS Metalurgy Division, in an induction furnace in beryllia crucibles and cold rolled to plates with three vacuum anneals at 1000°C for strain relief and homogenization. The last anneal was performed after all cold working and the sample allowed to furnace cool. Alloys of nominally X=0.20, 0.40, 0.525, 0.60 and 0.80 were prepared in the above method. Part of the alloy $Cu_{0.525}{}^{62}Ni_{0.475}$ was sent to Professor P. Beck of the University of Illinois to attempt a quench of the specimen from 1000°C. Neutron diffraction studies were performed on all the specimens. Diffraction data was taken with a 2.47Å neutron beam provided by the (002) reflection of an oriented pyrolytic graphite monochromator. A 2-1/2 inch filter of pyrolytic graphite was used to remove (<0.1%) the $\lambda/2$ contamination. Twenty minute collimators were used in-pile, after the monchromator, and before the counter. Diffraction data and background data were taken with counting statistics of 0.1%. Evidence for clustering is readily observed in the raw data in all the alloys, preliminary data anaylsis indicates little difference in clustering for the furnace cooled sample and the quenched sample at the identical composition. Data are being analyzed for the short-range-order parameters of these alloys.

Inelastic Neutron Scattering Study of Liquid Neon

Bernard Mozer
(Inorganic Materials Division)

and

L. A. de Graaf

The inelastic scattering of 2.44Å neutrons from liquid neon at a temperature of 35.05°K and a pressure of 21.4 atm has been studied on a time-of-flight spectrometer at the NBS reactor. The momentum transfer range covered in the experiment was $1.2 - 4.1\text{Å}^{-1}$. The total resolution for elastic scattering as measured at the detector position was 3.7% for $\Delta\lambda/\lambda$ which includes the resolution of the monochromator and collimation systems and 2.2% from the burst width of the chopper. The dynamic structure factor $S(Q,\omega)$ was obtained from the experimental data after applying corrections for detector efficiency, chopper transmission, background from the sample cell and cryostat, and multiple scattering effects. Special attention was given to the calculation of multiple scattering effects and to subtraction of the background scattering from the sample cell. The zeroth moment of $S(Q,\omega)$ has been compared with the static structure factor $S(Q)$ for liquid neon obtained from a recent neutron diffraction experiment at the same temperature and pressure as in the present experiments.

The full width at half maximum of $S(Q,\omega)$ and the dispersion curve $\omega_{max}(Q)$ were obtained and both corrected for the broadening due to the finite resolution of the spectrometer. $S(Q,\omega)$ and $\omega_{max}(Q)$ plotted versus Q show a behavior similar to that observed in liquid argon. A comparison was made between the experimental results and different theories and models for simple classical liquids.

Liquid Helium Structure Factor at Several Densities Above and Below the λ Transition

Bernard Mozer
(Inorganic Materials Division)

L. A. de Graaf

and

Bernard LeNeindre, Guest Worker
(Heat Division)

Preliminary neutron diffraction data has been taken on liquid helium at 12.53, 8.06, 4.15 and 3.97 atmospheres and at T = 2.91K. An attempt was also made to obtain neutron diffraction data below the transition at a temperature of roughly 2K for the same densities corresponding to the above pressures. The sample cell was thin walled Ti alloy with little coherent scattering. Although the alloy has almost flat neutron diffraction data it has poor thermal properties and we were unable to reach the same temperature below the λ transition for the several densities. Diffraction data in the λ region 1.5Å^{-1} to 4.60Å^{-1} had some data from 3.5Å^{-1} to 7.0Å^{-1} were taken. The data show density effects such that information on the three-body correlation function can be readily deduced from the density derivative of the structure factor at constant temperature.

A new sample cell has been constructed and tested. The new cell has titanium alloy walls with a base of good thermal transport properties at low temperatures. A good thermal path from the bath to the cell via this base is incorporated in a thermal radiation shield. We hope to achieve temperatures to 1.6K using this new cell and to obtain additional data for the density derivative of the structure factor at two temperatures well above and below the λ point.

High Resolution Fermi Chopper for Neutron Time of Flight Analysis

Bernard Mozer
(Inorganic Materials Division)

A curved slit Fermi chopper was designed to provide high resolution analysis at the instrument located at BT-5. The new chopper is similar to those currently in use and consists of aluminum disks bolted together holding a curved slit package of twelve plates of high strength maraging steel. The steel plates are 1/32" on a curve whose radius is 37.5 inches. The slits are 0.2 inches apart. The transmission function for this chopper is maximum at $\omega\lambda=20,000$ rpm-$\overset{\circ}{A}$, or for $2\overset{\circ}{A}$ neutrons and a rotor speed of 10,000 rpm. The rotor has been tested and was used in an experiment on Polyoxymethylene and to remeasure vanadium as a standard. The vanadium spectrum was quite sharp. The resolution of the chopper and the flight path is such as to allow 1% resolution at $1\overset{\circ}{A}$ for a rotor speed of 10,000 rpm.

Cold-Neutron Time of Flight Facility

R. S. Carter, A. A. Cinquepalma, L. A. de Graaf, F. J. Shorten, and J. B. Sturrock

The cold neutron time of flight facility is designed for inelastic neutron scattering studies of solids and liquids. A neutron beam from the reactor is filtered through poly-crystalline beryllium to eliminate neutrons with wavelengths smaller than $3.96\overset{\circ}{A}$. The beam then passes through a four rotor phased chopper system which chops the beam into short bursts of neutrons of selected energy. The sample to be studied is placed immediately after the choppers and the scattered neutrons are detected in a large bank of detectors arranged along the arc of a circle with a 2.4 meter radius. The detector information is fed into a multi-channel analyzer and the neutron energy determined from the flight time of the neutrons from the sample to the detectors.

The testing of the four rotor phased chopper system has been completed and the choppers have been installed at the beam hole. The steel framework for the detector shielding room has been completed and the walls and ceiling are under construction. The multiple detector bank is ready for installation in the shielding room when it is completed.

A temporary shielded flight path and a single bank of detectors have been installed to make use of the chopper system while the multiple-detector bank and shielding room are being completed. This temporary system is currently being checked out.

Cold-Neutron Source

R. S. Carter, E. M. Guglielmo, and L. A. de Graaf

The NBS reactor includes a special port, 22" in diameter, which penetrates the reflector up to the edge of the fuel elements. This large opening was designed to provide space for the insertion of a cold neutron source close to the core. By inserting a large, low temperature moderator it is possible to significantly increase the available intensity of very low energy neutron ($\Lambda > 4\text{Å}$) which are very useful for many types of molecular dynamic studies.

The moderator will be a cylinder of D_2O ice 14" in diameter by 12" long. A small amount of H_2O may be added to optimize the moderating property of the source. The source will have a reentrant hole 4" deep by 8" in diameter for beam extraction. The hole will be filled with beryllium which has the property of reflecting the higher energy neutrons back into the moderator while passing the lower energy neutrons. The moderator will be maintained at 25°K by cold helium gas passing through tubing in the moderator. The vacuum cryostat containing the moderator will be constructed of thin-walled aluminum to minimize heating by capture gamma rays. The cryostat will be mounted on a shielding plug and the whole assembly will roll inside a second plug and shield assembly which itself will roll into the special port of the NBSR. The shield mounted on the second plug surrounds the cryostat and consists of about 4" of lead and bismuth which grealy reduces the gamma ray heating of the moderator. Two 6" and two 2" diameter beams can be extracted from the cold source through the reactor shield. One of the 6" beams will be used for the cold neutron time-of-flight facility while the others await future development.

The helium for cooling is provided by an all helium refrigerator using a turbine expansion engine developed by the NBS cryogenic laboratory in Boulder. It has a one kilowatt capacity at 25°K which is sufficient to handle the heat generated in the moderator at twice the current reactor power.

The bismuth tip and the cryostat are the only items which remain to be installed. The cryostat has been received and extensive testing has just started. Of particular concern is the testing of the procedure for freezing the ice moderator in the cryostat without damaging it. The method consists of freezing the water in layers from the bottom up to prevent a thick layer of ice from forming over the freezing water. To make this possible the cryostat

has a water filled tube which is well enough insulated to use even after several layers of water have been frozen and the cryostat is quite cold. This procedure has been checked in a full scale mockup and refinement of the procedure is continuing.

The bismuth tip which was expected some time ago failed to pass its inspection at the plant. It had to be reworked and should be ready for another acceptance test shortly.

The installation procedures and the inplace testing program are being developed. After thorough testing, including measurements of radiation induced gas releases in the moderating material itself, application will be made to modify the technical specification so that it may be used at low temperature.

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"The Crystal and Molecular Structure of Nitratobis(dipyridine)Cobalt(III) Hydroxidenitrate Tetrahydrate, $[Co(C_{10}H_8N_2)_2(NO_3)](NO_3)(OH) \cdot 4H_2O$", C. W. Reimann, M. Zocchi, A. D. Mighell and A. Santoro, Acta Cryst. (in press).

Spin Structure of Erbium in High Magnetic Fields

J. J. Rhyne and S. J. Pickart
(Naval Ordnance Laboratory, White Oak, Md.)

The rare earth metals display a wide variety of magnetic spin states or structures depending upon a delicate balance of exchange, anisotropy and magneto-elastic interactions. The inter-dependence of exchange and anisotropy energies is not well understood, particularly in erbium. The results of high field magnetization experiments are complex and are not readily interperable without additional information. This requirement provided the motivation for a direct examination of the spin structure evolution in Er with fields up to 60 kOe and temperatures from 4K to 60K using the newly developed superconducting magnet facility.

The zero field spin state of erbium at 4K is a conical ferromagnet in which ferromagnetic order exists along the c crystal sxis with concomitant helical order of the basel plane moment component. Our studies show the resulting cone angle to be 30 degrees in zero field in accord with previous determinations. The periodicity of the helical order is not commensurate with the lattice and has a value $\tau = .24c*$. This wavevector remains unchanged to 60 kOe applied fields.

A computer program was developed which allows the automatic scanning and location of satellite and fundamental reflections along a prescribed path in reciprocal space.

A typical scan along the c axis using the arbitrary scan program is show in Fig. 1. The trajectory was from $(11\bar{2})$ to (112) in reciprocal space. The program automatically identifies peak positions during an initial coarse scan, then refines their exact coordinate, and finally executes a traditional 2:1 scan over each peak to permit comparison of integrated intensities.

The erbium results of Fig. 1a show the expected twin satellites of each principal nuclear peak arising from the helical order. Application of a magnetic field above 17 kOe along the [100] direction collapses the conical order as manifested by the dramatic decrease in $(100^{\pm})$ and $112^{\pm})$ satellite intensities as shown in Fig. 1b for data at 60 kOe. Such a transition to a stable of lower free energy results from the competition of exchange anisotropy and applied field energies. Analysis of the data has shown that the

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high field state is a "fan-type" moment structure. The angular width of the "fan" was observed to decrease with increasing field reaching 45 degrees at 60 kOe. The c axis moment component remains constant at 7.5 μ_B during the magnetization process. The moment associated with the fundamental (ferromagnetic) component of the fan structure is 3.8 μ_B. Harmonic satellites of all orders are theoretically possible with this structure; however those above first order were below our detection sensitivity. The data for the field dependence of the spin structure are to be evaluated in a theoretical model to obtain magnetic anisotropy and exchange energy information.
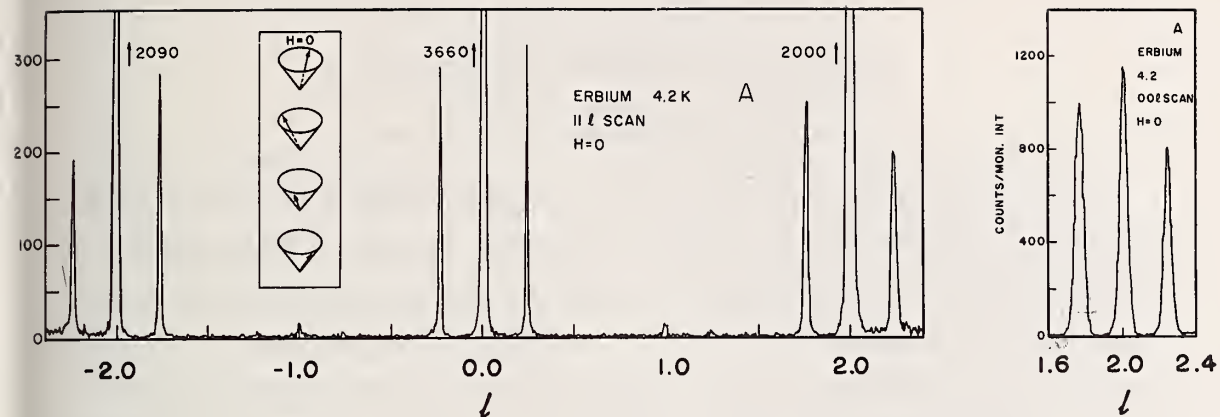
Fig. 1a. Abritrary reciprocal space scan a parallel to the c axis in erbium at 4K. Each allowed nuclear reflection is observed to have symetric first order magnetic satellites produced by the helical order. Magnetic intensity is also present on all nuclear reflections (except ool) from the c axis ferro-magnetic moment.

Note: Nuclear reflection amplitudes are truncated for clarity.
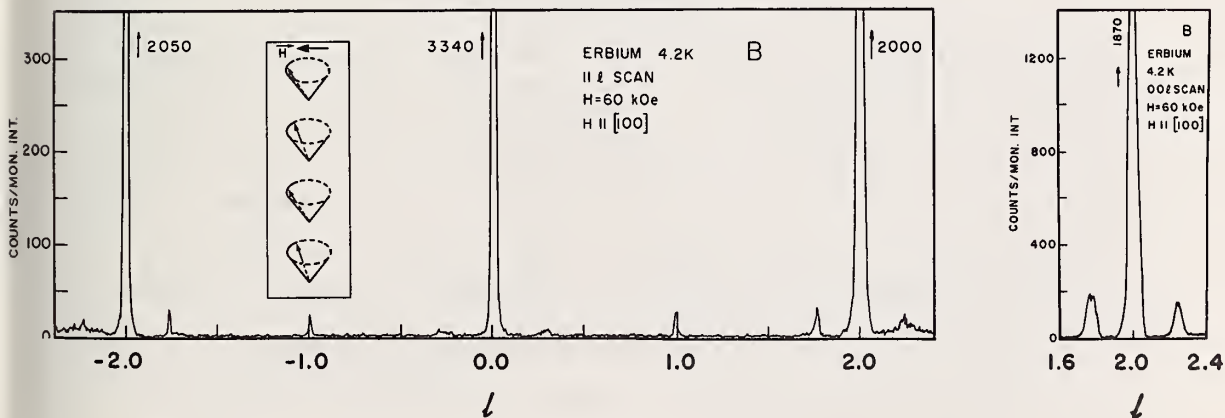
Fig. 1b. A similar scan at 4K with an applied magnetic field of 60 kOe along [100]. The satellite intensity is severly suppressed due to the development of a "fan" moment strucutre as discussed in the text. The <111> reflections are a result of double Bragg scattering.

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Rare Earth Lead Salt Magnetic Semiconductors

J. J. Rhyne
(Naval Ordnance Laboratory, White Oak, Md.)

A new study was undertaken of the doping of lead-salt, narrow-gap semiconductors with local moment rare earth ions. Such materials when ordered are known as magnetic semi-conductor properties (e.g. resistivity and optical band gap) by externally applied magnetic fields. We have used elastic neutron scattering to study the magnetic state of Gd-doped PbTe and SnTe using a powder dispersal method to reduce the effective Gd cross-section. Initial studies on 10% doped material showed no evidence of long range order to 2K. Work is presently underway using silver additions to the material to raise the effective carrier density and hence favor ordering by the indirect exchange process. Neutron investigation will be used to search for antiferromagnetic and periodic moment states whose detailed nature cannot be obtained by other methods.

Rare Earth - Cobalt Intermetallic Compounds

J. J. Rhyne and H. A. Alperin
(Naval Ordnance Laboratory, White Oak, Md.)

The magnetic structure of compounds of rare earths with cobalt is being examined throughout their range of magnetic order. Measurements to above 1000 K have been made using a modified high temperature vacuum furnance originally built for work on liquid aluminum. Principal compounds studied were of the RE_2Co_{17} and $RECo_3$ structures.

The neutron analysis allows the separation of the temperature dependence of the rare earth and cobalt sublattice magnetization. Of interest here is the possible modification of the rare earth exchange interaction by the presence of the cobalt.

Initial results on Tb_2Co_{17} confirm a ferromagnetic structure with a Cobalt Curie temperature near 800 K. A set of analysis programs is being developed to perform the decomposition of Co and rare earth moment dependences. $TbCo_3$ has an atomic arrangement quite similar to that of Tb_2Co_{17}. Neutron scans show, however, an unexpected anti-ferromagnetic moment component and a Curie temperature only a few degrees above room temperature. Additional investigations are planned of a series of other rare earth-cobalt and rare earth-iron intermetallic compounds.

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Structural Studies of Amorphous Solids
by Neutron Diffraction

G. A. Ferguson, J. Konnert and J. Karle
(U.S. Naval Research Laboratory, Washington, D.C.)

The interagency collaborative study between the Laboratory for the Structure of
Matter, U.S. Naval Research Laboratory and the Reactor Radiation Division, National
Bureau of Standards to determine the structure of amorphous solids of interest in modern
technology has included a precise measurement of the diffraction spectra of silica,
germania and vitreous arsenic selenide.

Previously published discrepancies appearing in reports of structural studies of
silica, using the neutron and x-ray diffraction techniques, have been investigated and
explained as part of an effort to understand this basic representative of the vitreous
state. This understanding has been obtained through newly devised data reduction
procedures applied to precisely measured diffraction data obtained over an extended
range of momentum change of incident particle (neutron and photon). These procedures
have revealed an ordering in the vitreous specimen which is greater than heretofore
suspected. A new interpretation of the structure of vitreous silica has been developed
which is based on maxima which occur in the radial distribution curve beyond 10A. This
interpretation suggests that when vitreous solids are formed, certain crystalline-like
arrangements of their atoms appear over limited spatial regions of the specimen. This
region, which was previously considered to be less than 5A for silica, is approximately
twice as large as reported by other investigators. The ordered arrangement is character-
istic of the vitreous state and is not to be considered as indicative of a partially
crystalline specimen.

The data reduction techniques which have been developed and applied successfully
in the silica are being employed to interpret new diffraction data obtained for germania
and vitreous arsenic triselenide.

The Lattice Dynamics of $NaNO_3$

K. W. Logan and S. F. Trevino
(Picatinny Arsenal, Dover, N. J.)

and

R. C. Casella

and

W. M. Shaw and J. D. Muhlestein
(University of Missouri, Columbia, Mo.)

and

R. D. Mical
(Massachusetts Institute of Technology
Cambridge, Mass.)

$NaNO_3$ crystallizes in a rhombohedral unit cell with two formula units in the primitive cell. Most of the dispersion curves have been measured in the high symmetry Λ direction and some in the lower symmetry A and B directions. The assignments of the observed Γ phonons was accomplished by the use of group theoretical selection rules developed by R. C. Casella and S. F. Trevino (see Casella and Trevino, Division Programs, Sec. A). These techniques proved invaluable in determining the reciprocal lattice point to use in observing the various phonons. The measurements resulted in a unification of the infrared and Raman assignments and the observation of the two Γ phonons which are optically inactive. In addition, a comparison with the results of a shell model calculation which successfully reproduced the observed optical data shows that it does not correctly predict all of the neutron measurements. The paper has been presented at the "International Conference on Phonons" and will be published in the proceedings of the conference.

The Structure of Ammonium Nitrate (IV)

E. Prince

and

C. S. Choi, and J. E. Mapes
(Explosives Laboratory, Picatinny Arsenal, Dover, N. J.)

The structure of ammonium nitrate (IV), the phase which is stable between -18°C and 32.C, has been refined by least-square methods with three-dimensional neutron diffraction data. Of 188 independent reflections, 29 were too weak to observe with sufficient accuracy, and 4 were affected by severe secondary extinction. The remaining 155 reflections were used in the refinement with anisotropic temperature factors and an isotropic extinction parameter. The final weighted R index was 0.028. The structure shown in Fig. 1, is orthorhombic $\underline{a}$ = 5.745Å, $\underline{b}$ = 5.438Å, $\underline{c}$ = 4.942Å, space group Pmmn, with two NH_4NO_3 formula units per unit cell. The ammonium and nitrate groups both occupy positions with mm symmetry. They are bound together by a two-dimensional network of hydrogen bonds, forming infinite sheets parallel to (001). The hydrogen bonds involve only one vertex of the triangular nitrate group, so the group is almost free to rotate around an N-) bond. Phase II, the tetragonal phase which is stable from 84.2°C to 125.2°C, is formed by a rotation around this bond. Phase III is stable from 32.3°C to 84.2°C, but both phase II and phase IV are metastable with respect to it. It is formed from phase IV by breaking the infinite sheets into double chains, followed by slight repacking.
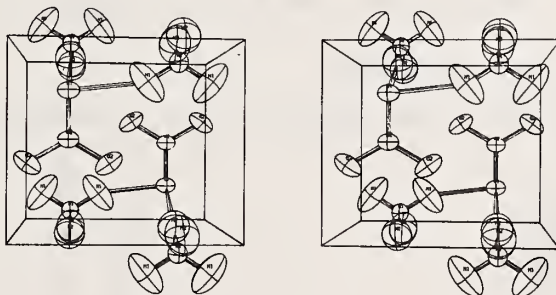
Fig. 1. Stereo pair showing the structure of ammonium nitrate, viewed along the b axis.

Precision Measurement of the Thermal Neutron
Scattering Amplitude for Silicon

C. S. Schneider, U. S. Naval Academy

The narrow wavelength bands of neutrons passing between two parallel perfect crystals include higher order wavelengths to the extent passed by the quartz filter at the reactor face. These neutron beams are simultaneously refracted by a right prism of silicon between the crystals, yielding an angular separation of the first and third orders to the second, or analyzer crystal:

$$\Delta\theta = \frac{8}{9} \frac{N\lambda^2 b}{2\pi} [\tan\phi + \cot\phi]$$

where N is the atomic density, λ is the primary neutron wavelength, and ϕ is the angle of the prism surface from the beam. The refraction peaks are shown in Fig. 1.

The beam port and counting electronics have been completed. The double perfect crystal spectrometer has been constructed as shown in Fig. 2, with calibrations completed for

(1) the wavelength slits (first order):

$$\lambda(\text{Å}) = 3.149\ 95(6) + (.250-\mu)0.0191$$

where μ is the slit center position in inches.

(2) the refractive goniometer:

$$\theta\left(\frac{\text{secs}}{\text{arc}}\right) = 1.3371(2)\mu - 0.036\left(\frac{\mu-50}{10}\right)^2$$

where μ is the computer angle in revolutions of the drive motor. This calibration is still under study.

(3) the prism goniometer:

$$\phi\ (\text{rads}) = 0.348\ 675(7)\ (\mu - .2868)\left[1 + \frac{(\mu - .2868)^2\ (\mu - .4743)}{141.54}\right]$$

where μ is the micrometer reading in inches.

One refractive separation has been measured and shows reproduceability within at least one part in 5000.

Fig. 1

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Fig. 2
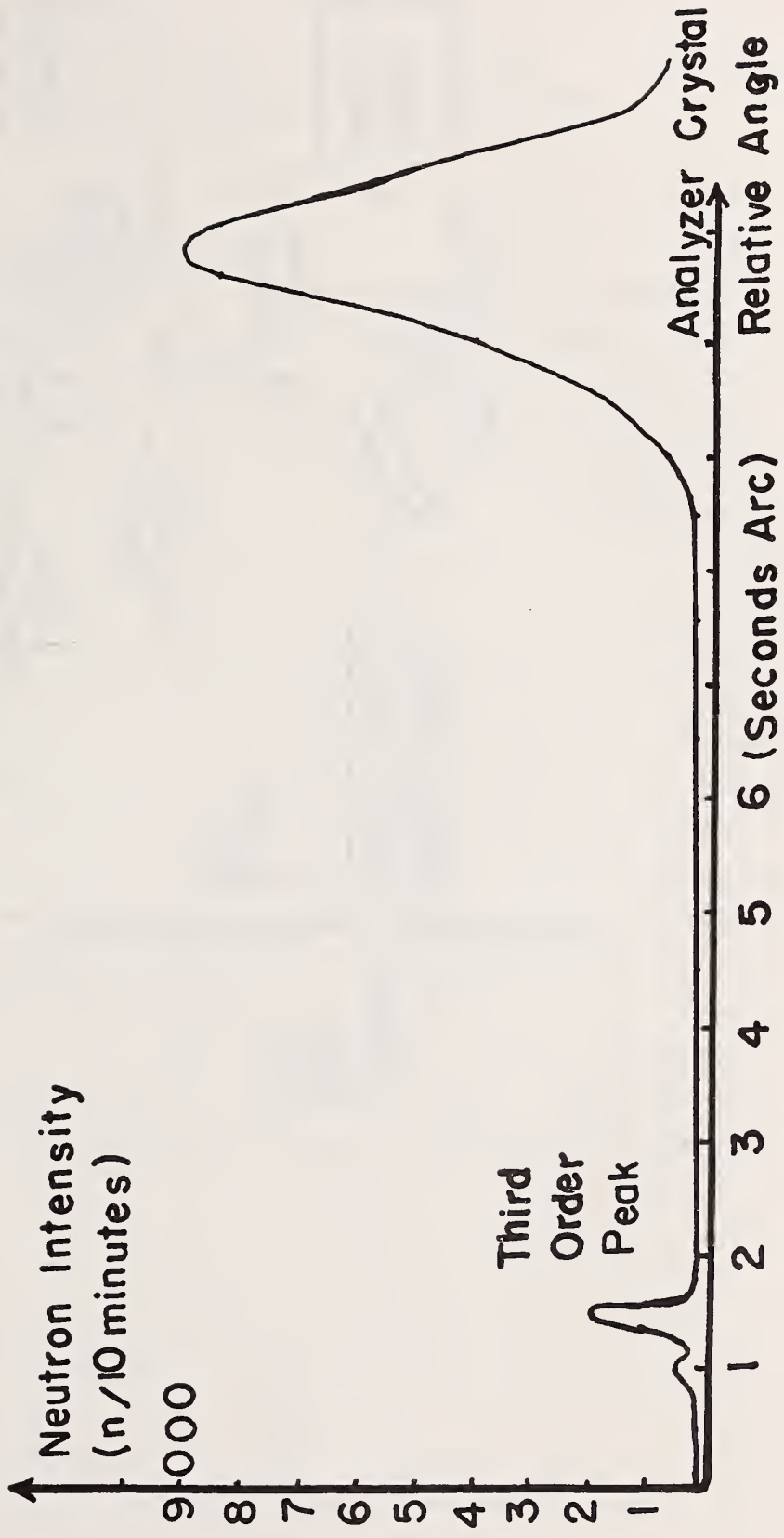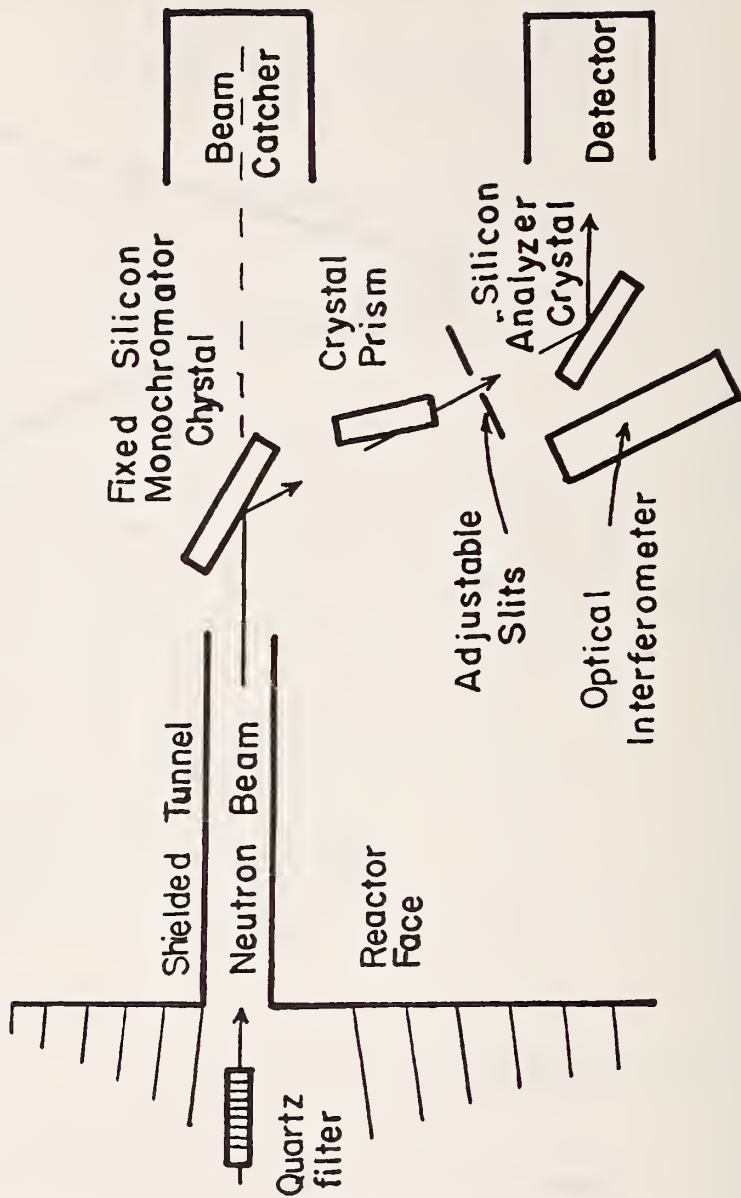
Equipment Development; NBS-NOL-NRL Joint Program

H. A. Alperin, J. J. Pickart, J. J. Rhyne and R. H. Williams
Naval Ordnance Laboratory, White Oak, Md.

V. J. Minkiewicz
University of Maryland, College Park, Md.

1. Triple-Axis Neutron Spectrometer

A three axis neutron spectrometer was completed and tested during the past year. Preliminary measurements have been made of phonons in PbTe at room temperature with an extension to low temperatures in progress. Initial results from the spectrometer indicated a need for additional shielding to reduce the background level to the range of one count per minute. This was accomplished with temporary facilities pending the completion and installation of a permanent shield late in 1971. Modifications have also been made to the counter and associated electronics to reduce noise and background.

2. Superconducting Magnet

A split-pair superconducting magnet and associated temperature-controlled dewar were completed, tested and used for the initial experimental investigation of the high field magnetic state of the erbium. The system performed well showing reasonable stability in the persistent operating mode and a field uniformity better than one percent over the width of the neutron beam. Helium losses from lead heating and thermal conduction were found to be minimal. One coil of the pair was found to have a low-resistance joint in the superconducting tape which precluded its operation above 60 kOe. This is in the process of being repaired and on completion the solenoid is expected to produce the full 100 kOe design field.

3. High Temperature Furnace

A vacuum furnace originally designed and used for a study of liquid aluminum was modified to accept conventional powder and single crystal specimens. Temperature stability was improved and an accurate calibration was performed to temperatures up to 1500 K.

Equipment Development; NBS-Picatinny Arsenal Joint Program

C. S. Choi, H. J. Prask and S. F. Trevino
Explosives Lab, Picatinny Arsenal, Dover, N. J.

and

J. J. Rush, R. S. Carter and A. Cinquepalma

The interest of the PA group has been the study of the structure and lattice dynamics of metastable and related materials, primarily by means of neutron scattering, with the aim of elucidating the cause of instability. Considerable effort has also been devoted to the study of stable materials such as polymers, hydrogen-bonded systems, ionic and molecular solids, molecular liquids, crystal hydrates and metal hydrides. The activities in the collaborative program began in September 1970 after the shutdown of the Army reactor at Watertown, Massachusetts.

The main effort of the collaborative program at NBS, thus far, has been the design and fabrication of new shielding and components for instruments used at Watertown. These instruments include a conventional triple-axis spectrometer and a neutron diffractometer. To achieve higher efficiency in neutron utilization, both of the above instruments will be mounted on a single beam-port. The neutron diffractometer will employ a thin aluminum single-crystal monochromator, and the three-axis instrument will utilize the neutrons transmitted by the diffractometer monochromator. Because large single crystals for coherent neutron inelastic scattering measurements are often difficult to obtain, considerable effort has been devoted to maximizing signal/background levels through shielding improvements. Two such improvements are a 360° counter-analyzer shield, fixed with respect to the scattered beam, in which the counter and analyzer are housed, and a fixed shield, circumferential to the main monochromator shield, which allows variation of incident energy without movement of shielding.

An existing fixed-crystal-monochromator, time of flight spectrometer will be mounted on a second beam tube along with a second three-axis instrument. The time of flight instrument incorporates a relatively simple mechanical phasing system between a coarse chopper - for order elimination and background suppression - and a "fine" Fermi chopper. Incident neutron burst times of 20-25 µs are obtained with this system.

The present status of the above-described development is the following. The diffractometer/triple-axis system is essentially completed, with fabrication of only a few,

relatively simple, shielding components remaining. Installation and operation is expected to be achieved in the next quarter. Modifications of the time of flight instrument are near to completion. A new support system for the five-meter flight path is being tested, and a modification of the chopper phasing control has been designed. New shielding - very little of which will be required for this instrument - is being designed. The second triple-axis instrument is in the conceptual design stage.

Publications

"The Lattice Dynamics of $NaNO_3$", K. W. Logan, S. F. Trevino, R. C. Casella, W. M. Shaw, L. D. Muhlestein and R. D. Mical, to be published in "Proceedings of the Conference on Phonons", Rennes, France, July 1971.

"Ice I - Lattice Dynamics and Incoherent Neutron Scattering", H. Prask, S. Trevino, J. D. Gault and K. W. Logan, submitted to J. Chem. Phys.

"Lattice Dynamics of Rhombohedral Sodium Azide", H. Rafizadeh, S. Yip and H. Prask, J. Chem. Phys., (in press).

"The Lattice Dynamics of KN_3", K. R. Rao, S. F. Trevino, H. Prask and R. D. Mical, Phys. Rev., (in press).

Measurement of Avergae Fission Cross Section Ratios

James A. Grundl
(Nuclear Radition Division)

A cavity fission neutron source at the reactor thermal column has been operated in conjunction with a fission ionization chamber to measure average fission cross section ratios. The ratios, $\bar{\sigma}_f(^{235}U)/\bar{\sigma}_f(^{238}U)$, have been measured for U-235 and Pu-239 fission neutrons. The cavity fission source, a single fission ionization chamber, and a redundant determination of fission foil weight ratios were employed for the measurements. The result for U-235 fission neutrons is 3.81±0.17, a value which confirms earlier integral microscopic measurements and remains discrepant with predictions based on differential microscopic data to the extent of 10 to 17%. The measured ratio of average cross section ratios, X_{235}/X_{239}, is 0.970±0.012. This value represents a departure from unity which is less than one-half that predicted by differential microscopic data. A paper describing these measurements was presented at a consultant's meeting, "Status of Prompt Fission Neutron Spectra", called by the Nuclear Data Center at the IAEA in Vienna. Refinement of these measurements is continuing.

Quality Assurance Irradiations for Fast Reactor Materials Dosimetry

W. N. McLroy, L. S. Kellogg, and W. Y. Matsumoto
(Dosimetry Center of the Hanford Engineering Development Laboratory)

Rabbit irradiations in the RT-4 pneumatic facility of the NBS reactor were performed with materials used for high flux, neutron fluence monitors by the Fast Reactor Materials Dosimetry Center (FRMDC) at the Hanford Engineering Development Laboratory (HEDL). The primary objective of these irradiations was quality assurance; in particular, the determination of the concentration and homogeneity of gold, silver, and tantalum in alloys of aluminum. A secondary objective was the search for small thermal and resonance energy cross sections in the exoergic threshold reactions $^{63}Cu(n,\alpha)$, $^{54}Fe(n,p)$, $^{47}Ti(n,p)$, and $^{58}Ni(n,p)$.

Arrangements for carefully positioning a multiplicity of small wires and foils, and also development of a technique for heat removal which made possible exposures with cadmium covers, were notable characteristics of the irradiations. The neutron flux for each irradiation was monitored with Au-Al and Co-Al activation detector wires; the latter is an NBS Standard Reference Material (SRM). The flux level and cadmium ratios given by the monitors for the RT-4 position were

$$(nv)_{th} = 1.26 \times 10^{13} \text{ (reactor at 10 MW)}$$

cadmium ratio for Cobalt = 368 (infinite dilution)

cadmium ratio for Gold = 38 (infinite dilution)

The necessity for such quality assurance irradiations was amply demonstrated. The silver and gold concentrations determined for the alloys differed by more than 50% and the tantalum alloy by 10% from the values certified by the vendor. The new values, obtained from the RT-4 irradiations, are based on comparisons with reference standards as well as on the NBS standard reference Co-Al wire and thermal neutron cross section values. The value for the Au-Al material was additionally verified by three independent analytical measurements. Needless to say, the reliability of this SRM is of considerable important now for the Fast Reactor Dosimetry Center at HEDL.

P. D. LaFleur
Analytical Chemistry Division

The Activation Analysis Section of the Analytical Chemistry Division is physically located in the NBS Reactor Building. A complete description of the Section's activities may be found in its annual progress report, NBS Technical Note 588, therefore only a brief summary will be included here.

1. Biological Standard Reference Materials

In addition to a comprehensive analytical program on two new Biological Standard Reference Materials, SRM 1571, Orchard Leaves and SRM 1577, Bovine Liver, Section personnel were responsible for the overall coordination of the analytical program for these important standard materials throughout the Analytical Chemistry Division. The first of these materials SRM 1571, was issued during the year and is certified for three major constituents, two minor constituents and 14 trace constituents. In addition, preliminary values were given for another 9 trace constituents. These materials are designed to give scientists working in the areas of nutrition, metabolic disease and pollution, as well as analytical chemists, materials which closely approximate the matrices with which they are concerned. The certification of these standard materials was a painstaking process so that the scientific community may be assured of the accuracy of the results obtained. As an example, the certified value for mercury in the orchard leaves was obtained only after approximately one man year of analytical effort had gone into this analysis in the Division.

Concurrent with the analysis of these materials, a program has been inaugurated to accurately determine the pressure buildup of a variety of organic materials upon irradiation in a nuclear reactor. Differences between pressure buildup in freeze-dried and nonfreeze-dried materials and as a function of fast to thermal neutron ratios are being pursued. Thus far over 100 samples have been irradiated and tested for pressure buildup inside a quartz ampoule using a water monometer system.

2. Mercury Analysis

Considerable effort was expended during the year on the development of a destructive analytical technique for determining mercury in biological matrices. The method consists of burning the sample after irradiation in an oxygen stream and trapping the mercury and other volatiles in a liquid nitrogen cold trap. Extensive testing for chemical yield was performed.

It was determined that the procedure is quantitative and that using high resolution lithium-drifted germanium solid state detectors, mercury may be determined at levels of approximately one part per billion without great difficulty. Using the procedure developed, mercury was determined in the orchard leaves and bovine liver Standard Reference Materials and Standard Reference Material 1630, Mercury in Coal. The Section also participated in a round robin analysis of mercury in coal sponsored by the United States Bureau of Mines.

3. Californium-252 Neutron Source

The use of Californium-252 neutron sources was investigated during the year and a procedure was developed for the determination of fluorine using the nuclear reaction $^{19}F(n,\alpha)^{16}N$. The procedure was applied to a number of fluorine containing organic materials with excellent accuracy and precision. In addition, hafnium was determined in zircoloys using the reaction $^{178}Hf(n,\gamma)^{179m}Hf$. Additional work is being done in the determination of macroconstituents in Standard Reference Material Metallo-organic compounds to evaluate high precision activation analysis using a stable neutron source such as californium-252.

4. The Nuclear Track Technique

The nuclear track technique has been applied to the determination of nitrogen and lithium in biological materials using protons from the reaction nitrogen $^{14}N(n,p)^{14}C$ in the case of the determination of nitrogen and alpha particles from the reaction $^{6}Li(n,p)^{4}He$ for the determination of lithium. Investigations are continuing for the application of this technique to the determination of thorium in geological and glass samples using a fast neutron, thermal neutron differential technique to correct for possible uranium contribution.

5. Mass Separator

A high throughput mass separator has been installed in the Section and its applications to activation analysis and nuclear chemistry are being evaluated at the present time. Resolution, yields and "memory" are being studied for the application to particularly difficult problems in activation analysis where an interference free source must be obtained. A separation by atomic number may be performed using chemistry and separation by mass number subsequently performed using the mass separator. In this way it is possible to obtain a pure isotope for radiation detection.

In addition to the subjects discussed above, the Section has participated in a large number of analyses for other Sections in the Division and other Divisions at the Bureau and

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for other Federal Agencies. Work is continuing in the application of computer techniques to
activation analysis and specifically in an interactive system using a minicomputer and multi-
channel pulse height analyzers. Work has also begun on the methods for the determination of
a number of other toxic metals in tissues and foodstuffs, for example selenium, cadmium, and
arsenic, and this effort will be continued during the coming year along with a number of
fundamental studies in the activation analysis technique.

Atomic Constants from x-ray and Positronium Decay γ-ray Measurements

R. D. Deslattes and W. C. Sauder
(Optical Physics Division)

The development of the necessary instrumentation for this experiment has continued
during the past year and shortly will be installed in the controlled environment chamber
in the reactor building.

Cobalt-60 Nuclear Thermometry

H. Marshak and R. J. Soulen, Jr.
(Heat Division)

The degree of orientation of a nuclear spin system is defined in terms of the popula-
tions a_m of the nuclear energy levels E_m by the Boltzman factor, viz

$$a_m = \frac{e^{-E_m/kT}}{\sum\limits_{m=-I}^{I} e^{-E_m/kT}}$$

where I is the nuclear spin with projection m, k is the Boltzman constant and T the absolute
temperature. A measurement of these populations would enable us to determine the absolute
temperature. The accuracy would depend upon the accuracies of E_m, k and the measurement of
a_m. Since the energies E_m are usually quite small - the order of fractions of a degree -
such a thermometer is only useful at very low temperatures.

The populations a_m can be determined by measuring the angular distribution of γ-rays
from a radioactive sample. If the multipolarity of the transition and the initial and final
states involved are known, the anisotropy of the radiation can yield the absolute temperature.

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A very favorable case is now being investigated; namely Co-60 in a hcp Co-59 single crystal. The Co-60 nuclei are produced in situ by irradiating the small Co-59 single crystal in the NBS reactor.

A preliminary comparison has been made of this thermometer and a Josephson Junction Noise Thermometer over the temperature range of 20 to 40 mK. The results showed agreement to within $\pm$ 0.5 mK. Further measurements are planned in order to improve the accuracy.

Decay Scheme Studies

F. J. Schima
(Nuclear Radiation Division)

Sources of Xe-129m have been prepared using the isotope separation at the CRR and the NBS reactor. Xe-128 was ion implanted into ultra pure Al foil. The foil was trimmed down to a strip, 4 mm x 2.5 cm and irradiated in RT-4 for 8 hour periods. The gamma activity was then observed by Si(Li) and Ge(Li) detectors. The energy of the isomeric transition was measured as 196.38 $\pm$ .11 keV, and the energy of the first excited state, as 39.50 $\pm$ .17 keV. The half-life of the 235.88 keV level was observed by the 196.38 keV gamma ray and was measured to be 8.87 $\pm$ .09 days. This is considerably greater than the 8.0 $\pm$ .2 day value adopted by the literature.

A source of Kr-81 was prepared by irradiating 7.6 ml sample of Kr gas (enriched to 50% Kr-80) in G-2 of the NBS reactor for 8 weeks. After a 6-month cool down, the gas was implanted in Al foil using the isotope separation. A gamma ray was found and its energy was measured to be 275.99 $\pm$ .01 keV. The K x-ray intensity was measured by an inter-comparison to the standard sources of Co-57 and Se-75. The ratio of the 275.99 keV gamma ray intensity to the K x-ray intensity was determined to be 6.8 $\pm$ .8%. This would indicate a branch to the first excite state of Br-81 of approximately 3.7%, depending on the value of the fluorescent yield that is taken. This branch in the Kr-81 decay has not been previously reported.

Preliminary work has been done on a measurement of the fission yields of Xe-131m and Xe-129. A noble gas handling system has been built and is undergoing tests. Several 2 mg $^{235}U_3O_8$ samples have been irradiated in RT-4. It has been estimated that a 30 minute irradiation would be least sensitive to the ingrowth of Xe-131m from I-131 decay and yet produce a detectable amount of Xe-131m from fission.

Parity Violation in Strong Interactions

J. L. Alberi and R. Wilson
(Harvard University, Cambridge, Mass.)

and

G. Scharff Goldhaber
(Brookhaven National Laboratory)

and

I. G. Schroder
(Nuclear Radiation Division)

The program to study parity violation due to the weak interaction in the overall nucleon-nucleon interaction (weak forces between nucleons) is continuing in collaboration with Harvard University and Brookhaven National Laboratory.

Preliminary data with a Cd-113 target has been obtained and a control experiment with an internal titanium target is underway. A search is being conducted for other nuclei which have neutron capture states which are no longer eigenstates of parity and, therefore, exhibit small parity impurities similar to those in cadmium.

Two-Photon Emission in (n,p) Capture

B. T. Chertok
(American University, Washington, D.C.)

and

J. L. Alberi
(Harvard University, Cambridge, Mass.)

and

I. G. Schroder
(Nuclear Radiation Division)

A search for two-photo emission in thermal (n,p) capture has been conducted with the aim of establishing a lower limit to the occurence of this phenomenon. The data both with a hydrogen (H_2O) and a deuterium (D_2O) target has been obtained and the data is being analyzed.

Fast Neutron ^{3}He Spectrometer

E. D. McGarry
(Nuclear Vulnerability Branch - HDL)

and

I. G. Schroder
(Nuclear Radiation Division)

In collaboration with HDL a program is underway to extend the region of application of the He-3 fast neutron spectrometer below the 500 KeV region by means of fast electronics and shaping circuitry to help eliminate the low-energy γ-ray sensitivity of the semi-conductor spectrometer. Several circuits have been used and γ-charged particle discrimination obtained in the 500 KeV region. Several tests have been performed with a Cf-252 source on loan from Savannah River (AEC).

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REACTOR OPERATIONS AND SERVICE PROGRAMS

A. NBSR Operating Summary

During the period October 1, 1970 through September 30, 1971, approximately 5100 hours of reactor time was provided to users. This is equivalent to about 51,000 megawatt-hours of energy produced. Operational problems discussed below and operations with minimum staffing reduced reactor on-line time to 58% for this period compared with 67% last year. Significantly improved average fuel burnup of 48% was achieved through utilization of heavier loaded elements. A summary of operating statistics together with those of the previous year for comparison are presented in the following table:

NBSR Operating Summary

	Nov. 1969 to Nov. 1970	Oct. 1970 to Oct. 1971
Reactor operations to date, Mwh	78213.3	123760.6
Reactor Operations for year, Mwh	59186.0	51175.3
Hours Reactor Critical	6000	5440
No. of Days at 10 Mw	247	213
On-line time at 10 Mw	67%	58%
Number of fuel elements used	44	24
Average U-235 Burnup	34%	48%
Number of Refueling Operations	11	6
Number of Unscheduled Shutdowns	3	10
Number of Irradiations	1500	3000
Number of Visitors	4000	4000

Four operational problems of significance were experienced over the last year. These problems collectively contributed to operating time losses of about four months. Some of this lost time was made up by reducing the number and frequency of subsequent scheduled shutdowns through advance performance of required maintenance operations. These problems are summarized below:

1. Repair of Primary System Piping Welds

    A small crack developed in each of two welds on small 1/2" drain lines in the primary
cooling system. Difficulty was experienced in rewelding the lines because they were aluminum
and old. In the process a 12" aluminum line had to be capped. Since all repair welds had
to be made to code, a welder had to be qualified and qualification welds prepared. The
final weld had to meet code requirements of inspection including 100% x-ray. Operating time
lost was approximately 22 days.

2. Failure of Primary System Inlet Strainer Basket

    The inlet strainer was opened to repair a leaking vent line at which time, it was
discovered that the entire wire mesh screen, two retaining bars and pieces of the bottom
plate had apparently torn loose from the strainer basket and had been washed downstream. The
search for the missing pieces required removal of the core into the storage pool and draining
of the vessel and primary piping. The entire area between the strainer were either visually
inspected or radiographed when visual inspection was not possible. Almost all of the missing
pieces were located and recovered intact. A few small pieces of the base could not be
accounted for but were definitely excluded from the area of concern. It is possible that
these pieces were removed from the basket during previous repairs prior to the operation of
the reactor. New strainers fabricated of heavier materials were installed in both the inlet
and outlet pipes (the outlet strainer was found intact). The new basket is recessed farther
from the pipe entrance permitting about 2-1/2 times the flow area and reducing the pressure
drop across the wire mesh. Total operating time lost was about 30 days.

3. Failure of the Locking Bar Pins of the Fuel Element Head Assembly

    During fuel reloading operations, two fuel elements were dropped in the transfer chute.
In both the fuel element head shaft separated from the locking bar by a type 410 stainless
steel roll pin. The roll pin purchased to Mil-STD 16562 is a 0.131" to 0.135" and is
pressed into a hole that is 0.125" to 0.129" in diameter. Subsequent examination indicated
that the two pins in question were either initially undersized or have become undersized
during installation or use. Both pins fell out and were recovered intact. They measured
0.125" and 0.126" respectively while the holes in the shaft measured 0.127". As a result of
this, fuel element heads were modified by replacing the type 410 stainless steel pin with a
bigger type 304 stainless steel #3 taper pin that is pressed in so that it is below the

surface of the bar. The pin is then staked to positively secure it in place. Operating time lost was about 10 days.

4. Leaks in the Primary System Main Heat Exchanger

Two separate leaks developed in the primary system main heat exchanger within a short time of each other. In each case the leak involved a single tube. One leak was due to local corrosion from the outside of the tube, the other was caused by the tube rubbing against a baffle. Very extensive examination, testing and inspection of the heat exchanger was performed since there is no backup exchanger. The results indicated numerous defects. On this basis 36 tubes out of 1285 were plugged. The entire problem was evaluated by an outside panel of experts. The panel recommended the prompt replacement of the present aluminum heat exchanger by one made of stainless steel. The specifications of a new stainless steel heat exchanger are almost completed and steps to obtain it will be taken shortly. It is expected that the new exchanger will be ready for installation in about 1-1/2 years. Total operating time lost was about 50 days.

B. SERVICE PROGRAMS

Service and irradiation programs have more than doubled over the last year. In all more than 3000 irradiations involving over 20,000 samples were irradiated. These again involved a wide range of people-oriented projects as well as research and application programs. Of significance among the new programs is the preparation of Fluorine-18 for scanning for bone cancer. Included among participating organizations are the major governmental medical and health centers and major university medical centers in the area. More specific summaries of the service and irradiation programs are outlined in succeeding sections.

Fluorine-18 Radiopharmaceutical for Bone Lesion Localization

Major George Dunson and Ira Godwin
Northern Virginia Pathology Laboratories, Fairfax, Va.

John Harbert
Georgetown University Hospital, Washington, D.C.

and

Edward Embree

Fluorine-18 (F-18) has been known for several years as the agent of choice for bone lesion imaging. The radioisotope administered orally or parenterally accumulates in bone in direct proportion to the bone flow and metabolic activity. F-18 scintographs demonstrate lesions far earlier than x-ray radiographs which require a 30% change in bone density in order to produce lesion images. F-18 gives a sufficiently low radiation dose to patients to permit its use in skeletal surveys and benign lesion imaging. The radiation dose of strontium-85, the readily available bone imaging agent, is so high that its use is restricted to patients with diagnosed primary carcinoma.

Fluorine-18 radiopharmaceutical was successfully prepared and used at Walter Reed General Hospital for about two years. However, due to the small amounts being produced and legal restrictions it could not be made available to the civilian medical community. F-18 could not be made commercially available to the Washington/Baltimore area because it has a two hour physical half life.

Due to the great need for F-18 for patient use and the potential of the National Bureau of Standards Reactor Radiation Division to make F-18 in large amounts several medical institutions joined in a group project to make F-18 radiopharmaceutical at the National Bureau of Standards. A goal was set of having 70 millicuries (mCi) F-18 in dispensing vials and quality control and assay testing completed by 7 a.m. on the day of use.

Legal and administrative problems included absolution of the U.S. government from patient responsibility, compliance with Atomic Energy Commission (AEC) radioactive materials licenses, assuring the Food and Drug Administration (FDA) of drug safety and efficacy and adequate records keeping, location of facilities and equipment for accomplishing the separation and testing of a two hour half life material, and the availability of a radiopharmacist to accomplish the project. The National Bureau of Standards Reactor Radiation Division provided laboratory facilities and equipment, materials and supplies, and health physics and

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reactor operations assistance and expertise. The Walter Reed General Hospital permitted its radiopharmacist to direct the project in an off-duty individual responsibility status. The FDA accepted the Investigational New Drug Application (IND) of the radiopharmacist, which assured drug safety, high standards of drug processing and testing and adequate records keeping. Relief of the government from patient liability was assured in three ways: the off-duty status of the radiopharmacist; physicians at the individual institutions using F-18 as investigators under the IND sponsorship of the radiopharmacist; and participating medical institutions executing statements of disclaimer of governmental responsibility. AEC license compliance was achieved by transfers of F-18 radioisotope from the NBS AEC license to AEC licenses of the participating hospitals.

Physical problems in the production of F-18 were solved according to the method described in the companion article "Fluorine-18 production in the NBS Reactor".

Problems involved in the radiopharmaceutical processing of high activities of F-18 include the protection of the worker from ionizing radiation, elimination of large amounts of tritium formed during the production reactions, preparing the drug for the desired route of administration, and testing for radiation activity, radioisotopic purity, chemical purity and drug stability.

The oral route of administration was chosen because it eliminated a requirement for pyrogen and sterility testing. Pyrogen testing of a two hour half life material would have necessitated maintaining a rabbit colony on site and a minimum of three hours delay in distribution of the radiopharmaceutical. Studies previously published by the radiopharmacist established the fact that for fasting patients oral and intravenous administration are equally effective. Pyrogen free chemicals and sterile techniques were used in the radiopharmaceutical processing of an isotonic product as a precaution in case the drug might be mistakenly given intravenously on some occasion.

Readily achievable standards of pharmaceutical components were established: $NaHCO_3$ up to 2% W/V; NaOH up to 3% W/V as an alternative to $NaHCO_3$; Na_2SO_4 up to 3%; total sodium less than 24 mg per mCi F-18 at completion of processing; tritium activity less than 20% of F-18 activity at completion of processing. All criteria were easily met and the tritium (H-3) activity was less than 1% of F-18 activity using the method described in this article.

Controls used in the processing and packing of the drug include:

1. sterility and pyrogen tested stock reagents

2. pH test for alkaline chemical stability

3. flame test for lithium

4. Determination of F-18 activity by ionization chamber or liquid scintillation assay, or both.

5. Determination of H-3 by liquid scintillation as an after packing control, prior to receipt by investigators.

6. Calculation of inert components on a W/V%.

7. Retention or recall of the product if it does not meet the standards given above.

8. Gamma spectrum analysis for nuclide purity of an irradiated control sample of each batch of targets prior to receipt by investigators.

Worker protection from external and internal radiation hazard was accomplished by working in a radioactive fume hood, using long handled tools behind lead brick barriers and uitlizing mirrors for vision. After developing a semiautomated technique and practice at working with mirror images a worker could accomplish the radiopharmaceutical processing three times weekly without approaching occupational radiation exposure limits.

The method of separation, purification and assay are as follows:

The basic equipment consists of a distillation flask with receiver, a survey meter and electric mantle as illustrated in Figure 1. A dry 300 ml distilling flask (A) was fitted with a dry funnel and placed over an electric heating mantle (B) with the probe (C) of a survey meter placed underneath. A porcelain dish (D) was placed under the condensor of the distilling flask which was separated by a lead partition (E). The whole set up was placed inside a fume hood with proper lead shielding.

The tubes were broken into 1-2 inch bits inside a flexible plastic tubing and the glass pieces with the powder were dropped into the flask. Forty ml of 50% H_2SO_4 was slowly added through the funnel. Effervescence was allowed to continue nearly to completion, and the flask was tightly stoppered. The heating mantle was turned on and the condensate was collected in the porcelain evaporating dish containing 10 ml of 1.5% sodium bicarbonate of parenteral quality. When the survey meter indicated a drop of about 95% of the radiation level, the flask was moved aside from the heat and the condensor was put over a beaker

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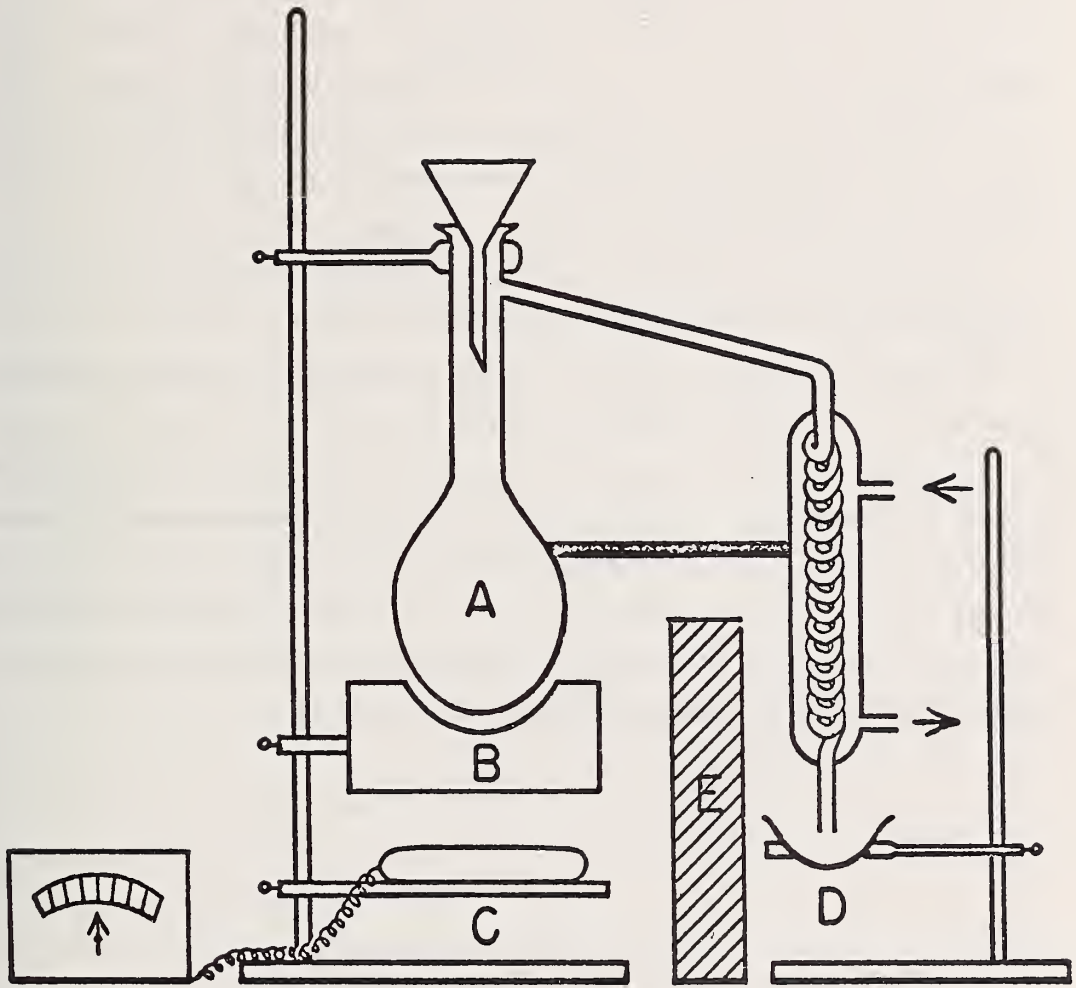
Fig. 1.

containing sodium bicarbonate solution to trap radioactive contamination. The porcelain
dish was then placed on the heater and the solution was tested for the alkalinity necessary
to form heat stable ionic sodium fluoride. The condensate was flame tested for the carmine
color of lithium. The dish was heated until the solution was boiled to near dryness.
Twenty-five ml of water for injection was then added, and the solution was passed through a
membrane filter into a vaccine vial. Assay was accomplished by ionization chamber for F-18
activity, using 1/2 S_H-85 as a standard, and by liquid scintillation for tritium. An initial
qualitative evaluation of H-3 can be done in the presence of F-18 when a narrow window is
used. Later a quantitative assay of H-3 can be performed.

Chemical assay was accomplished by calculation from volumes of solutions used. Biologi-
cal assays were done during preliminary phases of the project.

The result of the project is that 70 mCi F-18 radiopharmaceutical can be made ready for
distribution to participating medical institutions by 7 a.m.

Fluorine-18 produced and processed at the National Bureau of Standards Reactor Radiation
Division has been successfully used in patients at the Fairfax Hospital, Fairfax, Virginia.
Georgetown University Hospital, Washington, D.C., the Johns Hopkins Hospital, Baltimore, Md.,
National Institutes of Health, Bethesda, Md., National Naval Medical Center, Bethesda, Md.,
Northern Virginia Pathology Laboratories, Fairfax, Va., Veterans Administration Hospital,
Washington, D.C., and Washington Hospital Center, Washington, D.C.

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Fluorine-18 Production in the NBS Reactor

Major George Dunson
Walter Reed General Hospital, Washington, D.C.

William Crofford
Department of Army Civilians, Fort Belvoir, Va.

and

John Ring

Fluorine-18 (F-18) is a 110 minute half life positron emitter that has been used extensively, when available, for radioscintographic visualization of bone lesions.

F-18 has been produced in nuclear reactor and cyclotron facilities by a variety of nuclear reactions. However, due to its short physical half life, its use has been geographically restricted to institutions to which the radioisotope in pharmaceutical form can be quickly delivered to the using nuclear medicine clinic. There are no medical use cyclotrons or reactors in the Washington D.C. area so F-18 was undertaken in U.S. Army nuclear reactors.

F-18 was successfully produced in the Walter Reed Army Institute of Research (WRAIR) nuclear reactor and the Diamond Ordnance Radiation Facility (DORF) nuclear reactor. The WRAIR reactor was closed in February 1970. Due to the medium level thermal neutron flux and operation hours of the DORF reactor a maximum of six millicuries (mCi) of F-18 per day could be made available for patients. The F-18 could be available for pharmaceutical processing no earlier than 11:30 a.m. The expense per production run of F-18 in the medium power reactor was high because the reactor could not be used for other purposes for up to three hours and the non-reuseable target material and target cost about $45.00 for one irradiation with a maximum 6 mCi yield.

A project was undertaken at the National Bureau of Standards to produce F-18 in amounts exceeding fifty millicuries. Additional goals were that the production did not interfere with or prevent simultaneous reactor projects and the production could be completed by 6 a.m. on the morning of use.

The only reaction available for nuclear reactor production of F-18 is a two step reaction in which a thermal neutron splits a lithium-6 (Li-6) atom to give off a tritium atom which enters an oxygen nucleus and forms F-18 upon the ejection of a neutron. These reactions using enriched stable isotopic Li-6 as the carbonate are written as:

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$$1. \quad {}^6Li|n,\alpha|{}^3H \qquad\qquad 2. \quad {}^{16}O|{}^3H,n|{}^{18}F$$
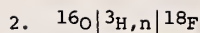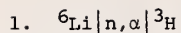
The cross section of the first reaction is 1000 barns and the cross section of the second reaction is one-half of a barn. The physical difficulties in the production of F-18 are primarily associated with the phenomena of heat generation when alpha particles are formed and contained during the first step of the reaction. Secondary problems are associated with using a target container and holder that neither becomes radioactive nor disintegrates in a high thermal neutron flux. Problems dependent on the characteristics of the nuclear reactor are the length and design of the target container and holder and method of placing them in and removing them from the desired flux region.

The method developed for the production of F-18 was encapsulating the Li-6 carbonate powder in thin quartz tubing, placing one to four of these tubes in a plastic holder, and lowering and raising the holder with nylon cord in the water filled radiation facility into the maximum flux area of the reactor. Characteristics of the quartz tubing are 3 mm inside diameter and 3-1/2 inch length. The plastic holder has spacer holes to maintain tube separation, holes for water circulation, lead sinker, and quartz wool for cushioning.

The results of the project far exceeded expectations. Each tube produces 34 mCi of F-18 at the end of two hours irradiation. A total of 136 mCi F-18 can be produced at any time upon two hours notice without disrupting reactor operations or interferring with other experiments or experimenters. The non-reuseable target and target material cost about $4.00 for one irradiation and a 34 mCi yield or about $13.00 for a 136 mCi F-18 yield.

The current status of the Army Fluorine-18 production project is that twice weekly F-18 is produced for a joint radiopharmacy operation of the Walter Reed General Hospital, National Institutes of Health and National Naval Medical Center. F-18 is used by these institutions for their patients and patients from other governmental hospitals.

The F-18 is extracted as sodium fluoride solution using a special separation process on equipment installed at the NBSR facility. The F-18 solution is provided then in a form suitable for oral administration.

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Studies of the Effects of Parenteral Copper Chelates on Bone Healing

G. C. Battistone

U. S. Army Institute of Dental Research, Walter Reed Army Hospital, Washington, D.C.

During the past year experiments have continued on the effect of injected copper chelates on bone healing in experimentally injured guinea pig bone. Neutron activation analysis was used to analyze for the trace metals. Doses of copper salts as low as 50 µg Cu/day given to experimental animals in previous experiments have demonstrated a high toxicity. In chelate form the copper is better tolerated but remains toxic. The daily intraperitoneal injection of 250 µg Cu/day as copper nitrilotriacetic acid has resulted in a noticeable acceleration of bone healing in the guinea pigs. However, copper accumulation was noted particularly in the liver and although animal deaths were not recorded the experimental animals did not show normal weight gains. Other copper chelates administered at doses containing 100 µg Cu/day did not produce toxic effects in the experimental animals but bone healing acceleration was either minimal or absent. Since it now appears that injected copper chelates can accelerate bone healing, and that copper toxicity can be diminished by administration in the chelate form, studies are being directed toward the possibility of finding the appropriate copper dose level and chelate which will produce optimal healing acceleration without toxic effects.

Activation Analysis Program of the Food and Drug Administration at the NBSR

J. T. Tanner, M. H. Friedman, M. Jaffee, L. A. Ford, and D. N. Lincoln
Food and Drug Administration, Washington, D.C.

1. Determination of Mercury in Common Foods and Total Diets

Data accumulated by the Food and Drug Administration and other government agencies have demonstrated that fish is one commodity which introduces mercury into the diet. Fish, however, only represents a small portion of the normal diet of the American public. It is necessary therefore, to determine if there are other foods besides fish that contribute significant amounts of mercury to the diet. Food samples of flour, sugar, non-fat dry milk, potatoes, hamburger, chicken breast, shrimp, lever, eggs, and whole milk were analyzed for mercury by neutron activation analysis. A modification of the Jervis procedure which involves separation of the mercury by anion exchanger chromatography and sulfide precipitation was used. The results of this study show that the common foods are well below 50 ppb Hg as used by the World Health Organization. In all foods except shrimp the mercury was found to be below 10 ppb.

2. Mercury Distribution in the Central Nervous System of Beagles

Mercury was determined by non-destructive neutron activation analysis in the central nervous system of Beagle dogs which had been fed a diet of MeHgCl. The data were collected by a low energy photon detector connected to a 1024 channel pulse height analyzer and then reduced by a computer program. Results of the study point out that a) mercury in the dog's brain is not distributed uniformly, b) from the scant data available we infer that the mercury in the occipital cortex does not decline as was the case with the other structures, and c) it appears that neutron activation analysis has the capability to determine mercury levels even in the smallest discernible samples dissected from the central nervous system of the dog.

3. Determination of Bromine in Pigs

This is a continuing program in support of FDA research of biological effects of brominated vegetable oil. Tissues from pigs dosed with brominated vegetable oil containing materials are being analyzed by non-destructive NAA.

4. Drug Analysis and Radioisotope Tracer Production

In addition to these projects several other problems were investigated. Thse include a) the determination of Na, I, Cl, Br in Ampicillian tablets from foreign manufacturers,

b) the determination of Hg in the hair and blood of a victim of mercury poisoning, c) the irradiation of nuts to use as a tracer to simulate mycrotoxin and d) the determination of Al in some drugs for regulatory purposes.

5. Analysis for Mercury in Fish, Flour and Standard Reference Orchard Leaves by Electrodeposition Radiochemistry

The use of electrolysis for the radiochemical separation of mercury-203 from other interfering radionuclides in neutron activated food samples yields mercury of high purity deposited on gold. However, modifications of published electrodeposition procedures were necessary before acceptable results could be obtained by this method.

Some of these modifications included shielding the thermometer against thermal shock, insulation of the reflux apparatus for uniform heating, and the use of hydrazine sulfate to remove chlorine interference.

Although the high variability of yield of the mercury carrier indicates the need for further modifications, the Hg-203 results were acceptable for a satisfactory comparison with data obtained by different methods. The data acquired from the test samples by electro-deposition of mercury were in good agreement with the data obtained by other investigators who used sulfide precipitation and gas chromatographic techniques.

Scandium Distribution in a Terrestrial Rock

John Barker and Hiroshi Nagasawa
Chemistry Department, University of Maryland

The bulk chemical composition of an igneous rock hides much of the history of formation· of the individual crystalline minerals in that rock. Silicate rocks, formed by cooling of melts, have similar minerals on Earth, the moon and in meteorites. A thorough study of any one of them should help in the understanding of all of them. Each and every element can provide unique information about the chemical evolution of the rock. Past results on the geochemistry of scandium have failed to notice the high preference of Sc for Mg and Fe containing minerals, relative to the residual liquid which is present in the rocks as a glassy matrix. This preference suggests that Sc behaves like some rare earth elements. The

following results were obtained by instrumental neutron activation analysis, INAA, for minerals from a dacite rock from a volcano in Japan:

Mineral Phase	Mineral Chemical Formula	Sc (μg/g)	Distribution Ratio (Mineral / Matrix)
Hypersthene	$(Mg,Fe)SiO_3$	39.0	15.6
Hornblende	$NaCa_2(Mg,Fe,Al)_5(Si,Al)_8O_{22}(OH)_2$	187.2	74.9
Matrix	glass	2.50	1.00

Distribution of Elements in Meteoritic Minerals

John Barker, David Nava, Philip Cressy and Patricia Buhl
Chemistry Department, University of Maryland

Minerals separated from a powdered sample of Bruderheim meteorite were analyzed for iron, chromium, and cobalt by instrumental neutron activation analysis, INAA. The National Bureau of Standards reactor was used, along with counting facilities at the University of Maryland. Preliminary numbers are given in the table below, along with a comparison to the literature which indicates that most of these elements have been accounted for with the phases that were analyzed. Additional work on this and other elements in more purified phases should yield information on the origin and evolution of meteorites. Work is in progress on the separation of minerals from the Allende meteorite, which is believed to be representative of the material from which the solar system originally condensed.

Phase	Formula	Modal Analysis	Fe	Cr	Co
			(microgram element per gram of phase)		
Pyroxene	$(Mg,Fe)SiO_3$	42.26%	113,300	1,350	13.7
Olivine	$(Mg,Fe)_2SiO_4$	40.67%	175,000	1,330	16.4
Metal	Fe,Ni	7.60%	783,000	–	5,610
Troilite	FeS	6.71%	587,000	26,400	33.1
+ Chromite	$FeOCr_2O_3$	0.85%			
Other		1.92%			
Totals (INAA for above phases)			222,000	3,100	430
Totals (Bulk from literature)			223,000	3,600	500

Trace Elements in the Environment

G. E. Gordon, W. H. Zoller, and W. B. Walters
University of Maryland, College Park, Maryland

Our major use of the NBS reactor to date has been the irradiation of atmospheric particulate material collected on various filter material. Using instrumental neutron activation analysis we routinely determine concentrations of approximately 20 elements by the use of Ge(Li) γ-ray detectors. An extensive study of the particulate material in the atmosphere of Boston has nearly been completed with the analysis of approximately 200 samples. High concentrations of vanadium (up to several $\mu g/m^3$) from the combustion of residual fuel have been observed. In addition, studies of the chemical composition with respect to size of the particles has indicated that some trace elements such as V, Sb, Zn and Br are predominantly attached to small particles where as others, such as Fe, Na and Cl, are on large particles. These studies group many of the elements into categories which may be indicative of the source of the various particulates.

Initial work on source materials such as residual fuels has shown that very high concentration of many elements are present and are presumably released into the atmosphere upon combustion. An example of a trace metal introduced into the atmosphere in high concentration is V, which has concentrations up to 800 ppm in residual fuel oil samples. We are presently extending these fuel analysis projects to distilled fuels such as kerosene, fuel oil and various lubricating oils.

We are presently performing the analysis of a group of atmospheric particulate samples from Antarctica. These samples are difficult to analyze due to the very low concentrations involved. We are detecting only small concentrations of various elements above the filter-material blanks. This project will be continued over the next three years in order to measure the concentration of the trace elements present in this very clean "background" area.

Several groups of samples from New Zealand have been analyzed to determine the contribution from the largest cities to the circulation of pollutants in the Southern Hemisphere. A set of samples from four sites will eventually be analyzed for several trace elements over a period of five years. The results of these analyses should indicate any changes in the chemical composition of the particulate material since implementation of anti-pollution laws in Auckland and Christchurch, New Zealand in the past few years.

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A project on the analysis of water samples for trace elements has been initiated. Samples can be analyzed directly for several elements that are present in high concentrations. Various pre-irradiation concentration steps are being tested in an attempt to increase the analytical capabilities of neutron activation analysis as applied to water samples. Some post-irradiation chemistry such as solvent extraction or sodium removal are also being tested in order to improve our ability to analyze for certain elements such as V and As. Samples have been collected from the Pautuxant and Potomac Rivers for the program. We will be studying the distribution of trace metals (transition metals) between the dissolved and particulate phase in a natural water system.

Work on new and better standards using lactose and cellulose pellets doped with various trace elements has produced encouraging results to date. Reproductability of some elements appears to be well within 1 to 2% on several standard pellets. This work is done in cooperation with Dr. Harry Rook of the Activation Analysis Section of NBS.

A number of isotopes have been produced for radioactive decay studies and for tracer studies of chemical reactions. Thirty-hour Te-131m has been produced by the ^{130}Te(n,γ) ^{131}Te reaction in order to study the decay of Te-131m and the levels of I-131. Preliminary studies have also been made on 25-min Se-83, 42-sec and 4.36 min Rh-104 isomers, and 4.44 hr Ru-105. Radioactive Se and Te nuclides have been prepared for use as tracers in studying separations of Se and Te in environmental pollutants. A number of radioactive isotopes have also been prepared for use as standards in calculating the energy response and efficiency of Ge(Li) γ-ray detectors.

The Use of Neutron Activation Analysis in Scientific Crime Detection

Forensic Staff, M. J. Pro, Chief
U. S. Treasury Department, Washington, D. C.

The value of NAA for examining physical evidence such as paints and soils is well documented. The findings obtained by NAA have been introduced as court evidence many times and its use has been upheld by Appellate Courts when tested by appeal.

The U. S. Treasury Department utilizes NAA extensively for the examination of a wide variety of physical evidence and the activation of specimens at the NBS Reactor is almost a daily event. In the past year 1850 samples from 300 criminal cases were processed by this method. The types of samples are quite diverse ranging from cotton swabs bearing traces of gunshot residue to heroin confiscated at a U. S. port of entry. While the NBS Reactor was undergoing repair this summer, the Treasury Laboratory used the Western New York Research Center Reactor in Buffalo, New York to examine 1500 gunshot residue samples in 190 criminal cases. This type of examination is requested by police departments across the country who have learned the value of NAA for detecting the presence of gunshot residue in cases involving the use of firearms.

The potential use of NAA for examining such unusual materials as coffee beans is being explored by the Treasury Laboratory in connection with U. S. Customs Bureau investigations. Beans from more than a dozen locations throughout the world are being examined to determine if meaningful differences exist in their trace elemental compositions. These data when correlated with other information may prove useful in establishing the country of origin of the coffee beans which is relevant to the import duty imposed on this commodity.

Since October, 1970 the cases in which the NBSR facility was used by the Treasury Laboratory are summarized below:

Type of Case	Number of Cases
Crimes of Violence (Gunshot)	225
Production of Illicit Spirits	40
Destructive Devices	20
Drug Abuse	5

The Treasury Laboratory's present NAA capabilities are being expanded by the aquisition of a new 60 cc Ge(Li) detector coupled to a Data General mini computer. It is anticipated

that once the system is operational a significant increase in the number of samples processed by NAA will be possible.

Federal Bureau of Investigation Activation Analysis Program

J. Kilty, J. Riley, G. Mahoney
Federal Bureau of Investigation, Washington, D.C.

The FBI Laboratory is conducting trace elemental analysis of copper wire in order to determine the homogeneity of the trace elements along the length of wire and to measure the variations in concentrations of the trace elements in different wires of the same gauge. The elements usually detected in copper wires are selenium, gold, silver and antimony. Due to the high cost of copper, thefts are a common occurrence and neutron activation analysis offers a method for the possible association of copper from the scene of a theft with copper recovered from the suspect or a salvage yard.

Many other types of evidence are received for analysis in the FBI Laboratory. Metals such as lead, steel and aluminum are analyzed on a routine basis for both the FBI's cases and for local law enforcement agencies. Examples of samples which are run on a less routine basis are biological samples which are analyzed for arsenic or other elements common in poisons, plastic-type materials and paint.

Paraffin and cotton used to remove gunpowder residues from the hands of a person suspected of having discharged a firearm are analyzed by NAA for the elements antimony and barium, which are components of most primer mixtures. This work has been conducted for the past four years and has formed the research upon which analyses are based today.

In April, 1970, investigators in a southern city were confronted with two slain individuals who were either the victim of a double murder or a murder – suicide. Analyses of the casts taken from the two slain individuals and the suspect's hand indicated that one of the slain persons had discharged a firearm. After all the facts were considered, the investigators ruled a murder – suicide had taken place and the suspect was released.

U. S. Postal Service Activation Analysis Program

Gordon Stangor, James Upton, and Michael Beckman
Crime Laboratory, U. S. Postal Service, Washington, D.C.

During the past year, the U. S. Postal Service Crime Laboratory continued using the NBS nuclear reactor facility for forensic applications. Types of evidence items irradiated at NBS and regularly analyzed by the neutron activation technique include glass, paint, hair, metals, tape, paper, and safe insulation. Much of this evidence is derived from investigations relating to post office burglaries and bomb mailings.

Presently, research is underway to make determinations as to the extent that compositional trace elements vary in safe insulation produced by a single manufacturer. It is hoped this will provide the basis for identification of a particular safe within a given manufacturer's production.

In an important decision relating to the results of neutron activation analysis, the Supreme Court on March 29, 1971, denied a petition for certiorari of an Appeals Court affirmation of the conviction of the mailer of a bomb parcel which had exploded and killed the addressee. Review of the case was petitioned on grounds that the process of neutron activation employed in making comparisons of certain bomb components (paper, metal, and plastic tape) was relatively new and had little legal precedent. The Appeals Court in upholding the admission of expert testimony concerning this method of analysis stated ".....neutron activation analysis has gained 'general acceptance in the particular field in which it belongs.'" and that it ".....meets the test of admissibility....."

Development of Analytical Methods and Determination of Constituents
of Geological Samples

J. J. Rowe
U. S. Geological Survey, Washington, D. C.

The Radioactivation and Radiochemistry projects of the U. S. Geological Survey has been
using the NBS reactor facilities for irradiation of rocks and minerals for the development
of analytical methods and to provide analytical services to geologists.

Some of the services and studies made during the year are:

1. Analysis for Gold in Rock Samples - J. J. Rowe

Rock samples have been analyzed for gold in studies of background levels and magnetic
fractionation. One gram samples were irradiated for one hour in RT-3, along with standards.
Irradiated samples are cooled for ten days to permit decay of short lived activity. Samples
are then processed, with carrier gold, using fire assay to obtain a gold bead which is
counted on NaI(Tl) detectors. The 0.413 meV peak of Au-198 is used for the determination.
This method permits the determination of gold at concentrations as low as 0.1 ppb. A
publication summarizing the data on more than 700 samples is in press (Gottfried, Rowe and
Tilling, "Distribution of gold in igneous rocks", U.S.G.S. Prof. Paper 727).

2. Gold as a Tracer in Thermal Waters - Rowe and Schwarz

Techniques have been studied for the concentration and collection of gold in thermal
waters using Au-195 as a tracer to monitor the efficiency of the ion exchange collection.
Resins are irradiated for 10 minutes, cooled one day, and processed by fire assay to destroy
the resin and collect Au-195 and Au-198 in a gold bead. The Au-195 is used to determine yield
from original sample, through ion exchanger concentration, irradiation and radiochemistry.
Concentrations of gold in thermal waters vary from 0.005 ppb to as high as 0.1 ppb. The
technique is being developed for use in field collections to avoid problems of gold plating
out on walls of containers. A similar procedure is being developed for collection and
concentration of mercury.

3. Trace Metals in Galena, Sphalerite, Pyrrhotite and Pyrite - Simon and Rowe

Samples of galena, sphalerite, pyrrhotite and pyrite were analyzed for Cu,Zn,Sb,Au,Ag
Mn,Cd, and Se using instrumental, coincidence and radiochemical methods. The results have
been published in Economic Geology v. 66, p. 602 (1971) "Fractionation of minor elements

between galena, sphalerite, Darwin Lead-Silver-Zinc Mine, Inyo County, California and its significance in geothermometry" by Hall, Rowe and Simon.

4. Analysis for La,Sc,Cr,Th, and Au - J. J. Rowe

Various samples submitted by geologists have been analyzed for La,Sc,Cr,Th, and gold by radiochemical and instrumental methods.

5. Determination of Platinum and Palladium in Geological Materials - J. J. Rowe

Samples from various localities were analyzed for platinum and palladium using fire assay preconcentration and radiochemistry. Ten to twenty gram samples are processed by fire assay to concentrate the platinum and palladium into a 3 mg silver bead. The silver beads are irradiated for two hours, and then platinum and palladium are radiochemically separated for beta counting following the procedure by Rowe and Simon, "Determination of platinum and palladium in geological materials by neutron activation analysis after fire assay preconcentration", Talanta, v. 18, p. 121-125 (1971). Concentrations of platinum as low as 3 ppb and palladium as low as 1.5 ppb were found in samples from the continental shelf. A study of platinum and palladium in various igneous rock provinces is being made.

6. Determination of Tantalam, Antimony, and Hafnium in Lunar Samples - Greenland

Lunar samples have been irradiated for the determination of tantalum, antimony and hafnium.

7. Background Levels of Arsenic in Rocks and Soils - J. J. Rowe

A radiochemical method is being tested for the determination of arsenic, using As-77 for the yield determination. A comprehensive study is being made of background arsenic levels in rocks and soils as part of the study of environmental pollution.

Study of Radiation Effects on the Transmission Characteristics

of Sapphire and Quartz Windows

R. E. Patrick
(U.S. Arms Control and Disarmament Agency, Washington, D.C.)

O. E. Frizzell and J. H. Ring

The purpose of this program was to measure the degradation in the light transmission characteristics of sapphire and quartz windows that had been exposed to a high neutron/gamma field. The general trends observed during the irradiating period were given primary consideration and little effort was made to insure high precision in the measurements. The general philosophy was based on the goal of the program which was to provide engineering support to the efforts to develop a unique identification system for arms control and international material safeguards application. The time frame under which this program operated prevented the experimenters from developing the sophistication and precision which one would normally strive to achieve.

The experimental procedure involved a cycle of events which include a specified irradiation period, cooling and measurement of the light transmission characteristics. The NBSR rabbit system was utilized in the irradiating period. This cycle was repeated until the desired total flux of $5.0 \times 10^{19} n/cm^2$ was attained. The characteristics of the flux field were approximately a thermal neutron flux of $5 \times 10^{13} n/cm^2$-sec and high energy (E>/Mev) gamma flux of about $6 \times 10^{13} \gamma/cm^2$-sec

The procedure used in measuring the light transmission characteristics involved the use of a beam splitter window and a red lens window. The red lens window was used to determine the degradation of transmitting red light through each window and the beam splitter was used as a standard to compare to. Figure 1 illustrates the experimental set-up.

In configuration B light passes through the beam splitter where part of it is absorbed. The remaining light then travels through the red lens and is measured by the light meter. In configuration A the light passes through either the sapphire or quartz window then through the red lens and is measured by the light meter. Readings without the red lens mounted in the configuration were also taken. There was also 3 different settings of the intensity of the light source.

The results of configuration A, with and without the red lens, could then be compared to the results of configuration B. The value of the measurement in configuration B should always remain constant thereby offering a benchmark to calibrate the experiment. The following table summarizes the results of this experiment.

PERCENT OF SOURCE LIGHT TRANSMITTED

Irradiation Level (n/cm^2)	Light Intensity	Saph	Saph/Red	Quartz	Quartz/Red	Beam Splitter	BS/Red
0	High	100	23.4	100	22.2	33.1	7.8
	Med	100	19.0	100	23.1	32.4	8.8
	Low	100	19.2	100	20.7	30.4	3.6
$7.2x10^{17}$	High	90.1	23.4	100	22.2	33.1	7.8
	Med	79.4	19.0	100	23.1	32.4	8.0
	Low	73.3	19.2	100	20.7	30.4	3.4
$1.44x10^{17}$	High	83.6	23.9	100	25.4	33.4	7.6
	Med	83.8	22.6	100	25.4	32.4	7.3
	Low	84.2	23.5	100	26.1	30.4	7.3
$5.0x10^{18}$	High	82.9	23.0	100	25.8	33.5	9.8
	Med	81.0	22.2	100	25.6	31.4	7.1
	Low	81.9	21.5	100	26.1	31.1	7.3
$3.5x10^{19}$	High	62.6	18.1	98.5	25.2	33.4	7.4
	Med	61.5	17.8	98.4	24.8	31.6	7.1
	Low	61.7	18.5	99.8	26.1	29.8	7.2
$5.0x10^{19}$	High	61.0	18.2	100	26.1	34.9	7.8
	Med	58.3	17.1	100	26.2	31.9	6.9
	Low	56.5	17.7	99.8	25.8	29.2	6.9

In addition to the above measuremsnts a more precise light transmission curve was made at the beginning, end and one intermediate point in the irradiation cycle. The following curves, Figs. 2 and 3, illustrate the results of these measurements.

Based on the results of this experiment the following conclusions were reached.

1. Sapphire Window

   a. There was a general degradation (30-40%) of the light transmission characteristics for "white light.

   b. The radiation effects on the transmission of light in the red region were (∿5%) when compared to the beam splitter.

   c. There was considerable degradation (70-80%) of light transmission characteristics in the blue region.

2. Quartz Window

   a. There was only limited degradation for "white" light.

   b. There was no predominant frequency (red or blue) in which this limited degradation occurred.

The results of this program were submitted to Sperry, an ACDA contractor, and will now be incorporated into the research and development program.
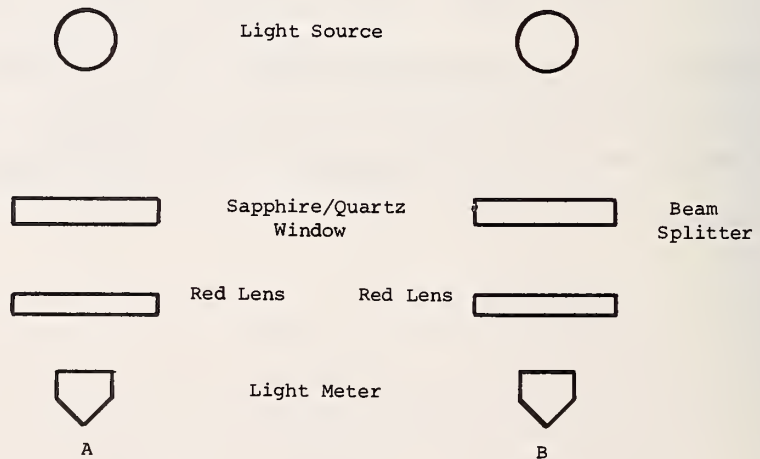

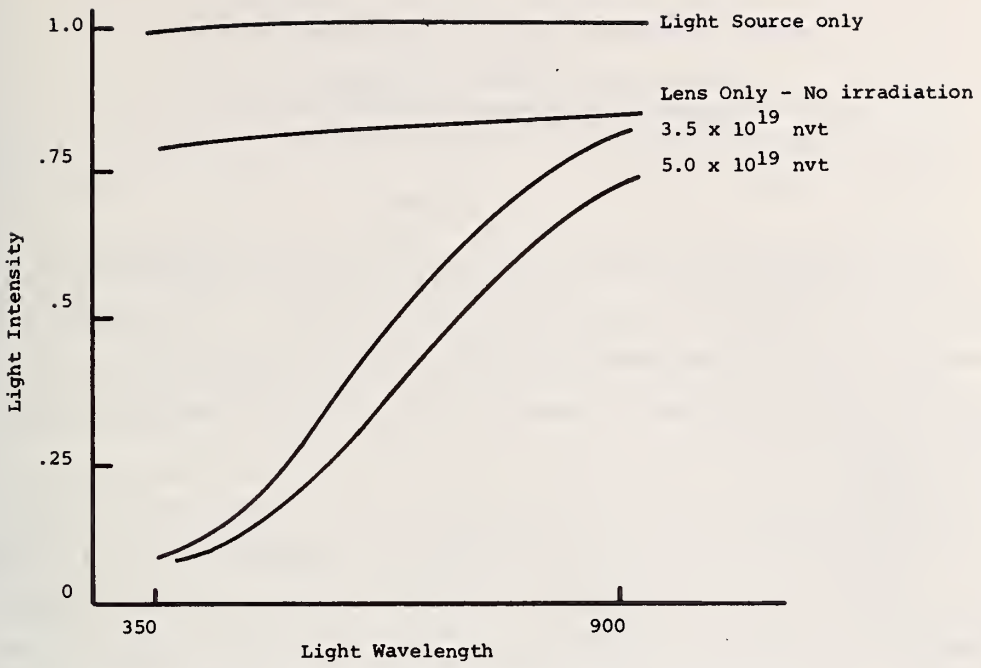Fig. 1. Experimental Configuration

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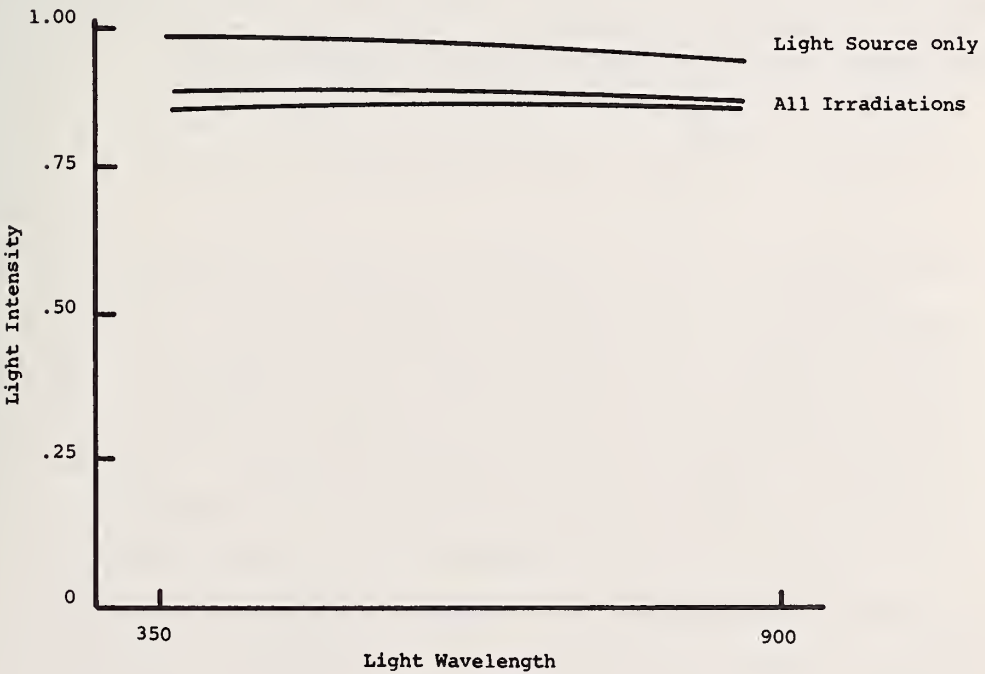Fig. 2. Sapphire Window Light Transmission Characteristics

Fig. 3. Quartz Window Light Transmission Characteristics

Feasibility Investigations of a Fuel Bundle Motion Monitor

J. P. Boright, R. E. Patrick
(U.S. Arms Control and Disarmament Agency, Washington, D.C.)

and

O. E. Frizzel

A joint Canadian-American group is developing un-attended instrumentation for IAEA safeguards (as required by the nuclear Non-Proliferation Treaty). One component under development is a pair of collimated ion chambers for monitoring irradiated reactor fuel. The difference signal from the two chambers can yield directional information on fuel bundle movement.

Some aspects of the proposed bundle monitor have been investigated using the spent fuel in the bay at the NBS reactor. The uniformity of the chambers, and their performance in high fields was measured. Attenuation and spatial smearing of the observed signals was investigated as a function of distance from the fuel, and of material between the fuel and the monitor. Also, effects of background signals in various geometrical configurations were investigated. With the results, qualitative estimates can be made of the signals expected in given monitoring siturations. These monitors are now being tested at a nuclear reactor and are being prepared for use at a reprocessing plant.

Investigation into Neutron Damage of PbS Films

R. M. Cullpepper
(Naval Ordnance Laboratory, White Oak, Maryland)

NOL has undertaken an investigation into the neutron induced damage in PbS semi-conducting films. The electrical properties of both n and p type films (the mobility, resistivity and the number of free carriers) have been measured from 4.5°K to 800°K at NOL before and after each of as many as five irradiations. The samples were irradiated in 100 minute steps in the RT-2 facility of the NBS reactor operating at 2Mw. The data obtained from these measurements was then used to determine free carrier removal rates, scattering mechanisms, and radiation induced energy lying near the band edges. The samples used in this experiment were grown in triplets on an NaCl substrate at NOL. Two of each set of three were then transfered to a plastic backing and the NaCl substrate disolved off, while the third remained on the NaCl. The effect of this transfer on the electrical properties was not measured, but the variation in electrical properties across the triplet samples after transfer was no greater than the variation in triplets still on an NaCl substrate. All three samples of each set were exposed to similar environments except one of the plastic backed films was subjected to irradiation. When the irradiated sample was measured after each irradiation, each of the other two was also measured. This was done because no data existed describing aging effects of such films, either on the NaCl substrate or on the plastic backing. It was discovered that aging lowers the carrier concentration of both types of unirradiated films, but not enough to be significant in the experiment.

The results indicate that the carrier concentration of both n and p type samples is lowered by irradiation. The carrier removal rate is approximately 4 free carriers/fast neutron per cm^2. There appears to be no irradiation induced energy levels within .02ev of the band edges. The mobility data indicates that the irradiation introduces no new scattering mechanisms. The use of the Mansfield-Dingle mobility equation to compute the number of defects introduced into the irradiated films leads to results that vary widely from sample to sample, and from the result predicted on the basis of various cascade displacement models. It is felt that the cause for such disagreement might be that the irradiated material is a new form of PbS, containing both interstitials and vacancies of Pb and S, whereas the as grown PbS has only vacancies of both elements. It is further hypothesized that these interstitials form some sort of amphoteric trap for the free

carriers. The nature of this trap is not known; it could be a system of donor and acceptor levels near the center of the band as has been shown to be the case in silicon, or a single level which may capture either a hole or electron, as does the level introduced by gold in germanium, or some even more complicated scheme not apparent from the data at hand.

Neutron Irradiation of InSb

John Stannard
(Naval Research Laboratory, Washington, D.C.)

The Naval Research Laboratory is studying the effects of band tailing in heavily doped indium-antimonide. Of particular interest is material doped with very nearly equal concentrations of donor and acceptor impurities. Transmutation of In-115 into the donor Sn-116 allows such closely compensated material to be produced when initially P-type material is irradiated with thermal neutrons. To date experiments have been performed to show the production rate of donors in the RT-4 facility. In addition a series of samples has been irradiated so as to become closely compensated. These samples are now being analyzed.

Neutron Activation Analysis of Silicon for MOS Devices

J. Repace and H. L. Hughes
(U.S. Naval Research Laboratory, Washington, D.C.)

As part of a continuing program in the Reliability Physics of MOS devices, experiments in our laboratory have established that the presence of sodium impurities in silicon devices substrates plays a crucial role in enhancing the failure of these devices upon exposure to ionizing radiation. We have therefore undertaken to measure the sodium impurity levels in a variety of silicon substrates, using neutron activation analysis. The presence of sodium is detected via the 1368.562 keV and 2753.92 keV gamma ray lines of Na-24 produced in the ^{23}Na (n,γ) ^{24}Na reaction; the gamma rays are detected by an 80 cc Ge(Li) detector with a 4096 channel pulse-height analyzer, and the data, on magnetic or paper tape, are processed by computer. Sodium levels of the order of 10^{15} atoms per cm^3 in unprocessed device wafers and of the order of 10^{13} atoms per cm^2 in processed wafers have been found. Further experiments to determine the sources and distribution of the sodium impurity are planned.

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Chemical Composition of Particulates Collected in the Marine Atmosphere

P. E. Wilkniss
(Naval Research Laboratory, Washington, D.C.)

Pollution of the marine atmosphere is steadily increasing, as indicated by increased lead concentrations in polar ice samples and ocean surface waters, by the observance of pesticides transported with wind-blown dust over the Atlantic and Indian Oceans and by a general increase in ambient aerosol concentrations probably of man-made origin. Pollution assessment in the marine atmosphere is very difficult for various reasons and the analytical problems encountered require use of the most sensitive analytical techniques known to date. We have investigated the use of neutron activation, to determine the elements Na, S, Cl, K, Ca, Mn, Br, Sr and I in particulate samples from the marine atmosphere.

Activation analysis of the aerosol samples was shown to be an excellent method for determining the listed elements. The implication for pollution studies is that, by determining elemental ratios such as F/Cl, Na/Cl, Na/K, Ca/S and Mn/Cl, one can determine whether an aerosol is of natural origin (the ocean, or volcanic activity) or of man-made origin.

Studies of Magnetic Properties in Rare Earth Alloys at Cryogenic Temperatures

R. Segnan and D. M. Sweger
(American University, Washington, D.C.)

A Mössbauer effect source of Dy-161 has been prepared in a matrix of Gd_2O_3 for use in magnetic rare earth alloys studies of cryogenic temperatures. A high velocity drive (±25 cm/sec) has been assembled for use in measuring resonant absorption in Dy-161.

Magnetic rare earth alloys offer promise of possible use as materials suitable for high energy density electromagnetic applications. Particularly promising are Dy-Gd and Dy-Y alloys, since these tend to have quite attractive magnetic properties (for example, dysprosium has a saturation magnetization of approximately 34 kilogauss at 4.2°K).

The alloy system Gd-Dy can be investigated by Mössbauer spectroscopy by using a suitable radioisotope of either Gd or Dy. An analysis of the properties and availability of possible radioactive sources led to the selection of a Dy-161 isotope. The γ-ray used in this case comes from the decay of the 25.6 KeV excited state to the ground state in Dy-161. The

excited state of Dy-161 can be populated by thermal neutron irradiation of Gd-160.

Gadolinium oxide, Gd_2O_3, enriched to 96% Gd-160 in the form of a fine powder is the source material. The source is activated in the RT-4 pneumatic facility of the National Bureau of Standards Reactor in a thermal neutron flux of 1.3 x 10^{13} neutrons/cm^2/sec. Such a flux will yield a source strength of about 3 millicuries/hr/gram of enriched Gd_2O_3.

The splitting of the saturation hyperfine field in Dy-161 is 43.7 cm/sec and thus the drive must be capable of peak velocities of about ±25 cm/sec. Velocities such as these are at least an order of magnitude larger than are required for most other Mössbauer isotopes. In addition, as for all drive systems, the velocity must be linear to within 1%. A modified version of the NBS Mössbauer drive was used to achieve the high velocities required for the Dy-161 resonance.

In order to test the Mössbauer source, absorbtion spectra were taken with the low velocity drive. Absorption vs. velocity curves were obtained for Gd_2O_3 and Dy_2O_3 against a Dy_2O_3 absorber. The full-width at half maximum of the Mössbauer absorption peaks obtained were 1.8 cm/sec and 2 cm/sec respectively. As one can see the linewidths are comparable. The relative resonant absorption is respectively 5% and 4%. All the measurements were taken at room temperature. Presently, measurements are being performed on Dy-Gd alloys at room temperature and liquid nitrogen temperature. Preliminary results on these alloys will be available in the near future.

STAFF ROSTER

A. DIVISION ORGANIZATION CHART

314.00

REACTOR RADIATION DIVISION

R. S. Carter, Chief
T. M. Raby, Deputy Chief

E. Maxwell, Admin. Officer
E. Simms, Receptionist

Technical Support

R. Casella
M. Ganoczy
V. Myers
F. Shorten

314.01

Reactor Operations
T. M. Raby, Chief
J. F. Torrence, Deputy

D. Ahalt, Sec'y.
J. Bowers, Admin. &
   Tech. Ass't.

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M. Bell
N. Bickford
H. Brake
D. Cea
A. Chapman
W. Crebs
H. Dilks
J. Ring
R. Scheide
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L. Clutter, Sec'y.

A. Cinquepalma
L. deGraaf*
D. Fravel
R. Livingston*
E. Prince
J. Rush
A. Santoro
A. Tudgay
W. Rymes

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*Temporary, Part-time

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B. Non-RRD NBS Staff Located at Reactor

Division 310.08

D. A. Becker
B. S. Carpenter
T. Gills
M. L. Kehrer
D. Ishii
W. D. Kinard
P. D. LaFleur
G. J. Lutz
L. T. McClendon
J. Olin (Smithsonian Inst.)
H. L. Rook
R. W. Shideler
J. E. Suddueth
B. A. Thompson
H. P. Yule

Division 313.07

B. Mozer

Division 240.01

A. L. Ahalt
P. R. Cassidy
H. E. DeSpain
E. J. Embree
J. J. Shubiak

Division 242.01

R. A. Dallatore
J. A. Grundl
I. A. Schroder

C. Guest Workers and Collaborators

Division 221.03

B. Dove
H. Marshak
R. J. Soulen, Jr.

Division 221.05

R. D. Mountain
H. J. Raveche

Division 232.06

R. D. Deslattes
W. C. Sauder

Division 242.02

L. D. Miller
F. J. Schima

Division 311.03

J. D. Barnes

Division 311.05

B. Dickens
L. W. Schroeder
R. M. Waterstrat

Division 313.01

C. W. Reimann

Division 313.06

A. D. Mighell

Argonne National Laboratory

H. Flotow
D. C. Price
J. M. Rowe

Department of Army Civilians

W. Crofford

Federal Bureau of Investigation

J. W. Kilty
J. M. Little
G. P. Mahoney
J. P. Riley

Food and Drug Administration

M. Friedman*
M. W. Heitzman
M. Jaffee
D. N. Lincoln
R. E. Simpson
J. T. Tanner*

Harry Diamond Laboratory

C. A. Morrison
D. B. Wortman

Northern Virginia Pathology Lab., Inc.

C. B. Cook

V. A. Hospital

B. J. L. Sauerbrunn

*Located full-time at NBS Reactor

Naval Ordnance Laboratory

H. Alperin*
R. M. Cullpepper
S. J. Pickart*
J. J. Rhyne*
R. H. Williams*
R. Youshaw

Naval Research Laboratory

G. A. Ferguson
H. L. Hughes
J. Karle
L. F. Paolella*
J. Repace
J. Stannard
P. E. Wilkniss

Picatinny Arsenal

C. S. Choi*
K. W. Logan
R. Munroe*
H. J. Prask*
R. S. Singh*
S. F. Trevino*

U. S. Arms Control Disarmament Agency

J. D. Boright
R. E. Patrick

U. S. Army Inst. of Dental Research

G. C. Battistone

U. S. Geological Survey

F. W. Brown
R. Finkelman
L. P. Greenland
J. J. Rowe
G. Sellers
F. O. Simon

U. S. Treasury Department

P. C. Buscemi
C. M. Hoffman
F. A. Lundgren
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