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Research Materials Developed Under the NBS Inorganic Materials Program

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Research Materials Developed Under the NBS Inorganic Materials Program

Edited by
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U. S. National Bureau of Standards. Special Publication 333.
Nat. Bur. Stand. (U.S.), Spec. Publ. 333, 71 pages (September 1970)
CODEN: XNBSA

Issued September 1970

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
(Order by SD Catalog No. C 13.10:333), Price 70 cents.

Library of Congress Catalog Card Number: 70-608227

Contents

Research Materials Developed Under the NBS Inorganic Materials Program

F. E. Brinckman and J. B. Wachtman, Jr.

The National Bureau of Standards develops many specialized materials in the process of carrying out research supporting its measurements, standards, and service activities. These materials include gases, liquids, glasses, single crystals, polycrystals, and various multiphase materials; their compositions (including trace elements in some cases) and physical characteristics are tailored to specific research needs, but the materials are often of use for other research purposes. Production is usually limited to immediate internal needs and samples are not generally available for distribution, but knowledge of production techniques and materials characteristics which may be helpful to other scientists is available. The present listing of research materials developed in the Inorganic Materials Division accordingly gives names of scientific staff members who may be contacted for this type of information as well as giving a brief summary of the nature, method of preparation and properties determined for each material.

Key words: Chemical properties; composition; gases; glasses; liquids; multiphase materials; physical properties; polycrystals; preparation; research.

1. Introduction

Improved materials are critically needed in many areas of technology. Attempts to provide such superior materials are frequently empirical and sometimes wasteful of time and funds [1].[1] An empirical component will probably always be present in any materials development program, but the establishment of a science of materials that increasingly affords predictable and reliable results in devising new materials for specific tasks is most desirable. Not only will this increase the efficiency of materials development programs, but also can ultimately lead to development of completely new materials or new applications. Much thought has been given to opportunities arising from recent developments in materials science [2] and to roadblocks preventing engineering achievement of scientifically feasible materials including the areas of characterization [1] and processing [3]. Advances in materials science assist materials development on the one hand, but depend upon development of materials suitable for research on the other. This interdependence of materials development and materials science is widely recognized and new programs on the properties of materials increasingly have as a major component a research material development subprogram. The experimental and theoretical competence needed to produce and characterize specimens is frequently quite different from that needed for the subsequent property study so that the decision to undertake a property study on a new material frequently means that a large investment of time and resources, perhaps comparable to the property measuring effort itself, must be made before the property study can begin. In these circumstances the availability of a few trial specimens, even if not of completely satisfactory quality for the final study, can be very valuable in permitting a test of the experimental feasibility of the contemplated measurements and perhaps in providing guidance on how closely the character of the specimen must be controlled for the final study. When trial specimens are not available, knowledge of previous preparation techniques and of the nature and quality of specimens which have been produced successfully is usually quite valuable in planning and carrying out the sample preparation aspect of a new research program. The present listing of research materials produced in the Inorganic Materials Division is offered as a means of promoting direct scientist-to-scientist exchange of information on production and characterization of materials for which the division has some special competence. In some cases, specimens may be on hand and available on request; in other cases, facilities and staff may be available to produce additional specimens for purposes coming within the NBS mission. Provision of information, rather than specimens, is the principal goal, however, because maintenance of a stock of the numerous and extremely varied materials produced in small batches or maintenance of specialized production facilities on a standby basis is not feasible.

Information concerning nationwide sources of many research materials, especially single crystals, is available from the Oak Ridge Research Materials Information Center [4].

The present listing of materials may be useful to scientists and engineers interested in the production of a material or its general availability for purposes other than research. No claim is made concerning exhaustive knowledge of the materials listed, but the staff members involved in the production or characterization of unusual materials sometimes have special knowledge relevant to other applications in addition to research.

2. Definition of Research Materials

2.1. General Definition

A research material is here considered to be one sufficiently well-characterized to be useful for a particular type of current research. Ideally, a fully characterized material is desirable; that is, the character (chemical composition, structure, microstructure, etc.) should be uniform and held within such narrow and known limits that all the chemical and physical properties of the material are well determined. Practical considerations usually limit the characterization to

[1] Figures in brackets indicate the literature references on page 3.

the factors thought to control the property being studied and limit the accuracy of the characterization. Thus, a material useful at a particular stage of research may no longer be useful for the same type of research as measurement techniques improve and finer details of behavior are studied or as new factors are discovered to have an influence on properties at the level being studied. A valid research material can cease to have this distinction as a field of study progresses but the same material can sometimes again become an important research material as new phenomena are investigated. For example, ruby grown by the Verneuil process for bearings, wear surfaces, or jewels suddenly became an exciting research material when the ruby laser was invented.

2.2. Differences in Degree of Characterization Needed for Research on Properties with Varying Degrees of Structure Sensitivity.

The characterization needed for certain research, such as phase equilibria, is sometimes limited primarily to the major element composition and such factors as trace impurities or surface condition are relatively unimportant. This fact is reflected in the listing, for example, of a number of crystalline materials developed for phase equilibria studies and a number of glasses developed for bulk optical or elasticity studies; major component characterization was sufficient for these purposes. Some of these materials have subsequently become the object of renewed research interest in connection with other properties dependent primarily upon major component composition such as electro-optic properties, photoelasticity, and mechanical properties under pressure. Certain structure-sensitive properties, such as transport, plastic deformation, and fracture depend upon trace impurities and upon small deviations from stoichiometry. Samples made for research in these areas, even though of the same major component composition as some of those mentioned previously, require more careful processing and characterization and are accordingly listed as separate entries with an indication of their special features.

2.3. Inhomogeneity

Among both the bulk composition and trace impurity types there are cases where a deliberate degree of inhomogeneity is required. Thus, a series of glasses with the same total composition but differing degrees of phase separation forms an interesting family of research materials. An example of an even finer scale of deliberately produced inhomogeneity is a series of crystals of CaF_2 all having the same level of Gd additive but differing in the degree of association of point defects with Gd atoms. This last example illustrates another feature of some of the listed materials; in this case specimens of given bulk composition were produced by a commercial supplier to NBS specifications. The NBS contribution was the development of special treatments and measurement techniques needed to produce and determine different degrees of association.

2.4. Metastable Materials

Still another category of research materials involves those which are not stable or have a very short lifetime under ordinary conditions but which occur as important components under special conditions such as high temperature vapor species, high pressure crystalline phases, or short-lived reactive intermediates. Such materials generally cannot be kept in stock but the technology of preparation and the techniques for measurement of concentration and properties during the brief lifetime of the material are important aspects of research material information. An overlapping family of research materials consists of those which are dangerous (toxic, explosive, or corrosive) and so require special handling procedures during processing, storage, or property measurement. An example of such a difficult research material is the high pressure polymorph of the detonator explosive lead azide.

3. Relation to Standard Reference Materials

A few of the research materials listed here have been developed into Standard Reference Materials and many of the Standard Reference Materials are used as research materials but the two categories should not be confused despite the fact that there is some overlap. Standard Reference Materials are kept in stock by the National Bureau of Standards and sold by the Office of Standard Reference Materials. Each Standard Reference Material is certified with respect to the aspects of chemical and/or physical properties relevant to the material's intended application. Perhaps the greatest use of these materials is for calibration of instruments and checking of measurement procedures, but the high degree of homogeneity and extensive characterization associated with Standard Reference Materials frequently makes them useful as research materials. A full listing of Standard Reference Materials is available [5].

4. Organization of the Tables

4.1. Rationale

As noted in the Introduction, entries have been tabulated into sections derived chiefly from gross physical state at ordinary conditions (e.g., gases, polycrystals, multiphases, etc.). Clearly a number of exceptions occur, particularly for research materials of low stability or transient existence, but their location will be fairly obvious to the reader.

Since this compilation tends to emphasize composition rather than properties, special care was taken in organizing the tables. Use of the widely accepted Chemical Abstracts Formula Index does not always generate familiar empirical chemical formulae, nor

does this system readily lend itself to classification of glasses, but we chose it for its rational basis. Moreover, signfiicant progress has been achieved for computer searching and retrieval; indeed, the parent program is now in use by the Patent Office.

4.2. Use of Tables—Key

a. The arrangement of symbols in formulae is alphabetic except that in carbon compounds C always comes first followed immediately by H if hydrogen is also present.

b. The arrangement of formulae or entries is also alphabetic, except that the number of atoms of any specific kind influences the order of listing; e.g., all C_1 compounds appear before C_2, thus CCl_2O, CCl_4, $CHCl_3$, CHN, CH_2O, CO, C_2Ca, $C_2H_4O_2$.

c. Water of hydration is not made a part of the formulae indexed.

d. Polymers having different names and recognized as different substances, e.g., acetaldehyde and paraldehyde, are all entered under their accepted formulae; but a definite compound for which different polymeric formulae are in use is entered under the simplest formula.

e. For series of compounds listed under a single entry heading "M" refers to metallic or metalloidal components (e.g., K, Li, Rb) and "X" denotes electronegative substituents such as Cl, F, O, S, etc.

f. Approximate compositions are indicated by enclosing listing in quotation marks, "—", such as in interstitial compounds or alloys.

g. Glasses, polyphases, and compounds of given stoichiometry but indefinite structure are cited alphabetically by M, followed by X; thus B_2O_3·$3Nb_2O_5$; Cr_2O_3-IrO_2; B_2O_3-BaO-SiO_2; etc.

h. Isotopic compositions are presumed to be of terrestrial abundance unless otherwise indicated.

i. Wherever practicable conventional formulae and/or nomenclature is listed to the right of the index formula.

j. Great effort has been made to maintain accuracy and provide current information. In some instances, through staff changes, some authors cited are no longer in the Inorganic Materials Division, yet inclusion of their recent efforts was deemed significant to this compendium. Hence, these individuals are signified by an asterisk. The reader may contact these individuals for further information, but it would probably be more expedient if the editors were contacted.

Many of the materials listed in this report were produced as part of programs sponsored by other agencies. Acknowledgements for specific materials are given in the references and are too numerous to repeat in detail here. The support by the Air Force Materials Laboratory, the Air Force Cambridge Research Laboratory, the Army Research Office (Durham), the Advanced Projects Research Agency (Materials Office), the Atomic Energy Commission (Research Division), the National Aeronautics and Space Administration, and the Office of Naval Research is gratefully acknowledged.

5. References

[1] Characterization of Materials, prepared by the Committee on Characterization of Materials of the Materials Advisory Board, H. W. Leverenz, Committee Chairman. National Academy of Sciences—National Academy of Engineering Publication MAB–229–M, March 1967.

[2] Research in Solid-State Sciences—Opportunities and Relevance to National Needs, prepared by a Committee of the Solid State Sciences Panel for the Coordinating Committee for Materials Research and Development of the Office of Science and Technology. R. Smoluchowski, Committee Chairman, Publication 1600, National Academy of Sciences, 1968.

[3] Ceramic Processing, prepared by the Committee on Ceramic Processing, of the Materials Advisory Board, Joseph A. Pask, Committee Chairman, Publication 1576, National Academy of Sciences, 1968.

[4] J. W. Cleland, Purity and Perfection of Research Specimens of Oxides in Mass Transport in Oxides, Nat. Bur. Stand. (U.S.), Spec. Pub. 296, (224 pages) edited by J. B Wachtman, Jr. and A. D. Franklin, issued August 1968. The address of the Research Materials Information Center is Building 3001, Oak Ridge National Laboratory, Box X, Oak Ridge, Tennessee 37830.

[5] Catalog of Standard Materials, Nat. Bur. Stand. (U.S.), Spec. Publ. 260, July 1970. See also supplements for price lists and changes.

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
BCl_nF_{3-n}	equilibrium soln. of all species, where n = 0-3, studied as liquids	$BCl_3 + BF_3$ ⇆ at < 25°C	NMR (^{11}B,^{19}F) spectrometry	attempt to determine chemical exchange rates in equilibrium	T. C. Farrar	
BF_2H HBF_2 difluoroborane	also studied as liquid (1,2)	(3) BF_3+ B_2H_6 at 250°C (4) BF_3 + $(CH_3O)_2BH$ B_2F_4 + $(CH_3)_3SiH$ + hν	mol. wt., infrared NMR (^1H,^{11}B,^{19}F) spectrometry, active H, chemical reactivity	qualitative structural detms; only known mono-boron fluoro-hydride; synth. reagent; air/ moisture sensitive	T. D. Coyle T. C. Farrar	(1) T. C. Farrar and T. D. Coyle, "Proton and Fluorine NMR Spectra of HBF_2", J. Chem. Phys., 41, 2612 (1964). (2) E. B. Whipple, T. H. Brown, T. C. Farrar, and T. D. Coyle, "Relative Signs of Nuclear Spin Coupling in $^1H^{11}B^{19}F_2$" J. Chem. Phys., 43, 1841 (1965). (3) T. D. Coyle, J. J. Ritter, and T. C. Farrar, "Preparation and Properties of Difluoroborane", Proc. Chem. Soc.,25, (1964). (4) T. D. Coyle, J. Cooper, and J. J. Ritter, "Preparation and Some Reactions of Difluoroborane", Inorg. Chem., 7, 1014 (1968).

B_2F_5OSi BF_2OSiF_3		electric discharge rxn of BF_3 in SiO_2 reactor	infrared, mass spectrometry using full isotopic anal.	novel Si-O-B molecular structure suitable for bond angle and dissn. studies; unstable at r.t.	F. E. Brinckman G. Gordon	(1) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Reactions of Covalent Main Group Fluorides in Electric Discharges", Fourth Int. Symp. on Fluorine Chem., Estes Park, Colo., July 1967, Abstr. of Papers, p. 132. (2) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Characterization of Transport Species in Electric Discharges", Proc. Int. Symp. on Decomposition of Organometallic Compounds to Refractory Ceramics, Metals, and Metal Alloys, Univ. Dayton Press (1967), p. 29.
B_2F_4 diboron tetrafluoride tetrafluorodiborane-4	also studied as liquid	B_2Cl_4 + SbF_3 at -80°C	infrared, mass, NMR (^{11}B,^{19}F) spectrometry	simplest polyboron fluoride; potential source of BF_2 function in free radical reactions; B-B bond dissn. energy	T. D. Coyle T. C. Farrar	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. XII. Boron Trifluoride and Diboron Tetrafluoride", Inorg. Chem.,7, 1742 (1968).
B_2F_4O BF_2OBF_2	obsd. as parent-daughter ions	microwave discharge of BF_3 in SiO_2 reactor	direct inlet mass spectrometry, isotopic anal.	novel B-O-B molecular structure suitable for bond angle and dissn. measurements; unstable even at low pressures	F. E. Brinckman	See refs. 1, 2 under BF_3OSi.

$^{n}B_2H_6$ diborane	studied as liquid	$^{n}BF_3 \cdot O(C_2H_5)_2$ + $LiAlH_4$ → (n = 10 or 11)	infrared (1), NMR (^{1}H,^{10}B,^{11}B) spectrometry (2)	parent material for synthesis of selected isotope (H, D,^{10}B,^{11}B) boranes; detailed interpretation of NMR spectra	T. D. Coyle T. C. Farrar R. B. Johannesen	(1) W. J. Lafferty, A. G. Maki, and T. D. Coyle, "High Resolution Infrared Spectrum and Structure of Diborane", J. Mol. Spectroscopy, 33, 345 (1970). (2) T. C. Farrar, R. B. Johannesen, and T. D. Coyle, "Magnetic Non-Equivalence in the High Resolution NMR Spectra of Diborane", J. Chem. Phys., 49, 281 (1968). See also $C_2H^{11}BD_6O_2$ in SECTION II.
CCl_3F Freon-11	also studied as liquid, solid	commercial	infrared, NMR (^{19}F,^{35}Cl,^{37}Cl) spectrometry, volatility, m.p.	model compound for structure in liquid; anisotropic motions in liquids; angular momentum cross-sections	T. C. Farrar	
CHN HCN		NaCN + H_2SO_4	infrared and mass spectrometry	used for photoionization studies: parent compound for X-CN series	T. D. Coyle	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. IX. Hydrogen Cyanide and Acetonitrile", J. Chem. Phys., 48, 4765 (1968).
CH_3NO_2 CH_3ONO methyl nitrite		$NaNO_2$ + CH_3OH + H_2SO_4	infrared, ultraviolet, mass spectrometry; vapor phase chromatography	prepd. for photoionization study	T. D. Coyle	

C_2HBCl_2 ethynyldichloroborane		trans-ClCH = $CHBCl_2$ + hν	mass, infrared spectrometry; cleavage with propionic acid → C_2H_2 + HCl	first ethynylboron chloride isolated; novel substituted acetylene; suitable for studies on coordination saturation of B; air/moisture sensitive	J. J. Ritter T. D. Coyle	J. J. Ritter, T. C. Coyle, and J. M. Bellama, "Synthesis of Ethynylboron Halides", Chem. Comm., 908 (1969).
C_2HBF_2 ethynyldifluoroborane	C_{2v} symmetry	$(CH_3)_3SnC{\equiv}CH$ + BF_3 at -80°C	mass, infrared, and microwave spectrometry, cleavage in glass with propionic acid → C_2H_2 + SiF_4	first ethynylboron fluoride isolated; suitable for extensive structural anal.; air/moisture sensitive	J. J. Ritter T. D. Coyle	See ref. under C_2HBCl_2. W. J. Lafferty, J. J. Ritter, "Microwave Spectrum, Structure, and Dipole Moment of Ethynyldifluoroborane, $HC{\equiv}CBF_2$", Chem. Comm., 909 (1969).
$ClFO_3$ $FClO_3$ perchloryl fluoride	also studied as liquid, solid	commercial	see CCl_3F	see CCl_3F	T. C. Farrar	
$Cl_nF_{4-n}Si$ chlorofluorosilanes	n = 1-3 also studied as liquids	$SiCl_4$ + SiF_4 + $AlCl_3$ (cat.) at 150°C (1) electric discharge in $SiCl_4$ + SiF_4 (2)	infrared, mass (full isotopic anal.), NMR [^{19}F,($^{19}F\{^{29}Si\}$)] spectrometry; volatility, mol. wt.; vapor phase chromatography	model compounds for structural, dipole moment (n = 1,3), and magnetic resonance properties; synthetic intermediates	F. E. Brinckman T. D. Coyle T. C. Farrar	(1) R. B. Johannessen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., 72, 660 (1968). (2) See refs. under BF_5OSi. (3) See ref. under F_4Si.

D D(atoms)		microwave discharge in D_2O(g)	measurement of electron g-factor relative to g_J of H atoms	experimental test of quantum electrodynamic calculations	M. Linzer	M. Linzer, "Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deuterium and Atomic Nitrogen", Bull. Am. Phys. Soc., 12, 507 (1967).
F_2HP HPF_2 difluorophosphine		PF_2I + HI (1)	mol. wt.; infrared NMR (^1H,^{19}F,^{31}P)(2) spectrometry	NMR relative sign measurements; reagent uses; air/moisture sensitive	T. D. Coyle	(1) R. W. Rudolph and R. W. Parry, "Fluorophosphine Ligands. I. The Preparation and Characterization of Difluorophosphine", Inorg. Chem., 4, 1339 (1965). (2) R. B. Johannesen, "NMR Studies of Inorganic Fluorides. IV. Relative Signs of Coupling Constants in CH_3SiF_3, $HSiF_3$, and HPF_2", J. Chem. Phys., 47, 3088 (1967).
F_3OP OPF_3 F_3P PF_3	studied as liquids also	commercial; fractional distillation	infrared, mass (1), NMR (^{19}F,^{31}P)(2) spectrometry; volatility	related to study of electric discharge rxns in PF_3 (1); model compounds for study of structures and electron anisotropies (2)	F. E. Brinckman T. C. Farrar (2)	(1) F. E. Brinckman, and G. Gordon, "Formation of μ-Oxyfluorophosphines and Polyphosphines in Gaseous Discharge Reactions", 4th Middle Atlantic Regional Mtg of the Am. Chem. Soc., Wash. D. C., February 1969, Abstr. of Papers, p. 41.
F_4Si SiF_4 tetrafluorosilane	Also obsd. as liquid (1)	commercial, fractional distillation	infrared, mass, NMR (^{19}F) spectrometry; volatility, mol. wt., vapor phase chromatography	Si-F coupling constant; ^{29}Si NMR by double resonance	T. D. Coyle T. C. Farrar	T. D. Coyle, R. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "NMR Studies of Inorganic Fluorides. II. Solvent Effects on $J_{(^{29}Si-^{19}F)}$ in SiF_4", J. Phys. Chem., 70, 1682 (1966).

F_6Ge_2O $(GeF_3)_2O$ F_6GeOSi GeF_3OSiF_3	obsd as parent and daughter ions	microwave discharge rxns. of GeF_4 and GeF_4 + SiF_4 in SiO_2 reactors	direct inlet mass spectrometry, full isotopic anal.	candidates for molecular structure and bond dissn. energy measurements; yields very small	F. E. Brinckman	See ref. 2 under BF_5OSi.
F_6OSi_2 Si_2OF_6 hexafluoro-disiloxane	also studied as liquid	Si_2OCl_6 + SbF_3(l); electric discharge rxn of SiF_4 with SiO_2(2)	NMR [^{19}F,($^{19}F\{^{29}Si\}$)] (1); mass (2) spectrometry	suitable for determination of Si-O-Si bond angle; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) R. B. Johannesen, T. C. Farrar, F. E. Brinckman, and T. D. Coyle, "NMR Studies of Inorganic Fluorides. I. High-Resolution ^{19}F Spectra of Si_2F_6 and $(SiF_3)_2O$", J. Chem. Phys., 44, 962 (1966). (2) See ref. 2 under BF_5OSi.
F_6Si_2 Si_2F_6 hexafluorodisilane	also studied as liquid	Si_2Cl_6 + SbF_3 (l)	NMR [^{19}F,($^{19}F\{^{29}Si\}$)] (1); spectrometry chemical reactivity (2)	parent compound for perfluoro-disilanyl chemistry; suitable for detm. of Si-Si bond energy; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) See ref. 1 under F_6OSi_2. (2) F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluoro-disilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Abstr. of Papers, p. D14.
Ga_2O		Ga_2O_3(s) heated to 1200-1500°C	mass spectrometry		R. C. Paule	
Ga_2O_4W Ga_2WO_4		Ga_2O_3(s) + W heated to 1200°C	mass spectrometry		R. C. Paule	
N N(atoms)		microwave discharge in N_2(g)	see D	see D	M. Linzer	See ref. under D.

II. LIQUIDS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author. Title, Journal)
$BD_{3-n}F_3H_nP$ $F_3P{\cdot}BH_nD_{3-n}$	n = 1-3 (2)	$PF_3+B_2H_6$ (deuterated)	synthesis, microwave spectrometry	rotational spectrum, barrier to internal rotation	T. D. Coyle	R. Kuczkowski and D. Lide, "Microwave Spectrum, Structure, Dipole Moment, and Barrier to Internal Rotation of Phosphorus Trifluoride-Borane", J. Chem. Phys., 46, 357 (1967).
BF_7Si_2 $SiF_3SiF_2BF_2$ BF_9Si_3 $n\text{-}Si_3F_7BF_2$		prepd. at Rice Univ. (a) $SiF_2(g)$ + $BF_3(g)$ (b) co-condensed at -196°C	infrared, NMR (^{11}B,^{19}F) spectro-metry	series of novel Si-B compounds; qualitative detm. of structure; air/moisture sensitive	F. E. Brinckman T. D. Coyle T. C. Farrar	P. L. Timms, T. C. Ehlert, J. A. Margrave, F. E. Brinckman, T. C. Farrar, and T. D. Coyle, "Silicon-Fluorine Chemistry. II. Silicon-Borane Fluorides, J. Am. Chem. Soc., 87, 3819 (1965).

B_2Cl_4 tetrachlorodiborane-4	unstable liquid	electric discharge reduction of BCl_3 (1)	infrared, mass (2) spectrometry; chemical reactivity (3)	parent material for chemistry of diboron compounds; detm. of B-B bond energy	J. J. Ritter	(1) T. Wartik, et. al., "Diboron Tetrachloride", Inorg. Syn. 10, 118 (1967); (2) V. H. Dibeler and J. A. Walker, "Mass Spectrometric Study of Photoionization. XIII. Boron Trichloride and Diboron Tetrachloride", Inorg. Chem., 8, 50 (1969); (3) T. D. Coyle and J. J. Ritter, "Reactions of Diboron Tetrahalides with Some Unsaturated Organometallic Compounds", J. Organomet. Chem., 12, 269 (1968).
$Br_nF_{3-n}Si$ bromofluorosilanes	n = 1-3 studied as liquids and gases	SiF_4 + $SiBr_4$ + $AlBr_3$ catalyst at 150°C	infrared, mass spectrometry; NMR $[^{19}F,(^{19}F\{^{29}Si\})]$ (1,2) spectrometry; volatility	dipole moments detmd. for SiF_3Br and $SiBr_3F$ (with A. Marryot, NBS)	F. E. Brinckman T. D. Coyle	(1) T. D. Coyle, R. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. II. Solvent Effects on $J_{(^{29}Si-^{19}F)}$ in Silicon Tetrafluoride", J. Phys. Chem., 70, 1682 (1966). (2) R. B. Johannesen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., 72, 660 (1968).

$CH_3B_2Cl_3$ methyltrichloro-diborane-4	unstable	B_2Cl_4 + $(CH_3)_4M$ (M = Ge,Sn, or Pb)	infrared, mass spectrometry; cleavage rxns. with Ag_2O + $NH_3(aq) \rightarrow CH_4$ + H_2	unsymmetrical diboron halide; useful for studies of additions to olefinic and and acetylenic systems; air/moisture sensitive	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Recent Studies in Organoboron Chemistry. Some Chemical Consequences of the Boron Subhalides", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. R1.
$^{13}CH_3I$ $^{13}CH_4O$ $^{13}CH_3OH$ [^{13}C]-methanol	also studied as gas	commercial, high-vacuum degassed	infrared, NMR (^{1}H,^{13}C) spectrometry; m. p.	model compounds for study of ^{13}C T_1 and T_2 values	T. C. Farrar	See also $C_2H_4O_2$.
$C_2H^nBD_6O_2$ $(CD_3O)_2{}^nBH$	n = 10,11; also studied as solid	CD_3OH + $^nB_2H_6$	infrared, NMR (^{1}H,^{11}B) spectrometry; mol. wt.; m. p.	isotopic composition provides selected nuclear spin properties only example of B-H bond distance measurement in 3-coordinate boron compounds; motional studies; precursors of HBF_2	T. D. Coyle T. C. Farrar	(1) T. C. Farrar, J. Cooper, and T. D. Coyle, "Proton Broad-Line NMR Study of [$^2H_6$]Dimethoxy-[^{11}B]borane", Chem. Comm., 610 (1966). (2) N. Boden, H. S. Gutowsky, J. R. Hansen, and T. C. Farrar, "Nuclear Magnetic Relaxation Studies of $(CD_3O)_2BH$", J. Chem. Phys., 46, 2849 (1967). (3) T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, 73A, 195 (1969).

$C_2H_2B_2Cl_4$ trans-(BCl$_2$)CH=CHCBCl$_2$)		cis-$(BCl_2)_2C_2H_2$ + hν	infrared, mass, NMR spectrometry; cleavage rxn of deutero-compound with Ag_2O + $NH_{3(aq)}$ → trans-dideuteroethylene	established the mode of addition of B_2Cl_4 to C_2H_2; pointed up the utility of ^1H NMR while irradiating at the boron frequency in distinguishing between 1,1 and 1,2 isomers while demonstrating the dangers of assigning structures on the basis of J_{H-H}; demonstrated utility of $Ag_2O/NH_{3(aq)}$ as B-C cleaving agent with retention of configuration: air/moisture sensitive	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Structure, Isomerization, and Cleavage of 1,2-Bis-(dichloroboryl)-ethylene", J. Am. Chem. Soc., 89, 5739 (1967).
$C_2H_2B_4Cl_4$ 1,1,2,2-tetrakis (dichloroboryl) ethane	white solid, melts at 30°C	$ClCH{=}CHBCl_2$+ $2B_2Cl_4$ or $HC{\equiv}CH+2B_2Cl_4$	elemental anal. mol. wt., rxn. ratios	further confirmation of the reaction between B_2Cl_4 and halo-olefins to produce a known compound; air/moisture sensitive	J. J. Ritter T. D. Coyle	(1) J. J. Ritter, T. D. Coyle, J. M. Bellama, "The Interaction of B_2Cl_4 with Halo-olefins", Abstracts, 156th National Meeting of Amer. Chem. Soc., Atlantic City, N. J., (1968) p.INOR 166; (2) C. Chambers, A. K. Holliday, and S. M. Walker, "1,2 Tetrakisdichloroboryl-ethane", Proc. Chem. Soc., 286 (1964).

$C_2H_3B_2Cl_2MX_3$ (M=C,Si,Ge,Sn; X=Cl,CH_3)	involatile liq. or oils	B_2Cl_4+CH_2=$CHMX_3$	elemental anal.; mol. wt., rxn. ratios	provides series of model compounds for structure-reactivity relationships in vicinal- and geminal-substituted organometal systems	J. J. Ritter T. D. Coyle	See ref. (3) under B_2Cl_4.
$C_2H_3B_3Cl_6$ 1,1,2-tris(dichloroboryl)-ethane		CH_2=$CHCl$ + $2B_2Cl_4$ or CH_2=$CHBCl_2$ + B_2Cl_4	complete elemental anal.; mol. wt.; infrared, NMR spectrometry, rxn. ratio	demonstrated a reaction pathway between B_2Cl_4 and halo olefins contrary to earlier reports of no reaction; suggested "BCl" as a possible "reactive intermediate" responsible for the observed product; air/moisture sensitive	J. J. Ritter T. D. Coyle	(1) See ref. under $C_2H_3B_2Cl_2MX_3$. (2) See ref. (1) under $C_2H_2B_4Cl_4$.
$C_2H_4O_2$ $CH_3{}^{13}COOH$ $\alpha[^{13}C]$-acetic acid		commercial; high-vacuum degassed	infrared, NMR (^1H,^{13}C) spectrometry; m.p.	model compound for study of ^{13}C T_1 and T_2 values	T. C. Farrar	See also $^{13}CH_3I$ and $^{13}CH_4O$.

$C_4H_{10}F_3NSi_2$ $(CH_3)_3SiNHSiF_3$		$[(CH_3)_3Si]_2NH$ + SiF_4	infrared, mass, NMR (^1H,^{19}F) spectrometry, elemental anal., subsequent rxns.	model compound for preparative route to perfluorosilazanes	F. E. Brinckman	See ref. 2 under $Br_nF_{3-n}Si$.
$C_3H_{10}F_5NSi_3$ $(CH_3)_3SiNHSi_2F_5$		$[(CH_3)_3Si]_2NH$ + Si_2F_6		model compound for derivative chemistry of perfluoropolysilanes based cleavage of Si-N		F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluoro-disilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. D14.
C_4HCoO_4 $HCo(CO)_4$	studied as solid; liquid unstable at > -45°C	see ref. (1)	NMR (^1H) spectrometry (1,2)	model compound for application of NMR in detm. metal-H bond distances; basis for reexamination of basic theory; see C_5HMnO_5	F. E. Brinckman T. D. Coyle T. C. Farrar	(1) T. C. Farrar, F. E. Brinckman, T. D. Coyle, A.Davidson and J. W. Faller, "A Broad-Line Proton Magnetic Resonance Study of Cobalt Tetracarbonyl Hydride", Inorg. Chem., 6, 161 (1967). (2) D. L. VanderHart, H. S. Gutowsky, and T. C. Farrar, "Dipole-Dipole Interactions of a Spin 1/2 Nucleus with a Quadrupole-Coupled Nucleus", J. Am. Chem. Soc., 89, 5056 (1967).
$C_4H_2FeO_4$ $H_2Fe(CO)_4$	studied as liquid		infrared, NMR (^1H) spectrometry	candidate compound for Fe-H distance detm., J_{Fe-H}	F. E. Brinckman T. C. Farrar	See C_5HMnO_5.

$C_4H_9B_3$ C,3-dimethyl-1,2-dicarbaclovo-pentaborane(5)		supplied by Prof. R. N. Grimes, Univ. of Virginia, Charlottsville, Va.	NMR (^1H,^{11}B) spectrometry	clarification of molecular structure	T. C. Farrar	R. N. Grimes, "A New Isomer of $C_2B_3H_5$,C,3-Dimethyl-1,2-dicarbaclovo-pentaborane(5)", J. Am. Chem. Soc., 88, 1070 (1966).
C_5HMnO_5 $HMn(CO)_5$ C_5HReO_5 $HRe(CO)_5$	also studied as solid	prepared at Mass. Inst, Tech., and at NBS by F. E. Brinckman	infrared, NMR (^1H) spectro-metry	model com-pound for application of NMR in detm. metal-H bond distance; see also C_4HCoO_4	T. C. Farrar	T. C. Farrar, Sister W. Ryan, A. Davidson, and J. W. Faller, "Manganese-Hydrogen Bond Distance in $HMn(CO)_4$", J. Am. Chem. Soc., 88, 184 (1966).
$C_5H_{15}N_3Si$	n = 3	(a) CH_3N_3 + CH_3MgCl (b) a + $(CH_3)_nSiCl_{4-n}$ at -40 to -80°C; see ref. (1)	infrared, NMR (^1H) spectro-metry (1,2), elemental anal., mol. wt.	parent com-pounds for synthesis of 1,3-dimethyl-triazeno-metal(loid) deriv.; novel bidentate ligand for coordination studies; air/moisture sensitive	F. E. Brinckman	(1) F. E. Brinckman, H. S. Haiss, and R. A. Robb, "Metal-Nitrogen Bonding, Covalent Complexes of 1,3-Dimethyltriazene with Elements of Groups I, II, III, IV, and V", Inorg. Chem., 4, 936 (1965).
$C_6H_{18}N_6Si$ $(CH_3)_nSi(CH_3N-N=NCH_3)_{4-n}$	n = 2					(2) F. E. Brinckman, K. Nelson, R. Barefoot, and H. Haiss, "Silicon-Nitrogen Bonding. Penta- and Hexa-coordinated Inter-mediates in Methyl-silazanes", in preparation.

C_6H_6 benzene	studied also as solid	commercial	NMR (^1H,^{13}C) spectrometry	application of Fast-Fourier transform NMR spectrometry to natural abundance ^{13}C	T. C. Farrar	See also C_6H_5D under SECTION IV.
C_6H_{12} cyclohexane		commercial, spectro-grade	NMR (^1H) spectrometry	measurement of ^1H g-factor relative to g_J of H atoms; secondary standard for g-factor detms; check on calculation of shielding parameter for H_2 molecule	M. Linzer	M. Linzer, "Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deuterium and Atomic Nitrogen", Bull. Am. Phys. Soc., 12, 507 (1967).
Cl_4V VCl_4	obsd. as neat liquid and in $TiCl_4$ soln.; also obsd. as polycrystalline solid	commercial, fractionally distilled	EPR spectrometry (as a function of frequency, temp.); magnetic susceptibility between 78-307°K; optical spectrophotometry of VCl_4 in $TiCl_4$	detm. magnetic parameters, Jahn-Teller distortion; paramagnetic relaxation time, T_1; air/moisture sensitive	R. B. Johannessen	R. B. Johannesen, G. A. Candela, and T. Tsang, "Jahn-Teller Distortion: Magnetic Studies of Vanadium Tetrachloride", J. Chem. Phys., 48, 5544 (1968).
Cl_5FSi_2 Si_2Cl_5F pentachlorofluoro-disilane		Si_2Cl_6 + SiF_4 + $AlCl_3$ (cat.) at 150°C	infrared, mass, NMR [^{19}F,($^{19}F\{^{29}Si\}$)] spectrometry	first example of mixed perhalogenated disilane; ^{19}F "tag" permits double-resonance NMR of Si nuclei	F. E. Brinckman T. D. Coyle	See ref. 2 under $Br_nF_{3-n}Si$. R. B. Johannesen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Some Fluorinated Mono- and Polysilanes", 153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers, p. L113.

Cl_6Si_2 hexachloro-disilane		commercial, fractional distillation; electric discharge in $SiCl_4$	infrared, mass (full isotopic anal.), spectrometry; vapor phase chromatography; volatility, cleavage rxn with $H_2O \rightarrow H_2+Cl-$	parent material for synthesis of compounds bearing Si-Si bond; suitable for Si-Si bond energy detm. air/moisture sensitive	F. E. Brinckman	F. E. Brinckman, J. Cooper, and T. D. Coyle, "Interactions of Some Halosilanes with Nitrogen Bases", 153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers, p. L112. See SECTION I under F_6Si_2.
H_2O "polywater"	polymeric	not available	infrared, NMR (^1H) spectrometry	attempt to establish nature of protons in sample (work with M. Malmberg, NBS)	T. C. Farrar E. D. Lippincott	

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
barium aluminoboro-fluorosilicate glasses; example: Al_2O_3 12 mol% B_2O_3 16 BaF_2 20 BaO 8 SiO_2 44	microspheres, 5-50 μ dia.	melting, chilling, size reduction, flame spherizing	refractive index, thermal expansion, sphere size	filler material for dental composites	G. W. Cleek	R. L. Bowen and G. W. Cleek, "X-Ray Opaque Reinforcing Fillers for Composite Materials", J. Dental Res., 48, 79 (1969).
glasses from Al_2O_3-BaO-SiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	
aluminosilicate glass, alkali-free	NBS Standard Reference Material # 715	melting, chilling	Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	ASTM draft in preparation.
glasses from B_2O_3-BaO-SiO_2 system	glass blocks	melting, annealing	chemical analysis; refractive index; density; liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	E. H. Hamilton, G. W. Cleek, and O. H. Grauer, "Some Properties of Glasses in the System Barium Oxide-Boric Oxide-Silica", Amer. Ceram. Soc., 41, 209 (1958).
glasses from B_2O_3-Ln_2O_3 system	small fragments	gravity separated from two immiscible liquids	polarizing microscopy	high-index solid state	E. M. Levin	E. M. Levin, "Liquid Immiscibility in the Rare Earth Oxide-Boric Oxide Systems", Phys.& Chem. Glasses, 7,90 (1966).

glasses from B_2O_3-Na_2O-SiO_2 system	glass blocks	melting, chilled	domain development kinetics; critical immiscibility temp.	specimens used for theoretical studies of immiscibility	W. Haller	W. Haller, D. H. Blackburn, F. E. Wagstaff, and R. J. Charles, "The Metastable Immiscibility Surface in the System Na_2O-B_2O_3-SiO_2", J. Am. Ceram. Soc. 53, 34 (1970).
^{10}B-enriched borate glasses doped with Co, Dy, or In	glass prisms	melting, chilling, grinding	neutron activation anal.	tested as possible method to detm. neutron flux	D. H. Blackburn	"Glass Beads for Neutron Flux Measurements", NBS Technical Highlights, 1965, p. 86
glasses from BaO-La_2O_3-SiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	
a base glass: BaO 5 mol% Na_2O 15 SiO_2 75 ZnO 5 doped with various rare earths: Eu, Er, Nd	blocks 3 x 3 x 3 cm.	melting, chilling, annealing	evaluation for laser use	specimens are being evaluated for fluorescence lifetime	G. W. Cleek	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Transfer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", J. Appl. Phys. 41, 364 (1970).
glasses from BaO-Nb_2O_5-SiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	
glasses from BaO-SiO_2-TiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	

glasses from BaO-SiO_2-ZnO system	glass blocks	melting, annealing	refractive index, transmittance, liquidus, temp.	specimens may be used for detm. of other properties	G. W. Cleek	
alkali-lime-silica glass: CaO 11.6 mol% K_2O 7.7 Na_2O 8.7 SiO_2 70.5	NBS Standard Reference Material # 710	melting, chilling	viscosity by beam bending	not NBS certified for this method but suitable as standard	A. Napolitano	(1) ASTM C598 and H. E. Hagy, "Calibration of Beam Bending Apparatus", J. Amer. Ceram. Soc., 46, 93 (1963).
			surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano	(2) D. F. Secrist, "Measurement of Surface Tension of Glasses by a Capillary Flow Technique", Bull. Amer. Ceram. Soc., 48, 563 (1969).
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	(3) ASTM draft in preparation.
			viscosity by penetrometer	not NBS certified for this method but suitable as standard for ASTM flow point detm.	A. Napolitano	(4) J. E. Kelley, T. D. Robert, and H. M. Hanes, "A Penetrometer for Measuring the Absolute Viscosity of Glass", U. S. Bureau of Mines RI 6358 (1964).
Fe-containing silicate glasses	30 x 30 x 2 mm	melting, chilling	Mossbauer spectrometry	specimens covering a wide range of Fe content may be used for characterization by Mossbauer spectroscopy	G. W. Cleek	

alkali-lead-silica glass: K_2O 5.6 mol% Na_2O 2.5 PbO 45.3 SiO_2 46.0	NBS Standard Reference Material # 711	melting, chilling	surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano	See ref. 2 under alkali-lime-silica glass.
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	ASTM draft in preparation.
glasses from Na_2O-SiO_2-TiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other glass properties	G. W. Cleek	E. H. Hamilton and G. W. Cleek, "Properties of Sodium Titanium Silicate Glasses", J. Res. NBS, 61, 89 (1958).
porous silica glass SiO_2	powders, 50-200 mesh particles	melting, chilling, thermal domain development, differential dissolution	mercury intrusion porosimetry, nitrogen absorption	possible surface area and porosity standards	W. Haller	(1) W. Haller, "Rearrangement Kinetics of the Liquid-Liquid Immiscible Microphases in Alkali-Borosilicate Melts", J. Chem. Phys., 42, 686, (1965). (2) N. M. Winslow and J. J. Shapiro, "An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration", ASTM Bull, 39 (Feb. 1959).
various	glass prisms	melting, chilling, annealing, polishing	refractive index	prisms covering range of refractive indices 1.5 to 1.9 measured by minimum deviation method for N_C, N_D, and N_F	G. W. Cleek	

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Al_2O_3 alumina	also studied as powder, see SECTION V	(1) commercial powder and crystals (2) purified powder by solar furnace	melting point	melting point detm. for SRM	S. J. Schneider	S. J. Schneider and C. L. McDaniel, "Effect of Environment upon the Melting Point of Al_2O_3", J. Res. NBS, 71A, 317 (1967).
						S. J. Schneider, "Cooperative Determination of the Melting Point of Al_2O_3", Pure and Applied Chemistry, to be published.
	single crystal bar	commercial	compressibility to 10Kb	see also under BH_3O_3, SECTION V	C. E. Weir*	C. E. Weir, "Compressibility of Eleven Inorganic Materials", J. Res. NBS, 69A, 29 (1965).
	single crystals and bicrystals, 3 x 8 x 20 mm	chemical vapor deposition, $AlCl_3$ + [O] → Al_2O_3 at 1700-1750°C	spectrochemical and activation anal., optical microscopy, x-ray diffractometry	material of high physical perfection and chemical purity; suitable for diffusion studies, physical properties	H. S. Parker	H. S. Parker and C. A. Harding, "Vapor Growth of Aluminum Oxide Bicrystals", Abstracts, ACCG-NBS Conference on Crystal Growth, Aug. 11-13, 1969, Washington, D. C., p. 42.
pure and with following dopants: Cr, Mg, Mg+Cr, Ti+Cr, Ti, V, Mn, Mn+Cr, Zn +Cr, Fe+Cr	∿ 1cm	flame fusion, prepd. by D. E. Roberts	optical, EPR spectrometry	laser properties (optical and x-ray damage)	R. F. Blunt T. Chang	C. M. Stickley, et al, "Color Centers and Ruby-Laser Output-Energy Degradation", J. Appl. Phys., 40, 1792 (1969).

$Al_{1-n}Cr_nKO_8S_2 \cdot 12H_2O$ $KAl_{1-n}Cr_n(SO_4)_2 \cdot 12H_2O$	n = 1 to 0.02	grown from aq. soln.	optical, magnetic	ferro-electric, see also $CrH_4NO_8S_2 \cdot 12H_2O$	G. A. Candela S. E. Stokowski*	
AsGa GaAs gallium arsenide	highly doped and compensated	vapor phase epitaxy on GaAs substrate, $GaCl_3$ + $AsCl_3$ →; prepared by Monsanto Co.	spectral examination of temp. dependence of radiative recombination induced by electrical injection across p-n junction or by optical excitation	understanding of band structure of GaAs responsible for unusual absorption-edge exhibited; GaAs semi-conductor injection lasers, im-provement of room temp. performance; present efficient use of GaAs as a laser necessitates cryogenic condi-tions, cause of large decrease in efficiency at room temp. not fully understood, such studies should lead to specific material useful for improved laser performance	A. H. Neill* N. Winogradoff*	(1) N. N. Winogradoff and H. Kessler, "White Emission and Electrical Characteristics of Epitaxial GaAs Lasers and Tunnel Diodes", Solid State Comm., 2, 119 (1964). (2) N. N. Winogradoff, K. Owen and R. Cutnutt "The Radiative Band Pinch Effect and Temperature Dependence of Radiative Recom-bination in GaAs", Int. J. Electronics, 22, 229 (1967). (3) N. Winogradoff, "Field Control of the Quantum Efficiency of Radiative Recombi-nation in Semicon-ductors", Phys. Rev., 138A, 1562 (1965). (4) N. Winogradoff and A. H. Neill, "Band Tailing Effects and the Temperature De-pendence of Radiation Recombination in Compensated Epitaxial GaAs Laser Junctions", IEEE J. Quant. Electronics, 4, 111 (1968).

B_2BaO_4 $BaO \cdot B_2O_3$	small, tenths of mm	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high-temp form, metastable at room temp.	A. Mirbelt A. Perloff	A. D. Mirbelt, A. Perloff and S. Block, "The Crystal Structure of the High Temperature Form of Barium Borate, $BaO \cdot B_2O_3$", Acta Cryst., 20, 819 (1966).
$B_2Bi_4O_9$ $2Bi_2O_3 \cdot B_2O_3$	~ 0.1 mm range	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.		A. Perloff	A. Perloff, paper in prepn. for submission to Acta Cryst.
B_4O_7Pb $PbO \cdot 2B_2O_3$	~ 0.1 mm range	crystallized from melt of stoichiometric composition	unit cell and diffraction symbol from single crystal data	structure by analogy to isomorphous $SrO \cdot 2B_2O_3$	A. Perloff	A. Perloff and S. Block, "The Crystal Structure of the Strontium and Lead Tetraborates, $SrO \cdot 2B_2O_3$ and $PbO \cdot 2B_2O_3$", Acta Cryst., 20, 274 (1966).
B_4O_7Sr $SrO \cdot 2B_2O_3$	needles; length ~ 0.1 mm, cross-section ~ .01 mm	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.	see B_4O_7Pb	A. Perloff	See ref. under B_4O_7Pb.
$4B_2O_3 \cdot BaO$ BaB_8O_{13}	twinned crystal, 1 x 1 x 2 mm	melt solidification, required seeding for crystallization	spectrochemical anal.; optical, x-ray powder diffractometry (from ambient to 850°C); differential thermal anal.	piezoelectric at ambient temp. (orthorhombic form); transforms reversibly to tetragonal form at 700°C	C. R. Robbins E. M. Levin	C. R. Robbins and E. M. Levin, "Phase Transformation in Barium Tetraborate", J. Res. NBS, 73A, 615 (1969).
$4B_2O_3 \cdot Na_2O$ $Na_2O \cdot 4B_2O_3$	small (~ 0.1 mm range)	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high temp. form, metastable at room temp.	A. Perloff	A. Hyman, A. Perloff, F. Mauer, and S. Block, "The Crystal Structure of Sodium Tetraborate", Acta Cryst., 22, 815 (1967).

$BaGe_4O_9$	1 x 1 x 3 mm	melt solidification of stoichiometric composition	optical and x-ray diffractometry (1), including single crystal structure detm. (2)	type structure for AB_4O_9 germanates, see also Ge_4O_9Pb and Ge_4O_9Sr in SECTION V	C. R. Robbins	(1) C. R. Robbins and E. M. Levin, "Tetragermanates of Strontium, Lead and Barium of Formula Type AB_4O_9", J. Res. NBS, 65A, 127 (1961). (2) C. Robbins, A. Perloff, and S. Block, "Crystal Structure of BaGe $[Ge_3O_9]$ and its Relation to Benitoite", J. Res. NBS, 70A, 385 (1966).
$Ba_2Ge_2O_8Ti$ $Ba_2TiGe_2O_8$	1 x 1 x 3 mm	melt solidification and melt solidification using TiO_2 as flux (1)	spectrochemical; optical and x-ray powder diffractometry; differential thermal anal.	crystals exhibit pronounced domain structure, show very strong piezoelectric response (1); phosphor (2)	C. R. Robbins	(1) Work in progress with particular interest in the crystal chemistry, domain structure and piezoelectric property of the compound. (C. R. Robbins, 1969). (2) G. Blasse, "Fluoresence of Compounds with Fresnoite ($Ba_2TiSi_2O_8$) Structure", J. Inorg. Nucl. Chem., 30, 2283 (1968).
$Ba_2O_8Si_2Ti$ $Ba_2TiSi_2O_8$	1 x 1 x 5 mm	melt solidification using TiO_2 as flux (1)	spectrochemical; optical, x-ray powder diffractometry (powder and single crystal), differential thermal anal. (1)	piezoelectric (1); phosphor (2); type structure	C. R. Robbins	(1) C. R. Robbins, "Growth of Fresnoite ($Ba_2TiSi_2O_8$) From a TiO_2 Flux and Relation to the System $BaTiO_3$ - SiO_2", J. Res. NBS, 74A, 229 (1970). (2) See ref. under $Ba_2Ge_2O_8Ti$.

$Be_2CuO_{10}Si_2Y_2$ $CuY_2Be_2Si_2O_{10}$ copper yttrium gadolinite	light blue, 1 mm; also studied as polycrystalline material	slow cooling from Na_2WO_4 or $Na_2W_2O_7$ flux	precession and x-ray powder diffraction anal.	see under $Be_2Ln_2MO_{10}Si_2$ (M = Mg,Ni,Zn, Co,Cu,Fe,Mn, Cd), SECTION V	H. S. Peiser	J. Ito, "The Synthesis of Gadolinite", Proc. Jap. Acad., 41, 404 (1965). J. Ito, "A Note on the Gadolinite Synthesis", Proc. Jap. Acad., 42, 634 (1966). J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., 52, 1523 (1967).
$Be_2O_4Si(V^{+4})$ Be_2SiO_4 doped with V^{+4}	bright blue, 2 mm	slow cooling from V_2O_5 or $Ba_3(VO_4)_2$ flux	precession and x-ray powder diffraction anal.		H. S. Peiser	C. Frondel and J. Ito, "Synthesis of the Scandium Analogues of Beryl", Amer. Mineral., 53, 943 (1968).
$Bi_{12}GeO_{20}$ $GeBi_{12}O_{20}$		see $Bi_{12}MO_{20}$	optical	see $Bi_{32}Ga_2O_{51}$	A. Feldman D. Horowitz	
$Bi_{12}MO_{20}$ $6Bi_2O_3 \cdot MO_2$	M = Ge, Si, Ti; 0.5 x 1 cm	melt grown	see also $Bi_{12}GeO_{20}$, $Bi_{32}Ga_2O_{51}$	Faraday rotation; piezoelectric electro-optic	W. S. Brower	E. L. Venturini, E. G. Spencer and A. A. Ballman, "Elasto-Optic Properties of $Bi_{12}GeO_{20}$, $Bi_{12}SiO_{20}$, $Sr_xBa_{1-x}Nb_2O_6$", J. Appl. Phys., 40, 1622 (1969).
$7Bi_2O_3 \cdot ZnO$	0.5 x 1 cm	melt grown			W. S. Brower	
$17Bi_2O_3 \cdot Ga_2O_3$	0.5 x 1 cm	melt grown			W. S. Brower	
$Bi_{32}Ga_2O_{51}$ $16Bi_2O_3 \cdot Ga_2O_3$		pulled from melt, prepd. by W. S. Brower	optical	optically active, high Faraday rotation; see also $Bi_{12}GeO_{20}$	A. Feldman D. Horowitz	

Br_2 bromine		from liquid at 20°C, 5Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	C. E. Weir, G. J. Piermarini, and S. Block, "Crystallography of Some High Pressure Forms of C_6H_6, CS_2, Br_2, CCl_4, and KNO_3", J. Chem. Phys., 50, 2089 (1969)
CCl_4	I structure	from liquid at 20°C, 1Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2.
	II structure	from CCl_4-I at 80°C, 8Kb				
	III structure	from CCl_4-I at 120°C, 10Kb				
CS_2		frozen from liquid at 20°C, 12Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2.
$C_5FeN_6Na_2O \cdot 2H_2O$ $Na_2Fe(CN)_5NO \cdot 2H_2O$ sodium nitroprusside	1 x 1 x 3 in.	controlled growth from aq. soln.	Mossbauer spectroscopy	crystal structure, Standard Reference Material #725; Mossbauer spectroscopy	A. T. Horton	J. J. Spijkerman, D. K. Snediker, F. M. Ruegg, and J. R. DeVoe, "Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds", NBS Misc. Publ. 260-13 (1967). "Standard Reference Material for Mossbauer Spectroscopy", NBS Tech. News Bull., 50, 73 (May 1966).
C_6H_6	I structure, see also SECTION II	frozen from liquid at 20°C, 0.6 Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2.

$C_{10}H_{10}Cl_2CuN_2O_2$ $Cu(C_5H_5NO)_2Cl_2$ with the following dopants: Ba,Cd,Ni,^{61}Ni (0.1-2%),Pb, and Zn bis(pyridine-N-oxide)copper (II) chloride	1 x 1 x 1 to 5 x 5 x 5 mm	grown from methanol soln.	optical and EPR spectrometry; chemical anal.	metal-metal interaction study	G. F. Kokoszka	R. W. Duerst and G. F. Kokoszka, "Hyperfine Fields in Dimeric Coordination Complexes", J. Chem. Phys., 51, 1673 (1969).
$C_{10}H_{10}Fe$ $(C_5H_5)_2Fe$ ferrocene	1 x 4 in	melt solidification	infrared and Raman spectrophotometry	infrared and Raman spectra vibrational transitions	A. T. Horton	
$C_{12}H_{12}N_{10}NiO_6 \cdot 2H_2O$ $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$		$C_6H_6N_4$ (2,2'-bi-imidazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	study of hydrogen bonding between co-ordinated water and biimidazole molecules with nitrate groups	C. W. Reimann	A. D. Mighell, C. W. Reimann, and F. A. Mauer, "The Crystal and Molecular Structure of Diaquo-bis(2,2'-Biimidazole) Nickel (II) Dinitrate, $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$", Acta Cryst., B25, 60 (1969).
$C_{12}H_{16}Br_2N_8Ni$ $(C_3H_4N_2)_4NiBr_2$ tetrakis (pyrazolo)-nickel (II) bromide		$C_3H_4N_2$-(pyrozole) + $NiBr_2$ + H_2O	x-ray anal.,single crystal structure detm.; optical spectrophotometry	role of internal hydrogen bond in detm. electronic energy levels of Ni^{+2}	C. W. Reimann	A. D. Mighell, C. W. Reimann, and A. Santoro, "The Crystal and Molecular Structure of Di-bromotetrapyrazole Nickel (II)", Acta Cryst., B25, 595 (1969).
$C_{12}H_{16}Cl_2N_8Ni$ $(C_3H_4N_2)_4NiCl_2$ tetrakis(pyrazolo)-nickel (II) chloride		$C_3H_4N_2$(pyrozole) + $NiCl_2$ + H_2O				C. W. Reimann, A. D. Mighell, and F. Mauer, "The Crystal and Molecular Structure of Tetrakis-pyrazole-nickel Chloride, $Ni(C_3H_4N_2)_4Cl_2$", Acta Cryst., 23, 135 (1967).

$C_{12}H_{18}$ $C_6(CH_3)_6$ hexamethyl-benzene	1" dia. x 3"	melt solidification	x-ray, neutron diffractometry; differential thermal anal.	crystal structure; phase transitions; lattice dynamics	A. T. Horton	
$C_{12}H_{18}N_{24}Ni_3O_{18}\cdot 8H_2O$ $[(H_2O)_3(C_2H_3N_3)_3Ni]_2$-$Ni(NO_3)_6(H_2O)_2$		$C_2H_3N_3$ (1,2,4-triazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	optical and magnetic properties of polynuclear species	C. W. Reimann	C. W. Reimann and M. Zocchi, "The Structure of the Trinuclear Cation Bis-[u-(tri-1,2,4-triazolo-N^1,N^2)-triaquonickel] nickel", Chem. Comm., 272 (1968).
$C_{12}H_{24}N_{14}NiO_6$ $(C_2H_4N_2)_6Ni(NO_3)_2$		$C_3H_4N_2$(pyrazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	study of competition between hydrogen bonding and coordinate bonding in cationic packing	C. W. Reimann	C. W. Reimann, A. Santoro, and A. D. Mighell, "The Crystal and Molecular Structure of Hexapyrazolenickel-(II) Nitrate, $Ni(C_3H_4)_6WO_3)_2$", Acta Cryst., B26, (1970).
$C_{18}H_{24}MN_{14}O_6$ $(C_3H_4N_2)_6Cd(NO_3)_2$ $(C_3H_4N_2)_6Co(NO_3)_2$ $(C_3H_4N_2)_6Ni(NO_3)_2$	M = Cd, Co, and Ni	$C_3H_4N_2$ (imidazole) + $M(NO_3)_2$ + H_2O	single crystal x-ray anal.	studies of single crystal visible and vibrational spectra	C. W. Reimann	A. Santoro, A. D. Mighell, M. Zocchi, and C. W. Reimann, "The Crystal and Molecular Structure of Hexakis (Imidazole) Nickel (II) Nitrate, $(C_3H_4N_3)_6Ni(NO_3)_2$", Acta Cryst., B25, 842 (1969).

$C_{20}H_{42}$ n-$C_{20}H_{42}$ n-eicosane	2" dia. x 4"	melt solidification	electrical, optical absorption measurements	detm. free carrier mobility and G values; radiolysis measurements by use of optical absorption techniques	A. T. Horton	
CaF_2 also Gd-doped (0.1 to 1.0%)	1 x 3 x 5 mm	commercially obtained; crystals annealed in He + HF at 500-1200°C, rapidly quenched	absorption (200 to 2500 nm), EPR spectrometry; chemical anal. for Gd and accidental impurities	controlled point-defect equilibria studies; oxygen free	A. Franklin	A. Franklin, "Mass Transport in Non-metallic Solids", Proc. of Mtg. of Basic Science Div. of British Ceramic Soc., London, Dec. 1969, in press. A. Franklin and S. Marzullo, "Orientation Kinetics of Gd^{3+}-F^- Interstitial Pairs in CaF_2", J. Physics C, British Institute of Physics, to be published.
$CaGeO_5Ti$ $CaTiGeO_5$	1 x 1 x 2 mm	melt solidification from stoichiometric composition	spectrochemical anal.; optical, x-ray diffractometry (powder and single crystal)	isostructural with synthetic CaO_5SiTi	C. R. Robbins	C. R. Robbins, "Synthetic $CaTiSiO_5$ and its Germanium Analogue ($CaTiGeO_5$)", Mat. Res. Bull., 3, 693 (1968).
$CaMoO_4$	see under $MMoO_4$					

CaO_5SiTi $CaTiSiO_5$	1 x 2.5 cm (1) 0.5 x 0.5 x 1.5 cm (2)	melt solidification (2), and Czochralski growth (1)	spectrochemical anal.; optical, x-ray diffractometry [powder and single crystal (2)]	serves as type structure for pure $CaTiSiO_5$ (space group P_{2_1}/n); mineral with impurities, $CaTiSiO_4(O,OH,F)$, has symmetry C_2/c; see also $CaGeO_5Ti$	C. R. Robbins W. S. Brower	(1) W. S. Brower and C. R. Robbins, "Growth of $CaTiSiO_5$ by the Czochralski Method", J. Cryst. Growth, 3, (1969). (2) See ref. under $CaGeO_5Ti$.
CaO_4W $CaWO_4$	see under MO_4W					
$Ca_3Fe_2MO_{12}Si_2$	M = Ti,Zr	slow cooling of Li_2MoO_4 flux	powder and precession x-ray diffraction anal.	crystallographic sites detm. for both Ti and Zr in garnet structure	H. S. Peiser	J. Ito and C. Frondel, "Synthetic Zirconium and Titanium Garnets", Amer. Mineral., 52, 773 (1967).
$Ca_3TiFe_2Si_2O_{12}$	titanium garnet, dark brown, 2 mm also polycrystalline					
$Ca_3ZrFe_2Si_2O_{12}$	zirconium garnet, chestnut brown, 0.55 mm, also polycrystalline					
CdF_2 also with dopings	1 cm	Bridgman growth	electrical		W. R. Hosler W. S. Brower	P. Eisenberger and P. S. Pershan, "Electron Spin-Resonance and Infrared Studies of Semiconducting, Rare-Earth-Doped CdF_2", Phys. Rev., 167, 292 (1968).

Liquid CuCl	0.7 cm	Vapor and Bridgman growth; prepd. by D. E. Roberts, also supplied by A. F. Armington AFCRL, Bedford, Mass., and by A. Linz, Mass. Inst. Tech., Cambridge, Mass.	optical, dielectric	optical modulator	A. Feldman D. Horowitz	F. Sterzer, et al., "Cuprous Chloride Light Modulators", J. Opt. Soc. Am., 54, 62 (1964). A. Feldman and D. Horowitz, "Refractive Index of CuCl", J. Opt. Soc. Am. 59, 1406 (1969). NBS Tech. Rpt. 10013.
ClD_4N ND_4Cl	3 x 3 x 13 mm see also H_4NX	evaporation aq. soln. ND_4Cl + deuterourea.	NMR (D, ^{14}N) spectrometry	measurement of quadrupole coupling const. for D on ND_4^+ configuration; torsional motion of ND_4^+; crystal structure	R. A. Forman M. Linzer	M. Linzer and R. A. Forman, "NMR Studies of Single Crystal ND_4Cl", J. Chem. Phys., 46, 4690 (1967).
ClO_3Na $NaClO_3$	1 x 1 in.	controlled soln. growth	optical microscopy (etch pits); x-ray topography	optical, x-ray measurements	A. T. Horton	R. D. Deslattes, B. G. Simsom, A. T. Horton, "Solution Polishing of Oriented Single Crystals", Rev. of Sci. Inst., 36, 944 (1965). R. Brooks, A. T. Horton, and J. L. Torgesen, "Occlusion of Mother Liquor in Solution-Grown Crystals", J. Cryst. Growth, 2, 279 (1968).

ClTl TlCl also with dopants Cd, Pb, Nd, T	~ 2 mm, very soft	Bridgman growth; prepd. by D. E. Roberts	optical	piezo-optical luminescent properties	A. Feldman L. H. Grabner	R. Z. Bachrach and F. C. Brown. "Exciton Structure of TlCl and TlBr", Phys. Rev. Ltrs., 21, 685 (1968). A. Feldman, "Anomalous Splitting of the Exciton Peak in the Reflectivity of TlCl and TlBr", Bull. Am. Phys. Soc., 14, 428 (1969). L. H. Grabner, "Edge Fluorescence in TlBr", Bull. Am. Phys. Soc., 14, 131 (1969).
Cl_3CsPb $CsPbCl_3$	yellow, 1 cm. dia. x 2 cm.	grown by W. S. Brower, Bridgman method	dielectric constant	measured from 4.2K through phase transition at 320K	K. F. Young	C. K. Moller, "Structure of Perovskite-like Ce Pb Trihalides", Kgel. Danske Videnskab. Selskab, Mat. Fys. Medd., 32, 1 (1959);also see Nature, 182, 1436 (1958).
Cl_6K_2Re K_2ReCl_6	~ mm size	controlled evaporation, from soln.	ultra-sonic attenuation	second and higher order phase transitions	A. T. Horton	
$CoM_2O_7Si_2$ $Ca_2CoSi_2O_7$ $Sr_2CoSi_2O_7$	M = Ca,Sr; dark blue, 2 mm. Ca- and Sr cobalt akermanite; also poly-crystalline analogues with M = Cd,Cu,Fe,Mg, and Mn	slow cooling, flux Na_2WO_4	x-ray powder diffractometry		H. S. Peiser	J. Ito and H. S. Peiser, "Distorted Tetrahedra in Strontium Copper Akermanite", J. Res. NBS, 73A, 69 (1969).

$CrH_6Mo_6Na_3O_{24}\cdot nH_2O$ $Na(CrMo_6O_{24}H_6)\cdot nH_2O$	n = 8, prepn. leads to mm size crystals, no commercial supply available	aq. soln. of $Na_2MoO_4\cdot 2H_2O$ + $Cr(NO_3)_3\cdot 9H_2O$ at pH = 4.5	complete single crystal x-ray structure anal.	readily dehydrates to a composition approximating $(Na_3(CrMo_6O_{24}H_6)\cdot 2H_2O$	A. Perloff	(1) A. Perloff, "The Crystal Structure of Sodium Hexamolybdochromate (III) Octahydrate: $Na_3(CrMo_6O_{24}H_6)\cdot 8H_2O$", Inorg. Chem., in press. (2) A. Perloff, Doctoral Dissertation, Georgetown Univ. (1966). (3) G. A. Tsigdinos, Doctoral Dissertation, Boston Univ. (1961).
	n = 13, prepn. leads to mm size crystals, no commercial supply available	crystallizes on evaporation of aq. soln. of octahydrate at room temp.	complete single crystal x-ray structure anal.	readily dehydrates to a lower hydrate		(1) A. Perloff, paper in prepn.
$CrH_4NO_8S_2\cdot 12H_2O$ $NH_4Cr(SO_4)_2\cdot 12H_2O$		from aq. soln.	optical, magnetic	ferroelectric see also $Al_{n-1}Cr_n-KO_8S_2\cdot 12H_2O$	G. A. Candela S. E. Stokowski*	
$CuO_4S\cdot 5H_2O$ $CuSo_4\cdot 5H_2O$	1 cm^2	controlled evaporation from soln.	NMR spectrometry	NMR spectroscopy, wave guide experiments	A. T. Horton	

Cu_2O	1.5 x 6 cm	grown from melt		optical and electrical measurements; air sensitive	W. S. Brower H. S. Parker	W. S. Brower and H. S. Parker, "Melt Growth of Large Cuprous Oxide Single Crystals", Abstracts, ACCG-NBS Conf. on Crystal Growth, August 11-13, 1969, Washington, D. C., p. 115.
		prepd. by W. S. Brower	electrical	oxide-semiconductor dielectric constant	R. A. Forman W. R. Hosler K. F. Young	
$F_2M_8Na_2O_{24}Si_6$ M = Gd La Nd Pr Sm	largest crystal 2 x 2 x 6 cm colorless colorless purple green yellow	flux (NaF) isothermal evaporation	x-ray powder diffraction anal.; visible emmission spectrometry	strong red and green fluorescence when activated by Eu and Tb in Gd analogues	H. S. Peiser	J. Ito, "Silicate Apatites and Oxyapatites", Amer. Mineral., 53, 890 (1968).
F_2Mg MgF_2	$\sim 1\ cm^3$	pulled from melt, D. E. Roberts	optical; EPR spectrometry	color centers, optical coatings	M. I. Cohen	R. F. Blunt and M. I. Cohen, "Irradiation-Induced Color Centers in Magnesium Fluoride", Phys. Rev., 153, 1031 1967).
F_2Pb PbF_2 also Y, rare-earth dopings		Bridgeman growth			W. R. Hosler W. S. Brower	D. A. Jones, "Growth of Lead Fluoride Crystals from the Melt", Proc. Phys. Soc., 68B, 165 (1955).

H_2O - VII ice VII	structure VII	from liquid at 100°C and 30 Kb	x-ray diffractometry	unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weir*	C. E. Weir, S. Block, and G. Piermarini, "Single Crystal X-ray Diffraction at High Pressures", J. Res. NBS, 69C, 275 (1965).
H_4NX NH_4X X = Br,Cl,I also with Tl doping	~ 1 cm^3	from aq. soln. by evaporation	optical	relation to alkali halides	R. A. Forman	
H_6NO_4P $NH_4H_2PO_4$ "ADP"	3/4 x 3 in	controlled growth from soln.	optical microscopy (etch pits); x-ray topography	monochrometers, electro-optics, x-ray reference crystals	A. T. Horton	R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Preliminary Studies on the Characterization of Solution-Grown ADP Crystals", Adv. in X-ray Anal., 8, p. 315 (1965). R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Observation of Dislocations in ADP: Production of Dislocation-Free Crystals", J. Appl. Phys., 37, 541 (1966).
$InNaO_6Si_2$ $NaInSi_2O_6$	colorless indium aegirine, 1 cm; also obtained as polycrystalline	slow cooling from Na_2WO_4 or Na_2MoO_4 flux	powder and precession x-ray diffraction anal.	see also NaO_6ScSi_2	H. S. Peiser	J. Ito, "Synthetic Indium Silicates and Hydrogarnet", Am. Mineral., 53, 1663 (1968).

KNO_3 III	structure III	obtained from KNO_3 II at elevated temp. and pressure	x-ray diffractometry	detm. unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weir*	See ref. under Br_2.
KNO_3 IV	structure IV	from saturated aq. soln. KNO_3 at 100°C, 20 Kb				
KO_3Ta $KTaO_3$, also doped with Mn		prepd. by W. S. Brower; also obtained with doping from A. Linz, Mass. Inst. Tech., Cambridge, Mass., flux growth	electrical, optical	oxide semiconductor	M. I. Cohen W. R. Hosler	M. I. Cohen, et al., "Lattice Absorption in Strontium Titanate", Bull. Am. Phys. Soc., 14, 498 (1969). W. R. Hosler and H. P. R. Frederikse, "Magnetoresistive Effects in $KTaO_3$", Sol. State Comm., 7, 1443 (1969).

$MMoO_4$	M = Ca,Pb, Sr			see also $MnMoO_4$		W. S. Brower and P. H. Fang, "Dielectric Constants of $PbMoO_4$ and $CaMoO_4$", Phys. Rev., 149, 640 (1966).
$CaMoO_4$ $PbMoO_4$ $SrMoO_4$	1 x 3 cm	melt grown	x-ray topography; dielectric constant, etch pits	acousto-optic devices	W. S. Brower	W. S. Brower and P. H. Fang, "Dielectric Constants of $PbWO_4$ and $CaWO_4$", J. Appl. Phys., 38, 2391 (1967).
			mechanical	elastic constants of $CaMoO_4$	J. Wachtman, Jr.	J. B. Wachtman, Jr., W. S. Brower, Jr. and E. N. Farabaugh, "Elastic Constants of Single Crystal Calcium Molybdate ($CaMoO_4$)", J. Am. Ceram. Soc., 51, 341 (1968).
						W. A. Bonner, "Growth of Large Single Crystal Lead Molybdate for Acousto-Optic Applications", Abstr. ACCG-NBS Conf., August, 1969, p. 116.
MO_4W $CaWO_4$ $PbWO_4$ $SrWO_4$ $ZnWO_4$	M = Ca,Pb, Sr,Zn; 1 x 3 cm	melt grown	x-ray topography; dielectric constant; etch pits	acousto-optical devices	W. S. Brower	See refs. under $MMoO_4$.
$ZnWO_4$	1 cm		optical	magneto-optical properties	R. F. Blunt	

$MnMoO_4$	1 cm	pulled from melt; prepd. by D. E. Roberts	optical	magneto-optical properties	R. F. Blunt	See also under $MMoO_4$ and MO_4W for additional refs. to Scheelite crystals.
MnO_4W $MnWO_4$	1 cm	pulled from melt; prepd. by D. E. Roberts.	optical	magneto-optical properties	R. F. Blunt	See also MO_4W.
MoO_4Pb MoO_4Sr	see $MMoO_4$					
NaO_6ScSi_2 $NaScSi_2O_6$	colorless, scandium aegirine, 0.5 cm; also as poly-crystalline	slow cooling from Na_2WO_4 or Na_2MoO_4 flux	powder and pre-cession x-ray diffraction anal.	see also $InNaO_6Si_2$	H. S. Peiser	J. Ito and C. Frondel, "Synthesis of Scandium Analogues of Aegirine, Andradite and Melano-tekite", Am. Mineral., 53, 1276 (1968).
NiTi TiNi	wire	obtained from NOL, White Oak, Md.	electrical and magnetic	exhibits martinsitic transition; i.e., shows mechanical memory through transition	W. R. Hosler	F. E. Wang, B. F. DeSavage, W. J. Buehler, and W. R. Hosler, "The Ir-reversible Critical Range in the TiNi Transition", J. Appl. Phys. 39, 2166 (1968).
O_2Th ThO_2	2 x 1 x 1 cm	selected from arc melt by G. R. Findlay, Norton Co.	Laue diffraction	elastic constants	J. Wachtman, Jr.	P. B. Macedo, W. Capps, and J. B. Wachtman, Jr., "Elastic Constants of Single Crystal ThO_2 at 25°C", J. Am. Ceram. Soc., 47, 651 (1964).

[illegible] TiO_2	single crystal boule	Verneuil process, re-heated in O_2	compressibility to 10 Kb		C. E. Weir*	See ref. under Al_2O_3.
TiO_2 pure, also with dopants: Al, Cr, Cu, Fe, Mo, Nb, rare earths, V, W, Zn	1 cm	flame fusion, prepd. by D. E. Roberts	EPR spectrometry; electrical, optical	maser, high dielectric constant, oxide semi-conductor	R. F. Blunt T. Chang T. C. Ensign* W. R. Hosler	T. Chang, "Electron Spin Resonance of Mo^{5+} in Rutile", Phys. Rev., 136, A1413 (1964); "Paramagnetic Resonance Spectrum of W^{5+} in Rutile (TiO_2)", Phys. Rev., 147, 264 (1966). H. P. R. Frederikse, "Recent Studies on Rutile", J. Appl. Phys. Suppl., 32, 2211 (1961).
TiO_2 pure, also with dopants: Cr, Ni; oxidized and reduced conditions	single crystal rod	flame fusion	Laue diffraction patterns	elastic constants; internal friction	J. Wachtman, Jr.	J. B. Wachtman, Jr., W. E. Tefft, and D. G. Lam, Jr., "Elastic Constants of Rutile (TiO_2)", J. Res. NBS, 66A, 465 (1962). S. Spinner and J. B. Wachtman, Jr., "Some Elastic Compliances of Single Crystal Rutile from 25 to 1000°C", J. Res. NBS, 68A, 669 (1964). J. B. Wachtman, Jr. and L. R. Doyle, "Internal Friction in Rutile Containing Point Defects", Phys. Rev., 135, A276 (1964). J. B. Wachtman, Jr., S. Spinner, W. S. Brower, T. Fridinger, and R. W. Dickson, "Internal Friction in Rutile Containing Ni or Cr", Phys. Rev., 148, 811 (1966).

O_2U UO_2	3 x 1 x 1 cm	selected from arc melt by H. J. Anderson of Hanford Atomic Products Operation	density, coulometric titration, spec. analysis, Laue diffraction	elastic constants	J. Wachtman, Jr.	J. B. Wachtman, Jr., M. L. Wheat, H. J. Anderson and J. L. Bates, "Elastic Constants of Single Crystal UO_2 at 25°C", J. Nuclear Materials, 16, 39 (1965).
O_3PbSi $PbSiO_3$	alamosite, colorless, 2 mm; also prepd. as polycrystal-line	hydrothermal, spontaneous nucleation isothermal	x-ray structural anal. (M. Mydlar, Ceramic Engr. Dept., Univ. of Mo.)	structure	H. S. Peiser	J. Ito, "Synthesis of Some Lead Calcium Zinc Silicates", Amer. Mineral., 53, 231 (1968).

O_3SrTi	1 cm	flame fusion;	optical, electrical;	insulator;	T. Chang	A. H. Kahn and A.
$SrTiO_3$ pure, also with dopants: Al,Ga,Nb		also supplied by D. Beals, National Lead Co., South Amboy, N. J.	EPR spectrometry	semiconductor, superconductor; pseudo-ferroelectric; photochromic	M. I. Cohen A. Feldman L. H. Grabner W. R. Hosler A. H. Kahn	J. Leyendecker, "Electronic Energy Bands in Strontium Titanate", Phys. Rev., 135, A1321 (1964).
						H. P. R. Frederikse, W. R. Hosler, and W. R. Thurber, "Experimental Evidence Concerning the Conduction and Band of $SrTiO_3$", J. Phys. Soc. Japan, 21, Suppl., 32 (1966).
						J. F. Schooley and W. R. Thurber, "Superconductivity in Semiconducting $SrTiO_3$", J. Phys. Soc. Japan, 21, Suppl., 639 (1966).
						A. Feldman and D. Horowitz, "Stress-Induced Dichroism at the Absorption Edge of Strontium Titanate", Sol. State Comm., 6, 607 (1968).
$SrTiO_3$	1 - 2.5 mm	melt solidification using SiO_2 as flux	spectrochemical, optical and x-ray powder diffractometry	prepd. in absence of fluorides, etc.	C. R. Robbins	C. R. Robbins, "Growth of Strontium Titanate from a Silica Flux", J. Cryst. Growth, 2, 402 (1968).

V. POLYCRYSTALS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Ag	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials, SRM # 748	Knudsen, torque Knudsen, and mass spectrometric detm. of vapor pressure as function of temperature; detm. heat of sublimation	calibrn. of vapor pressure apparatus and techniques (10^{-8} to 10^{-3} atm., 1000-1600°K); this and other elements (see Au, Cd, Pt, and W) will each cover a different temperature range; these five materials cover 400-3000°C	R. C. Paule	R. C. Paule and J. Mandel, NBS Special Publication # 260.
			vapor pressure, Knudsen effusion collection	develop vapor pressure standard	E. R. Plante	See ref. above.
$AgNO_3$ "high-$AgNO_3$"	polycrystalline surface, $\alpha \sim 50\mu$	high temperature x-ray diffractometer furnace	x-ray powder diffractometry	unstable at room temp.	E. M. Levin	E. M. Levin, "X-Ray Determination of the Thermal Expansion of Silver Nitrate", J. Am. Ceram. Soc., 52, 53 (1969).
AlN aluminum nitride	100-300 mesh	four commercial sources	x-ray diffractometry	parent compound for study of transport chemistry	F. E. Brinckman	
Al_2BaO_4 $BaAl_2O_4$		γ-Al_2O_3 + $BaCO_3$ at 1200°C for 1.5 hr.	x-ray diffractometry; petrographic microscopy		H. Swanson J. deGroot H. McMurdie	H. Swanson et al., "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 5, p. 11 (1967).

Al_2O_3	See also IV. SINGLE CRYSTALS	(a) commercial powder and crystals (b) powder purified by solar furnace	melting point detm.	melting point standards; Standard Reference Material	S. J. Schneider	S. J. Schneider and C. L. McDaniel, "Effect of Environment Upon the Melting Point of Al_2O_3", J. Res. NBS, 71A, 317 (1967). S. J. Schneider, "Cooperative Determination of the Melting Point of Al_2O_3", to be published in Pure and Applied Chemistry (1970).
Au	high purity wire, 0.055 in. dia. x 6 in; Standard Reference Material # 745	special prepn. for Office of Standard Reference Materials, SRM # 745	see under Ag	temp. range 1300-2100°K; see also under Ag	R. C. Paule	R. C. Paule and J. Mandel, "Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold", NBS Special Publication # 260-19.
	0.055" wire	available as SRM # 745	see under Ag	see under Ag	E. R. Plante	See ref. above.
BF_4M KBF_4 $NaBF_4$	M = K, Na; obsd. also in aq. soln.	commercial	NMR (^{11}B, ^{19}F) spectrometry	model compounds for high resolution and broad-line NMR; J_{B-F} as related to solvents	F. E. Brinckman T. C. Farrar	
BH_3O_3 H_3BO_3		commercial	compressibility to 10 Kb	see also Al_2O_3 under Section IV.	C. E. Weir*	C. E. Weir, "Compressibility of Eleven Inorganic Materials", J. Res. NBS, 69A, 29 (1965).

BH_4M KBH_4 $LiBH_4$ $NaBH_4$	M = Li, K, Na; obsd. also in aq. soln.	commercial	NMR (^1H,^{11}B) spectrometry	model compounds, detm. phase transitions and motions in solids	T. C. Farrar	T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, 73A, 195 (1969).
BO_3Sc $ScBO_3$	∿50μ particles, calcite type	solid state rxn. of Sc_2O_3 + B_2O_3 in sealed tube	x-ray powder diffractometry, polarizing microscopy	phase diagram	E. M. Levin	E. M. Levin, "System Sc_2O_3-B_2O_3", J. Am. Ceram. Soc., 50, 53 (1967).
B_2O_3		controlled dehydration of H_3BO_3	compressibility to 10 Kb		C. E. Weir*	See ref. under BH_3O_3.
$B_2O_3 \cdot 3Nb_2O_5$ "$3Nb_2O_5 \cdot B_2O_3$"	∿50μ	solid state rxn. of B_2O_3 + Nb_2O_5 in sealed Pt tube	x-ray powder diffractometry	phase diagram	E. M. Levin	E. M. Levin, "Phase Equilibria on the System Niobium Pentoxide-Boric Oxide", J. Res. NBS, 70A, 11 (1966).
Borate minerals, anhydrous	∿80 in number, fine powders	metal oxide + H_3BO_3 at elevated temp.	infrared (2000-300 cm^{-1}) spectrometry	correlation of anion and spectra	C. E. Weir*	C. E. Weir and R. A. Schroeder, "Infrared Spectra of the Crystalline Inorganic Borates", J. Res. NBS, 68A, 465 (1965).
Borate minerals, hydrated	∿42 natural and synthetic	obtained from the Smithsonian Inst. U. S. Geological Survey, Washington, D. C. and Menlo Park, Calif.	infrared spectrometry, principally in 2000-300 cm^{-1} region	evaluation of limitations of spectra in identification, effects of treatment on spectra	C. E. Weir*	C. E. Weir, "Infrared Spectra of the Hydrated Borates", J. Res. NBS, 70A, 153 (1966).
BaF_3Li $LiBaF_3$		fuse stoichiometric amt. LiF + BaF_2 at 800°, annealed at 500° to remove BaF_2 ppt.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 35.

$BaGe_3O_9Ti$ $BaTiGe_3O_9$	20-40μ	solid state rxn. of $BaTiO_3$ + GeO_2 in sealed Pt tube at 1160°C	optical and x-ray powder diffractometry	stable from 1132° to 1235°C but readily obtainable by quenching to ambient temp.; serves as a structural intermediate between $BaGe_4O_9$ and $BaTiSi_3O_9$	C. R. Robbins	C. R. Robbins, "The Compound $BaTiGe_3O_9$", J. Am. Ceram. Soc., 43, 610 (1960).
$Be_2CuO_{10}Si_2Y_2$ $CuY_2Be_2Si_2O_{10}$	polycrystalline for all possible combinations with $MLn_2Be_2Si_2O_{10}$, where M=Mg, Ni, Zn, Co, Cu, Fe, Mn, and Cd; single crystal (1mm) also studied	slow cooling of solute in Na_2WO_4 or $Na_2W_2O_7$ flux	precession and powder x-ray diffractometry		H. S. Peiser	J. Ito, "The Synthesis of Gadolinite", Proc. Jap. Acad., 41, 404 (1965). J. Ito, "A Note on the Gadolinite Synthesis", Proc. J. Acad., 42, 634 (1966). J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., 52, 1523 (1967).
$BiNbO_4$	see also: BiO_4Ta BiO_4V NbO_4Sb O_4SbTa O_4SbV	solid state rxn. of Bi_2O_3 + Nb_2O_5 in sealed Pt tubes at 950 - 1250°C	x-ray powder diffractometry		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Synthesis and Stability of Bismutotantalite, Stibiotantalite and Chemically Similar ABO_4 Compounds", Am. Mineral., 48, 1348 (1963).
BiO_4Ta $BiTaO_4$		solid state rxn. of Bi_2O_3 + Ta_2O_5 in sealed Pt tubes at 845 - 1150°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.
BiO_4V $BiVO_4$		solid state rxn. of Bi_2O_3 + V_2O_5 in sealed Pt tubes at 500-1000°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.

Bi_2O_3	cubic	high-temp. x-ray diffractometer furnace; programmed heating and cooling	differential thermal anal.; x-ray diffractometry	obsd. at room temp. as metastable phase	E. M. Levin	E. M. Levin and R. S. Roth, "Polymorphism of Bismuth Sesquioxide. I. Pure Bi_2O_3", J. Res. NBS, 68A, 189 (1964); Ibid, II, 197-206.
	tetragonal					
$Br_2O_2Pb_3$ $Pb_3O_2Br_2$		heat stoichiometric PbO (red) + $PbBr_2$ in Au boat, in vacuo, at 350°C for 45 min.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 32.
Br_3Ti $TiBr_3$	dark purple, fine powder or small crystals; see also Cl_3Ti	$TiBr_4+H_2 \rightarrow$ using hot Ir filament	elemental anal. for Ti, Br	sublimes at ~ 500°C with disproportionation, strong reducing agent; catalyst for olefin polymerization; air/moisture sensitive	R. B. Johannesen	J. M. Sherfey, "Titanium-(III) Chloride and Titanium (III) Bromide", Inorg. Synth., 6, 57 (1960).
Br_4Ti $TiBr_4$	yellow, m.p. 38°C	$TiCl_4+HBr \rightarrow$ at b.p. of $TiCl_{4-n}Br_n$	elemental anal., m.p., b.p.	strong Lewis Acid properties, air/moisture sensitive	R. B. Johannesen	R. B. Johannesen and G. L. Gordon, "Titanium (IV) Bromide", Inorg. Synth., 9, 46 (1967).
$Br_6H_8N_2Os$ $(NH_4)_2OsBr_6$	mixed crystals (solid soln.) of $(NH_4)_2$-(Os-Pt)Br_6, also prepd. where Os/Os +Pt = 0.0763	(a) OsO_4 + HBr(aq) (b) (a) + NH_4Br	elemental anal., x-ray diffractometry	parent compounds for magnetic susceptibility studies	R. B. Johannesen	R. B. Johannesen and G. A. Candela, "Magnetic Susceptibilities and Dilution Effects in Low-Spin d^4 Complexes: Osmium (IV)", Inorg. Chem., 2, 67 (1963).

CSi SiC	cubic, ~200 mesh	Si + C above m.p. of Si	x-ray diffractometry, vapor pressure	other polymorphs present, C-contaminated	E. R. Plante	
$C_2Na_2O_4$ $Na_2C_2O_4$ sodium oxalate	NBS standard sample	NBS standard sample No. 40d	assay 99.9% $Na_2C_2O_4$; x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	H. Swanson, et al, "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 6, p. 70 (1968).
C_3Al_4	large crystallites embedded in polycrystalline matrix	Al(l) + C at 1400°C	x-ray diffractometry, vapor pressure	usually contains excess C; moisture sensitive		E. R. Plante and C. H. Schreyer, "Dissociation Pressure of Aluminum Carbide Using a Rotating Knudsen Cell", J. Res. NBS, 70A, 253 (1966).
C_4HCoO_4 $HCo(CO)_4$	unstable liquid, studied as solid	see SECTION II	see SECTION II	see SECTION II	T. C. Farrar	See SECTION II.
$C_4H_{12}F_2O_4W$ $(CH_3O)_4WF_2$	colorless crystals m.p. 81-83°C cis-stereoisomer only	WF_6 + $4CH_3OSi(CH_3)_3$ in C_6F_6	NMR(^1H,^{19}F) mol. wt. elemental analysis	one of a series of stable tungsten (VI) compounds; easily purified by sublimation; cis-difluoro configuration; provides basis for W (VI) stereochemistry; air/moisture sensitive.	F. E. Brinckman L. B. Handy*	L. B. Handy, F. E. Brinckman, "Chemistry of the Methoxyfluorotungsten (VI) Series", Chem. Comm., 214 (1970).

$C_6H_{10}CuO_4 \cdot H_2O$ Copper (II) propionate monohydrate	also Zn-doped	heat $Cu(Zn)CO_3$ + $[CH_3CH_2CO]_2O$ + H_2O	EPR spectrometry	model compounds for detm. magnetic parameters in dimeric Cu(II) alkanoates from EPR studies of polycrystalline samples	M. Linzer	G. F. Kokoszka, M. Linzer, G. Gordon, "Electron Paramagnetic Resonance Spectra of Polycrystalline Dimeric Complexes. Copper Propionate Monohydrate and Zinc-Doped Copper Propionate Monohydrate", Inorg. Chem., 7, 1730 (1968).
$C_6H_{18}HgSi_2$ $[(CH_3)_3Si]_2Hg$	yellow	$(CH_3)_3SiX$ + Na/Hg at 55°C (X=Cl,Br) (1)	NMR (^1H) spectrometry	source of trimethylsilyl group or radical (2); toxic, volatile Hg compound, soln. light-sensitive	F. E. Brinckman L. B. Handy*	(1) E. Wiberg, et. al, "Recent Developments in the Chemistry of Metal Silyls of the Type $M(SiR_3)n$", Angew. Chem. internat. Edit., 2, 507 (1963). (2) C. Eaborn, R. A. Jackson, and R. W. Walsingham, "The Reduction of Organosilicon Radicals in Solution", Chem. Comm., 1965, 300.
$C_{12}H_{36}LiN_2NaSi_4$ $[(CH_3)_3Si]_2N<^{Li}_{Na}>$ $>N[Si(CH_3)_3]_2$		metallation of N-H on disilazanes; sample supplied by Prof. U. Wannagat, T. U. Carolo-Wilhelmina, Braunschweig, Germany	see ref., NMR (^1H, ^7Li, ^{14}N, ^{23}Na) spectrometry	first example of NMR measurement of a mixed dimeric electron-deficient compound in the solid state	M. Linzer	D. Mootz, A. Zinnius, and B. Bottcher, "Association in the Solid State of Bis(trimethylsilyl) amidolithium and Methyltrimethylsilanatoberyllium", Angew. Chem., 81, 398 (1969).
$C_{20}H_{16}Cu_2N_{20} \cdot 4H_2O$ $Cu_2(C_5H_4N_5)_4 \cdot 4H_2O$ $C_{20}H_{20}Cl_4Cu_2N_{20}O_{16} \cdot 3H_2O$ $Cu_2(C_5H_5N_5)_4-(ClO_4)_4 \cdot 3H_2O$	(C_5H_5N)= adenine moiety	$Cu(ClO_4)_2$(aq) + adenine, adjust pH	chemical anal., optical and EPR spectroscopy	metal-metal bond interactions pertinent to biochemical processes involving nucleic acids	G. F. Kokoszka	R. W. Duerst, S. J. Baum, and G. F. Kokoszka, "Exchange Coupling in Two Dimeric Copper Adenine Complexes", Nature, 222, 665 (1969).

$CaCl_3Cs$ $CsCaCl_3$		$CsCl + CaCl_2$ at ~ 900°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 21.
$CaNa_2O_8S_2$ $Na_2Ca(SO_4)_2$	glauberite	$CaCl_2$+Na_2SO_4 in aq. soln.; intermediate $CaSO_4$ forms, desired product after several hr., washed with alcohol	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 59.
Cd	high purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials; SRM # 746	see Ag	temp. range 400-600°K; see also under Ag	R. C. Paule	See ref. under Ag.
$CdCl_3Cs$ $CsCdCl_3$		stoichiometric $CsCl+CdCl_2$ fusion	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 19.
$CdCl_3H_4N$ NH_4CdCl_3		stoichiometric $NH_4Cl+CdCl_2$ (aq. soln.) → ppt.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 6.
$CdCl_3K$ $KCdCl_3$		fuse KCl + $CdCl_2 \cdot 2.5H_2O$ at 550°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 38.
$CdCl_3Rb$	orthorhombic	ppt. from aq. soln. RbCl + $CdCl_2$	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 41.
	tetragonal	fuse RbCl + $CdCl_2$ (anhydr.) at ~ 500°C				Same, p. 43.

$CdCr_2O_4$		(a) CdO + Cr_2O_3 heated in vacuo at 300°C for 17 hr. (b) ground, pelletized, heated at 1050°C for 1.5 hr.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 16.
$CdO_4S \cdot H_2O$ $CdSO_4 \cdot H_2O$		(a) crystallized from aq. soln. at 95°C; (b) also prepd. from $CdSO_4$ or $CdSO_4 \cdot 8H_2O$ on prolonged exposure to air of RH = 50%.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 10.
$3CdO_4S \cdot 8H_2O$		obtained from Johnson, Mathey & Co., Ltd.		high humidity necessary to prevent formation of monohydrate		same, p. 8.
ClH_4NO NH_3OHCl		obtained from Naval Ordnance Plant, Indian Head, Md.	NMR (^1H) spectrometry, vapor pressure	study of phase changes and motions in solids	T. C. Farrar	
Cl_2P PCl_2 (free radical)	at 77K in PCl_3 matrix, C_{2v} symmetry	$PCl_3 + h\nu \rightarrow PCl_2 + PCl_4 + Cl$	EPR spectrometry	formation of PCl_4 by sequential rxns.; evaluation of molecular bonding, relation to chlorophosphorylation via radical intermediates	F. E. Brinckman G. F. Kokoszka	G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", Chem. Comm., 349 (1969). G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", J. Am. Chem. Soc., 92, 1199 (1970).
Cl_4P PCl_4 (free radical)	C_{2v} symmetry					

Cl_3CoCs $CsCoCl_3$		co-pptd. CsCl + $CoCl_2$ heated in sealed glass tube at 500°C	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 11.
Cl_3CoH_4N NH_4CoCl_3		co-pptd. NH_4Cl + $CoCl_2$ heated in sealed glass tube at 500°C	x-ray diffracto-metry, petrographic microscopy	hydrates readily in moist air	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 5.
Cl_3CoRb $RbCoCl_3$		co-pptd. RbCl + $CoCl_2$ heated in sealed glass tube at 500°C	x-ray diffracto-metry, petrographic microscopy	moderately hygroscopic	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 57.
Cl_3CsCu $CsCuCl_3$	dark red hex. prisms terminated by bipyramids	crystallized from aq. CsCl + $CuCl_2$	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 22.
Cl_3CsNi $CsNiCl_3$		co-pptd. CsCl + $NiCl_2$ heated in sealed glass tube at 500°C	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 12.
Cl_3CsPb $CsPbCl_3$		fuse CsCl + $PbCl_2$ at ~ 500°C	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 24. See also in SECTION IV.
Cl_3CsSr $CsSrCl_3$		$SrCl_2$ + CsCl melted at 900°C	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 13.
Cl_3H_4HgN NH_4HgCl_3		pptd. from soln. of NH_4Cl + $HgCl_2$	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 9.

Cl_3H_4NNi NH_4NiCl_3	(a) $NiCl_2 \cdot 2H_2O$ + HCl at 150°C → $NiCl_2$ (b) (a) + NH_4Cl in sealed glass tube at 300°C for 72 hr.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 6.
$Cl_3HgNa \cdot 2H_2O$ $NaHgCl_3 \cdot 2H_2O$	crystallization from aq. soln. of equimolar amts of NaCl + $HgCl_2$	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 66.
Cl_3NiRb $RbNiCl_3$	co-pptd. RbCl + $NiCl_2$ heated in sealed glass tube at 500°C		moderately hygroscopic	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 58.
Cl_4P	see Cl_2P				see Cl_2P
CoF_3K $KCoF_3$	pptd. from mixt. KF + CoF_2 soln., washed	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 37.
$CoNa_2O_8S_2 \cdot 4H_2O$ $Na_2Co(SO_4)_2 \cdot 4H_2O$	crystallized from aq. soln. Na_2SO_4 + $CaSO_4$ at room temp.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 61.
CoO_6Sb_2 $CoSb_2O_6$	CoC_2O_4 + Sb_2O_5 → at 1000°C for 30 min. in Au boat	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 26.
$Co_2K_2O_{12}S_3$ $K_2Co_2(SO_4)_3$	K_2SO_4 + $CoSO_4$ melted at 600°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 35.

CuF_3K $KCuF_4$		add $CuCl_2$ to excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 38.
CuO_6Sb_2 $CuSb_2O_6$		solid state rxn. of CuO + Sb_2O_5 in Ag boat at 945°C in vacuo for 2 hr.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 27.
ErO_4V $ErVO_4$		ErC_2O_4 + V_2O_5 heated at 1100°C for 30 min.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 29.
$FMPO_3$ $BaPO_3F$ $CaPO_3F$ K_2PO_3F Na_2PO_3F	also studied in soln.	Na_2PO_3F + MCl_2; M= Ba,Ca,K	x-ray diffractometry, NMR (^{19}F,^{31}P) spectrometry	first detm. of absolute sign of a spin-coupling constant; high resolution, pulsed and wide-line NMR	T. C. Farrar A. Perloff D. VanderHart	D. L. VanderHart, H. S. Gutowsky, and T. C. Farrar, "NMR Study of $BaFPO_3$: ^{31}P and ^{19}F Chemical-Shift Anisotropies and the Absolute Sign of the F-P Coupling Constant", J. Chem. Phys., 50, 1050 (1969).
F_3FeK $KFeF_3$		aq. soln. $FeCl_2$ + KF → ppt., washed, heated to 400°C in vacuo	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 39.
F_3KMg $KMgF_3$		HF + slurry of K_2CO_3 + $MgCO_3$, evaporated to dryness; x-ray pattern sharpened by heating sample to m.p.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 42.

F_3KMn $KMnF_3$		aq. $MnCl_2$ + excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 45.
F_3MnNa $NaMnF_3$		aq. $MnCl_2$ + excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 65.
F_3NaZn $NaZnF_3$		soln. $ZnCl_2$ + conc. soln. NaF → ppt., washed, annealed at 500°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 74.
$FeNbO_4$		solid state rxn. of Fe_2O_3 + Nb_2O_5 in sealed Pt tube at 1000-1475°C	x-ray powder diffractometry	polymorphism	R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Ixiolite and Other Polymorphic Types of $FeNbO_4$", Am. Mineral., 49, 242 (1964).
GdO_4V $GdVO_4$		$Gd_2(C_2O_4)_3$ + V_2O_5 at 800°C for 15 min.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 30.
$GeO_2 \cdot 9Nb_2O_5$	~ 50μ	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	stable below 1420°C, phase diagram	E. M. Levin	E. M. Levin, "Phase Equilibria in the System Niobium Pentoxide-Germanium Dioxide", J. Res. NBS, 70A, 5 (1966).
Ge_4O_9Pb $PbGe_4O_9$	20-40 μ see $BaGe_4O_9$ under SECTION IV	solid state rxn. of PbO + GeO_2 in sealed Pt tube at 700°C	optical and x-ray powder diffractometry	isostructural with $BaGe_4O_9$ $SrGe_4O_9$	C. R. Robbins	C. R. Robbins and E. M. Levin, "Tetragermanates of Strontium Lead and Barium of Formula Type AB_4O_9", J. Res. NBS, 65A, 127 (1961).

Ge_3O_7Sr $SrGe_3O_7$	20-40 μ	solid state rxn. of SrO + GeO_2 in sealed Pt tube at 1200°C	optical and x-ray powder diffractometry	isostructural with $BaGe_4O_9$, $PbGe_4O_9$	C. K. McMinn	see ref. under Ge_4O_9Pb.
$H_2M_{10}O_{26}P_6$ $Ba_{10}P_6O_{24}(OH)_2$ $Pb_{10}P_6O_{24}(OH)_2$ $Sr_{10}P_6O_{24}(OH)_2$	1-25 μ M = Ba,Pb,Sr	solid state rxn. of BaO, PbO, SrO + P_2O_5 in air at elevated temp.	x-ray diffractometry; infrared, broad-line NMR spectrometry; gravimetric anal.	illustrate nature of non-stoichiometric phases	T. Negas R. S. Roth	T. Negas and R. S. Roth, "High Temperature Dehydroxylation of Apatitic Phosphates", J. Res. NBS, 72A, 783 (1968).
H_4PI PH_4I		PH_3 + HI →	infrared, NMR (^1H, ^{31}P) spectrometry, vapor pressure	phase transitions and molecular motions in solids; toxic, air/moisture sensitive	T. C. Farrar	T. C. Farrar and J. J. Rush, "Nuclear Magnetic Resonance and Neutron-Scattering Studies of Molecular Motions in Phosphomium Iodide", Proc. Second IMR Symp. (Molecular Dynamics and Structure of Solids), NBS Special Publ. 301 (1969), p. 245.
H_4PX PH_4Br PH_4Cl PH_4I	X=Br,Cl,I; volatile solid	PH_3 + HX →	NMR (^1H, ^{31}P), laser Raman spectrometry; vapor pressure, neutron inelastic scattering	model compounds, phase transitions and motions in solids	T. C. Farrar	T. Tsang, T. C. Farrar, and J. J. Rush, "Proton Magnetic Resonance and Hindered Rotation in Phosphonium Halides and Ammonium Iodide", J. Chem. Phys., 49, 4403 (1968). J. J. Rush, A. J. Melveger, T. C. Farrar and T. Tsang, "Laser-Raman Spectra and Hindered Rotation in the Phosphonium Halides", Chem. Phys. Letters, 2, 621 (1968).

H_3K_2Re K_2ReH_9		supplied by A. P. Ginsberg, Bell Telephone Labs., Murray Hill, N. J.	neutron, x-ray diffractometry, NMR (^1H) spectrometry; elemental anal.	study of molecular dynamics of two crystallographically different anions; illustrates different reorientation energies	T. C. Farrar R. B. Johannesen	T. C. Farrar, T. Tsang, and R. B. Johannesen, "Internal Reorientation in K_2ReH_9 via Wide-Line and Pulsed Proton Magnetic Resonance Studies", J. Chem. Phys., 51, 3595 (1969).
IrO_2	powder	oxidation of Ir by heat treatment in air and/or O_2	x-ray diffractometry, phase equilibrium	illustrates behavior of container materials; dissociates at 1020°C in air to $Ir+O_2$; see also O_2Ru	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider,"Phase Relations of the Ru-Ir-O_2 System", J. Res. NBS, 72A, 213 (1968).
$KNaO_4S$ $KNaSO_4$		melt equimolar $K_2SO_4+Na_2SO_4$, annealed overnight at 600°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 50.
$K_2Mg_2O_{12}S_3$ $K_2Mg_2(SO_4)_3$		melt K_2SO_4 + $MgSO_4$ at 1000°C	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 40.
$K_2Mn_2O_{12}S_3$ $K_2Mn_2(SO_4)_3$		melt K_2SO_4 + $MgSO_4$, annealed at 500°C for 15 hr.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 43.
$K_2Na_4O_{12}S_3$ $K_{.67}Na_{1.33}(SO_4)$		melt stoichiometric amts. K_2SO_4 + Na_2SO_4	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 48.

$K_2Ni_2O_{13}S_3$ $K_2Ni_2O_{12}S_3$		K_2SO_4+$NiSO_4$ heated at 750°C, cooled slowly, ground, annealed at 550°C for 30 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 46.
$K_2O_4S_3Zn_2$ $K_2Zn_2(SO_4)_3$		K_2SO_4+$ZnSO_4$ melted, ground, remelted	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 54.
$K_3NaO_8S_2$ $K_3Na(SO_4)_2$	glaserite	stoichiometric K_2SO_4 + Na_2SO_4 melted, annealed at 700°C for 72 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 52.
$LiNaO_4S$ $LiNaSO_4$		equimolar Li_2SO_4 + Na_2SO_4 melted, annealed at 500°C overnight	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 24.
$LiNbO_3$		obtained from CIBA, Rare Metals Div., Summit, N.J.; recrystallized by W. S. Brower, pulled from melt, annealed under O_2 at 1100°C for 10 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 22.
Li_2O_4S Li_2SO_4		$Li_2SO_4 \cdot H_2O$ heated at 600°C for 24 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 26.

LuO_4V $LuVO_4$		soln. Na_3VO_4 + $Lu_2(SO_4)_3$ → ppt., annealed at 1300°C for 15 hr.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 37.
$Na_2NiO_8S_2 \cdot 4H_2O$ $Na_2Ni(SO_4)_2 \cdot 4H_2O$		crystallized from aq. soln. of Na_2SO_4 + $NiSO_4$ at room temp.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 68.
$Na_2O_8S_2Zn \cdot 4H_2O$ $Na_2Zn(SO_4)_2 \cdot 4H_2O$		crystallized from soln. of Na_2SO_4 + $ZnSO_4$ at room temp.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 72.
NbO_4Sb $SbNbO_4$		solid state rxn. of Sb_2O_3 + Nb_2O_5 in sealed Pt tube at 1000-1100°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.
"NbO_5P" "$NbPO_5$" $22Nb_2O_5 \cdot P_2O_5$	"high-low" ~ 50μ	solid state rxn. of Nb_2O_5+P_2O_5 in sealed Pt. tube	x-ray powder diffractometry		E. M. Levin	E. M. Levin and R. S. Roth, "The System Niobium Pentoxide-Phosphorus Pentoxide", J. Solid State Chemistry 2, (1970).
$Nb_{24}O_{62}Ti$ $TiNb_{24}O_{62}$	10-25 μ	solid state rxn. of TiO_2 + Nb_2O_5 at 1450°C	x-ray powder and single crystal diffractometry; crystal structure detm.	systematics of "non-stoichiometry"	R. S. Roth	R. S. Roth and A. D. Wadsley, "Mixed Oxides of Titanium and Niobium: The Crystal Structure of $TiNb_{24}O_{62}$ ($TiO_2 \cdot 12Nb_2O_5$)", Acta Cryst., 18, 724 (1965).

O_2Ru RuO_2	powder	oxidation of Ru by heat treatment in air	x-ray diffractometry, phase equilibrium	illustrates behavior of container materials, dissociates at 1045°C in air to Ru + O_2; see also IrO_2	C. L. McDaniel S. J. Schneider	See ref. under IrO_2.
$(1-x)O_2Th$ + $xCaO$ $0 \leq x \leq 0.10$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	spectroscopic analysis	character of point defect; electrical and mechanical relaxation	J. Wachtman, Jr.	J. B. Wachtman, Jr., "Mechanical and Electrical Relaxation in ThO_2 Containing CaO", Phys. Rev., 131, 517 (1963).
$(1-x)O_2Zr$ + $xCaO$ $.04 \leq x \leq .20$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	porosity, grain size	spans range of stability of cubic solid solution and includes 2-phase region below x=.10 as test of change in character of point defects	J. Wachtman, Jr.	J. B. Wachtman, Jr. and W. C. Corwin, "Internal Friction in ZrO_2 Containing CaO", J. Res. NBS, 69A, 457 (1965).
O_4PrV $PrVO_4$		$Pr_2(C_2O_4)_3$ + V_2O_5 heated at 850°C for 30 min.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 40.
O_4SbTa $SbTaO_4$		solid state rxn. of Sb_2O_3 + Ta_2O_5 in sealed Pt tube at 1000-1200°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.
O_4SbV $SbVO_4$		solid state rxn. of Sb_2O_3 + V_2O_5 in sealed Pt tube at 700-865°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.

O_4SmV $SmVO_4$		$Sm_2(C_2O_4)_3$ + V_2O_5 heated at 850°C for 45 min.	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 47.
O_4TbV $TbVO_4$		$Tb_2(C_2O_4)_3$ + V_2O_5 heated at 1400°C for 1 hr.	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, P. 56.
O_4TmV $TmVO_4$		$Tm_2(C_2O_4)_3$ + V_2O_5 heated at 1400°C for 1 hr.	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 57.
O_4VYb $YbVO_4$		$Yb_2(C_2O_4)_3$ + V_2O_5 heated at 1400°C for 1 hr.	x-ray diffracto-metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4, p. 58.
Pt	high-purity rod, 1/8 in. dia. x 1.5 in.	special prepn. for Office of Standard Reference Materials, proposed SRM # 747	Langmuir detm. of vapor pressure as function of temp.; detm. of heat of sublimation	temp. range 1800-2500K; see also under Ag	R. C. Paule	See ref. under Ag.
		SRM # 747 SRM # 680	vapor pressure Langmuir method	evaluated as container for high-temp. studies (up to 2000K); vapor pressure standard	E. R. Plante	See above ref.
Re	rod, .25 cm. dia. x 1.5 cm.	commercial, zone refined	vapor pressure	container for high-temp. studies	E. R. Plante	E. R. Plante and R. Szwarc, "Vapor Pressure and Heat of Sublimation of Rhenium", J. Res. NBS, 70A, 175 (1966).

$V_2O_5 \cdot 4Y_2O_3$ $4Y_2O_3 \cdot V_2O_5$ $V_2O_5 \cdot 5Y_2O_3$ $5Y_2O_3 \cdot V_2O_5$	~ 50 μ	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	phosphors	E. M. Levin	E. M. Levin, "System Y_2O_3-V_2O_5", J. Am. Ceram. Soc., 50, 381 (1967).
W	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials, proposed SRM # 749	see Pt	see Pt	R. C. Paule	See ref. under Pt.
	polycrystalline rod	commercial	vapor pressure, Langmuir method	vapor pressure standard; container for high-temp. studies	E. R. Plante	R. Szwarc, E. R. Plante, and J. J. Diamond, "Vapor Pressure and Heat of Sublimation of Tungsten", J. Res. NBS, 69A, 417 (1965).

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Al_2O_3-WO_3 entire system	polycrystalline, 1-25 μ	solid state rxn. of components in sealed Pt tubes	phase equilibria; x-ray powder and single crystal diffraction studies	phase equilibria	J. L. Waring	J. L. Waring, "Phase Equilibria in the System Aluminum Oxide-Tungsten Oxide", J. Am. Ceram. Soc., 48, 493 (1965). D. C. Craig and N. C. Stephenson, "A Structural Study in the System Al_2O_3. WO_3", Acta. Cryst., B24, 1250 (1969).
As_2O_5-$9Nb_2O_5$ partial system	polycrystalline, 1-25 μ	solid state rxn. in sealed Pt tube	phase equilibria; x-ray diffraction powder and single crystal structure	illustrates nature of apparent isostructural phases in related systems; see also: As_2O_5-$9Ta_2O_5$, GeO_2-$9Nb_2O_5$, $9Nb_2O_5$-P_2O_5, $9Nb_2O_5$-V_2O_5, $2Nb_2O_5$-Ta_2O_5, P_2O_5-$9Ta_2O_5$, $9Ta_2O_5$-V_2O_5	J. L. Waring R. S. Roth	J. L. Waring and R. S. Roth, "Tetragonal Phases of the General Type $10M_2O_5 \cdot 90M_2O_5$ apparently isostructtural with $Ta_2O_5 \cdot Nb_2O_5$", Acta Cryst., 17, 455 (1964). R. S. Roth and A. D. Wadsley, "The Crystal Structure of PNb_9O_{25} ($P_2O_5 \cdot 9Nb_2O_5$)", Acta Cryst., 18, 643 (1965).
As_2O_5-$9Ta_2O_5$	partial system, see comments and ref. under As_2O_5-$9Nb_2O_5$					
BO_3M MBO_3	solid solns. of rare-earth borates, M = Dy,Er,Eu,Gd, Ho,La,Lu,Nd, Sm,Tm,Y,Yb; polycrystalline	solid state rxns. of mixtures of M_2O_3 + B_2O_3 in sealed Pt tubes	x-ray powder diffractometry; high-temp. x-ray phase equilibria	most specimens exhibit polymorphic inversions	R. S. Roth J. L. Waring E. M. Levin	R. S. Roth, J. L. Waring, and E. M. Levin, "Polymorphism of ABO_3-Type Rare Earth Borate Solid Solutions", Rare Earth Research II, Gordon & Breach, N.Y., (1964), p. 153.

$BaFeO_{3-n}$ BaO· "iron oxide"	$0 \leq n \leq 0.5$; system in the vicinity of 1:1 cation ratio; polycrystalline, 1-25 μ	solid state rxn. in Au tubes under O_2	phase equilibria; x-ray diffractometry	illustrates influence of stoichiometry on structure	T. Negas R. S. Roth	T. Negas and R. S. Roth, "Synthesis of Barium Ferrates in Oxygen", J. Res. NBS, 73A, (1969).
$BaO-Nb_2O_5$ entire system	polycrystalline	solid state rxns. of components in sealed Pt tubes	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Phase Equilibrium Relations in the Binary System Barium Oxide-Niobium Pentoxide", J. Res. NBS, 65A, 337 (1961).
BaO-Pt entire system	powders	solid state rxn. conducted in air in Pt and Au containers	x-ray diffractometry, phase equilibria	all intermediate phases dissociate; illustrates behavior of container material	S. J. Schneider C. L. McDaniel	S. J. Schneider and C. L. McDaniel, "The BaO-Pt System in Air", J. Am. Ceram. Soc., 52, 518 (1969).
$BaSi_2O_5-Ba_2Si_3O_8$ entire system	polycrystalline	solid state rxn.	x-ray powder diffractometry; phase equilibria; single crystal x-ray diffractometry		R. S. Roth E. M. Levin	R. S. Roth and E. M. Levin, "Phase Equilibria in the System Barium Disilicate-Dibarium Trisilicate", J. Res. NBS, 62, 193 (1959). R. S. Roth and E. M. Levin, "Polymorphism in Barium Disilicate", Am. Mineral., 44, 452 (1959).
$Bi_2O_3-Nb_2O_5$ entire system	polycrystalline	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Phase Equilibrium Relations in the Binary System Bismuth Sesquioxide-Niobium Pentoxide", J. Res. NBS, 66A, 451 (1962).

Cr_2O_3-IrO_2 entire system	powders	solid state rxns. in Pt, Vycor, Ir containers, conducted in air	x-ray diffractometry; phase equilibria	illustrates behavior of container materials	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider, "Phase Relations between Cr_2O_3 and IrO_2 in Air", J. Am. Ceram. Soc., 49, 285 (1966).
"CrO_n"-SrO "chromium oxide" - SrO entire system	polycrystalline, 1-25 μ	solid state rxns. in air and O_2	x-ray diffractometry; gravimetric anal.; phase equilibria	illustrates redox behavior of Cr in system	T. Negas R. S. Roth	T. Negas and R. S. Roth, "The System SrO-"chromium oxide" in Air and Oxygen", J. Res. NBS, 73A (1969).
Dy_2O_3-TiO_2	partial system, see comments and ref. under Gd_2O_3-TiO_2					
Er_2O_3-$2TiO_2$	partial system, see comments and ref. under Gd_2O_3-TiO_2					
Eu_2O_3-TiO_2 Eu_2O_3-$2TiO_2$	partial system, see comments and ref. under Gd_2O_3-TiO_2					
Gd_2O_3-TiO_2 entire system	polycrystalline, 1-25 μ	solid state rxn. in unsealed Pt tubes	x-ray diffractometry; polarizing microscopy; phase equilibria	illustrates nature of phases form and relationship to analogous systems; see also: Dy_2O_3-TiO_2, Er_2O_3-$2TiO_2$, Eu_2O_3-TiO_2, Eu_2O_3-$2TiO_2$, Ho_2O_3-$2TiO_2$, Lu_2O_3-$2TiO_2$, Sm_2O_3-TiO_2, $2TiO_2$-Tm_2O_3	J. L. Waring S. J. Schneider	J. L. Waring and S. J. Schneider, "Phase Equilibrium Relationships in the System Gd_2O_5-TiO_2", J. Res. NBS, 69A, 255 (1965).
GeO_2-$9Nb_2O_5$	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$; also see $GeO_2 \cdot 9Nb_2O_5$ in SECTION V					
Ho_2O_3-$2TiO_2$	partial system, see comments and ref. under Gd_2O_3-TiO_2					

IrO_2-SnO_2 entire system IrO_2-TiO_2 entire system	powders	solid state rxns. in Pt, Vycor, and Ir containers, conducted in air	x-ray diffractometry, phase equilibria	illustrates behavior of container materials	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider, "Phase Relations in the Systems TiO_2-IrO_2 and SnO_2-IrO_2 in Air", J. Res. NBS, 71A, 119 (1967).
Ln_2O_3-PdO entire system	powders	solid state rxns. in Pt, Vycor containers	x-ray diffractometry; phase equilibria	all intermediate phases dissociate; illustrates behavior of container materials	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider, "Phase Relations between Palladium Oxide and the Rare-Earth Sesquioxides in Air", J. Res. NBS, 72A, 27 (1968).
Lu_2O_3-$2TiO_2$	partial system, see comments and ref. under Gd_2O_3-TiO_2					
$9Nb_2O_5$-P_2O_5	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$; also see "NbO_5P" in SECTION V					
$2Nb_2O_5$-Ta_2O_5	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$					
Nb_2O_5-V_2O_5 entire system	polycrystalline, 1-25 μ	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; phase equilibria	illustrates nature of multi-phase formation and non-stoichiometric phases; see also $9Nb_2O_5$-V_2O_5	J. L. Waring R. S. Roth	J. L. Waring and R. S. Roth, "Phase Equilibria in the System Vanadium Oxide-Niobium Oxide", J. Res. NBS, 69A, 119 (1964).
$9Nb_2O_5$-V_2O_5	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$; also see Nb_2O_5-V_2O_5					
P_2O_5-$9Ta_2O_5$	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$					
Sm_2O_3-TiO_2	partial system, see comments and ref. under Gd_2O_3-TiO_2					

Ta_2O_5-TiO_2 entire system	polycrystalline	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; high-temp. x-ray powder diffractometry; phase equilibria	illustrates various stable and metastable phases formed in the system	J. L. Waring R. S. Roth	J. L. Waring and R. S. Roth, "Effects of Oxide Additions on the Polymorphism of Tantalum Pentoxide (System Ta_2O_5-TiO_2)", J. Res. NBS, 72A, 175 (1967).
$9Ta_2O_5$-V_2O_5	partial system, see comments and refs. under As_2O_5-$9Nb_2O_5$					
$2TiO_2$-Tm_2O_3	partial system, see comments and ref. under Gd_2O_3-TiO_2					

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