

# THE STRUCTURE OF CRYSTALS

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BY  
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## Preface

This supplement is a summary of new X-ray structure determinations published during the past four years. As such it follows strictly the form used in Part II of the second edition of "The Structure of Crystals" and aims to include all new studies that lead at least to cell dimensions. In order to facilitate comparison, the figure numbers and paragraph designations are continuations of those in the book. As before, reference numbers, with the year in bold-face, apply to the appended bibliography. The grouping of compounds is identical with that previously used except that in the chapter covering the type  $RX_3$  a separate table has been created for crystals of the composition  $R_x(MX_2)_y$ .

The writer is indebted to R. B. Corey and K. Pestrecov for much help in making the illustrations and to A. A. Murtland for assistance in preparing the bibliography.

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## Table of Contents

The chapters in this supplement are all lettered A and correspond in content to the similarly numbered chapters in Part II of the book—second edition.

CHAPTER XA. STRUCTURES OF THE ELEMENTS . . . . .	7
CHAPTER XIA. STRUCTURES OF THE TYPE $RX$ . . . . .	15
CHAPTER XIIA. STRUCTURES OF THE TYPE $RX_2$ . . . . .	23
CHAPTER XIII A. STRUCTURES OF THE TYPE $R_2X_3$ . . . . .	34
CHAPTER XIVA. STRUCTURES OF THE TYPE $RX_3$ , OF HIGHER COM- POUNDS $R_mX_n$ , AND OF NEW COMPOUNDS OF THE TYPE $R_x(MX_2)_y$	37
CHAPTER XVA. STRUCTURES OF THE TYPE $R_x(MX_3)_y$ . . . . .	52
CHAPTER XVIA. STRUCTURES OF THE TYPE $R_x(MX_4)_y$ . . . . .	63
CHAPTER XVII A. STRUCTURES OF THE TYPE $R_x(MX_6)_y$ . . . . .	77
CHAPTER XVIII A. STRUCTURES OF HYDRATES AND AMMONIATES AND OF MISCELLANEOUS INORGANIC COMPOUNDS . . . . .	81
CHAPTER XIX A. STRUCTURES OF THE SILICATES . . . . .	106
CHAPTER XX A. STRUCTURES OF ORGANIC COMPOUNDS . . . . .	130
APPENDIX. A BIBLIOGRAPHY OF CRYSTAL STRUCTURE DATA . . . . .	165



## Chapter XA. Structures of the Elements

Most of the new data of Table I are accurate determinations of the cell dimensions of the metallic elements. For some metals which can easily be prepared in a state of great chemical purity the edge lengths of the units are now known with an accuracy of 0.0002 to 0.0003 Å. The error for most elements is, however, about ten times greater. Spacings to the fourth decimal place have real significance only if the purity is precisely known, if the sample is sufficiently outgassed and otherwise prepared for measurement and if the temperature is determined. It is not always possible to be sure from the published data that all these conditions have been properly met; the accuracy limits stated in Table I are therefore for the most part those set by the investigators themselves.

New information about the atomic arrangements in elements are recorded in the paragraphs that follow.

(v) In place of the previously described tetragonal structure for **gallium** there has recently been given an orthorhombic (pseudo-tetragonal) arrangement based on  $V_h^{18}$  and having its eight atoms in the special positions:

$$(f) \frac{1}{4}uv; \frac{3}{4}, u, \frac{1}{2}-v; \frac{3}{4}, u+\frac{1}{2}, v; \frac{1}{4}, u+\frac{1}{2}, \frac{1}{2}-v; \\ \frac{1}{4}\bar{u}\bar{v}; \frac{3}{4}, \bar{u}, v+\frac{1}{2}; \frac{3}{4}, \frac{1}{2}-u, \bar{v}; \frac{1}{4}, \frac{1}{2}-u, v+\frac{1}{2}$$

with  $u=0.159$ ,  $v=0.080$ . As is evident from Figure 275 (drawn for comparison with Figure 168) this arrangement is very different from the earlier one.

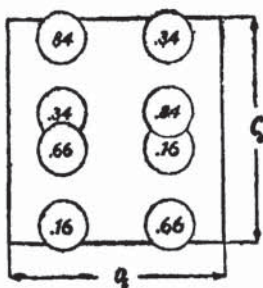


FIG. 275a —(left) The unit cell of the new orthorhombic arrangement found for metallic gallium as projected on its b-face.

FIG. 275b.—(right) A packing drawing of the gallium atoms shown in a.



(w) The rhombohedral structure of **mercury** has recently been confirmed by single crystal measurements at  $-50^{\circ}$  C. At the same time it is shown that the diffraction data upon which a false hexagonal arrangement was based (1922, 1) apparently were a mixture of the lines of mercury and of solid  $\text{CO}_2$ .

(x) Three recent determinations agree with the original in giving **indium** a face-centered tetragonal arrangement with atoms at  $000$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$ .

(y) The X-ray patterns from the  $\beta$ -form of **nitrogen** are thought to indicate that it is a close-packed assemblage of spherical (rotating)  $\text{N}_2$  molecules. The parameter chosen for the center of gravity of these molecules is 0.22.

(z) Hexagonal **nickel** prepared by a glow discharge in  $\text{N}_2$  has the dimensions stated in the table; prepared in  $\text{H}_2$  its unit is larger:  $a_0 = 2.66$  A,  $c_0 = 4.29$  A. It reverts to cubic nickel if heated to  $300^{\circ}$  C.

(aa) This modification of **tungsten**, said to be obtained by electrolyses under various conditions, is thought to have 8 atoms in its unit. The atoms have been placed at (2a) and (6g) (of 1930, 352) though no intensity data are given in support of this assignment.

(ab) The recorded unit cube of  $\alpha$ -**rhodium** is considered to contain 48 atoms. This modification is produced by the reduction of rhodium salts. mixed with the usual form it is also said to result from electrolytic reduction;

(ac) Plastic **sulfur** is rubber-like in that it crystallizes on stretching. The monoclinic unit recorded in the table contains 112 atoms; its space group is given as  $C_{2h}^2$ . As monoclinic sulfur (either stretched or not) ages, the diffraction lines of the orthorhombic form gradually appear.

(ad) The monoclinic cell of  $\alpha$ -**Se** contains 32 atoms; its space group is reported to be  $C_{2h}^2$ . Crystals of a solid solution of selenium and sulfur (55.2 wt. % Se), which presumably are isomorphous with a second modification of monoclinic Se, have been assigned a unit different from that of the  $\beta$ -Se reported in the table. The unit of this mixed crystal has  $a_0 = 8.48$  A,  $b_0 = 13.34$  A,  $c_0 = 8.33$  A,  $\beta = 67^{\circ}30'$ .

(ae) The two **uranium** atoms in the centered unit having the dimensions of the table are said to be at  $000$ ;  $0\frac{1}{2}\frac{1}{2}$ . Another determination has made uranium body-centered cubic with  $a_0 = 3.43$  A (1930, 424).

(af) A second form of **beryllium** is reported to be present to the extent of about 10% in samples which have been heated for some time in vacuo at  $600^{\circ}$ — $800^{\circ}$  C. It is described as hexagonal with  $a_0 = 7.1$  A,  $c_0 = 10.8$  A and with ca 60 atoms in this unit cell (1933, 224).

(ag) Electrodeposition at high  $p_H$  yields  $\beta$ -**cobalt**, at low  $p_H$  a mixture of  $\beta$  and  $\alpha$  forms (1932, 262). The  $\beta$ -cobalt becomes cubic above  $450^{\circ}$  C without change in crystal boundaries; it reverts only on cold rolling or through high temperature annealing (1932, 463).



TABLE I. THE CRYSTAL STRUCTURES OF THE ELEMENTS

<i>Element</i>	<i>Crystal system</i>	<i>Type of structure</i>	$a_0$	$c_0$ or $\alpha$	<i>References</i>
Ag Silver	Cubic	F.c. (a)	4.0772± 0.0002 <sup>1</sup>		1932, 348; 1933, 342; 1934, 199.
Al Aluminum	Cubic	F.c. (a) <sup>2</sup>	4.0406± 0.0002		1931, 8; 1932, 336, 348; 1933, 342, 420.
As Arsenic	Hexagonal	As (e)			1934, 295.
Au Gold	Cubic	F.c. (a)	4.0699± 0.0003 <sup>3</sup>		1932, 348; 1933, 342, 343; 1934, 124, 357.
Be Beryllium	Hexagonal	C.p. (b), (af)	2.2680± 0.0002	3.5942± 0.0003	1932, 329; 1933, 224, 319.
Bi <sup>4</sup> Bismuth	Hexagonal	As (e)			1930, 369, 434; 1931, 420; 1932, 162.
C <sup>5</sup> Graphite	Hexagonal	(h)			1931, 208.
$\beta$ -Ca (>450° C) Calcium	Hexagonal	(ah)	3.98	6.52	1933, 121, 156; 1934, 93.
Cb Columbium	Cubic	B.c. (c)	3.294± 0.001 <sup>6</sup>		1931, 323, 324; 1932, 371; 1934, 43.
Cd Cadmium	Hexagonal	C.p. (b)	2.9736± 0.0005	5.6058± 0.0005 <sup>7</sup>	1931, 238; 1932, 428a.
$\alpha$ -Ce Cerium	Cubic	F.c. (a)	5.143± 0.004		1932, 372.
$\beta$ -Co Cobalt		(ag)			1932, 262.
$\alpha$ -Cr Chromium	Cubic	B.c. (c)	2.8787		1931, 367a; 1932, 369; 1934, 125.
Cu Copper	Cubic	F.c. (a)	3.6077± 0.0002 <sup>8</sup>		1932, 348; 1933, 328, 342; 1934, 357.
$\alpha$ -Fe Iron	Cubic <sup>9</sup>	B.c. (c)	2.8607± 0.0002		1930, 439; 1931, 16; 1932, 62, 336, 369; 1933, 128, 342; 1934, 124, 137.

<sup>1</sup> At 600° C,  $a_0 = 4.1276$  A (1934, 199).

<sup>2</sup> There is no allotropic change up to 600° C (1931, 8; 1933, 420).

<sup>3</sup> At 475° C,  $a_0 = 4.1010$  A.

<sup>4</sup> The thermal expansion from room temperature to the melting point has been carefully measured by X-ray means (1931, 127; 1932, 163; 1934, 120, 121a).

<sup>5</sup> Photographs of incandescent electrodes show that expansion is all normal to the basal plane.

<sup>6</sup> From a very ductile preparation of Cb made by thermal decomposition of  $\text{CbCl}_6$  in vacuo. Other  $a_0$ 's are ca 0.01 A larger.

<sup>7</sup> The other precision measurement (1931, 238) gives lower values:  $a_0 = 2.9724$ ,  $c_0 = 5.6042$ .

<sup>8</sup> At 475° C,  $a_0 = 3.6514$  A.

<sup>9</sup> Measurements have been made up to 1100° C (1930, 439; 1933, 128; 1934, 137).

<i>Element</i>	<i>Crystal system</i>	<i>Type of structure</i>	$a_0$	$c_0$ or $\alpha$	<i>References</i>
Ga Gallium	Ortho- rhombic	( <i>j</i> ), ( <i>v</i> )	4.506	4.506 $b_0=7.642$	1932, 282; 1933, 281.
Hg Mercury	Hexagonal	( <i>k</i> ), ( <i>w</i> )	2.999	70°32' at -46° C	1929, 218; 1932, 209; 1933, 321.
In Indium	Tetragonal	( <i>m</i> ), ( <i>x</i> )	4.583	4.936	1932, 134; 1933, 429, 527.
Ir Iridium	Cubic	F.c. ( <i>a</i> )	3.8312± 0.0005		1932, 348; 1933, 342.
Kr Krypton	Cubic <sup>1</sup>	F.c. ( <i>a</i> )	5.69 at 88° K		1930, 428; 1932, 392.
$\alpha$ -La Lanthanum	Hexagonal	C.p. ( <i>b</i> )	3.75	6.06	1930, 425; 1932, 372.
$\beta$ -La <sup>2</sup> Lanthanum	Cubic	F.c. ( <i>a</i> )	5.296		1933, 528; 1934, 233.
Mg Magnesium	Hexagonal	C.p. ( <i>b</i> )	3.2022± 0.0002	5.1991± 0.0004	1932, 428a.
Mn ( $\alpha$ , $\beta$ , $\gamma$ ) Manganese					1931, 411.
Mo Molybdenum	Cubic	B.c. ( <i>c</i> )	3.140± 0.001		1932, 348.
$\alpha$ -N <sub>2</sub> (<35° K) Nitrogen	Cubic	( <i>o</i> )	5.67		1932, 391.
$\beta$ -N <sub>2</sub> (>35° K) Nitrogen	Hexagonal	( <i>y</i> )	4.039	6.670	1932, 391, 456; 1934, 272.
Nd Neodymium	Hexagonal	C.p. ( <i>b</i> )	3.65 <sub>7</sub>	5.88	1932, 372.
$\alpha$ -Ni Nickel	Cubic	F.c. ( <i>a</i> )	3.5175 <sup>3</sup>		1931, 60; 1932, 62, 348; 1934, 122, 125, 198.
$\beta$ -Ni Nickel	Hexagonal	C.p. ( <i>b</i> ), ( <i>z</i> )	2.60	4.15	1931, 60.
O <sub>2</sub> Oxygen	Ortho- rhombic				1932, 316, 391.
Os Osmium	Hexagonal	C.p. ( <i>b</i> )	2.716	4.331	1932, 434.
Pb Lead	Cubic	F.c. ( <i>a</i> )	4.9396± 0.0003		1931, 420; 1932, 120, 348; 1933, 327, 342; 1934, 192.
Pd Palladium	Cubic	F.c. ( <i>a</i> )	3.8823 <sub>8</sub>		1931, 427; 1932, 348; 1933, 342, 343.

<sup>1</sup> There is no X-ray evidence for a structural transition.

<sup>2</sup> This form appears as a surface layer on the  $\alpha$ -material after vacuum annealing at 350° for several days.

<sup>3</sup> Another determination, on 99.88% Ni, gives  $a_0=3.5143$  A.

<i>Element</i>	<i>Crystal system</i>	<i>Type of structure</i>	$a_0$	$c_0$ or $\alpha$	<i>References</i>
Pr Praseodymium	Hexagonal	C.p. (b)	3.657	5.924	1932, 93, 390.
Pt Platinum	Cubic <sup>1</sup>	F.c. (a)	3.9161± 0.0003		1933, 342, 343; 1934, 199.
Re Rhenium	Hexagonal	C.p. (b)	2.7553± 0.0004	4.4493± 0.0003	1931, 3, 4, 305; 1932, 428a.
$\alpha$ -Rh Rhodium	Cubic	(ab)	9.211		1931, 225, 226.
$\beta$ -Rh Rhodium	Cubic	B.c. (c)	3.7955 <sub>9</sub>		1931, 225; 1932, 348; 1933, 342, 343.
S Sulfur (Plastic form stretched)	Monoclinic	(ac)	26.4 $b_0=9.26, \beta=79^\circ 15'$	12.32	1930, 119a; 1931, 448; 1932, 447; 1934, 175.
Sb Antimony	Hexagonal	As (e) <sup>2</sup>			1932, 263.
Se ( $\alpha$ -form) Selenium	Monoclinic	(ad)	8.992 $b_0=8.973, \beta=91^\circ 34'$	11.52	1931, 152, 153; 1934, 136.
Se ( $\beta$ -form) Selenium	Monoclinic	(ad)	12.74 $b_0=8.04, \beta=93^\circ 4'$	9.25	1934, 136.
Sn (white) Tin	Tetragonal	(s)	5.8194± 0.0003	3.1753± 0.0009	1932, 428a; 1933, 429.
Ta Tantalum	Cubic	B.c. (c)	3.296 <sup>3</sup>		1932, 348, 371; 1934, 43.
Tl ( $\alpha, \beta$ ) Thallium					1931, 411.
U Uranium	Monoclinic	(ae)	2.829 $b_0=4.887, \beta=63^\circ 26'$	3.308	1930, 424; 1933, 507.
W Tungsten	Cubic	B.c. (c)	3.1589		1932, 348; 1933, 318; 1934, 187.
W (second form) Tungsten	Cubic	(aa)	5.038		1933, 318.
X Xenon	Cubic	F.c. (a)	6.24 at 88° K		1930, 220a; 1932, 392.
Y Yttrium	Hexagonal	C.p. (b)	3.66 <sub>2</sub>	5.81 <sub>4</sub>	1932, 370.
Zn <sup>4</sup> Zinc	Hexagonal	C.p. (b)	2.6589	4.9349	1932, 52, 428a; 1933, 337, 339, 493; 1934, 199.
Zr Zirconium	Cubic	B.c. (c)	3.61 near 862° C		1932, 84, 85.

<sup>1</sup> At 600° C,  $a_0=3.9383$  A.

<sup>2</sup> "Explosive" Sb is amorphous; it becomes crystalline on exploding.

<sup>3</sup> This measurement was made upon a very ductile sample prepared by the thermal dissociation of TaCl<sub>5</sub> in vacuo. Results on other material are higher (3.311 A in 1932, 348).

<sup>4</sup> At 415° C,  $a_0 = 2.6792$  A,  $c_0 = 5.0481$  A. There is no structural change up to the melting point.

(*ah*) The evidence concerning the structure of  $\beta$ -calcium (stable above 450° C) was at first contradictory. It is now known that if the metal is pure it is hexagonal close-packed (*b*); if impure it may be either hexagonal or body-centered cubic (*c*) with  $a_0 = ca$  4.33 Å.

### Alloys

A bibliography of new papers describing X-ray measurements on alloy systems is contained in Table II. The structures that occur in metallic systems are of three kinds: (1) solid solutions of one metal in the lattice of another, (2) definite chemical compounds with atoms combined together in stoichiometric proportions, (3) phases with atoms in fixed geometric array but with compositions that can vary over wide limits. Many examples of the second type are described in succeeding chapters but no attempt has been made to summarize the data about structures (1) and (3).

A few non-metallic compounds such as pyrrhotite (FeS) can contain an excess of one or the other of their atomic components; similar compounds, which we often erroneously, from a structural standpoint, describe as being capable of taking one or both of their constituents into solid solution, are common amongst intermetallic compounds. Within recent years another kind of intermetallic compound, the so-called superlattice compound, has become familiar. A superlattice is a relatively complicated atomic arrangement which arises, as an equilibrium state, through the prolonged annealing of an alloy of stoichiometric atomic composition. Such alloys before annealing are usually solid solutions having their atoms in haphazard distribution. Especially simple superlattices are illustrated by the compounds AuCu and AuCu<sub>3</sub>; Sb<sub>2</sub>Tl<sub>7</sub> is a more complicated example.

TABLE II. BIBLIOGRAPHY OF ALLOY SYSTEMS

<i>Alloy system</i>	<i>References</i>	<i>Alloy system</i>	<i>References</i>
Ag-Al	1932, 364; 1933, 2, 29; 1934, 1, 139.	Ag-Sn	1931, 326.
Ag-As	1931, 66.	Ag-Zn	1932, 426, 465; 1933, 341.
Ag-Au	1933, 116, 283, 502.	Al-Au	1931, 244.
Ag-Bi	1931, 66.	Al-Co	1931, 105.
Ag-Cd	1931, 436; 1932, 430; 1933, 450.	Al-Cu	1931, 334, 365; 1933, 328, 357, 358, 436, 447, 546; 1934, 211, 282.
Ag-Cu	1930, 3a; 1931, 96, 418; 1932, 306, 477; 1933, 414, 505.	Al-Fe	1932, 63, 64; 1933, 334; 1934, 313, 313a.
Ag-Hg	1931, 313, 367; 1933, 494.	Al-Li	1931, 348.
Ag-Li	1931, 348.	Al-Mg	1931, 396; 1932, 407; 1933, 414; 1934, 238a, 340.
Ag-Pd	1931, 427; 1933, 275.	Al-Mn	1931, 52.
Ag-Rh	1933, 118.	Al-Ti	1931, 114.
Ag-Sb	1931, 66.		

<i>Alloy system</i>	<i>References</i>	<i>Alloy system</i>	<i>References</i>
Al-Zn	1932, 148, 313, 409, 415; 1934, 197, 351.	Cu-Mg	1934, 242.
As-Cu	1929, 205a, 205b.	Cu-Mn	1931, 411.
As-Sn	1934, 295.	Cu-Ni	1931, 474; 1934, 198.
Au-Cd	1932, 442.	Cu-Pd	1932, 292.
Au-Cu	1931, 366, 386; 1932, 178; 1933, 419; 1934, 85a, 357.	Cu-Si	1931, 19, 389.
Au-Fe	1934, 124.	Cu-Sn	1927, 313; 1932, 94, 229, 422, 426; 1933, 253, 268; 1934, 39, 118.
Au-Mn	1934, 40.	Cu-Zn	1930, 370; 1931, 51; 1932, 264, 268, 349, 350, 432, 465; 1933, 141, 168, 339, 340; 1934, 2, 335, 349.
Au-Pd	1931, 427; 1934, 181.		
Au-Pt	1931, 428.	Fe-H <sub>2</sub>	1933, 501.
Au-Rh	1933, 118.	Fe-Hg	1932, 77.
Au-Sb	1931, 326; 1932, 58.	Fe-Mn	1930, 388; 1931, 125, 342, 406; 1933, 486.
Au-Sn	1931, 426; 1932, 58.	Fe-N	1931, 69; 1933, 324, 510; 1934, 44.
*B-Co	1933, 47.	Fe-Ni	1931, 358; 1932, 234; 1933, 76; 1934, 67.
B-Fe	1933, 47.	Fe-Si	1933, 516.
B-Ni	1933, 47.	Fe-Sn	1933, 125.
Be-Cu	1933, 464.	Fe-V	1930, 456; 1934, 148.
Bi-Pb	1931, 420; 1932, 246; 1934, 82.	Fe-W	1931, 343; 1932, 435.
Bi-Sb	1932, 59; 1934, 76.	Fe-Zn	1931, 105.
Bi-Se	1930, 434.		
Bi-Sn	1931, 420; 1932, 246.	H <sub>2</sub> -La	1934, 233.
Bi-Tl	1931, 411; 1934, 193.	H <sub>2</sub> -Pd	1933, 274, 384.
		H <sub>2</sub> -Ta	1931, 144; 1934, 212.
C-Cr	1930, 455; 1931, 468; 1932, 197; 1933, 401.	H <sub>2</sub> -Ti	1931, 144.
C-Fe	1931, 339, 340, 341, 414; 1932, 225, 281, 474; 1933, 128, 325; 1934, 44, 97, 98, 259, 354.	H <sub>2</sub> -V	1931, 144.
C-Ni	1931, 509; 1933, 415.	H <sub>2</sub> -Zr	1931, 144.
Cd-Cu	1931, 51; 1933, 338.	Hg-Ni	1932, 77.
Cd-Hg	1932, 438.	Hg-Sn	1933, 439.
Cd-Li	1933, 27; 1934, 11, 310.		
Cd-Mg	1930, 72a.	Ir-Os	1932, 434.
Cd-Ni	1931, 105.		
Cd-Pt	1931, 371.	Li-Sn	1932, 20.
Cd-Sb	1930, 379; 1932, 1; 1933, 167.		
Cd-Sn	1931, 295.	Mg-Mn	1931, 397.
Cd-Zn	1932, 52.	Mg-Zn	1932, 407; 1933, 238a, 414, 465.
Ce-H <sub>2</sub>	1934, 233.	Mn-N	1933, 411.
Co-Mn	1934, 137.	Mn-Si	1933, 58; 1934, 337.
Co-W	1932, 4; 1933, 459.	Mn-Zn	1930, 435; 1931, 346; 1932, 353.
Co-Zn	1930, 454; 1931, 105; 1932, 353.	Mo-Ni	1934, 137.
Cr-Fe	1931, 16, 367a; 1932, 369; 1933, 196.		
Cr-Ni	1930, 376; 1934, 125.		
Cu-Fe	1932, 95.		
Cu-Li	1930, 247a.		

<i>Alloy system</i>	<i>References</i>	<i>Alloy system</i>	<i>References</i>
Na-Pb	1933, 451.	Al-C-Fe	1934, 341.
Na-Tl	1932, 500.	Al-Cr-Fe	1932, 399.
Ni-Zn	1930, 454; 1931, 105; 1932, 200; 1933, 85; 1934, 258.	Al-Cu-Mn	1933, 192, 193; 1934, 26, 107.
		Al-Fe-N	1934, 190.
		Au-H <sub>2</sub> -Pd	1934, 181.
Pb-Sb	1933, 327.		
Pb-Sn	1933, 327.	Bi-S-Te	1933, 142.
Pb-Tl	1931, 411; 1934, 192.		
Pd-Zn	1931, 105.	C-Co-Fe	1932, 451.
Pt-Zn	1931, 105.	C-Cr-Fe	1931, 468; 1932, 475.
		C-Cr-Ni	1934, 248.
Rh-Zn	1931, 105.	C-Fe-Mn	1932, 15; 1934, 65.
		C-Fe-W	1931, 343.
Sb-Sn	1931, 49, 224; 1933, 425.	Co-Fe-Mn	1933, 263; 1934, 137.
Sb-Tl	1931, 411; 1934, 180.		
Sb-Zn	1933, 167.	Fe-Ni-V	1934, 148.
Sn-Tl	1931, 411; 1933, 215.	Fe-P-Si	1933, 401.
Ag-Cu-Ni	1934, 210.	Al-C-Cr-Mo	1932, 230.
Ag-H <sub>2</sub> -Pd	1930, 237a; 1933, 275.		

## Chapter XIA. Structures of the Type RX

(ac) For some time there was debate as to whether the rhombohedral unit of **AgCN** contains one or two molecules and whether the correct space group is  $C_{3v}^6$  or  $C_{3v}^6$ . A recent recalculation proves that the cell of Table I is monomolecular with  $C_{3v}^6$  as space group and that the atoms are all on trigonal axes with the coordinates (a)  $uuu$ . Parameters have not been determined.

. **AgBr** and **AgCN** form cubic solid solutions; by extrapolation from measurements on them, it can be concluded that cubic **AgCN** would have  $a_0 = 5.69$  Å if it were stable.

(ad) The low temperature modification of **AuCd**, stable at room temperature, has been assigned a distorted **CsCl** structure based on the orthorhombic space group  $V_h^6$ . The two molecules in its unit have atoms in the positions:

$$\begin{aligned} \text{Cd: (e)} & \quad 0u\frac{1}{4}; 0\bar{u}\frac{3}{4} \text{ with } u = ca \frac{5}{16} \text{ [or } 000; 0\frac{5}{8}\frac{1}{2}] \\ \text{Au: (f)} & \quad \frac{1}{2}v\frac{1}{4}; \frac{1}{2}\bar{v}\frac{3}{4} \text{ with } v = ca \frac{1}{8} \text{ [or } \frac{1}{2}\frac{1}{2}0; \frac{1}{2}\frac{1}{8}\frac{1}{2}]. \end{aligned}$$

(ae) The data on **CdLi** are contradictory. One determination gives it the cubic body-centered **CsCl** structure (a) with  $a_0 = 3.32$  Å; the other assigns to it the **NaTl** superstructure [(ao), below] with  $a_0 = 6.687$  Å.

(af) No diffraction lines have been found to give **CsCN** a unit larger than the one-molecule cell. The available experimental data thus indicate that in this cyanide, as in the other alkali cyanides, the CN group functions geometrically as a single atom.

(ag) It has recently been concluded that the triclinic symmetry previously assigned to **CuO** is unnecessarily low. The proposed arrangement, developed from  $C_{2h}^6$ , has atoms in the following positions:

$$\begin{aligned} \text{Cu: (c)} & \quad \frac{1}{4}\frac{1}{4}0; \frac{3}{4}\frac{1}{4}\frac{1}{2}; \frac{3}{4}\frac{3}{4}0; \frac{1}{4}\frac{3}{4}\frac{1}{2} \\ \text{O: (e)} & \quad 0u\frac{1}{4}; 0\bar{u}\frac{3}{4}; \frac{1}{2}, u+\frac{1}{2}, \frac{1}{4}; \frac{1}{2}, \frac{1}{2}-u, \frac{3}{4} \text{ with } u=0.08. \end{aligned}$$

(ah) A new structure, based on photographic data, has been proposed for **covellite, CuS**. It differs from the earlier one (q) mainly in transferring the two sulfur atoms, which were in (a), to the coordinates (c)  $\frac{1}{2}\frac{2}{3}\frac{1}{2}$ ;

TABLE I. THE CRYSTAL STRUCTURES OF THE COMPOUNDS RX

Substance	Symmetry	Structure type	$a_0$	$c_0$ or $\alpha$	References
AgCN	Hexagonal	( <i>k</i> ), ( <i>ac</i> )	3.88	101°11'	1933, 317; 1934, 288.
AgI	Hexagonal	ZnO ( <i>d</i> ) <sup>1</sup>	4.580	7.494	1931, 46; 1934, 141.
AgI (low)	Cubic <sup>2</sup>	ZnS ( <i>c</i> )	6.473		1931, 46; 1934, 141.
AgI (high)	Cubic	( <i>aw</i> )	5.034		1931, 46; 1934, 257.
AgLi	Cubic	CsCl ( <i>a</i> )	3.168		1930, 247; 1931, 348; 1933, 523.
AlLi	Cubic		3.23		1931, 348.
AlNd	Cubic	CsCl ( <i>a</i> )	3.73		1934, 255.
AuCd (low)	Ortho- rhombic	( <i>ad</i> )	3.144	4.745 $b_0=4.851$	1932, 341.
AuCd (high)	Cubic	CsCl ( <i>a</i> )	3.34 at 400° C		1932, 341.
AuSn	Hexagonal	NiAs ( <i>e</i> )	4.314	5.512	1931, 426; 1932, 58; 1933, 223.
BaNH	Cubic	NaCl ( <i>b</i> )	5.84		1934, 103.
BaO	Cubic	NaCl ( <i>b</i> )	5.523		1933, 80.
$\beta$ -CO	Hexagonal	( <i>at</i> )	4.11	6.79	1934, 272.
CaNH	Cubic	NaCl ( <i>b</i> )	5.006		1933, 137; 1934, 103.
CaTi	Cubic	CsCl ( <i>a</i> )	3.847		1933, 523.
CdLi	Cubic	( <i>ae</i> )	[6.687]		1933, 27, 523.
CdO	Cubic	NaCl ( <i>b</i> )	4.689		1931, 272; 1933, 158.
CdSb	Ortho- rhombic	CdSb ( <i>n</i> ) <sup>3</sup>			1930, 379; 1932, 1.
CoS	Hexagonal	NiAs ( <i>e</i> )	3.38	5.20	1932, 90.
CsCN	Cubic	CsCl ( <i>a</i> ), ( <i>af</i> )	4.25		1931, 319.
CsCl (low)	Cubic	CsCl ( <i>a</i> )	4.20 at ca 450° C		1934, 286.
CsCl (high, >456° C)	Cubic	NaCl ( <i>b</i> )	7.10		1933, 485; 1934, 286.
CsHS	Cubic	CsCl ( <i>a</i> )	4.29		1934, 287.
CuF	Cubic	ZnS ( <i>c</i> )	4.25 <sub>6</sub>		1933, 122.
CuO (Tenorite)	Monoclinic	( <i>o</i> ), ( <i>ag</i> )	4.66	5.09 $b_0=3.40, \beta=99^\circ 30'$	1933, 479.
CuS (Covellite)	Hexagonal	( <i>q</i> ), ( <i>ah</i> )	3.76	16.26	1930, 371; 1932, 342.
(Cu, Fe, Mo, Sn) <sub>4</sub> (S, As, Te) <sub>8-4</sub> (Colusite)	Cubic	ZnS ( <i>c</i> )	5.304		1933, 517.
FeO	Cubic	NaCl ( <i>b</i> ), ( <i>ai</i> ), ( <i>ax</i> )	4.33 <sub>2</sub>		1933, 234, 235; 1934, 77.
FeS	Hexagonal	NiAs ( <i>e</i> ), ( <i>af</i> )			1932, 253; 1933, 162, 165; 1934, 176.
FeSi	Cubic	( <i>t</i> )			1930, 340a.

<sup>1</sup>  $u = 0.37_1$ .<sup>2</sup> Precipitates with excess of Ag ions.<sup>3</sup> According to one analysis there are two forms of CdSb, both hexagonal with identical unit cells (1930, 379).



Substance	Symmetry	Structure type	$a_0$	$c_0$ or $\alpha$	References
GeS	Ortho-rhombic	(ak)	4.29	3.64 $b_0=10.42$	1932, 489.
IIBr (low)	Ortho-rhombic (pseudo-cubic)	(al)	5.555	6.063 $b_0=5.64$	1931, 316, 317; 1932, 392; 1933, 315.
HBr (high)	Cubic	HCl (h)	5.76 at $-170^\circ$ C		1931, 316, 317; 1933, 315.
HCl (low)	Ortho-rhombic	(al)	5.03	5.71 $b_0=5.35$	1931, 316, 317; 1933, 315.
HCl (high)	Cubic	HCl (h)	5.46		1931, 316, 317; 1933, 315.
HI	Tetragonal	(am)	6.19	6.68 at $125^\circ$ K	1931, 316, 317; 1932, 392.
HgF	Tetragonal	Hg <sub>2</sub> Cl <sub>2</sub> (g)	3.66	10.9	1933, 122.
HgLi	Cubic	CsCl (a)	3.287		1933, 523.
KCN	Cubic	NaCl (b), (an)	6.51		1931, 319.
KHS (low)	Hexagonal	NaHS (az)	4.374	$68^\circ 51'$	1934, 287.
KHS (high, above ca $170^\circ$ C)	Cubic	NaCl (b)	6.60		1934, 287.
LiGa	Cubic	NaTl (ao)	6.195		1933, 523.
LiH	Cubic	NaCl (b)			1932, 48.
LiIn	Cubic	NaTl (ao)	6.786		1933, 523.
LiOH	Tetragonal	PbO (f), (ap)	3.546	4.334	1932, 144; 1933, 127.
LiTl	Cubic	CsCl (a)	3.424		1933, 523.
LiZn	Cubic	NaTl (ao)	6.209		1933, 523.
MgPr	Cubic	CsCl (a), (?)	3.88		1933, 387.
MgTl	Cubic	CsCl (a)	3.628		1933, 523.
MgZn	Hexagonal	(aq)	5.33	17.16	1933, 465.
MnO	Cubic	NaCl (b), (ax)			1934, 77.
MnS (red precipitate)	Cubic	ZnS (c)	$5.600 \pm 0.002$		1932, 411; 1933, 417.
MnS (green precipitate)	Cubic	NaCl (b), (ax)	$5.212 \pm 0.002$		1933, 417; 1934, 77.
MnS (red precipitate)	Hexagonal	ZnO (d)	$3.976 \pm 0.002$	$6.432 \pm 0.004$	1932, 411; 1933, 417.
MoC	Hexagonal	(ar)	2.901	2.786	1932, 450.
$\gamma$ -NH <sub>4</sub> Br (at $-100^\circ$ C)	Tetragonal	(ap)	6.007	4.035	1934, 132.
NH <sub>4</sub> HS	Tetragonal	PbO (f), (ap)	6.01	4.01	1934, 287.
NaBi	Tetragonal*		3.46	4.80	1932, 499.
NaCN	Cubic	NaCl (b)	5.83		1931, 319.
NaHS (low)	Hexagonal	NaHS (az)	3.986	$68^\circ 5'$	1934, 287.

\* Said to contain one molecule and probably to be body-centered.

Substance	Symmetry	Structure type	$a_0$	$c_0$ or $\alpha$	References
NaHS (high, above ca 90° C)	Cubic	NaCl (b)	6.05		1934, 287.
NaIn	Cubic	NaTl (ao)	7.297		1933, 526.
NaTl	Cubic	NaTl (ao)	7.473		1932, 500.
NiAs	Hexagonal*	NiAs (e)	3.602	5.009	1933, 130.
NiO	Cubic	NaCl (b)	4.1684± 0.0001		1931, 37, 272; 1933, 87.
NiS	Hexagonal				1931, 264.
PbO (red)	Tetragonal	PbO (f)	3.968	5.011	1932, 120.
PbO (yellow)	Ortho-rhombic	(x)	5.459	5.859 $b_0=4.723$	1932, 120.
PtS (Cooperite)	Tetragonal	(av)	3.47	6.10	1932, 17.
(Pt, Pd, Ni)S (Braggite)	Tetragonal	(as)	6.37	6.58	1932, 17.
PtSn	Hexagonal	NiAs (e)	4.103	5.428	1932, 236; 1933, 223.
RbCN	Cubic	NaCl (b)	6.82		1931, 319.
RbHS (low)	Hexagonal	NaHS (az)	4.525	69°20'	1934, 287.
RbHS (high)	Cubic	NaCl (b)	6.93 at ca 200° C		1934, 287.
SbZn	Ortho-rhombic	CdSb (n)	6.17	3.94 $b_0=8.27$	1933, 167.
SiC (II)	Hexagonal	(y), (au)	3.076	15.07	1932, 205; 1933, 59.
SnAs	Cubic	NaCl (b), (ay)	5.681		1934, 295.
SnO	Tetragonal	PbO (f)			1932, 469.
SrNH	Cubic	NaCl (b)	5.45		1934, 103.
SrO	Cubic	NaCl (b)	5.144		1933, 80.
SrTl	Cubic	CsCl (a)	4.024		1933, 523.
TaC	Cubic	NaCl (b)	4.4460± 0.0005		1933, 424; 1934, 42.
TiC	Cubic	NaCl (b)	4.320		1931, 59; 1932, 414; 1934, 42.
TiCN	Cubic	CsCl (a)	3.82		1934, 256a.
TiCl	Cubic	CsCl (a)	The value $a_0=3.380$ in 1933, 302 is undoubtedly a misprint.		
VO	Cubic	NaCl (b)	4.08		1932, 302.
WC	Hexagonal		2.910	2.838	1931, 343.
ZnO	Hexagonal†	ZnO (d)	3.248	5.203	1933, 218.
ZrC	Cubic	NaCl (b)	4.687		1934, 42.

\* There is no change in structure below 600° C.

† No change in structure between 110° and 1300° C.

$\frac{2}{3}\frac{1}{3}\frac{3}{4}$ . The other atoms are similarly placed in both structures. The new copper parameter  $u=0.107$  in (f)  $\frac{1}{3}\frac{2}{3}u$ ; etc. is nearly the same as the old; the sulfur parameter  $v=0.062$  in (e)  $00v$ ; etc. is considerably different. This new structure has the atomic separations characteristic of neutral atoms (Cu-S=2.20–2.35 Å, S-S=2.05 Å).

(ai) The values of  $a_o$  for specimens of **FeO**, which invariably are deficient in iron, increase with the amount of iron present. For an oxide containing 76.08% Fe,  $a_o=4.2816$  A; for a sample with 76.72% Fe,  $a_o=4.3010$  A. By extrapolation pure FeO (77.73% Fe) would have  $a_o=4.332$  A.

(aj) **Pyrrhotite** has the composition  $Fe_1S_{(1+x)}$  not by reason of the presence of an excess of sulfur but because some of the iron atoms are missing from their structural positions. It is said that pure FeS gives evidence of a superlattice containing 12 molecules; the  $a_o$  of this lattice is the diagonal of  $a_o$  for the simple cell, its  $c_o$  is twice as great:  $a_o=5.946$  A,  $c_o=11.720$  A.

Ordinary pyrrhotite ( $a_o=3.41$  A,  $c_o=5.72$  A) becomes ferromagnetic if heated above 200° C; this form, giving  $a_o=3.47$  A,  $c_o=5.84$  A at room temperature, reverts to the non-magnetic form if heated above 450° C (1934, 176).

(ak) From photographic data it has been concluded that the four molecules of **GeS** are in special positions (c) of the space group  $V_h^{16}$ :

$$\text{Ge: (c) } uv0; \bar{u}, \frac{1}{2}-v, \frac{1}{2}; u+\frac{1}{2}, \bar{v}, \frac{1}{2}; \frac{1}{2}-u, v+\frac{1}{2}, 0$$

with  $u=0.167$ ,  $v=-0.125$

$$\text{S: (c) } u'v'0; \text{ etc. with } u'=v'=0.111.$$

The axes  $X'Y'Z'$  of 1930, 352 bear the following relation to the axes  $abc$  of this description:  $a=Z'$ ,  $b=Y'$ ,  $c=X'$ . The kind of packing that prevails is illustrated by Figure 276a and b.

FIG. 276a.—The unit cell of the structure of **GeS** projected on an  $a$ -face. The large circles are sulfur.

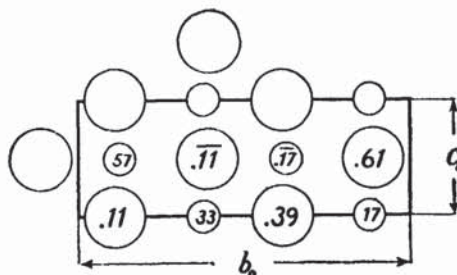
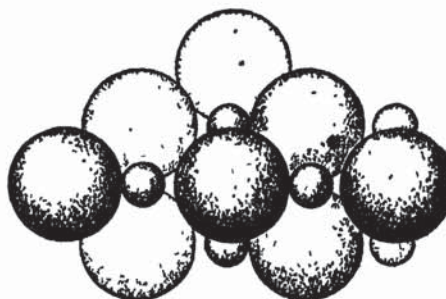


FIG. 276b.—A packing drawing of  $a$  with Ge and S atoms given their ionic sizes. In making these packing drawings it is sometimes better to show an atom at a height  $1+x$  instead of the equivalent atom of height  $x$ . Thus in this figure the central sulfur atom is at  $1-0.11=0.89$  (and not at  $-0.11$ ). Similarly its neighboring Ge is at  $0.83$  instead of  $-0.17$ , as in  $a$ .

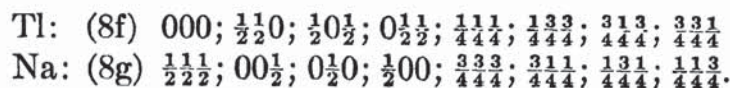


(al) Earlier work described the low temperature modifications of **HCl** and **HBr** as tetragonal. The more recent experiments, however, make them orthorhombic pseudo-cubic with four molecules in the unit. The space group of low **HBr** is thought to be either  $V^7$  or  $V_h^{23}$ .

(am) Cubic **HI** is not cubic, as first stated, but tetragonal. It is said that its transitions do not involve atomic rearrangements.

(an) A reexamination of **KCN** has failed to supply data indicating a unit larger than that of the four-molecule **NaCl**-like grouping.

(ao) The **NaTl** superlattice, found for a number of alloys, contains eight molecules with atoms in the following special positions of  $O_h^7$ :



(ap) Three additional crystals have been found to have the **PbO** (*f*) type of structure but with parameters and cells so different that other atomic relationships are produced.

Of these substances **LiOH** is most like **PbO**. Lithium atoms are in (a)  $000; \frac{1}{2}\frac{1}{2}0$ , OH groups are at (c)  $0\frac{1}{2}u; \frac{1}{2}0\bar{u}$  with *u* said to be 0.20. If *u* is really so small there is a surprisingly large separation (ca 3.5 Å) between the OH ions of adjacent layers.

The other two crystals with this grouping, **NH<sub>4</sub>HS** and the  $\gamma$ -form of **NH<sub>4</sub>Br**, photographed at  $-100^\circ\text{C}$ , have identical units. Since in most compounds the HS ion has practically the same size as the bromide ion, it might be expected that the parameters defining them would be little different. In view of this fact it will be interesting to learn from future work whether the unlike parameters found for the bromide and hydro-sulfide are both right. Expressing the atomic positions in the coordinates used for **LiOH** (above) and for **PbO**,  $u(\text{HS})=0.66$  for **NH<sub>4</sub>HS**; for  $\gamma$ -**NH<sub>4</sub>Br**,  $u(\text{Br})=0.53$ . A drawing of the bromide is reproduced in Figure 277a and b for comparison with the **PbO** packing illustrated in Figure 176.

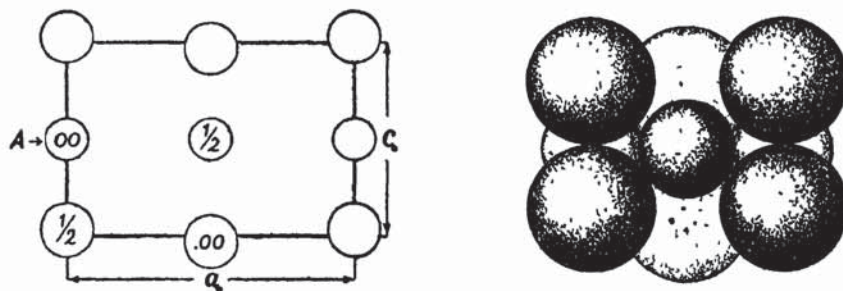


FIG. 277a.—(left) The variant of the tetragonal **PbO** (*f*) arrangement provided by the  $\gamma$ -form of **NH<sub>4</sub>Br**. Small circles are **NH<sub>4</sub>** groups; the origin is in the **NH<sub>4</sub>** ion at A.

FIG. 277b.—(right) A packing drawing of  $\gamma$ -**NH<sub>4</sub>Br** with the **NH<sub>4</sub>** group shown as the smaller sphere. It is evident that good packing results if, as in this drawing, the atoms are given their usual ionic sizes.

(aq) It is said that the structure of **MgZn** resembles that of **MgZn<sub>2</sub>** [see Chapter XII, (ab)] with two magnesium atoms in place of zinc atoms and with  $c_0$  doubled in length.

(ar) The molybdenum atom of the single **MoC** molecule in the unit is at the origin (000); the carbon atom is thought to be at  $\frac{1}{3}\frac{2}{3}\frac{1}{2}$ .

(as) The cell of **braggite** is reported to contain eight molecules and to have an arrangement based on  $D_{4h}^2$ . The sample that was studied contained 20% Pd and 5% Ni.

(at) The  $\beta$ -form of **CO**, stable above 61.5° K, has the same structure as  $\beta$ -nitrogen. If the arrangement found for  $\beta$ -N<sub>2</sub> is correct, this modification of CO, too, has rotating molecules in close-packed array.

(au) A Fourier analysis has been made of the basal reflections from an unspecified type of **SiC** (1932, 205).

(av) The tetragonal cell of the mineral **cooperite**, **PtS**, has been described as containing two molecules. Its atoms are thought to be in the following special positions of  $D_{4h}^9$ : Pt: (c)  $0\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ , S: (e)  $00\frac{1}{4}$ ;  $00\frac{3}{4}$ .

(aw) The unit cube of the modification of **AgI** stable above 146° C is supposed to contain two molecules. Iodine atoms are reported to be at 000;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ ; silver is described as occupying two of the 30 largest holes resulting from this iodine packing. Such an unusual structure needs further confirmation.

(ax) Measurements of  $a_0$  for cubic MnO at low temperatures show that there is no change in structure around 160° K where an anomalous heat change occurs. At this point there is, however, a minimum in the cubic edge length. Magnetite, Fe<sub>3</sub>O<sub>4</sub>, behaves similarly. MnS and FeO likewise do not have different crystal structures above and below the temperatures at which they show thermal anomalies. Neither do they have a region in which they contract on warming; instead they show two different rates of thermal expansion. The data for these conclusions are given in Table II.

TABLE II. VALUES OF  $a_0$  AT SEVERAL TEMPERATURES

Temperature	$a_0$ for Compound			
	MnO	Fe <sub>3</sub> O <sub>4</sub>	MnS	FeO
299° K	4.436	8.363	5.210	4.290
200	—	—	—	4.286
186	—	—	—	4.284
160	4.409	8.357	5.204	4.283
143	—	—	5.204	—
138	—	—	5.197	—
130	—	—	5.192	—
114	4.416	8.363	—	—
104	4.419	8.363	—	—

(ay) The compound **SnAs** is especially interesting in being an example of the NaCl grouping which is capable of "dissolving" both components, the As-rich limit being at 49% As, the Sn limit at 34.5% As. The pure compound has the lowest value of  $a_0$ .

(az) The one-molecule rhombohedron of the form of the alkali hydro-sulfides stable at room temperatures is very different in shape from that found for AgCN. Atomic parameters are not known for any of these crystals.

## Chapter XIIA. Structures of the Type $RX_2$

(as) The monoclinic unit of  $Ag_2Te$  has been said to contain three molecules; this is improbable. A sample heated to  $250^\circ C$  and cooled in nitrogen showed no change in pattern.

(at) The diffraction lines of  $BeF_2$ , which were not very sharp, are reported to be those of a tetragonal high cristobalite-like ( $ae, bd$ ) structure containing eight molecules.

(au) The atomic arrangement assigned to  $COS$  on the basis of low temperature powder photographs is developed from  $C_{3v}^5$ . The atoms in its single molecule rhombohedron are on three-fold axes with the coordinates  $uuu$ . For C,  $u=0$ ; for S,  $u'=0.33_6$ ; for O,  $u_1=-0.18_7$ . The resulting interatomic distances within the molecule are C-O=1.10 A, C-S=1.96 A; between different molecules O-S=2.78 A.

(av) The astonishingly large unit assigned to  $Cu_2S$  would contain 160 molecules.

(aw) Marcasite ( $FeS_2$ ) and löllingite ( $FeAs_2$ ) have been given smaller unit cells. These cells contain two molecules with atoms arranged according to  $V_h^{12}$ . Iron atoms are in (a)  $000; \frac{1}{2}\frac{1}{2}\frac{1}{2}$ , sulfur (or arsenic) atoms in (g)  $0uv; 0\bar{u}\bar{v}; \frac{1}{2}, \frac{1}{2}-u, v+\frac{1}{2}; \frac{1}{2}, u+\frac{1}{2}, \frac{1}{2}-v$ . The axes,  $abc$ , of this description and  $X'Y'Z'$  of 1930, 352 are connected by the relation  $a=Z'$ ,  $b=X'$ ,  $c=Y'$ . For  $FeS_2$ ,  $u=0.203$ ,  $v=0.375$ ; for  $FeAs_2$ ,  $u'=0.175$ ,  $v'=0.361$ ; for the more recently studied phosphide  $FeP_2$ ,  $u''=0.16$ ,  $v''=0.37$ . The grouping of marcasite, as typical of this structure, is illustrated in Figure 278a and b. It consists of open meshworks of iron atoms and

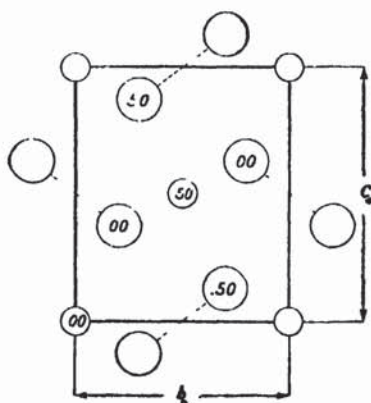


FIG. 278a.—(left) The new atomic arrangement deduced for marcasite,  $FeS_2$ , as projected upon the  $a$ -face of its orthorhombic cell. The small circles are Fe atoms.

FIG. 278b.—(right) A packing drawing of  $FeS_2$  if Fe and S are shown with the radii of their neutral atoms. The line-shaded atoms are Fe.



TABLE I. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $RX_2$ 

Substance, symmetry and structure type			$a_o$	$c_o$ or $a$	References
Ag <sub>2</sub> S (Acanthite)	Ortho- rhombic	(bf)	4.77	6.88	1931, 124, 345.
				$b_o=6.92$	
Ag <sub>2</sub> S (Argentite)	Cubic	Cu <sub>2</sub> O (j), above 180° C (bf)	4.90		1931, 124, 345.
Ag <sub>2</sub> Te	Monoclinic	(as)	5.98	5.56	1932, 444.
				$b_o=6.31, \beta=75^\circ 2'$	
Al <sub>2</sub> Au	Cubic	CaF <sub>2</sub> (a)	6.00		1932, 292.
AlCuMg	Hexagonal	MgZn <sub>2</sub> (ab)	5.09	8.35	1934, 339.
Au <sub>2</sub> Pb	Cubic	MgCu <sub>2</sub> (q)	7.91		1934, 209a.
AuSb <sub>2</sub>	Cubic	FeS <sub>2</sub> (f)	6.647		1931, 326; 1932, 58; 1933, 223.
BaF <sub>2</sub>	Cubic	CaF <sub>2</sub> (a)	6.187		1933, 422.
Be <sub>2</sub> C	Cubic	CaF <sub>2</sub> (a)	4.33		1931, 425; 1934, 354b.
Be <sub>2</sub> Cu	Cubic	MgCu <sub>2</sub> (q)	5.94		1934, 339.
BeF <sub>2</sub>	Tetragonal	(at)	6.60	6.74	1932, 70.
Bc <sub>2</sub> Fe	Hexagonal	MgZn <sub>2</sub> (ab)	4.22	6.83	1934, 339.
CO <sub>2</sub>	Cubic	CO <sub>2</sub> (g), (bg)	5.575 at -190° C		1931, 458, 459; 1934, 130, 130a.
COS	Hexagonal	(au)	4.08	98°58'	1931, 457.
				at liquid air temp.	
CaF <sub>2</sub>	Cubic	CaF <sub>2</sub> (a)	5.451		1933, 422.
CaI <sub>2</sub>	Hexagonal	CdI <sub>2</sub> (c), (bi)	4.48	6.96	1933, 52.
CdBr <sub>2</sub>	Hexagonal	(bn)			1933, 43.
CdCl(OH)	Hexagonal	(bh)	3.66	10.27	1934, 110.
CdI <sub>2</sub>		(c), (bi)			1932, 8; 1933, 174.
CoCl <sub>2</sub>	Hexagonal*	CdCl <sub>2</sub> (e)	6.16	33°26'	1934, 95.
CuF <sub>2</sub>	Cubic	CaF <sub>2</sub> (a)	5.406		1933, 122.
Cu <sub>2</sub> Mg	Cubic	(q), (bs)			1934, 242.
Cu <sub>2</sub> O	Cubic	Cu <sub>2</sub> O (j)	4.252		1931, 321, 322; 1932, 483.
Cu <sub>2</sub> S	Ortho- rhombic	(av)	11.8	22.7	1930, 371.
				$b_o=27.2$	
FeAs <sub>2</sub>	Ortho- rhombic	FeAs <sub>2</sub> (i), (aw)	2.85	5.92	1932, 83.
				$b_o=5.25$	
Fe <sub>2</sub> B	Tetragonal	(t), (bp)			1930, 116a; 1931, 146.
FeOCl	Ortho- rhombic	(bj)	3.75	3.3	1934, 84.
				$b_o=7.95$	
FeP <sub>2</sub>	Ortho- rhombic	FeAs <sub>2</sub> (aw)	2.725	5.657	1934, 173.
				$b_o=4.975$	
FeS <sub>2</sub>	Cubic	FeS <sub>2</sub> (f), (bk)	5.405		1932, 352.
FeS <sub>2</sub>	Ortho- rhombic	FeAs <sub>2</sub> (i), (aw)	3.37	5.39	1931, 71.
				$b_o=4.44$	
GeO <sub>2</sub>	Tetragonal†	SnO <sub>2</sub> (b)	4.390	2.895	1932, 164.

("Insoluble" form)

\* For CoCl<sub>2</sub>,  $u=0.25$ .† Parameter  $u=0.3$ .



Substance, symmetry and structure type			$a_o$	$c_o$ or $a$	References
H <sub>2</sub> O	Hexagonal	( <i>x</i> ), ( <i>bl</i> )	4.508	7.338	1933, 257; 1934, 16.
			at -66° C		
H <sub>2</sub> S	Cubic	( <i>al</i> ), ( <i>ax</i> )			1931, 315, 456.
H <sub>2</sub> Se	Cubic	( <i>al</i> ), ( <i>ax</i> )			1931, 315, 456.
HgBr <sub>2</sub>	Ortho- rhombic	( <i>ay</i> )	6.85	12.45	1931, 461; 1932, 66.
			$b_o=4.67$		
HgCl <sub>2</sub>	Ortho- rhombic	( <i>y</i> ), ( <i>bm</i> )	5.963	4.324	1932, 332; 1934, 316.
			$b_o=12.735$		
HgF <sub>2</sub>	Cubic	CaF <sub>2</sub> ( <i>a</i> )	5.54		1933, 122.
HgI <sub>2</sub>	Ortho- rhombic	HgBr <sub>2</sub> ( <i>ay</i> )	7.32	13.76	1934, 85.
			$b_o=4.674$		
KBi <sub>2</sub>	Cubic	Cu <sub>2</sub> Mg ( <i>q</i> )	9.501		1932, 501.
K <sub>2</sub> O	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.436		1934, 309.
K <sub>2</sub> S	Cubic	CaF <sub>2</sub> ( <i>a</i> )	7.391		1934, 287, 309.
K <sub>2</sub> Se	Cubic	CaF <sub>2</sub> ( <i>a</i> )	7.676		1934, 309.
K <sub>2</sub> Te	Cubic	CaF <sub>2</sub> ( <i>a</i> )	8.152		1934, 309.
Li <sub>2</sub> O	Cubic	CaF <sub>2</sub> ( <i>a</i> )	4.619		1934, 309.
Li <sub>2</sub> S	Cubic	CaF <sub>2</sub> ( <i>a</i> )	5.708		1934, 309.
Li <sub>2</sub> Se	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.005		1934, 287, 309.
Li <sub>2</sub> Te	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.504		1934, 287, 309.
Mg <sub>2</sub> Ge	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.378		1933, 525.
MgI <sub>2</sub>	Hexagonal	CdI <sub>2</sub> ( <i>c</i> ), ( <i>bi</i> )	4.14	6.88	1933, 52.
MgNi <sub>2</sub>	Hexagonal	MgZn <sub>2</sub> ( <i>ab</i> )	4.81	7.95	1934, 339.
MgNiZn	Cubic	MgCu <sub>2</sub> ( <i>q</i> )	6.96		1934, 339.
Mg <sub>2</sub> Pb	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.836		1933, 525.
Mg <sub>2</sub> Sn	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.765		1933, 525.
MgZn <sub>2</sub>	Hexagonal	( <i>ab</i> )			1934, 260.
MnS <sub>2</sub> (Hauerite)	Cubic	FeS <sub>2</sub> ( <i>f</i> )	6.097*		1932, 346, 411; 1933, 417; 1934, 204, 346.
Mo <sub>2</sub> C	Hexagonal	( <i>az</i> )	2.994	4.722	1932, 450.
N <sub>2</sub> O	Cubic	CO <sub>2</sub> ( <i>g</i> )	5.656		1931, 458, 459.
NO <sub>2</sub>	Cubic	( <i>an</i> ), ( <i>ba</i> )			1931, 170, 454, 455.
Na <sub>2</sub> S	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.526		1934, 309.
Na <sub>2</sub> Se	Cubic	CaF <sub>2</sub> ( <i>a</i> )	6.809		1934, 309.
Na <sub>2</sub> Te	Cubic	CaF <sub>2</sub> ( <i>a</i> )	7.314		1934, 309.
NdC <sub>2</sub>	Tetragonal	CaC <sub>2</sub> ( <i>k</i> )	3.81	6.36	1931, 425.
NiBr <sub>2</sub>	Hexagonal	CdCl <sub>2</sub> ( <i>e</i> ), ( <i>bn</i> )	6.46	33°20'	1934, 134.
NiI <sub>2</sub>	Hexagonal	CdCl <sub>2</sub> ( <i>e</i> )	6.92	32°40'	1934, 134.
Ni(OH) <sub>2</sub>	Hexagonal	CdI <sub>2</sub> ( <i>c</i> )	3.114	4.617	1933, 87.
OsS <sub>2</sub>	Cubic	FeS <sub>2</sub> ( <i>f</i> )	5.6075± 0.0006		1934, 174.
PbBr <sub>2</sub>	Ortho- rhombic	PbBr <sub>2</sub> ( <i>y</i> ), ( <i>bb</i> )			1932, 332.
PbFBr	Tetragonal	PbFBr ( <i>bc</i> )	4.18	7.59	1932, 331.
PbCl <sub>2</sub>	Ortho- rhombic	( <i>y</i> ), ( <i>bb</i> )	4.525	9.030	1931, 302; 1932, 69.
			$b_o=7.608$		
PbFCl	Tetragonal	( <i>bc</i> )	4.09	7.21	1932, 330; 1933, 323; 1934, 9.

\*  $u=0.4012$ .

Substance, symmetry and structure type			$a_o$	$c_o$ or $a$	References
$\alpha$ -PbF <sub>2</sub>	Ortho-rhombic	(y), (bb)	3.80	7.61	1932, 269; 1933, 422.
$\beta$ -PbF <sub>2</sub>	Cubic*	CaF <sub>2</sub> (a)	5.942± 0.002	$b_o=6.41$	1933, 422.
PbO <sub>2</sub>	Tetragonal	SnO <sub>2</sub> (b)	4.931	3.367	1932, 120.
PdF <sub>2</sub>	Tetragonal	SnO <sub>2</sub> (b)	4.93	3.38	1931, 100.
PrC <sub>2</sub>	Tetragonal	CaC <sub>2</sub> (k)	3.85	6.41	1931, 425.
PtAs <sub>2</sub> (Sperrylite)	Cubic	FeS <sub>2</sub> (f), (br)			1932, 17.
RuS <sub>2</sub> (Laurite)	Cubic	FeS <sub>2</sub> (f), (br)	5.59		1932, 17.
SaC <sub>2</sub>	Tetragonal	CaC <sub>2</sub> (k)	3.75	6.28	1931, 425.
SiO <sub>2</sub> ( $\alpha$ -Quartz, low)	Hexagonal	(l)	4.9029	5.3933	1930, 375; 1933, 62, 232, 277; 1934, 121a.
SiO <sub>2</sub> ( $\alpha$ -Cris- tobalite, low)	Ortho-rhombic	(bq)	7.00	7.00	1932, 25.
SiO <sub>2</sub> ( $\beta$ -Cris- tobalite, high)	Cubic	(ae), (bd)		$b_o=7.00$	1932, 24.
SrC <sub>2</sub>	Tetragonal	CaC <sub>2</sub> (k)	4.11†	6.68	1930, 299.
SrF <sub>2</sub>	Cubic	CaF <sub>2</sub> (a)	5.784		1933, 422.
Ta <sub>2</sub> C	Hexagonal	(az)	3.091	4.93	1934, 42.
YC <sub>2</sub>	Hexagonal	(bo)	3.79	6.58	1931, 425.
Zn(OH) <sub>2</sub>	Hexagonal	CdI <sub>2</sub> (c), (?)	3.14	ca 5.12	1932, 146a.
Zn(OH) <sub>2</sub>	Ortho-rhombic	(ai), (be)	8.53	4.92	1933, 104.
ZrW <sub>2</sub>	Cubic	Cu <sub>2</sub> Mg (q)	7.61	$b_o=5.16$	1933, 95.

\* Transition between 220°–280° C.

†  $a_o=5.81$  A as given in book, p. 239, applies to a larger diagonal cell.

sulfur pairs layered normal to the a-axes. The atomic contacts are perfect if iron and sulfur are given their radii as neutral atoms.

(ax) The patterns of H<sub>2</sub>S and H<sub>2</sub>Se indicate that the sulfur and selenium atoms are in face-centered array; nothing can of course be told about the hydrogen positions.

(ay) The four molecules in the unit of HgBr<sub>2</sub> are arranged according to the space group C<sub>2v</sub><sup>12</sup>. All atoms are in special positions (a)  $u0v; \bar{u}, 0, v+\frac{1}{2}; u+\frac{1}{2}, \frac{1}{2}, v; \frac{1}{2}-u, \frac{1}{2}, v+\frac{1}{2}$ , with the parameters: for Hg,  $u=0.33_4, v=0$ ; for Br',  $u'=0.05_6, v'=0.13_2$ , for Br'',  $u''=0.38_9, v''=0.36_8$ . As Figure 279 shows, this arrangement is a typical layer-like structure, contact between the layers being maintained through bromine atoms. If the atoms are given their usual ionic sizes, as in the packing drawing, excellent contacts result.

Yellow mercuric iodide apparently has this  $HgBr_2$  grouping.

(az) The two molybdenum atoms in the unit cell of  $Mo_2C$  are said to be at  $\frac{1}{3}\frac{2}{3}\frac{1}{4}$ ;  $\frac{2}{3}\frac{1}{3}\frac{3}{4}$ ; it is thought that the carbon atom may be at the origin 000. A tantalum carbide,  $Ta_2C$ , has the same hexagonal close-packing of its metal atoms; like  $W_2C$  it has a second modification.

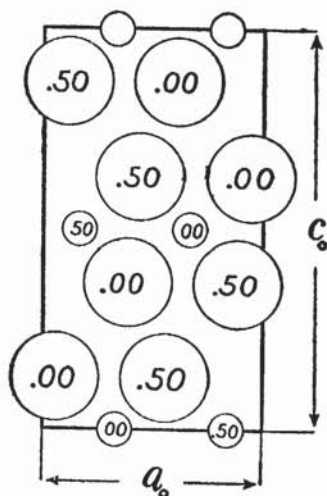
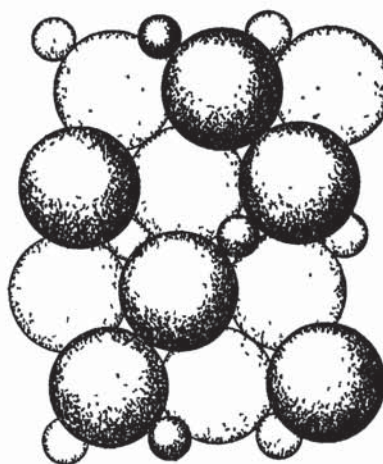


FIG. 279a.—(left) The layer structure of  $HgBr_2$  projected on the b-face of its orthorhombic unit. The large circles are Br.

FIG. 279b.—(right) A packing drawing of  $HgBr_2$  with the atoms having their ionic sizes.



(ba) The unit cube of solid  $NO_2$  contains 12 molecules. Two conflicting structure types have been proposed using the same data (1931, 454). In one of these, based on  $T^5$ , the nitrogen atoms are in  $(12c)$   $u\frac{1}{2}$ ; etc. with  $u=0.40_3$ , the oxygen atoms in general positions  $xyz$ ; etc. with  $x=0.17_8$ ,  $y=0.25$ ,  $z=0.40_3$ . This gives  $NO_2$  molecules with an N-O separation of 1.38 Å. The other discussion proceeds on the assumption that the solid ought to show  $N_2O_4$  molecules. It is pointed out (1931, 170) that this can result if the space group is  $T^3$  with oxygen atoms in general positions and nitrogen atoms in  $(12a)$  or  $(12b)$ . The atomic positions have not been determined for such an arrangement but it is considered that the evidence favors planar molecules.

(bb) In the structures found for  $PbBr_2$  and  $PbCl_2$  all the atoms are in special positions (c) of  $V_h^{18}$ :  $0uv$ ;  $\frac{1}{2}$ ,  $\frac{1}{2}-u$ ,  $\bar{v}$ ;  $0$ ,  $u+\frac{1}{2}$ ,  $\frac{1}{2}-v$ ;  $\frac{1}{2}$ ,  $\bar{u}$ ,  $v+\frac{1}{2}$ . In  $PbBr_2$ ,  $u(Pb)=0.01_6$ ,  $v(Pb)=0.08_7$ ; for  $Br'$ ,  $u'=0.61$ ,  $v'=0.07_6$ , for  $Br''$ ,  $u''=0.23$ ,  $v''=-0.17$ . The different parameters given to the atoms in  $PbCl_2$  are to be attributed to the choice of another origin. These parameters are: for Pb,  $u=0.25_4$ ,  $v=0.09_5$ ; for  $Cl'$ ,  $u'=0.65$ ,  $v'=0.07$ , for  $Cl''$ ,  $u''=0.55$ ,  $v''=0.67$ . The extreme layer-like nature of the structure possessed by both salts is illustrated by Figure 280; in b the atoms have been drawn with their customary ionic sizes. The packing is far less perfect than that found for  $HgBr_2$  (Figure 279); but very possibly more accurate intensity data upon these lead salts would alter the chosen parameters enough to give them better ionic contacts.

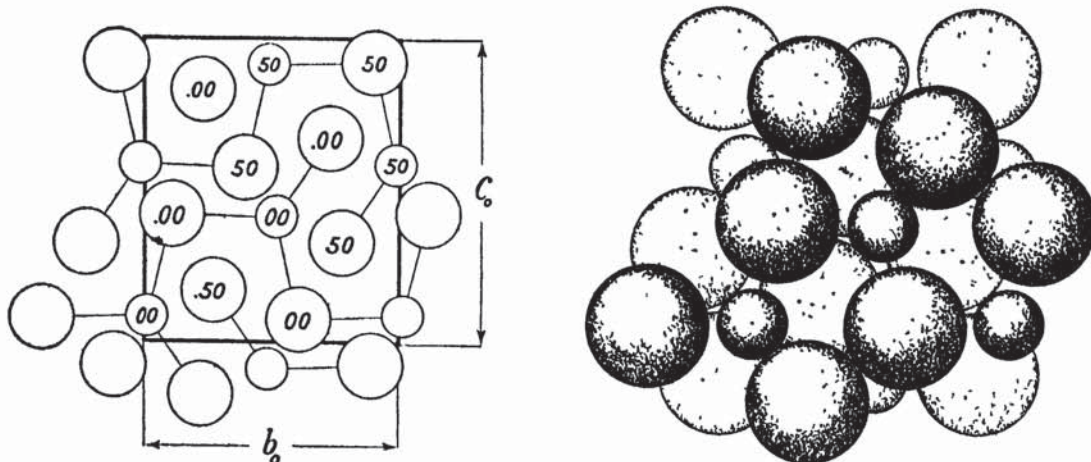


FIG. 280a.—(left) An *a*-face projection of the  $\text{PbBr}_2$  arrangement. The small circles are the Pb atoms. All atoms are in layers normal to the *a*-axis.

FIG. 280b.—(right) A packing drawing of  $\text{PbBr}_2$  with the atoms given their ionic sizes.

(bc) Photographic data have been used to show that the atoms in the two-molecule units of  $\text{PbFBr}$  and  $\text{PbFCl}$  are in the following special positions of  $D_{4h}^7$ :

$$\text{Pb: (c) } 0\frac{1}{2}u; \frac{1}{2}0\bar{u}, \quad \text{Br(or Cl): (c) } 0\frac{1}{2}v; \frac{1}{2}0\bar{v}, \quad \text{F: (a) } \frac{1}{2}\frac{1}{2}0; 000.$$

For  $\text{PbFBr}$ ,  $u=0.19_5$ ,  $v=0.65$ ; for  $\text{PbFCl}$ ,  $u'=0.20$ ,  $v'=0.65$  (Figure 281).

The mineral matlockite is not  $\text{Pb}_2\text{Cl}_2\text{O}$  but  $\text{PbFCl}$  with the structure described above.

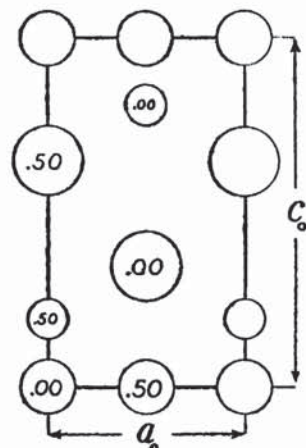


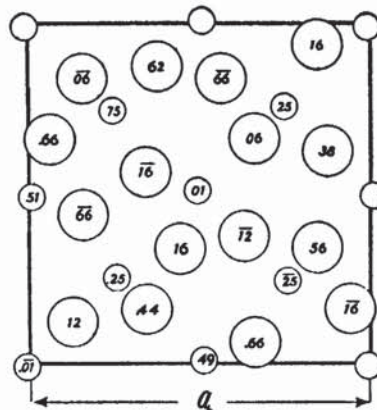
FIG. 281a.—(left) The unit cell of the  $\text{PbFCl}$  arrangement projected upon one of its tetragonal *a*-faces. The atom at the origin is F. The largest circles represent Cl atoms, the smallest Pb atoms.

FIG. 281b.—(right) A packing drawing of  $\text{PbFCl}$  if the atoms have their ionic sizes. The line-shaded spheres are Pb ions.

(bd) It is said that certain faint lines occur in the pattern of **high cristobalite**,  $\text{SiO}_2$ , which are not predicted by the holohedral structure (*ae*). A tetartohedral variant of this arrangement has accordingly been proposed in which the silicon atoms are in two sets of special positions (4f) of  $T^4$ :  $uuu; u+\frac{1}{2}, \frac{1}{2}-u, \bar{u}; \bar{u}, u+\frac{1}{2}, \frac{1}{2}-u; \frac{1}{2}-u, \bar{u}, u+\frac{1}{2}$  with  $u=0.25_5$  and  $u'=-0.00_8$ .

The oxygen atoms are in another set of (4f) with  $u''=0.125$  and in general positions  $xyz$ ; etc. (see p. 267 of book) with  $x=y=0.66$ ,  $z=0.06$ . The difference between this grouping and (ae) may be seen by comparing Figure 282 with Figure 194a. If the observed faint lines really are due to high cristobalite then this distortion of (ae), or a similar one, is necessary, but the proposed parameters do not provide better agreement with the strong lines than that given by (ae) itself. It is clear that more quantitative experimental data are required for an accurate placing of the atoms.

FIG. 282.—The structure recently proposed for high ( $\beta$ ) cristobalite (cf. book, Figure 194a). The small circles are  $Si$  atoms.



(be) The dimensions previously determined (1927, 104) do not refer to the orthorhombic form of  $Zn(OH)_2$  that has recently been analyzed using quantitative spectrometric data. The unit of the table, in which the  $a$  and  $b$ -axes of the original crystallographic description have been interchanged, contains four molecules. All its atoms are in general positions of  $V^4$ :  $xyz$ ;  $x+\frac{1}{2}$ ,  $\frac{1}{2}-y$ ,  $\bar{z}$ ;  $\bar{x}$ ,  $y+\frac{1}{2}$ ,  $\frac{1}{2}-z$ ;  $\frac{1}{2}-x$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ , with the parameters of Table II. In this structure (Figure 283) each zinc atom is at the center

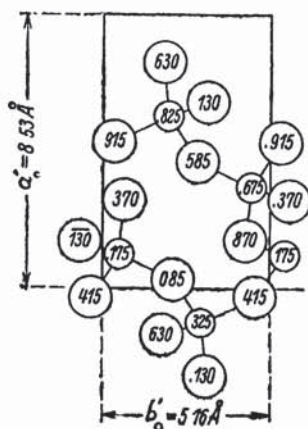


FIG. 283a.—(left) The structure of orthorhombic  $Zn(OH)_2$  projected upon its  $c$ -face. The large circles are  $OH$  groups.

FIG. 283b.—(right) The packing of the  $OH^-$  (large) and  $Zn^{++}$  ions in orthorhombic  $Zn(OH)_2$ .

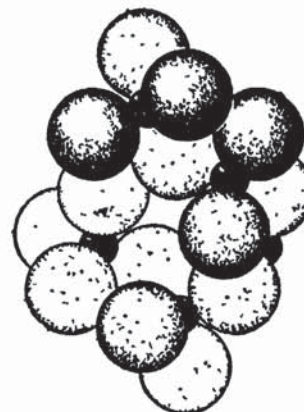


TABLE II. PARAMETERS OF THE ATOMS IN  $Zn(OH)_2$

Atom	$x$	$y$	$z$
Zn	0.125	0.100	0.175
O(1)	.025	.430	.085
O(2)	.325	.125	.370

of a distorted tetrahedron of (OH) groups; each hydroxyl belongs to two such  $Zn(OH)_4$  sphenoids. The result is a tetrahedral network somewhat resembling those found for the various forms of silica.

A hexagonal modification of  $Zn(OH)_2$  has been said to exist with a  $CdI_2$  (*c*) structure (1932, 146a). The available experimental evidence is not, however, satisfactory.

(*bf*) Above  $180^\circ C$   $Ag_2S$  is cubic with the  $Cu_2O$  (*j*) structure; below this temperature the observed pattern is variously described as orthorhombic, like acanthite (1931, 124) and as a mixture of the acanthite and cubic patterns (1931, 345). Four molecules are contained in the acanthite unit described in Table I.

(*bg*) A reinvestigation of solid  $CO_2$  leads, as before, to the pyrite-like arrangement (*f*) with  $u=0.116$ . This gives a C-O distance of 1.13 Å. Recent measurements of  $a_0$  between  $20^\circ$  and  $114^\circ K$  can be expressed by the equation  $a_0 = 5.540 + (4.679 \times 10^{-6})T^2$ .

(*bh*) The two-molecule cell of  $CdCl(OH)$  has an atomic arrangement based on  $C_{6v}^4$ . Cadmium and chlorine are in special positions (b)  $\frac{1}{3}\frac{2}{3}u$ ;  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $u+\frac{1}{2}$  with  $u$  (Cd)=0 and  $u'$ (Cl)=0.337. Hydroxyl groups are in (a)  $00v$ ;  $0, 0, v+\frac{1}{2}$  with  $v=0.100$ . This gives rise to the interionic contacts pictured in Figure 284a, b and c.

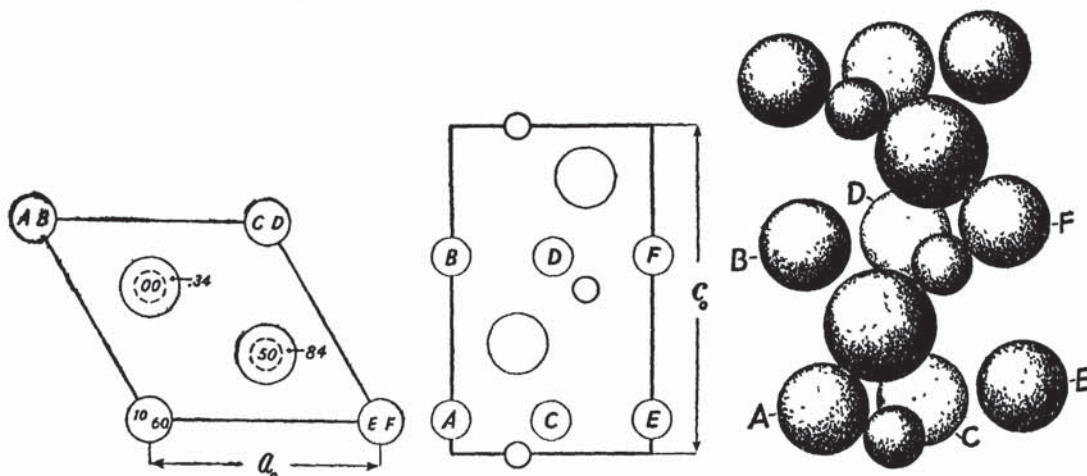


FIG. 284a.—(left) A basal projection of the  $Cd(OH)Cl$  arrangement. Cd, (OH) and Cl are represented by circles of increasing size. Letters refer to corresponding atoms in Figures 284b and 284c.

FIG. 284b.—(center) A diagonal (11·0) face projection of  $Cd(OH)Cl$ .

FIG. 284c.—(right) A packing drawing of *b* with the atoms of  $Cd(OH)Cl$  having their ionic sizes.

(*bi*) Lines have been found on powder and rotation photographs of  $CdI_2$  which indicate that the *c*-axis of the one-molecule cell should be doubled. It has been concluded that the atoms in this two-molecule cell are in the positions: Cd:  $000$ ;  $\frac{1}{3}\frac{2}{3}\frac{1}{2}$ , I:  $\frac{1}{3}\frac{2}{3}u$ ;  $\frac{2}{3}\frac{1}{3}\bar{u}$ ;  $0, 0, u+\frac{1}{2}$ ;  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $\frac{1}{2}-u$ . It would seem to be proved that under certain circumstances  $CdI_2$  can crystallize with this larger unit, but it is not clear whether it always has such a

complicated structure and the existing data are insufficient to establish the new atomic arrangement. Additional work is especially needed since the original one-molecule structure (c) has been found to satisfy the diffraction data from a large number of different compounds. The additional lines calling for a larger unit have not been recorded from these other  $CdI_2$ -like crystals: some should show them clearly, others would not be expected to do so because of the relative scattering powers of their atoms.

(bj) It has been proposed that the atoms in the two-molecule unit of  $FeOCl$  are arranged according to the demands of the space group  $V_h^{13}$ . Oxygen and chlorine are placed in (a)  $0u0$ ;  $\frac{1}{2}\bar{u}\frac{1}{2}$  [interchange of Y and Z from 1930, 352] and iron in (b)  $0v\frac{1}{2}$ ;  $\frac{1}{2}\bar{v}0$  with  $u(O)=-0.083$ ,  $u(Cl)=0.305$ ,  $v=0.097$ . So many crystals have in the past been incorrectly assigned to  $V_h^{13}$  that data far more complete and convincing than those yet published for  $FeOCl$  are highly desirable. It is also probable that the true atomic arrangement will be found to provide interatomic distances that differ somewhat from those of the structure outlined above.

(bk) A Fourier analysis has been made of quantitative intensity data from crystals of pyrite ( $FeS_2$ ). This leads to a parameter  $u(S)=0.386$ . The resulting atomic separations are  $S-S=2.14$  A,  $S-Fe=2.26$  A.

(bl) Within the limits of experimental error ( $ca\pm 0.004$ ) the dimensions of ice composed of heavy hydrogen are identical with those of ordinary ice. Structures have been proposed for ice which assign positions to its hydrogen atoms (see 1933, 257); the results of X-ray determinations of course have nothing to say about such speculations.

(bm) From photographic data  $HgCl_2$  has recently been given a structure which is considered to be essentially molecular. Like  $PbBr_2$  and  $PbCl_2$  (bb), all atoms are in special positions (c) of  $V_h^{16}$ :  $0uv$ ;  $\frac{1}{2}$ ,  $\frac{1}{2}-u$ ,  $\bar{v}$ ;  $0$ ,  $u+\frac{1}{2}$ ,  $\frac{1}{2}-v$ ;  $\frac{1}{2}$ ,  $\bar{u}$ ,  $v+\frac{1}{2}$ . The parameters found for them are  $u(Hg)=0.376$ ,  $v=0.053$ ;  $u'(Cl')=0.517$ ,  $v'=0.375$ ;  $u''(Cl'')=0.742$ ,  $v''=0.778$ . The

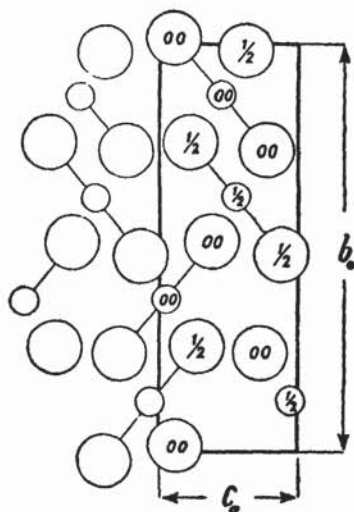
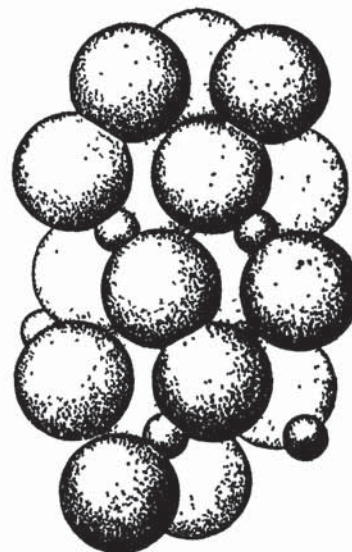


FIG. 284d.—(left) The structure found for  $HgCl_2$  projected upon an a-face. Large circles are Cl atoms.

FIG. 284e.—(right) The type of packing that prevails in the  $HgCl_2$  arrangement if atoms are given their ionic radii. In this drawing the atomic layer at  $\frac{1}{2}$  in  $d$  is on top.



type of packing provided by this arrangement if the atoms have their usual ionic sizes is illustrated in Figure 284d and e. The nearest approach of Hg and Cl atoms is 2.25 Å; Cl-Cl is 3.4 Å.

(bn) NiBr<sub>2</sub> obtained by sublimation has the CdCl<sub>2</sub> arrangement (e) with  $u(\text{Br})=0.255$ . The compound made by dehydration, by driving NH<sub>3</sub> from the hexammoniate or by recrystallization from alcohol is a "Wechselstruktur." The pattern for this gives  $a_0=2.11$  Å,  $c_0=6.08$  Å, a hexagonal cell which would contain only a third of a molecule. It has been proposed that these results can be interpreted in terms of an intimate twinning of CdCl<sub>2</sub> and CdI<sub>2</sub> structures—a few layers of each together. CdBr<sub>2</sub> has been found to give a similar "Wechselstruktur" with  $a_0=2.30$  Å,  $c_0=6.23$  Å; NiI<sub>2</sub> on the other hand seems always to have the CdCl<sub>2</sub> arrangement.

(bo) The structure of YC<sub>2</sub> is different from that of the other carbides studied. It is supposed to be hexagonal with a two-molecule cell.

(bp) A new structure has been proposed for Fe<sub>2</sub>B which differs from the previous one (t) in the parameters assigned to the Fe atoms at (i) of  $V_d^{11}$  and in the positions thought probable for the boron atoms. Borons are placed at (c)  $\frac{1}{2}00$ ;  $0\frac{1}{2}\frac{1}{2}$ ;  $0\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$  instead of (a) and (b), and for Fe,  $u=\frac{1}{6}$  and  $v=\frac{1}{4}$  instead of  $\frac{1}{3}$  (Figure 285). If  $v=\frac{1}{4}$  is exactly correct the structure is identical with the one found for CuAl<sub>2</sub> (n), with a change of origin to  $0\frac{1}{2}0$ .

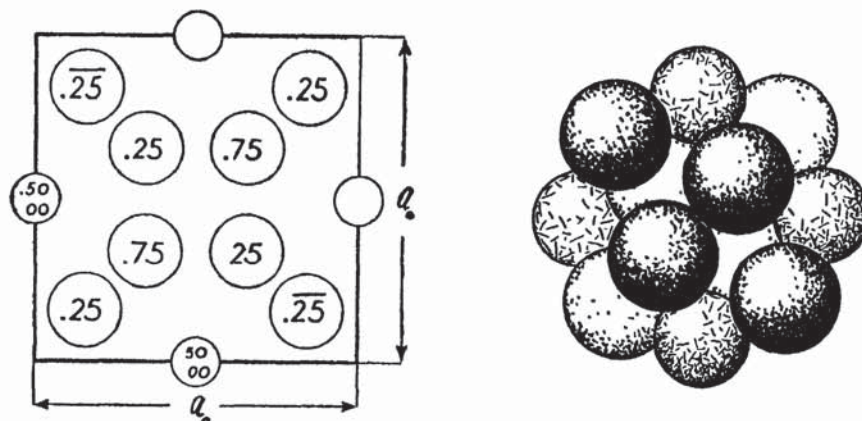


FIG. 285a.—(left) A basal projection of the new arrangement proposed for Fe<sub>2</sub>B. The large circles are the Fe atoms.

FIG. 285b.—(right) A packing drawing of  $a$  if the Fe atoms are assumed to be neutral and if the (line-shaded) boron is given a size probable for its neutral atoms.

(bq) It is suggested that the unit cell of the room temperature (low) modification of cristobalite contains eight molecules and that the atomic arrangement, based on  $V^4$ , is a distortion of that of high cristobalite (compare Figure 286 with Figures 282 and 194a). All atoms are in general positions:  $xyz$ ;  $x+\frac{1}{2}$ ,  $\frac{1}{2}-y$ ,  $\bar{z}$ ;  $\bar{x}$ ,  $y+\frac{1}{2}$ ,  $\frac{1}{2}-z$ ;  $\frac{1}{2}-x$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ . Parameters which



are considered approximately correct are listed in Table III. As in the case of  $\beta$ -cristobalite, more quantitative data are needed to fix these atomic positions with any certainty.

FIG. 286.—A  $c$ -face projection of the structure assigned to low temperature ( $\alpha$ ) cristobalite,  $SiO_2$ . The small circles are Si atoms.

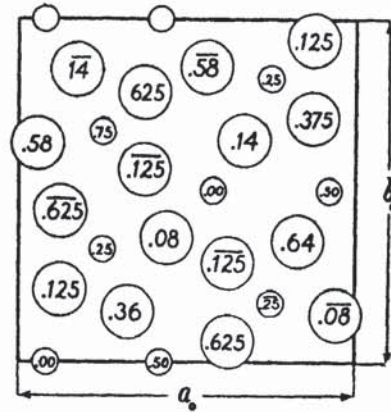


TABLE III. PARAMETERS GIVEN TO THE ATOMS IN LOW CRISTOBALITE

Atom	$x$	$y$	$z$
O(1)	0.67	0.65	0.14
O(2)	.06	.64	.58
O(3)	.625	.06	.625
O(4)	.125	.21	.125
Si(1)	.25	.33	.25
Si(2)	.08	.00	.00

(br) The mineral laurite has been shown to be  $RuS_2$  with the same pyrite structure ( $f$ ) previously established for the synthetic compound. The sulfur parameter is  $0.39 < u < 0.395$ .

A further study of speryllite, also isomorphous with pyrite, has shown that  $0.385 < u < 0.390$ .

(bs) The compound  $Cu_2Mg$  has only a narrow range of homogeneity. On the  $\alpha$ -side  $a_0$  varies from 7.0087 Å to 7.0185 Å with annealing temperatures between 600° C and 400° C; two samples in the  $\beta + \gamma$  region annealed at 500° C and 380° C gave  $a_0 = 7.0518$  Å and  $a_0 = 7.0343$  Å.

## Chapter XIII A. Structures of the Type $R_2X_3$

(m) A complex arrangement which includes a place for one sodium atom per cell has been proposed for  $\beta\text{-Al}_2\text{O}_3$ . Based on  $D_{6h}^4$  it has atoms in the following positions:

- 1 Na+1 Al: (a)  $000; 00\frac{1}{2}$   
 4 Al: (f)  $\frac{1}{3}\frac{2}{3}u; \frac{2}{3}, \frac{1}{3}, u+\frac{1}{2}; \frac{2}{3}\frac{1}{3}\bar{u}; \frac{1}{3}, \frac{2}{3}, \frac{1}{2}-u$  with  $u=0.022$   
 3 Al:  $\frac{3}{4}$  of positions of (f) with  $u'=0.178$ ,      3 Al: same with  $u''=-0.178$   
 12 Al: (k)  $u\bar{v}$ ; etc. (see 1930, 352, p. 169) with  $u=\frac{1}{6}, v=-0.106$   
 12 O: (k)  $u_1\bar{u}_1v_1$ ; etc. with  $u_1=\frac{1}{6}, v_1=0.05$   
 12 O: (k)  $u_2\bar{u}_2v_2$ ; etc. with  $u_2=\frac{1}{2}, v_2=0.144$   
 4 O: (f)  $\frac{1}{3}\frac{2}{3}u_3$ ; etc. with  $u_3=-0.05$   
 4 O: (e)  $00w; 00\bar{w}; 0, 0, \frac{1}{2}-w; 0, 0, w+\frac{1}{2}$  with  $w=0.144$   
 $\frac{3}{2}$  O:  $1\frac{1}{2}$  of two equivalent positions (c)  $\frac{1}{3}\frac{2}{3}\frac{1}{4}; \frac{2}{3}\frac{1}{3}\frac{3}{4}$ ,  
 $\frac{3}{2}$  O: same for (d)  $\frac{1}{3}\frac{2}{3}\frac{3}{4}; \frac{2}{3}\frac{1}{3}\frac{1}{4}$ .

This distribution of atoms among the equivalent positions of the space group is so bizarre that a confirmation of the structure is much to be desired.

(n) The atomic arrangement in  $\gamma\text{-Al}_2\text{O}_3$  has not yet been satisfactorily established.

TABLE I. PARAMETERS ASSIGNED TO THE ATOMS IN  $\text{Sb}_2\text{S}_3$

Atom	$u$	$v$
Sb(1)	0.328	0.031
Sb(2)	-.039	-.149
S(1)	.883	.047
S(2)	-.439	-.125
S(3)	.194	.208

(o) Spectral photographs of **stibnite** indicate that the four  $\text{Sb}_2\text{S}_3$  molecules in its orthorhombic unit are arranged according to  $V_h^{16}$ . All atoms are said to be in special positions (c)  $uv\frac{1}{4}; \bar{u}\bar{v}\frac{3}{4}; \frac{1}{2}-u, v+\frac{1}{2}, \frac{1}{4}; u+\frac{1}{2}, \frac{1}{2}-v, \frac{3}{4}$  with the parameters of Table I. If  $abc$  are the axes of this description and  $X'Y'Z'$  are those of 1930, 352:  $a=Z', b=Y', c=X'$ , the origin being moved to a center of symmetry. Within the limit of experimental error the Bi parameters in  $\text{Bi}_2\text{S}_3$  are the same as those of Sb.

This structure consists of chains of the composition  $(Sb_2S_3)_n$ . If the atoms have their uncharged radii, there is good contact within these chains (Figure 287). Whether the atoms are charged or neutral the atoms in adjacent chains are unexpectedly far apart. In view of this fact it will be important to see whether future work confirms this arrangement.

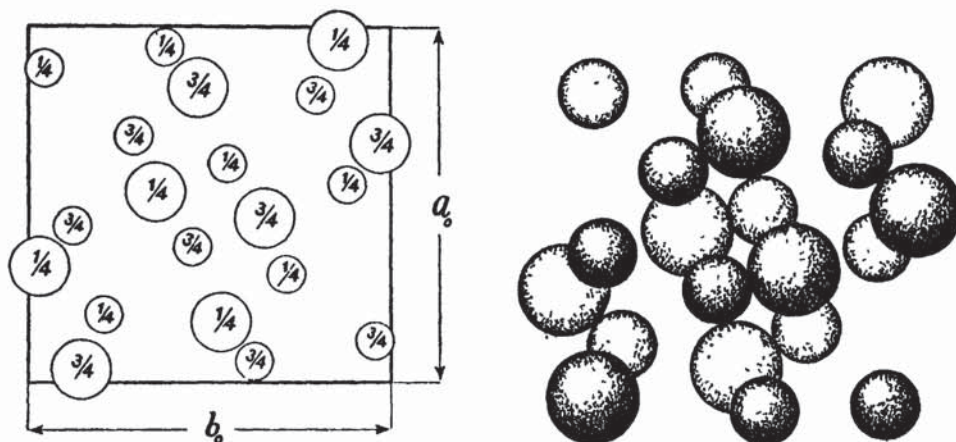


FIG. 287a.—(left) The arrangement proposed for stibnite,  $Sb_2S_3$ , projected on its *c*-face. The large circles are Sb atoms.

FIG. 287b.—(right) A packing drawing of *a* if Sb and S are given their neutral radii. The small spheres thus are the sulfur atoms. Packing is not improved by assuming that the atoms are charged.

(*p*) A reexamination of  $Mg_3P_2$  shows that its correct structure is identical with that of  $Tl_2O_3$  (*b*). The selected parameters are the same as those found for bixbyite,  $(Fe, Mn)_2O_3$ . This atomic arrangement also prevails for  $Be_3P_2$ ,  $Be_3N_2$ ,  $Mg_3N_2$  and  $\alpha-Ca_3N_2$ .

It is said that  $Zn_3P_2$ ,  $Cd_3P_2$  and  $Zn_3As_2$ , though likewise possessed of 16-molecule cubic units, have different structures.

The earlier choice of a 12-molecule cube for  $Mg_3N_2$  was due to a faulty estimate of its density.

Probably the small unit previously determined for  $Cd_3As_2$  is equally wrong.

(*q*) The monoclinic unit assigned to  $Cd_3Sb_2$  contains four molecules.

(*r*) The structure first suggested for the magnetic  $\gamma-Fe_2O_3$  was the same as that of magnetite [(*k*) of Chapter XVI] with four oxygen atoms added. It has recently been shown that if these additional atoms are put in either of the two sets of positions originally proposed, the observed intensities cannot be explained. Better intensity agreement can be obtained by placing these atoms in (4*g*)  $uuu$ ; etc. with  $u = \frac{1}{8}$ . The smallest O-O separations in this structure are greater than those existing in the previous arrangements; nevertheless they still have the improbably small value 2.14 Å.

(*s*) Eight molecules of  $Fe_3W_2$  are contained in the large cell found for the  $\epsilon$ -phase of the Fe-W system.

TABLE II. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $R_2X_3$ 

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
$Al_2Mg_3$	Cubic	( <i>t</i> )	10.54		1934, 340.
$\alpha-Al_2O_3$	Hexagonal	$Fe_2O_3$ ( <i>a</i> )	5.13	55°6'	1930, 246b.
$\beta-Al_2O_3$	Hexagonal	( <i>f</i> ), ( <i>m</i> )	5.56	22.55	1931, 56.
$\gamma-Al_2O_3$	Cubic	( <i>n</i> )	7.90		1932, 18, 75.
$As_2O_3$	Cubic	$As_2O_3$ ( <i>e</i> )	11.0457± 0.0002		1932, 290.
$Be_3N_3$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>p</i> )	8.134		1933, 443.
$Be_3P_2$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>p</i> )	10.15		1933, 443.
$Bi_2S_3$ (Bismuthinite)	Ortho- rhombic	$Sb_2S_3$ ( <i>k</i> ), ( <i>o</i> )	11.13	3.97 $b_o=11.27$	1933, 207.
$\alpha-Ca_3N_2$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>p</i> )	11.40		1933, 137, 443.
$Cd_3As_2$	Cubic	( <i>p</i> )	12.58 (?)		
$Cd_3P_2$	Cubic	( <i>p</i> )	12.26		1933, 443.
$Cd_3Sb_2$	Monoclinic	( <i>q</i> )	7.20	6.16 $b_o=13.51, \beta=100^\circ 14'$	1933, 167.
$Cr_3C_2$	Ortho- rhombic	( <i>g</i> )			1931, 468.
$Cr_2O_3$	Hexagonal	$Fe_2O_3$ ( <i>a</i> )	5.38	54°50'	1930, 246b.
$Fe_2O_3$ (Hematite)	Hexagonal	$Fe_2O_3$ ( <i>a</i> )	5.4135	55°17'	1930, 246b; 1932, 75; 1933, 247; 1934, 34.
$Fe_2O_3$ (Magnetic)	Cubic	( <i>h</i> ), ( <i>r</i> )			1931, 117, 445.
$Fe_3W_2$ ( $\epsilon$ -phase)	Hexagonal	( <i>s</i> )	4.738	25.726	1931, 343.
$Mg_3As_2$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>p</i> )	12.33		1933, 443, 524.
$Mg_3Bi_2$	Hexagonal	$La_2O_3$ ( <i>c</i> )	4.666	7.401	1933, 524.
$Mg_3N_2$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>i</i> ), ( <i>p</i> )	9.95		1932, 185; 1933, 443.
$Mg_3P_2$	Cubic	$Tl_2O_3$ ( <i>b</i> ), ( <i>p</i> )	12.03		1933, 443, 524.
$Mg_3Sb_2$	Hexagonal*	$La_2O_3$ ( <i>c</i> )	4.573	7.229	1933, 524.
$Sb_2S_3$ (Stibnite)	Ortho- rhombic	$Sb_2S_3$ ( <i>k</i> ), ( <i>o</i> )	11.20	3.83 $b_o=11.28$	1933, 207.
$Zn_3As_2$	Cubic	( <i>p</i> )	11.74		1933, 443.
$Zn_3P_2$	Cubic	( <i>p</i> )	11.42		1933, 443.

\* The parameters are the same as those of  $La_2O_3$ .

(*t*) The intermetallic phase  $Al_2Mg_3$  gives the cubic pattern of  $\alpha$ -Mn. It is therefore concluded that the true composition is  $Mg_{17}Al_{12}$  with two molecules per cell. In such a structure Mg atoms presumably are in (2a), (8a) and (24g) (book, p. 270) with  $u=0.356$ ,  $v=0.012$ ; the Al atoms, also in (24g), have  $u'=0.089$ ,  $v'=0.278$ .

## Chapter XIVA. Structures of the Type $RX_3$ , of Higher Compounds $R_mX_n$ and of New Compounds of the Type $R_x(MX_2)_y$

### The Compounds $R_mX_n$

(*al*)  $AlF_3$  was earlier described as hexagonal with a unit containing three molecules. An atomic arrangement more recently found for it is rhombohedral with two molecules in the unit (corresponding to a six-molecule hexagonal cell). The structure, based on  $D_3^7$ , has atoms in the special positions:

$$\begin{aligned} Al: & \text{ (c) } uuu; \bar{u}\bar{u}\bar{u} \text{ with } u=0.237 \\ F: & \text{ (d) } u_1\bar{u}_10; \bar{u}_10u_1; 0u_1\bar{u}_1 \text{ with } u_1=0.430 \\ F: & \text{ (e) } u'\bar{u}'\frac{1}{2}; \bar{u}'\frac{1}{2}u'; \frac{1}{2}u'\bar{u}' \text{ with } u'=0.070. \end{aligned}$$

(*am*) **Arsine,  $AsH_3$ , and phosphine,  $PH_3$ ,** when solidified give patterns corresponding to four-molecule cubic cells. They are said to be face-centered but a further study of them is desirable to be sure that they do not have structures like ammonia (*t*).

(*an*) A previous determination has given  $AsI_3$ ,  $SbI_3$  and  $BiI_3$  hexagonal unit cells containing six molecules. Atomic positions, said to be developed from  $C_3^1$ , were stated for  $BiI_3$  [see (*b*)]. More recent work on  $AsI_3$  has shown that its space group really is  $C_3^2$ . The atoms in the two-molecule rhombohedron that is its true unit have the coordinates:

$$\begin{aligned} As: & \text{ (c) } \pm(uuu) \text{ with } u=\frac{1}{6} \\ I: & \text{ (f) } \pm(xyz); \pm(zxy); \pm(yzx) \text{ with } x=0.42, y=0.08, z=0.75. \end{aligned}$$

If, as is presumably the case,  $SbI_3$  and  $BiI_3$  are isomorphous with  $AsI_3$  then their two-molecule rhombohedral units will have the dimensions of Table I.

Though the space groups and the unit cell suggested for  $CrBr_3$  are hexagonal, it is reported to be isomorphous with  $BiI_3$ . Its true unit is thus without doubt rhombohedral, the dimensions being those stated in Table I.

TABLE I. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $R_mX_n$ 

Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References	
<b>Compounds <math>RX_3</math></b>					
$AlF_3$	Hexagonal	( <i>a</i> ), ( <i>al</i> )	5.029	58°31'	1931, 256; 1933, 255.
$Al_2Fe$	Ortho- rhombic	( <i>bs</i> )	11.87 (?)	15.80 $b_o=8.09$	1933, 334; 1934, 313, 313a.
$AsH_3$	Cubic	( <i>am</i> )	6.40 at -170° C		1930, 429.
$AsI_3$	Hexagonal	$AsI_3$ ( <i>b</i> ), ( <i>an</i> )	8.25	51°20'	1931, 205, 206.
$BiI_3$	Hexagonal	$AsI_3$ ( <i>b</i> ), ( <i>an</i> )	8.13	54°50'	
$CaPb_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.891		1933, 529.
$CaSn_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.732		1933, 529.
$CaTl_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.794		1933, 529.
(Ce, La, . . .)F <sub>3</sub> (Tysonite)	Hexagonal	( <i>ap</i> )			1931, 335.
$CeMg_3$	Cubic	$LaMg_3$ ( <i>bc</i> )	7.373		1934, 234.
$CePb_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.864		1933, 529.
$CeSn_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.711		1933, 529.
$CoF_3$	Hexagonal	( <i>aq</i> )	3.66 <sub>4</sub>	87°20'	1931, 100.
$CrBr_3$	Hexagonal	$AsI_3$ ( <i>b</i> ), ( <i>an</i> )	7.05	52°36'	1932, 68.
$CrO_3$	Ortho- rhombic (?)	( <i>as</i> )	8.50	5.72 $b_o=4.73$	1931, 53, 487.
$Fe_3C$	Ortho- rhombic	( <i>o</i> ), ( <i>av</i> )	4.626	6.633 $b_o=5.107$	1931, 343; 1932, 474.
$FeCl_3$	Hexagonal	$AlF_3$ (?), ( <i>al</i> )	6.69	52°30'	1932, 481.
$FeF_3$	Hexagonal	( <i>aq</i> )	3.75 <sub>6</sub>	88°14'	1931, 100, 256; 1933, 511.
KCNS	Ortho- rhombic	( <i>aw</i> )	6.66	6.635 $b_o=7.58$	1933, 261; 1934, 38.
$LaMg_3$	Cubic	$LaMg_3$ ( <i>bc</i> )	7.478		1934, 234.
$LaPb_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.893		1933, 386.
$LaSn_3$	Cubic	$AuCu_3$ ( <i>e</i> ), ( <i>ao</i> )	4.772		1933, 386.
$LiCd_3$		( <i>at</i> )			1933, 27; 1934, 310.
$MoO_3$	Ortho- rhombic	( <i>ax</i> )	3.954	3.694 $b_o=13.825$	1931, 53, 484, 485.
$NH_3$	Cubic	( <i>t</i> )	5.08 at -170° C		1930, 429.
$NaCNO$	Hexagonal	$CsCl_2I$ ( <i>d</i> )	5.45	38°16'	1934, 287.
$NaN_3$	Hexagonal	$CsCl_2I$ ( <i>d</i> ), ( <i>ar</i> )	5.45	38°48'	1934, 287.

Substance, symmetry and structure type			$a_0$	$c_0$ or $\alpha$	References
NaPb <sub>3</sub>	Cubic	AuCu <sub>3</sub> (e), (ao)	4.873		1931, 505.
PH <sub>3</sub>	Cubic	(am)	6.31 at -170° C		1930, 429.
PI <sub>3</sub>	Hexagonal	(ay)	7.11	7.42	1933, 64.
PdF <sub>3</sub>	Hexagonal	(aq)	3.75 <sub>8</sub>	84°29'	1931, 100.
PrMg <sub>3</sub>	Cubic	LaMg <sub>3</sub> (bc)	7.373		1934, 234.
RbN <sub>3</sub>	Tetragonal	KN <sub>3</sub> (c)			1931, 72.
ReO <sub>3</sub>	Cubic	(az)	3.734		1931, 44; 1932, 308; 1933, 45.
RhF <sub>3</sub>	Hexagonal	(aq)	3.62	84°48'	1931, 100.
SbI <sub>3</sub>	Hexagonal	AsI <sub>3</sub> (b), (an)	8.18	54°14'	
SrPb <sub>3</sub>	Tetragonal	(ba)	4.955	5.025	1933, 529.
TiAl <sub>3</sub>	Tetragonal	(au)	5.424	8.574	1931, 114.
TiCNS	Ortho- rhombic	(aw)	6.80	6.78	1934, 38, 256.
WO <sub>3</sub>	Triclinic	(bb)	7.28	3.82 b <sub>0</sub> =7.52 b <sub>0</sub> =7.48*	1931, 53.

Compounds  $RX_4$ 

Cl <sub>4</sub>	Cubic	(bd)	9.14		1931, 164.
Cr <sub>4</sub> C	Cubic	(x)			1931, 468.
Fe(CO) <sub>4</sub>	Monoclinic	(be)	13.00	11.41	1931, 62.
			b <sub>0</sub> =11.41, $\beta$ =85°35'		
HfF <sub>4</sub>	Monoclinic	(bt)	9.45	7.62	1934, 240a.
			b <sub>0</sub> =9.84, $\beta$ =94°29'		
LaAl <sub>4</sub>	Tetragonal	(bf)	13.2	10.2	1933, 385.
SiF <sub>4</sub>	Cubic	(bg)			1930, 218a.
SiI <sub>4</sub>	Cubic	SnI <sub>4</sub> (aa)	11.986		1931, 163.
TiBr <sub>4</sub>	Cubic	SnI <sub>4</sub> (aa)	11.250		1932, 194.
TiI <sub>4</sub>	Cubic	SnI <sub>4</sub> (aa)	12.002		1932, 194.
ZrF <sub>4</sub>	Monoclinic	(bt)	9.46	7.64	1934, 240a.
			b <sub>0</sub> =9.87, $\beta$ =94°30'		

Compounds  $RX_5$ 

MgZn <sub>5</sub>	Hexagonal	(bh)	9.92	16.48	1933, 465.
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Compounds  $RX_6$ 

BaB <sub>6</sub>	Cubic	ThB <sub>6</sub> (ac), (bj)	4.28		1931, 425; 1932, 6, 428.
B <sub>6</sub> C (?)	Hexagonal	(bu)	5.62	12.12	1934, 338.
CaB <sub>6</sub>	Cubic	ThB <sub>6</sub> (bj)	4.145		1931, 425; 1932, 6, 428; 1934, 207.
CeB <sub>6</sub>	Cubic	ThB <sub>6</sub> (bj)	4.129		1931, 425; 1932, 6, 428.
ErB <sub>6</sub>	Cubic	ThB <sub>6</sub> (bj)	4.102		1932, 6, 428.
GdB <sub>6</sub>	Cubic	ThB <sub>6</sub> (bj)	4.12		1932, 6.
LaB <sub>6</sub>	Cubic	ThB <sub>6</sub> (bj)	4.145		1931, 425; 1932, 6, 428.

\* All angles close to 90°.

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
NdB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.118		1932, 6, 428.
PrB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.121		1932, 428.
SrB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.19		1931, 425; 1932, 6, 428.
Te(OH) <sub>6</sub>	Cubic	( <i>ab</i> ), ( <i>bi</i> )			1934, 88.
Te(OH) <sub>6</sub> (second form)	Monoclinic	( <i>bi</i> )	5.54	9.74	1934, 88.
			$b_o=9.30, \beta=104^{\circ}30'$		
ThB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.15		1932, 6.
YB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.07		1932, 6.
YtB <sub>6</sub>	Cubic	ThB <sub>6</sub> ( <i>bj</i> )	4.13		1932, 6.

Higher Compounds RX<sub>n</sub>

CsC <sub>8</sub>	Hexagonal	KC <sub>8</sub> ( <i>bk</i> )	4.94	23.76	1932, 405.
KC <sub>8</sub>	Hexagonal	KC <sub>8</sub> ( <i>bk</i> )	4.94	21.34	1932, 405.
RbC <sub>8</sub>	Hexagonal	KC <sub>8</sub> ( <i>bk</i> )	4.94	22.73	1932, 405.
CsC <sub>16</sub>	Hexagonal	KC <sub>16</sub> ( <i>bl</i> )	4.94	18.51	1932, 405.
KC <sub>16</sub>	Hexagonal	KC <sub>16</sub> ( <i>bl</i> )	4.94	17.45	1932, 405.
RbC <sub>16</sub>	Hexagonal	KC <sub>16</sub> ( <i>bl</i> )	4.94	17.95	1932, 405.

Miscellaneous Compounds R<sub>m</sub>X<sub>n</sub>

Ag <sub>3</sub> Hg <sub>4</sub>	Cubic	( <i>bn</i> )	10.09		1933, 494.
Al <sub>4</sub> C <sub>3</sub>	Hexagonal	( <i>bv</i> )	8.53	22°28'	1934, 354c.
B <sub>10</sub> H <sub>14</sub>	Ortho- rhombohedral	( <i>bo</i> )	14.46	5.69	1931, 304.
			$b_o=20.85$		
Co <sub>4</sub> S <sub>3</sub>	Cubic		9.91		1932, 90.
Cr <sub>7</sub> C <sub>3</sub>	Hexagonal	( <i>af</i> )	13.98	4.52	1931, 468.
Cu <sub>5</sub> Cd <sub>8</sub>	Cubic	Cu <sub>5</sub> Zn <sub>8</sub> ( <i>ad</i> )			1931, 51.
Cu <sub>16</sub> Si <sub>4</sub>	Cubic	( <i>bq</i> )	9.694		1934, 179.
Cu <sub>5</sub> Zn <sub>8</sub>	Cubic	Cu <sub>5</sub> Zn <sub>8</sub> ( <i>ad</i> )			1931, 51.
Na <sub>31</sub> Pb <sub>8</sub>	Cubic	( <i>bp</i> )	13.27		1933, 451.
Sb <sub>2</sub> Tl <sub>7</sub>	Cubic	( <i>bm</i> )	11.59		1934, 180.
W <sub>4</sub> O <sub>11</sub>	Tetragonal	( <i>br</i> )	7.56	3.735	1934, 74.

TABLE II. NEW CRYSTAL STRUCTURES OF THE COMPOUNDS R<sub>x</sub>(MX<sub>2</sub>)<sub>y</sub>

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
AgClO <sub>2</sub>	Pseudo- tetragonal	( <i>ca</i> )	12.17	6.69	1931, 282.
AgSbS <sub>2</sub> (Miargyrite)	Monoclinic	( <i>cb</i> )	13.17	12.82	1932, 219.
			$b_o=4.39, \beta=98^{\circ}37\frac{1}{2}'$		
(Ag, Cu) <sub>2</sub> Sb <sub>2</sub> S <sub>4</sub> (Polybasite)	Ortho- rhombohedral (?)	( <i>cc</i> )	7.50	11.95	1934, 89.
			$b_o=12.99$		
CaB <sub>2</sub> O <sub>4</sub>	Ortho- rhombohedral	( <i>cd</i> )	6.19	4.28	1931, 494; 1932, 494.
			$b_o=11.60$		
Ca(ClO <sub>2</sub> ) <sub>2</sub>	Pseudo- cubic	( <i>ce</i> )	5.80		1931, 282.
CuBiS <sub>2</sub> (Emplectite)	Ortho- rhombohedral	( <i>cf</i> )	6.12	14.51	1932, 219; 1933, 206.
			$b_o=3.89$		



Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References
$Cu_2Fe_2O_4$	Hexagonal	6.06	2.82	1934, 281.
$CuFeS_2$ (Chalcopyrite)	Tetragonal [XI, (aa)], (cg)	5.24	10.30	1932, 359.
$CuSbS_2$ (Wolfsbergite)	Ortho- rhombic (cf)	6.01	14.46 $b_o=3.78$	1932, 219; 1933, 206.
$KAg(CN)_2$	Hexagonal (ch)	7.384	17.55	1933, 199.
$K_2Fe_2O_4$	Cubic (ci)	7.958		1933, 197.
$KFeS_2$	Hexagonal (cj)	13.03	5.40	1933, 329.
$Li_2Fe_2O_4$	Cubic (ck)	4.141		1931, 362.
$NH_4ClO_2$	Tetragonal (cl)	6.30	3.73	1931, 282.
$NH_4HF_2$	Ortho- rhombic (cm)	8.33	3.68 $b_o=8.14$	1932, 196; 1933, 349.
$NH_4H_2PO_2$	Ortho- rhombic (cr)	3.98	11.47 $b_o=7.57$	1934, 307.
$Na_2Fe_2O_4$	Hexagonal (cn)	5.59	$35^\circ 20'$	1933, 149.
$NaNO_2$	Ortho- rhombic (co)	3.55	5.37 $b_o=5.56$	1931, 504.
$Pb(ClO_2)_2$	Pseudo- tetragonal (cp)	4.14	6.25	1931, 282.
$PbFe_2O_4$	Cubic	7.81		1933, 197.
$TlAsS_2$ (Lorandite)	Monoclinic (cq)	15.02	6.10 $b_o=11.31, \beta=127^\circ 45'$	1932, 219.

(ao) Several intermetallic compounds, of which  $NaPb_3$  is typical, have been found to have the simple cubic arrangement (e) which occurs as a superlattice in alloys of copper with gold, platinum and palladium. For  $NaPb_3$  the atomic coordinates are: Na: (1a) 000, Pb: (3a)  $0\frac{1}{2}\frac{1}{2}$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$  (Figure 288a and b).

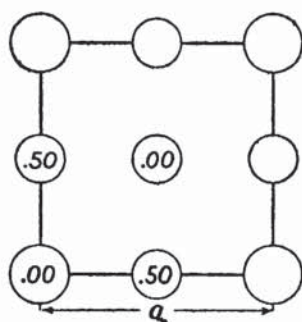
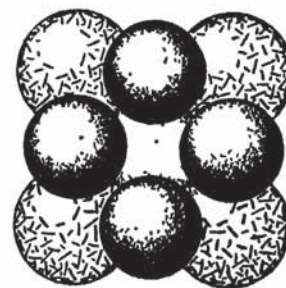


FIG. 288a.—(left) A cube face projection of the simple  $NaPb_3$  grouping. Na atoms are at the origin.

FIG. 288b.—(right) A packing drawing of a with the atoms having the (neutral) radii found in the metals themselves. Na atoms are line-shaded.



(ap) A new structure has been suggested for tysonite  $(Ce, La, \dots)F_3 = R'F_3$ . It has atoms of its six-molecule unit in the following special positions of  $D_{6h}^3$ :

- R: (g)  $uu0$ ; etc. (1930, 352, p. 168) with  $u = ca\ 0.34$   
 F: (a) 000;  $00\frac{1}{2}$ , at (c)  $\frac{1}{3}\frac{2}{3}0$ ;  $\frac{2}{3}\frac{1}{3}\frac{1}{2}$ ;  $\frac{2}{3}\frac{1}{3}0$ ;  $\frac{1}{3}\frac{2}{3}\frac{1}{2}$  and at (k)  $u'u'v$ ; etc.  
 with  $u' = ca\ \frac{2}{3}$  and  $v = ca\ 0.175$ .

This arrangement is not definitely established by the existing data.

(aq) The data on  $\text{FeF}_3$  are conflicting. According to 1931, 256 it is hexagonal with a three-molecule unit. The analysis of 1931, 100 gives it a one-molecule rhombohedron having the dimensions recorded in Table I. A recent discussion suggests that neither of these is correct but that the arrangement really resembles that of  $\text{WO}_3$  (bb). Whatever structure may ultimately be established for  $\text{FeF}_3$ , it probably is possessed by  $\text{CoF}_3$ ,  $\text{RhF}_3$  and  $\text{PdF}_3$  as well. The one-molecule rhombohedral units, or pseudo-units, of these substances are listed in Table I.

(ar) The cell dimensions of  $\text{NaN}_3$  have been determined at  $200^\circ \text{C}$  as well as at room temperature. At the higher temperature the edge length is unchanged,  $a_0 = 5.45 \text{ \AA}$ ; the rhombohedral angle, however, has become slightly less acute,  $\alpha = 39^\circ 14'$ . The parameter  $u$  for nitrogen has not been established for any of the sodium compounds showing this arrangement; for  $\text{CsCl}_2\text{I}$  it was 0.31.

(as) The unit cell of  $\text{CrO}_3$  contains four molecules. A structure based on  $V_h^{17}$  has been suggested but not proved.

(at) The data on  $\text{LiCd}_3$  are conflicting. According to one investigator it is cubic with a cell apparently holding 6 molecules; others state that the arrangement is hexagonal close-packed.

(au) The tetragonal unit of  $\text{TiAl}_3$  contains four molecules. An atomic arrangement has been described which, based on  $V_d^8$ , has atoms in the following special positions:

$$\begin{array}{ll} \text{Ti:} & \text{(a) } 000; \frac{1}{2}0\frac{1}{2}; \frac{1}{2}\frac{1}{2}0; 0\frac{1}{2}\frac{1}{2} \\ & \text{Al(1): (b) } \frac{1}{2}\frac{1}{2}\frac{1}{2}; 0\frac{1}{2}0; 00\frac{1}{2}; \frac{1}{2}00 \\ \text{Al(2):} & \text{(c) } \frac{1}{4}\frac{1}{4}\frac{1}{4}; \frac{3}{4}\frac{1}{4}\frac{3}{4}; \frac{3}{4}\frac{3}{4}\frac{1}{4}; \frac{1}{4}\frac{3}{4}\frac{3}{4} \\ & \text{Al(3): (d) } \frac{3}{4}\frac{3}{4}\frac{3}{4}; \frac{1}{4}\frac{3}{4}\frac{1}{4}; \frac{1}{4}\frac{1}{4}\frac{3}{4}; \frac{3}{4}\frac{1}{4}\frac{1}{4}. \end{array}$$

(av) It has been found that the agreement between observed and calculated intensities from  $\text{Fe}_3\text{C}$  can be improved by altering very slightly the parameters of the iron atoms and by placing the carbon atoms in positions different from those previously suggested. As before, the iron atoms are in the following special positions of  $V_h^{16}$ :

$$\begin{array}{l} \text{Fe: (c) } uv\frac{1}{4}; \text{ etc. (see p. 266 of book) with } u=0.833, v=0.04 \\ \text{Fe: (d) } xyz; \text{ etc. with } x=0.333, y=0.175, z=0.065. \end{array}$$

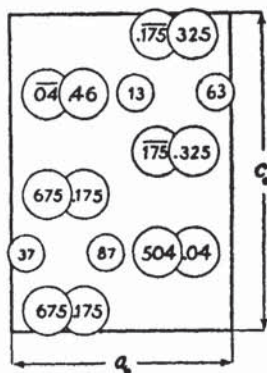


FIG. 289a.—(left) The improved structure for  $\text{Fe}_3\text{C}$  projected on the  $b$ -face of its orthorhombic cell. The large circles are the iron atoms.

FIG. 289b.—(right) A packing drawing of  $\text{Fe}_3\text{C}$  if the (larger) Fe atoms have their neutral (metallic) radii and the C atoms have the radius suggested by the diamond.



Instead of being in symmetry centers the carbon atoms are in another set of (c)  $u'v'\frac{1}{4}$  with  $u'=0.43$ ,  $v'=0.87$ . The resulting interatomic distances give iron the radius found in the metal, 1.25 Å; the radius of carbon, 0.76 Å, is that which occurs in the diamond (Figure 289a and b).

(aw) The orthorhombic unit of **KCNS** contains four molecules. A structure which gives fairly good agreement between calculated and photographically observed intensities is obtained by putting atoms in the following special positions of  $V_h^{11}$ :

$$\begin{aligned} \text{K: (c) } & u'0\frac{3}{4}; \bar{u}'\frac{1}{2}\frac{1}{4}; \bar{u}'0\frac{1}{4}; u'\frac{1}{2}\frac{3}{4} \text{ with } u'=0.212 \\ \text{N, C, S: (d) } & u\frac{1}{4}v; \bar{u}\frac{3}{4}\bar{v}; \bar{u}, \frac{1}{4}, v+\frac{1}{2}; u, \frac{3}{4}, \frac{1}{2}-v \text{ with} \\ & u(\text{S})=0.400, v(\text{S})=0.095. \end{aligned}$$

The suggested parameters for nitrogen are  $u(\text{N})=0.080$ ,  $v(\text{N})=0.400$ ; for carbon  $u(\text{C})=0.205$ ,  $v(\text{C})=0.280$ . To derive the axes of this description (abc) from those of 1930, 352 ( $X'Y'Z'$ ) the transformations  $a=Z'$ ,  $b=X'$ ,  $c=Y'$  are necessary. The kind of packing provided by this arrangement is illustrated by Figure 290.

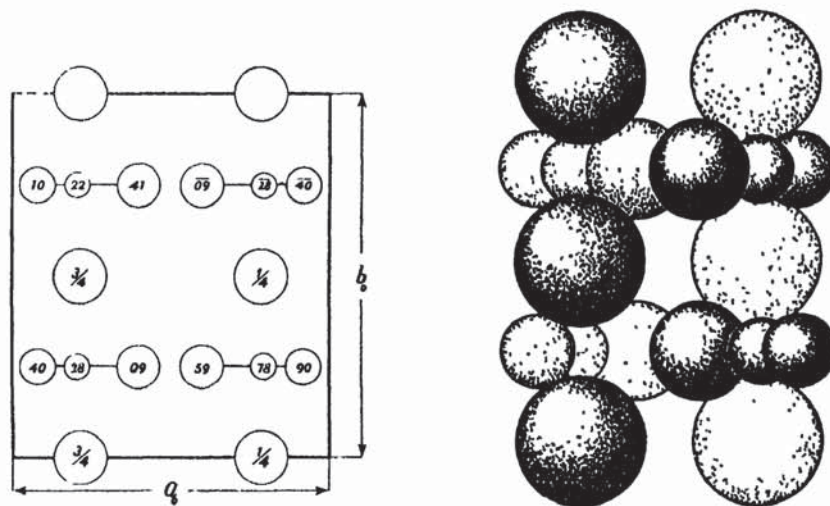


FIG. 290a.—(left) The structure assigned to **KCNS** as projected upon the  $c$ -face of its orthorhombic unit. The largest circles are K atoms; the others, in order of decreasing size, are S, N, and C.

FIG. 290b.—(right) A packing drawing of  $a$ . In this figure K atoms have their ionic size but for lack of better knowledge the other atoms have been assigned their neutral radii.

The thallium salt, **TlCNS**, probably has the same atomic grouping as **KCNS**. In fact one of the two studies (1934, 38) of **TlCNS** makes it orthorhombic with a similarly shaped unit and the same space group  $V_h^{11}$ . Another determination, which presumably is wrong, found it to be tetragonal with a unimolecular cell.

(ax) The orthorhombic unit of **MoO<sub>3</sub>** contains four molecules. Two determinations agree in placing the molybdenum atoms in special positions (c) of  $V_h^{16}$ :  $uv\frac{1}{4}$ ; etc. (see p. 266 of book). The parameters found for

these atoms are practically identical:  $u=0.086$  (0.088),  $v=0.099$  (0.101). According to one study (1931, 484, 485) the oxygen atoms likewise are in three sets of these special positions (c) with  $u_1=0.086$ ,  $v_1=0.25$ ,  $u_2=0.586$ ,  $v_2=0.099$ ,  $u_3=0.086$ ,  $v_3=0.070$ .

(ay) The unit cell of  $\text{PI}_3$  contains two molecules. It is said that the space group is  $C_6^6$  with a structure similar to that of iodoform,  $\text{CHI}_3$  [see p. 372 (z) of book]. The parameters chosen for the iodine atoms are  $x=0.30$ ,  $y=0.35$ ;  $z$  presumably being zero.

(az) No evidence has been obtained that the unit cube of  $\text{ReO}_3$  is larger than the one-molecule cell of the table. The Re atom is at the origin 000; it is thought that the three oxygen atoms are at  $(3b)$   $00\frac{1}{2}$ ;  $0\frac{1}{2}0$ ;  $\frac{1}{2}00$ . This arrangement is said to resemble that of  $\text{WO}_3$  which according to (bb) is triclinic.

(ba) The structure assigned to  $\text{SrPb}_3$  is a slight distortion of the  $\text{NaPb}_3$  grouping (ao). The tetragonal unit contains one molecule with atoms in the following special positions: Sr: 000, Pb:  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$ .

(bb) The cell of  $\text{WO}_3$  listed in the table contains four molecules. The following atomic arrangement, based on  $C_1^1$ , has been reported for its atoms:

W: (i)  $xyz$ ;  $\bar{x}\bar{y}\bar{z}$  with  $x'=\frac{1}{4}$ ,  $y'=\frac{1}{32}$ ,  $z'=\frac{1}{16}$ , and  $x''=\frac{1}{4}$ ,  $y''=\frac{1}{32}$ ,  $z''=-\frac{1}{16}$   
 O: (a) 000, (d)  $\frac{1}{2}00$ , (c)  $0\frac{1}{2}0$ , (e)  $\frac{1}{2}\frac{1}{2}0$ ,  
 (i)  $xyz$ ;  $\bar{x}\bar{y}\bar{z}$  with  $x_1=\frac{1}{4}$ ,  $y_1=\frac{9}{32}$ ,  $z_1=0$ ;  $x_2=\frac{1}{4}$ ,  $y_2=-\frac{7}{32}$ ,  $z_2=0$ ;  $x_3=\frac{1}{4}$ ,  
 $y_3=\frac{1}{32}$ ,  $z_3=\frac{9}{16}$ ;  $x_4=\frac{1}{4}$ ,  $y_4=\frac{1}{32}$ ,  $z_4=\frac{7}{16}$ .

A more thorough study of  $\text{WO}_3$  is obviously needed; whether this arrangement is correct or not, the unit described above is undoubtedly not the simplest one possible.

(bc) The cubic arrangement found for  $\text{LaMg}_3$  and several intermetallic compounds like it has four molecules in the special positions:

La: (4b) 000;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$   
 Mg: (4c)  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ ;  $00\frac{1}{2}$ ;  $0\frac{1}{2}0$ ;  $\frac{1}{2}00$   
 Mg: (4d)  $\frac{1}{4}\frac{1}{4}\frac{1}{4}$ ;  $\frac{1}{4}\frac{3}{4}\frac{3}{4}$ ;  $\frac{3}{4}\frac{1}{4}\frac{3}{4}$ ;  $\frac{3}{4}\frac{3}{4}\frac{1}{4}$   
 Mg: (4e)  $\frac{3}{4}\frac{3}{4}\frac{3}{4}$ ;  $\frac{3}{4}\frac{1}{4}\frac{1}{4}$ ;  $\frac{1}{4}\frac{3}{4}\frac{1}{4}$ ;  $\frac{1}{4}\frac{1}{4}\frac{3}{4}$ .

(bd) A new study of  $\text{Cl}_4$  found diffraction lines incompatible with an  $\text{SnI}_4$ -like grouping. It is said that this new pattern corresponds to a four-molecule unit cube but no structure has been deduced.

(be) The large monoclinic cell of  $\text{Fe}(\text{CO})_4$  described in the table is said to be built upon  $C_{2h}^6$  and to contain 12 molecules. Making the doubtful assumption that this is the true unit it is concluded that the molecule of iron carbonyl is  $[\text{Fe}(\text{CO})_4]_3$ .

(bf) The tetragonal cell of  $\text{LaAl}_4$  is thought to contain 16 molecules.

(bg) An analysis which is undoubtedly wrong makes  $\text{SiF}_4$ , solidified at  $-170^\circ \text{C}$ , cubic with a two-molecule unit having  $a_0 = 5.41 \text{ \AA}$ . Faulty interatomic distances prevail in the suggested arrangement.

(bh) The unit chosen for  $\text{MgZn}_5$  is said to contain 16 molecules. A structure derived from that given to  $\text{MgZn}$  has been discussed.

(bi) A recent study of telluric acid,  $\text{Te}(\text{OH})_6$ , contains evidence which is thought to show that the unit of its cubic modification is not the large 32-molecule cell previously found. This new cube contains four molecules and has half the edge length,  $a_0 = 7.83 \text{ \AA}$ .

Four molecules are also to be found in the monoclinic unit of the second form of  $\text{Te}(\text{OH})_6$ ; the space group is reported to be  $C_{2h}^6$ .

(bj) The unit cube of the  $\text{ThB}_6$  arrangement contains a single molecule and is based on  $O_h^1$ . Placing the metal atom at the origin 000, the boron atoms form an octahedron with the coordinates (Figure 291a):

$$\text{B: (6d) } \frac{1}{2}\frac{1}{2}u; \frac{1}{2}u\frac{1}{2}; u\frac{1}{2}\frac{1}{2}; \frac{1}{2}\frac{1}{2}\bar{u}; \frac{1}{2}\bar{u}\frac{1}{2}; \bar{u}\frac{1}{2}\frac{1}{2}.$$

In  $\text{CaB}_6$ , which has been studied more fully than the other compounds of this type,  $u = 0.207$  giving a B-B separation of  $1.716 \text{ \AA}$ . In Figure 291b where the origin has been translated to a  $\text{B}_6$  center at  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ , the structure appears as a body-centered CsCl packing of metal atoms and boron octahedra.

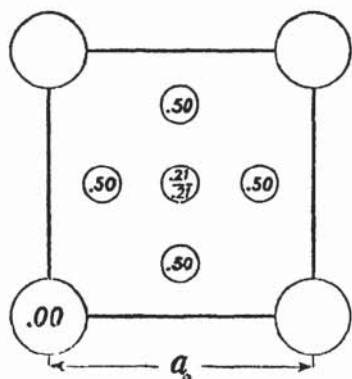
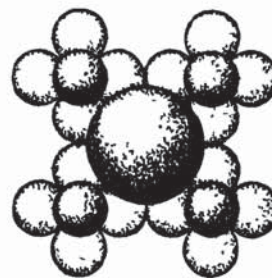


FIG. 291a.—(left) The unit of the  $\text{CaB}_6$  grouping projected on a cube face. Small circles are B atoms.

FIG. 291b.—(right) A packing drawing of  $\text{CaB}_6$  giving the atoms their neutral radii. The calcium atom at the origin of  $\alpha$  has been translated to the cube center of this drawing.



(bk) The brown alkali graphites have been given the composition  $\text{RC}_8$ . Their four-molecule hexagonal cells have  $a_0 = 4.94 \text{ \AA}$ , twice that of graphite. The alkali atoms are between the graphite layers in positions which have not been exactly fixed.

(bl) The black alkali graphites are said to be  $\text{RC}_{16}$ . Their units containing two molecules also have bases with twice the edge length and four times the area of graphite. It is considered that they are derived from the brown graphites by allowing alternate layers of alkali atoms to distill away.

(bm) Crystals of the intermetallic compound  $\text{Sb}_2\text{Tl}_7$  provide an example of a body-centered cubic superlattice. Atomic positions in the

four-molecule cube, as determined from photographic data, are the following special positions of  $O_h^9$  (1930, 352, p. 148):

12 Sb: (12a)  $\pm(u00)$ ;  $\pm(0u0)$ ;  $\pm(00u)$  and 6 similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ , with  $u=0.29$

2 Tl: (2a)  $000$ ;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$

16 Tl: (16d)  $\pm(u'u'u')$ ;  $\pm(u'\bar{u}'\bar{u}')$ ;  $\pm(\bar{u}'u'\bar{u}')$ ;  $\pm(\bar{u}'\bar{u}'u')$  and 8 similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  with  $u'=0.167$

24 Tl: (24j)  $\pm(u_1u_10)$ ;  $\pm(u_1\bar{u}_10)$ ;  $\pm(0u_1u_1)$ ;  $\pm(0u_1\bar{u}_1)$ ;  $\pm(u_10u_1)$ ;  $\pm(\bar{u}_10u_1)$  and 12 similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  with  $u_1=0.35$ .

(bn) The atomic arrangement given to  $Ag_3Hg_4$  has a four-molecule unit cube with atoms in the following special positions of  $O_h^9$ :

Ag: (12h)  $\frac{1}{2}0\frac{1}{4}$ ;  $\frac{1}{2}0\frac{3}{4}$ ;  $\frac{1}{4}\frac{1}{2}0$ ;  $\frac{3}{4}\frac{1}{2}0$ ;  $0\frac{1}{4}\frac{1}{2}$ ;  $0\frac{3}{4}\frac{1}{2}$  and 6 similar coordinates about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$

Hg: (16d)  $uuu$ ;  $u\bar{u}\bar{u}$ ;  $\bar{u}u\bar{u}$ ;  $\bar{u}\bar{u}u$ ;  $\bar{u}\bar{u}\bar{u}$ ;  $\bar{u}uu$ ;  $u\bar{u}u$ ;  $uu\bar{u}$  and 8 similar coordinates about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  with  $u=0.192$ .

(bo) The orthorhombic unit assigned to  $B_{10}H_{14}$  would contain eight molecules; its space group is said to be  $V_h^{21}$ .

(bp) The so-called  $Na_4Pb$  phase of the Na-Pb system has been said to be actually  $Na_{31}Pb_8$  with a cubic structure like that of  $Cu_{31}Sn_8$  (ad).

(bq) The complete structure found for the cubic intermetallic compound  $Cu_{15}Si_4$  is developed from  $T_d^6$ . Silicon atoms in its four-molecule cell are in (1930, 352, p. 131):

$$(16f) \quad uuu; u, \bar{u}, \frac{1}{2}-u; \frac{1}{2}-u, u, \bar{u}; \bar{u}, \frac{1}{2}-u, u; u+\frac{1}{4}, u+\frac{1}{4}, u+\frac{1}{4}; \\ \frac{1}{4}-u, u+\frac{1}{4}, \frac{3}{4}-u; u+\frac{1}{4}, \frac{3}{4}-u, \frac{1}{4}-u; \frac{3}{4}-u, \frac{1}{4}-u, u+\frac{1}{4}$$

and similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ , with  $u=0.208$ . Copper atoms are in:

$$(12k) \quad \frac{3}{8}0\frac{1}{4}; \frac{1}{8}0\frac{3}{4}; \frac{1}{4}\frac{3}{8}0; \frac{3}{4}\frac{1}{8}0; 0\frac{1}{4}\frac{3}{8}; 0\frac{3}{4}\frac{1}{8}$$
 and similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$

and in

$$(e) \quad xyz; x, \bar{y}, \frac{1}{2}-z; \frac{1}{2}-x, y, \bar{z}; \bar{x}, \frac{1}{2}-y, z; \\ zxy; \frac{1}{2}-z, x, \bar{y}; \bar{z}, \frac{1}{2}-x, y; z, \bar{x}, \frac{1}{2}-y; \\ yzx; \bar{y}, \frac{1}{2}-z, x; y, \bar{z}, \frac{1}{2}-x; \frac{1}{2}-y, z, \bar{x}; \\ y+\frac{1}{4}, x+\frac{1}{4}, z+\frac{1}{4}; \frac{1}{4}-y, x+\frac{1}{4}, \frac{3}{4}-z; y+\frac{1}{4}, \frac{3}{4}-x, \frac{1}{4}-z; \frac{3}{4}-y, \frac{1}{4}-x, z+\frac{1}{4}; \\ x+\frac{1}{4}, z+\frac{1}{4}, y+\frac{1}{4}; x+\frac{1}{4}, \frac{3}{4}-z, \frac{1}{4}-y; \frac{3}{4}-x, \frac{1}{4}-z, y+\frac{1}{4}; \frac{1}{4}-x, z+\frac{1}{4}, \frac{3}{4}-y; \\ z+\frac{1}{4}, y+\frac{1}{4}, x+\frac{1}{4}; \frac{3}{4}-z, \frac{1}{4}-y, x+\frac{1}{4}; \frac{1}{4}-z, y+\frac{1}{4}, \frac{3}{4}-x; z+\frac{1}{4}, \frac{3}{4}-y, \frac{1}{4}-x$$

and similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  with  $x=0.12$ ,  $y=0.16$ ,  $z=-0.04$ .

(br) A tetragonal tungsten oxide, of the apparent composition  $W_4O_{11}$ , has been found to have a unit of almost the same size and shape as the triclinic unit assigned to  $WO_3$ . One  $W_4O_{11}$  molecule is contained in this cell and it is thought that its atomic arrangement is practically the same as that of  $WO_3$  with one oxygen atom per cell removed.

(bs) The unit prism of  $Al_3Fe$  (Table I) is said to contain 24 molecules. A recent study makes  $a_0$  four times as big (47.43 Å) with  $V_h^{23}$  as space group. Such a cell is reported to have in it 400 atoms.

(bt) The large cells of  $HfF_4$  and  $ZrF_4$  of Table I would enclose 12 molecules. The space group is  $C_{2h}^6$ .

(bu) If boron carbide is  $B_6C$ , calculation would give it 2.19 molecules per cell. Its composition is therefore considered to be in doubt. The space group is  $D_{3d}^5$ .

(bv) A rhombohedral unit containing one molecule has been found for aluminum carbide,  $Al_4C_3$ . It is said that the Al atoms are in two sets of special positions: (c)  $\pm(uuu)$  of  $D_{3d}^5$  with  $u_1=0.293$  and  $u_2=0.128$ . Two of the three C atoms are in another set with  $u'=0.217$ ; the third is at the origin (a) 000.

### New Structures of the Type $R_x(MX_2)_y$

(ca) The pseudo-tetragonal unit assigned to  $AgClO_2$  is supposed to include 16 molecules.

(cb) The cell described for the monoclinic sulfide **miargyrite**,  $AgSbS_2$ , contains eight molecules. The space group is reported to be  $C_{2h}^6$ .

(cc) The possibly orthorhombic mineral **polybasite**  $(Ag, Cu)_2Sb_2S_4$ , has been given an eight-molecule cell. A space group assignment is  $V_h^{19}$ .

(cd) The orthorhombic unit of  $CaB_2O_4$  contains four molecules. An atomic arrangement found from photographic data places atoms in the following positions of  $V_h^{14}$ :

Ca: (c)  $\frac{1}{4}0u$ ;  $\frac{3}{4}0\bar{u}$ ;  $\frac{1}{4}, \frac{1}{2}, u+\frac{1}{2}$ ;  $\frac{3}{4}, \frac{1}{2}, \frac{1}{2}-u$  with  $u=0.26$

B: (d)  $xyz$ ;  $x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z$ ;  $\bar{x}, y+\frac{1}{2}, \frac{1}{2}-z$ ;  $\frac{1}{2}-x, \bar{y}, z$ ;  $\bar{x}\bar{y}\bar{z}$ ;  $\frac{1}{2}-x, y+\frac{1}{2}, z+\frac{1}{2}$ ;  
 $x, \frac{1}{2}-y, z+\frac{1}{2}$ ;  $x+\frac{1}{2}, y, \bar{z}$  with  $x=0.12, y=0.20, z=0.88$

O<sub>1</sub>: (d)  $x_1y_1z_1$ ; etc. with  $x_1=0.125, y_1=0.21, z_1=0.19$

O': (d)  $x'y'z'$ ; etc. with  $x'=0.11, y'=0.09, z'=0.75$ .

The axes of this description are the same as those of 1930, 352; the origin is in a symmetry center at  $\frac{1}{4}0\frac{1}{4}$ . The linked B-O tetrahedra which make up the framework of this crystal can be seen from Figure 292a and b.

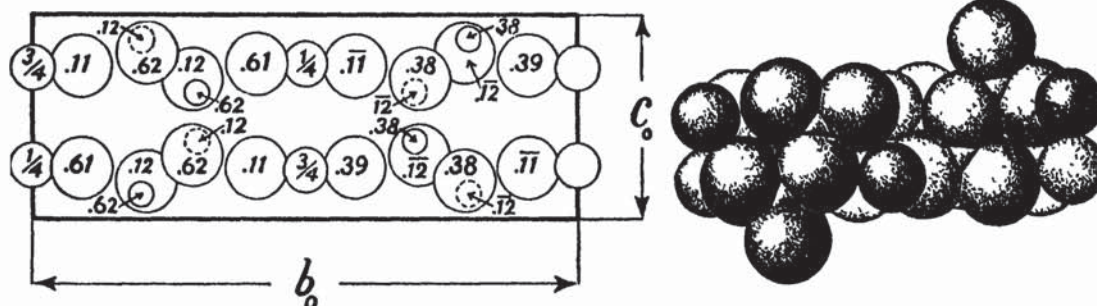


FIG. 292a.—(left) Atoms in the unit prism of  $CaB_2O_4$  projected on the  $a$ -face. The largest circles are O atoms; intermediate ones are Ca.

FIG. 292b.—(right) A packing drawing of  $a$  with Ca and O atoms having their ionic radii. The way the  $BO_4$  tetrahedra are linked together by sharing oxygen atoms is clearly seen.

(ce) The pseudo-cube ascribed to  $\text{Ca}(\text{ClO}_2)_2$  is bimolecular.

(cf) The orthorhombic unit of **emphlectite**,  $\text{CuBiS}_2$ , contains four molecules; its space group is  $V_h^{16}$ . The corresponding antimony compound wolfsbergite,  $\text{CuSbS}_2$ , is structurally isomorphous.

(cg) A redetermination of the structure of **chalcopyrite**,  $\text{CuFeS}_2$ , has led to a somewhat more complicated arrangement. The unit prism, with the same base, has twice the previous height and contains four molecules. Atoms, in special positions of  $V_d^{12}$ , are at:

Cu: (a)  $000$ ;  $\frac{1}{2}0\frac{1}{4}$ ;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ ;  $0\frac{1}{2}\frac{3}{4}$ , Fe: (b)  $00\frac{1}{2}$ ;  $\frac{1}{2}0\frac{3}{4}$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $0\frac{1}{2}\frac{1}{4}$   
 S: (d)  $\frac{1}{4}u\frac{1}{8}$ ;  $u\frac{3}{8}\frac{7}{8}$ ;  $\frac{3}{4}\bar{u}\frac{1}{8}$ ;  $\bar{u}\frac{1}{4}\frac{7}{8}$ ;  $\frac{3}{4}$ ,  $u+\frac{1}{2}$ ,  $\frac{5}{8}$ ;  $u+\frac{1}{2}$ ,  $\frac{1}{4}$ ,  $\frac{3}{8}$ ;  $\frac{1}{4}$ ,  $\frac{1}{2}-u$ ,  $\frac{5}{8}$ ;  $\frac{1}{2}-u$ ,  $\frac{3}{4}$ ,  $\frac{3}{8}$

with  $u=0.27$ . The resulting atomic separations,  $\text{Cu-S}=2.32$  A,  $\text{Fe-S}=2.20$  A, are those to be expected from neutral atoms (Figure 293a and b).

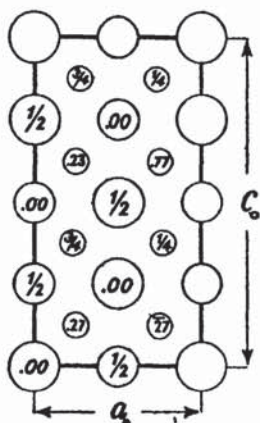


FIG. 293a.—(left) The new arrangement found for chalcopyrite,  $\text{CuFeS}_2$ , as projected upon an  $a$ -face. Cu atoms are at the origin; intermediate circles are the Fe and the smallest circles the S atoms.



FIG. 293b.—(right) A packing drawing of  $\text{CuFeS}_2$  giving the atoms their neutral radii. The line-shaded spheres are Cu.

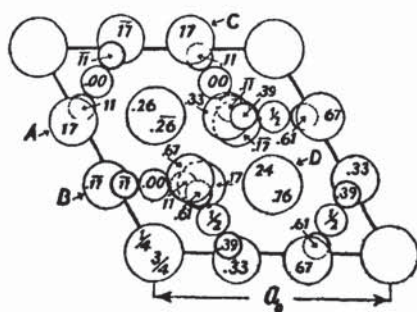


FIG. 294a.—(left) A basal projection of the atoms in the hexagonal unit of the structure found for  $\text{KAg}(\text{CN})_2$ . The largest circles are K atoms, the small heavy ones are C; of the intermediate circles the smaller represent the Ag atoms.

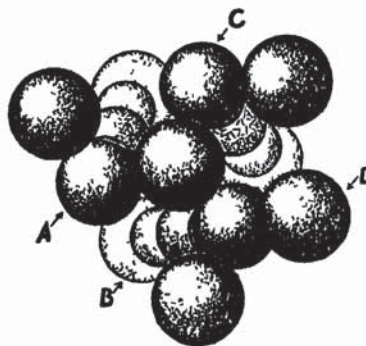


FIG. 294b.—(right) A packing drawing showing half the contents of the unit prism of  $\text{KAg}(\text{CN})_2$ . Corresponding atoms in  $a$  and  $b$  are designated by the same letters. Potassium and silver (line-shaded) atoms have their ionic radii but the sizes of C and N are probably without real significance.

(ch) The six-molecule unit found for  $\text{KAg}(\text{CN})_2$  has an arrangement based on  $D_{3d}^2$ . Its atomic positions have been given as (Figure 294):



K: (b)  $00\frac{1}{4}; 00\frac{3}{4}$  K': (f)  $\frac{1}{3}\frac{2}{3}u; \frac{1}{3}\frac{2}{3}\bar{u}; \frac{2}{3}, \frac{1}{3}, \frac{1}{2}-u; \frac{2}{3}, \frac{1}{3}, u+\frac{1}{2}$  with  $u=0.260$   
 Ag: (h)  $u\bar{u}0; 2\bar{u}, \bar{u}, 0; u, 2u, 0; \bar{u}u\frac{1}{2}; 2u, u, \frac{1}{2}; \bar{u}, 2\bar{u}, \frac{1}{2}$  with  $u=0.167$   
 C: (i)  $xyz; y-x, \bar{x}, z; \bar{y}, x-y, z; x, x-y, \bar{z}; \bar{y}\bar{x}\bar{z}; y-x, y, \bar{z}; \bar{x}, \bar{y}, \frac{1}{2}-z;$   
 $x-y, x, \frac{1}{2}-z; y, y-x, \frac{1}{2}-z; \bar{x}, y-x, z+\frac{1}{2}; y, x, z+\frac{1}{2}; x-y, \bar{y}, z+\frac{1}{2}$

with  $x=0.295, y=\frac{1}{3}, z=0.109$

N: (i)  $x'y'z'$ ; etc. with  $x'=0.365, y'=\frac{1}{3}, z'=0.167$ .

In all other cyanides it has not been possible to establish the separate positions of carbon and nitrogen. Instead the cyanide radical seems to have the spatial characteristics of a sphere with a radius substantially that of the bromide ion. For this reason it is not clear how much significance is to be attached to the C and N parameters stated above and to the short K-N separation (2.56 Å) that results.

(ci) The unit cube assigned to  $K_2Fe_2O_4$  is reported to contain four molecules.

(cj) Eight molecules are associated with the hexagonal prism of  $KFeS_2$ .

(ck) Lithium ferrite,  $Li_2Fe_2O_4$ , is anisotropic if prepared below ca  $600^\circ$  C; above this temperature a cubic modification is produced which does not invert on cooling. The curious fact has been observed that its powder lines correspond to a unit containing one molecule. The intensities of these lines are explicable in terms of an NaCl arrangement [XI, (b)] of O atoms in (4c)  $\frac{1}{2}\frac{1}{2}\frac{1}{2}; 00\frac{1}{2}; 0\frac{1}{2}0; \frac{1}{2}00$  and of Fe and Li atoms irregularly distributed among the positions (4b)  $000; \frac{1}{2}\frac{1}{2}0; \frac{1}{2}0\frac{1}{2}; 0\frac{1}{2}\frac{1}{2}$ .

(cl) Crystals of  $NH_4ClO_2$  are said to have a tetragonal unit holding two molecules. The proposed atomic arrangement places atoms in the following special positions of  $C_{4v}^2$ :

$NH_4$ : (a)  $00u; \frac{1}{2}\frac{1}{2}u$  with  $u=0$

Cl: (b)  $0\frac{1}{2}u'; \frac{1}{2}0u'$  with  $u'=\frac{1}{4}$

O: (c)  $u_1, \frac{1}{2}-u_1, v; u_1+\frac{1}{2}, u_1, v; \bar{u}_1, u_1+\frac{1}{2}, v; \frac{1}{2}-u_1, \bar{u}_1, v$  with  $v=\frac{1}{2}$ .

A more detailed study of this structure would be instructive.

(cm) The original investigation of  $NH_4HF_2$  (1932, 196) gave it the symmetry of  $V_h^{13}$  but failed to find an atomic arrangement. Recently the same data have been shown to be consistent with the following structure developed from  $V_h^7$ :

N: (g)  $\frac{1}{4}\frac{1}{4}u; \frac{1}{4}\frac{3}{4}\bar{u}; \frac{3}{4}\frac{3}{4}\bar{u}; \frac{3}{4}\frac{1}{4}u$  with  $u=0.560$

F: (e)  $u'00; \bar{u}'00; \frac{1}{2}-u', \frac{1}{2}, 0; u'+\frac{1}{2}, \frac{1}{2}, 0$  with  $u'=0.142$

F: (h)  $\frac{1}{2}u_1v; \frac{1}{2}\bar{u}_1\bar{v}; 0, \frac{1}{2}-u_1, v; 0, u_1+\frac{1}{2}, \bar{v}$  with  $u_1=0.132, v=0.135$ .

These axes,  $abc$ , and  $X'Y'Z'$  of 1930, 352 are connected by the relations  $a=X', b=Z', c=Y'$ . The pairs of fluorine atoms belonging to an  $HF_2$  ion are, as should be expected, especially close together with  $F-F=2.37$  Å

(Figure 295a and b). It is customary to assume that the hydrogen atom in these acid fluorides lies midway between the two fluorine atoms on a line joining their centers; such an assumption cannot of course be proved by means of X-rays.

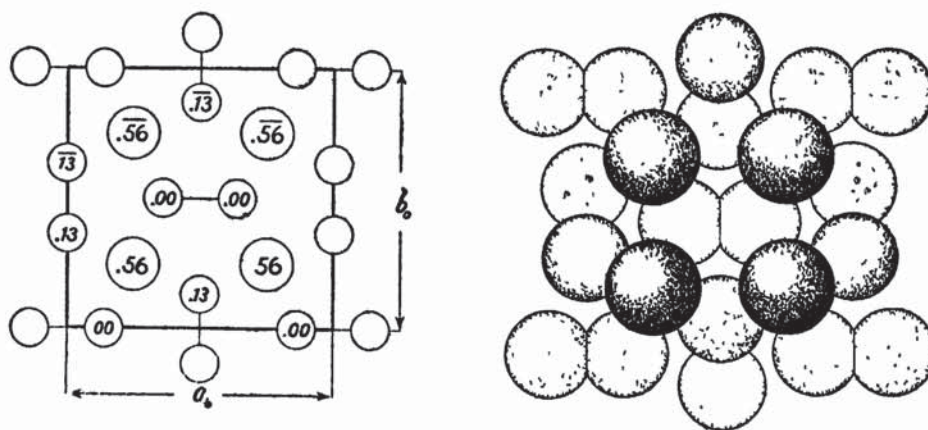


FIG. 295a.—(left) The structure found for  $\text{NH}_4\text{HF}_2$  projected upon the  $c$ -face of its orthorhombic unit. The larger circles are the  $\text{NH}_4$  groups.

FIG. 295b.—(right) A packing drawing of the  $\text{NH}_4$  and  $\text{HF}_2$  ions in  $\text{NH}_4\text{HF}_2$ .

(cn) **Sodium ferrite** is rhombohedral with the  $\text{CsCl}_2\text{I}$  structure ( $d$ ). Atoms of the single  $\text{NaFeO}_2$  molecule in the unit rhombohedron have the coordinates: Na at 000; Fe at  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ ; O at  $uuu$ ;  $\bar{u}\bar{u}\bar{u}$  with  $u=0.22$ .

(co) From photographic data it has been concluded that the atoms in the two-molecule orthorhombic unit of  $\text{NaNO}_2$  are in the following special positions of  $C_{2v}^{20}$ :

Na: (a)  $0u0$ ;  $\frac{1}{2}$ ,  $u+\frac{1}{2}$ ,  $\frac{1}{2}$  with  $u=0.583$

N: (a)  $0u'0$ ;  $\frac{1}{2}$ ,  $u'+\frac{1}{2}$ ,  $\frac{1}{2}$  with  $u'=0.083$

O: (d)  $0u_1v$ ;  $0u_1\bar{v}$ ;  $\frac{1}{2}$ ,  $u_1+\frac{1}{2}$ ,  $v+\frac{1}{2}$ ;  $\frac{1}{2}$ ,  $u_1+\frac{1}{2}$ ,  $\frac{1}{2}-v$  with  $u_1=0$  and  $v=0.194$ .

The coordinates of this description can be derived from those of 1930, 352, p. 56 by an interchange of  $Y'$  and  $Z'$ . The simple structure outlined above is illustrated in Figure 296a and b. The N-O separation in its non-linear  $\text{NO}_2$  ion is 1.13 Å; the Na-O distance, ca 2.48 Å, is substantially that found in  $\text{NaNO}_3$ .

(cp) The supposed pseudo-tetragonal unit of  $\text{Pb}(\text{ClO}_2)_2$  is reported to contain one molecule.

(cq) A monoclinic cell for  $\text{TlAsS}_2$  with the dimensions of the table would enclose eight molecules. The space group has been given as either  $C_{2h}^4$  or  $C_{2h}^5$ .

(cr) Photographic observations have been used to assign an atomic arrangement to crystals of **ammonium hypophosphite**,  $\text{NH}_4\text{H}_2\text{PO}_2$ . Ac-

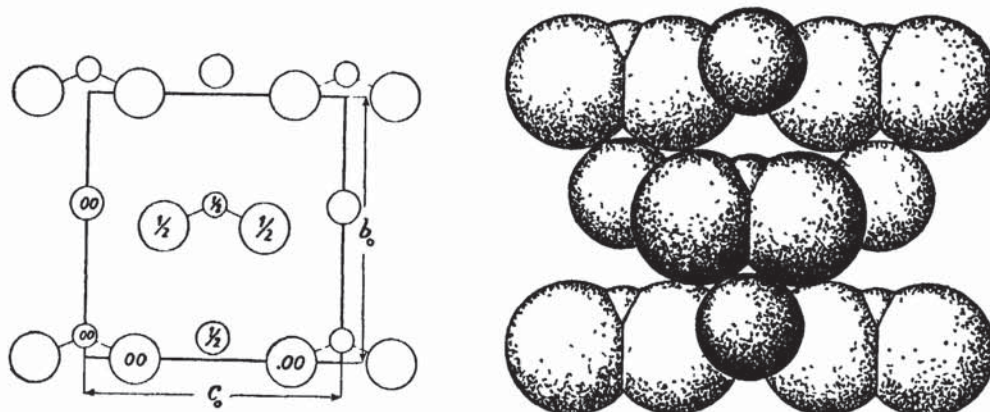


FIG. 296a.—(left) The orthorhombic grouping found for  $\text{NaNO}_2$  projected on its  $a$ -face. Atoms of the non-linear  $\text{NO}_2$  groups are joined by light lines; intermediate circles designate the Na atoms.

FIG. 296b.—(right) A packing drawing of  $a$ .

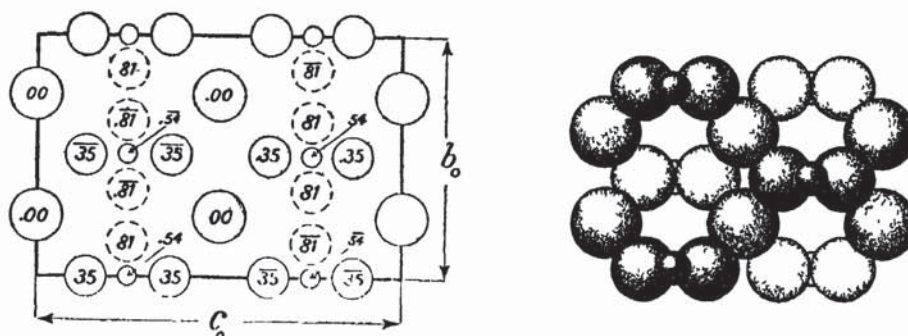


FIG. 297a.—(left) The structure chosen for  $\text{NH}_4\text{H}_2\text{PO}_2$  projected on the  $a$ -face of its orthorhombic cell. K, O and P atoms are shown as large, intermediate and small circles. Proposed positions for the hydrogen atoms are indicated by the dashed circles.

FIG. 297b.—(right) A packing drawing of  $a$  showing the positions of the  $\text{NH}_4$  and  $\text{PO}_2$  groups.

cording to this structure (Figure 297) which places four molecules in the orthorhombic unit, atoms are in the following special positions of  $V_b^{21}$ :

$$\begin{aligned} \text{NH}_4: & (a) \pm(0\frac{1}{4}0); \pm(0\frac{3}{4}\frac{1}{2}) & \text{P:} & (g) \pm(u0\frac{1}{4}); \pm(u\frac{1}{2}\frac{3}{4}) \\ \text{O:} & (m) \pm(u'0v'); \pm(u'\frac{1}{2}\bar{v}'); \pm(u', 0, \frac{1}{2}-v'); \pm(u', \frac{1}{2}, v'+\frac{1}{2}). \end{aligned}$$

The axes of this description ( $abc$ ) arise from those of 1930, 352, p. 67 ( $X'Y'Z'$ ) by transferring the origin to a center of symmetry and using the transformation  $a=Z'$ ,  $b=X'$ ,  $c=Y'$ .

The chosen parameters  $u(\text{P})=0.541$ ,  $u'(\text{O})=0.347$ ,  $v'=0.136$  give an  $\text{NH}_4\text{-O}$  separation (2.81 Å) which is unusually short. This has been considered to show that the  $\text{NH}_4$  groups are not rotating; such an interpretation could be convincing only if the correctness of the selected parameters were supported by more quantitative evidence than is now available.

Suggested hydrogen positions, which would bind each atom to two  $\text{NH}_4$  groups and one phosphorus atom are  $(m) u_10v_1$ ; etc. with  $u_1=0.805$ ,  $v_1=0.142$ ; they cannot of course be checked by X-ray observations.

## Chapter XVA. Structures of the Type $R_x(MX_3)_y$

(ab) The parameters of Table I have been assigned to the atoms in divalent nitrates having the structure (s) characteristic of  $Ba(NO_3)_2$ . It has been suggested (1931, 265) that at ordinary temperatures the nitrate groups in  $Ca(NO_3)_2$  are rotating but data in support of this idea have not been published.

TABLE I. PARAMETERS FOR CRYSTALS OF THE ALKALINE EARTH NITRATES

<i>Crystal</i>	$u(N)$	$x(O)$	$y(O)$	$z(O)$
$Ba(NO_3)_2$	0.150	0.220	0.204	0.026
$Ca(NO_3)_2$	.161	.247	.207	.033
$Pb(NO_3)_2$	.156	.234	.209	.033
$Sr(NO_3)_2$	.159	.236	.209	.032

(ac) The unit prism of **bromlite** (alstonite),  $BaCa(CO_3)_2$ , contains two molecules. It is similar in shape to the orthorhombic cells of barite and aragonite with dimensions lying between them. Nevertheless this mineral is thought to be a compound rather than a solid solution.

(ad) The monoclinic **barytocalcite**, also  $BaCa(CO_3)_2$ , has been assigned a two-molecule unit. The space group is reported as  $C_2^2$ .

(ae) The three hexagonal carbonates **synchisite**,  $CaCO_3 \cdot RFeCO_3$ , **parisite**,  $CaCO_3 \cdot 2RFeCO_3$ , and **cordylite**,  $BaCO_3 \cdot 2RFeCO_3$  (R is a mixture of trivalent rare earth atoms, Ce, La, etc.) have unit prisms with bases of about equal size but with very different heights. Closely related atomic arrangements, which however need further confirmation, have been proposed for these minerals. The following atomic coordinates are necessary for their description:

$$\begin{array}{ll}
 \text{(a) } 00w; 0, 0, w+\frac{1}{2} & \text{(b) } 00v; 00\bar{v}; 0, 0, v+\frac{1}{2}; 0, 0, \frac{1}{2}-v \\
 \text{(c) } \frac{1}{3}\frac{2}{3}u; \frac{2}{3}\frac{1}{3}\bar{u}; \frac{2}{3}, \frac{1}{3}, u+\frac{1}{2}; \frac{1}{3}, \frac{2}{3}, \frac{1}{2}-u & \text{(d) } \frac{2}{3}\frac{1}{3}t; \frac{1}{3}, \frac{2}{3}, t+\frac{1}{2}.
 \end{array}$$

Oxygen atoms have not been located; the other atomic positions together with the corresponding parameters (in parentheses) are listed in Table II.

TABLE II. ATOMIC POSITIONS AND PARAMETERS GIVEN TO THE ATOMS IN SYNCHISITE, PARISITE AND CORDYLITE

Substance	$z$ Ca (or Ba)	$z$ F	$z$ R	$z$ CO <sub>3</sub>	$4$ R	$4$ F	$4$ CO <sub>3</sub>
Synchisite	a	d	a	—	—	—	c
CaCO <sub>3</sub> ·RFeCO <sub>3</sub>	(w=0)	( $\frac{2}{3}$ )	( $\frac{1}{3}$ )	—	—	—	(-0.117)
Parisite	a	—	—	d	b	c	c
CaCO <sub>3</sub> ·2RFeCO <sub>3</sub>	(0)	—	—	( $\frac{1}{3}$ )	(0.163)	(0.163)	(-0.076)
Cordylite	a	—	—	d	c	b	c
BaCO <sub>3</sub> ·2RFeCO <sub>3</sub>	(0)	—	—	( $\frac{1}{3}$ )	( $\frac{1}{7}$ )	( $\frac{1}{7}$ )	(-0.07)

(af) A new structure has been proposed for **bastnäsite**, (Ce, La, . . .) FCO<sub>3</sub>, based on  $D_{3h}^4$  instead of  $D_{3h}^3$ . Its atoms have been put in the positions:

- 6 R: (g) uu0; etc. of 1930, 352, p. 159, with  $u = \frac{2}{3}$   
 2 F: (a) 000;  $00\frac{1}{2}$       4 F: (f)  $\frac{1}{3}\frac{2}{3}u$ ; etc. with  $u = ca\ 0$ .  
 12 O: (i) xyz; etc. with  $x=y=ca\ \frac{1}{3}$ ,  $z=ca\ \frac{1}{7}$   
 6 O: (h)  $uv\frac{1}{4}$ ; etc. with  $u$  and  $v$  undetermined  
 6 C: (h)  $u'v'\frac{1}{4}$ ; etc. with  $u'$  and  $v'$  undetermined.

(ag) Parameters have been determined for the atoms in **KNO<sub>3</sub>** and **PbCO<sub>3</sub>**. As Table III indicates they are almost identical with one another and with those previously found for aragonite (b) (see p. 272 of book).

TABLE III. PARAMETERS OF THE ATOMS IN KNO<sub>3</sub>, PbCO<sub>3</sub> AND ARAGONITE

Atom	KNO <sub>3</sub>			PbCO <sub>3</sub>			CaCO <sub>3</sub>		
	x	y	z	x	y	z	x	y	z
K, Pb, Ca	0	0.416	0	0	0.417	0	0	0.417	0
N, C, C	0	.75	$\frac{1}{6}$	0	.764	0.153	0	.75	$\frac{1}{6}$
O(1)	0	.883	$\frac{1}{6}$	0	.908	.153	0	.917	$\frac{1}{6}$
O(2)	0.194	.686	$\frac{1}{6}$	0.205	.692	.153	0.23	.67	$\frac{1}{6}$

(ah) A structure based on photographic data has been deduced for the iodine and oxygen atoms in **LiIO<sub>3</sub>**. These atoms in the two-molecule unit are placed in the following special positions of  $D_6^6$ :

I: (c)  $\frac{1}{3}\frac{2}{3}\frac{1}{4}$ ;  $\frac{2}{3}\frac{1}{3}\frac{3}{4}$       O: (g) uu0; 0 $\bar{u}$ 0;  $\bar{u}$ 00;  $\bar{u}\bar{u}\frac{1}{2}$ ;  $0u\frac{1}{2}$ ;  $u0\frac{1}{2}$  with  $u = \frac{1}{3}$ .

If the lithium atoms are in (b)  $00\frac{1}{4}$ ;  $00\frac{3}{4}$  a reasonable Li-O separation, 2.23 Å, is obtained. It should be noticed (Figure 298) that this arrange-

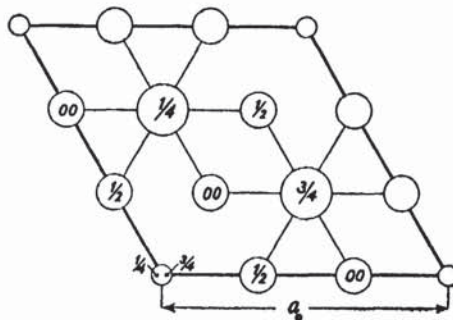


FIG. 298.—A basal projection of the arrangement proposed for **LiIO<sub>3</sub>**. The Li atoms are represented by the largest, the I by the smallest circles. The absence of IO<sub>3</sub> ions in this grouping is evident.

TABLE IV. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $R_x(MX_3)_y$ 

Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References
AgFO <sub>3</sub>	Tetragonal (?)	5.33	6.08	1933, 117.
BaCO <sub>3</sub>	Ortho- rhombic	KNO <sub>3</sub> (b) 5.2556	6.5490 b <sub>o</sub> =8.8345	1931, 89.
BaCa(CO <sub>3</sub> ) <sub>2</sub> (Bromlite)	Ortho- rhombic	(ac) 8.77	6.11 b <sub>o</sub> =4.99	1930, 111b.
BaCa(CO <sub>3</sub> ) <sub>2</sub> (Barytocalcite)	Monoclinic	(ad) 8.15	6.58 b <sub>o</sub> =5.22, $\beta=83^\circ52'$	1930, 111c.
BaCO <sub>3</sub> ·2RFeCO <sub>3</sub> (Cordylite)	Hexagonal	(ae) 4.35	22.8	1931, 337.
BaCeO <sub>3</sub>	Cubic	CaTiO <sub>3</sub> (d) 4.377		1934, 112.
Ba(NO <sub>3</sub> ) <sub>2</sub>	Cubic	Ba(NO <sub>3</sub> ) <sub>2</sub> (s), (ab)		1931, 460.
BaThO <sub>3</sub>	Cubic	CaTiO <sub>3</sub> (d) 4.480		1934, 112.
BaZrO <sub>3</sub>	Cubic	CaTiO <sub>3</sub> (d) 4.176		1934, 112.
CaCO <sub>3</sub> (Calcite)	Hexagonal	(ap)		1931, 34, 35, 122; 1934, 283.
CaCO <sub>3</sub> ·RFeCO <sub>3</sub> (Synchisite)	Hexagonal	(ae) 4.094	18.20	1931, 337.
CaCO <sub>3</sub> ·2RFeCO <sub>3</sub> (Parisite)	Hexagonal	(ae) 4.094	27.93	1931, 337.
CaMg(CO <sub>3</sub> ) <sub>2</sub> (Dolomite)	Hexagonal	(v) 6.050± 0.004	46°54'	1930, 398.
Ca(NO <sub>3</sub> ) <sub>2</sub>	Cubic	Ba(NO <sub>3</sub> ) <sub>2</sub> (s), (ab)		1931, 265, 460.
CaSn(BO <sub>3</sub> ) <sub>2</sub> (Nordenskiöldite)	Hexagonal	(v) 6.24	45°44'	1934, 219.
CdTlO <sub>3</sub> (low temp. form)	Hexagonal	FeTiO <sub>3</sub> (ax) 5.82	53°36'	1934, 216.
(Ce, La . . .). FeCO <sub>3</sub> (Bastnäsite)	Hexagonal	(af) 7.094	9.718	1931, 336.
CoCO <sub>3</sub>	Hexagonal	NaNO <sub>3</sub> (a)		1932, 14.
CoTiO <sub>3</sub>	Hexagonal	FeTiO <sub>3</sub> (ax) 5.49	54°42'	1934, 216.
CsNO <sub>3</sub>	Hexagonal	(aw) 10.74	7.68	1934, 273.
Cs <sub>2</sub> S <sub>2</sub> O <sub>6</sub>	Hexagonal	(au) 6.326	11.535	1932, 187.
FeCO <sub>3</sub> * (Siderite)	Hexagonal	NaNO <sub>3</sub> (a) 5.754	47°25'	1932, 167.
FeTiO <sub>3</sub> (Ilmenite)	Hexagonal	FeTiO <sub>3</sub> (ax) 5.52	54°50'	1934, 14, 216.
H <sub>3</sub> BO <sub>3</sub>	Triclinic	(ay) 7.04	6 56† b <sub>o</sub> =7.04	1934, 304.
InBO <sub>3</sub>	Hexagonal	NaNO <sub>3</sub> (a) 5.841	48°10'	1932, 167.
KCbO <sub>3</sub>	Cubic (?)	CaTiO <sub>3</sub> (d) 4.005		1932, 371.
KNO <sub>3</sub>	Ortho- rhombic	KNO <sub>3</sub> (b), (ag) 5.43	6.45 b <sub>o</sub> =9.17	1931, 102.

\* 92.5% FeCO<sub>3</sub>, 6.1% MnCO<sub>3</sub>.† For H<sub>3</sub>BO<sub>3</sub>,  $\alpha=92^\circ30'$ ,  $\beta=101^\circ10'$ ,  $\gamma=120^\circ$ .

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
$K_2S_2O_6$	Hexagonal	( <i>av</i> )	9.756	6.274	1931, 30, 220; 1932, 186, 201; 1933, 210, 211.
$KTaO_3$	Cubic (?)	$CaTiO_3$ ( <i>d</i> )	3.981		1932, 371.
$LaBO_3$	Ortho-rhombic	$KNO_3$ ( <i>b</i> )	5.10	5.83	1932, 167.
				$b_o=8.22$	
$LiIO_3$	Hexagonal	( <i>ah</i> )	5.469	5.155	1931, 499.
$MgTiO_3$	Hexagonal	$FeTiO_3$ ( <i>ax</i> )	5.54	$54^\circ 39'$	1934, 216.
$Mn_3As_2O_6$ (Armangite)	Hexagonal	( <i>ai</i> )	13.44	8.72	1933, 5.
$MnTiO_3$	Hexagonal	$FeTiO_3$ ( <i>ax</i> )	5.62	$54^\circ 16'$	1934, 216.
$NH_4IO_3$	Cubic	$CaTiO_3$ ( <i>d</i> )	4.5		1932, 158.
$NH_4NO_3$ (I) ( $169.5^\circ$ to $125.2^\circ$ C range)	Cubic	( <i>aj</i> )	4.40		1931, 265; 1932, 204.
$NH_4NO_3$ (II) ( $125.2^\circ$ to $84.2^\circ$ C range)	Tetragonal	( <i>ak</i> )	5.75	5.00	1931, 265; 1932, 204.
$NH_4NO_3$ (III) ( $84.2^\circ$ to $32.3^\circ$ C range)	Ortho-rhombic	( <i>al</i> )	7.06	5.80	1932, 204.
				$b_o=7.66$	
$NH_4NO_3$ (IV) ( $32.3^\circ$ to $-18^\circ$ C range)	Ortho-rhombic	( <i>am</i> )	5.75	4.96	1932, 204, 470.
				$b_o=5.45$	
$NH_4NO_3$ (V) (below $-18^\circ$ C)	Hexagonal	( <i>an</i> )	5.75	15.9	1932, 204.
$NaClO_3$	Cubic (?)	$CaTiO_3$ ( <i>d</i> )	3.889		1932, 371.
$NaHCO_3$	Monoclinic	( <i>ao</i> )	7.51	3.53	1933, 518.
				$b_o=9.70, \beta=93^\circ 19'$	
$NaNO_3$	Hexagonal	$NaNO_3$ ( <i>a</i> ), ( <i>ap</i> )			1931, 266; 1932, 49; 1933, 492; 1934, 235.
$Na_2SO_3$	Hexagonal	( <i>aq</i> )	5.441	6.133	1931, 500.
$NaSbO_3 \cdot 4BcO$ (Swedenborgite)	Hexagonal	( <i>y</i> ), ( <i>as</i> )			1933, 3.
$NaTaO_3$	Cubic (?)	$CaTiO_3$ ( <i>d</i> )	3.881		1932, 371.
$NaWO_3$ (Cubic Na-W Bronze)	Cubic	$CaTiO_3$ ( <i>d</i> ), ( <i>ar</i> )	3.83		1932, 250.
$Na_2(WO_3)_5$ (?) (Blue Na-W Bronze)	Tetragonal	( <i>ar</i> )	17.5	3.80	1932, 251.
(Na, Ce, Ca). (Ti, Cb) $O_3$ (Loparite)	Cubic (?)	$CaTiO_3$ ( <i>d</i> )	3.854		1930, 391.
$NiTiO_3$	Hexagonal	$FeTiO_3$ ( <i>ax</i> )	5.45	$55^\circ 8'$	1934, 216.
$PbCO_3$ (Cerussite)	Ortho-rhombic	$KNO_3$ ( <i>b</i> ), ( <i>ag</i> )	5.166	6.146	1933, 101.
				$b_o=8.468$	
$Pb(NO_3)_2$	Cubic	$Ba(NO_3)_2$ ( <i>s</i> ), ( <i>ab</i> )			1931, 460.
$RbNO_3$	Ortho-rhombic	( <i>at</i> )	18.08	7.38	1933, 351.
				$b_o=10.45$	
$Rb_2S_2O_6$	Hexagonal	$K_2S_2O_6$ ( <i>av</i> )	10.144	6.409	1931, 220; 1932, 186.
$ScBO_3$	Hexagonal	$NaNO_3$ ( <i>a</i> )	5.78?	$48^\circ 28'$	1932, 167.

Substance, symmetry and structure type			$a_0$	$c_0$ or $\alpha$	References
SrHfO <sub>3</sub>	Cubic	CaTiO <sub>3</sub> ( <i>d</i> )	4.069		1933, 204.
Sr(NO <sub>3</sub> ) <sub>2</sub>	Cubic	Ba(NO <sub>3</sub> ) <sub>2</sub> ( <i>s</i> ), ( <i>ab</i> )			1931, 460.
SrZrO <sub>3</sub>	Cubic	CaTiO <sub>3</sub> ( <i>d</i> )	4.089		1933, 204.
YBO <sub>3</sub>	Hexagonal	NaNO <sub>3</sub> ( <i>a</i> )	6.44	46°17'	1932, 167.
ZnCO <sub>3</sub>	Hexagonal	NaNO <sub>3</sub> ( <i>a</i> )	5.669	48°26'	1932, 167.

ment does not provide either simple or complex iodate ions such as would be expected on chemical grounds; instead each iodine atom is made equidistant from six oxygen atoms (I-O=2.23 Å=Li-O). For this reason a further study of LiIO<sub>3</sub> must sometime be made.

(*ai*) The hexagonal unit of **armangite**, **Mn<sub>3</sub>As<sub>2</sub>O<sub>6</sub>**, recorded in Table IV would contain nine molecules. It is thought that the true unit is probably rhombohedral, with a space group that is  $C_{3v}^5$ ,  $D_3^7$  or  $D_{3d}^5$ .

(*aj*) The **highest** temperature modification of **NH<sub>4</sub>NO<sub>3</sub>** seems to give the simple diffraction pattern required by a one-molecule cube in which N atoms and NO<sub>3</sub> groups have a body-centered CsCl grouping [XI, (*a*)]. Individual crystals of this modification grow so fast that good intensity data could not be obtained but the single molecule unit has been taken as evidence for a rotating NO<sub>3</sub> group.

(*ak*) The unit of the **second**, tetragonal, form of **NH<sub>4</sub>NO<sub>3</sub>** contains two molecules. Even at 100° C these crystals grew too fast to yield good diffraction data and no *z* parameters could be established. The *x* and *y* parameters are said to be the following:

$$\begin{array}{lll} \text{NH}_4: & 00?; \frac{1}{2}\frac{1}{2}? & \text{N: } 0\frac{1}{2}?; \frac{1}{2}0? & \text{O: } 0\frac{1}{2}?; \frac{1}{2}0? \\ \text{O: } & xy?; \bar{x}\bar{y}?; \bar{y}x?; y\bar{x}? & \text{with } x=0.14, y=0.36. \end{array}$$

(*al*) The **third** modification of **NH<sub>4</sub>NO<sub>3</sub>** has a four-molecule orthorhombic prism and a structure based on  $V_h^{16}$ . Choosing the same axial orientation that was used for cementite [XIV, (*o*)] atoms have been found to be in the positions:

$$\begin{array}{ll} \text{NH}_4: & \text{(c) } uv\frac{1}{4}; \text{ etc. (book, p. 266) with } u=0.30, v=0.52 \\ \text{N: } & \text{(c) } u'v'\frac{1}{4}; \text{ etc. with } u'=-0.09, v'=-0.19 \\ \text{O: } & \text{(c) } u_1v_1\frac{1}{4}; \text{ etc. with } u_1=-0.19, v_1=-0.05 \\ \text{O: } & \text{(d) } xyz; \text{ etc. with } x=-0.07, y=-0.27, z=0.06. \end{array}$$

This arrangement is illustrated in Figure 299a and b.

(*am*) Two separate determinations have shown that the two molecules in the orthorhombic unit of **NH<sub>4</sub>NO<sub>3</sub>** which is stable at ordinary temperatures are arranged according to the unusual space group  $V_h^{13}$ . With axes chosen as in Table IV they agree in placing atoms in the following special positions:



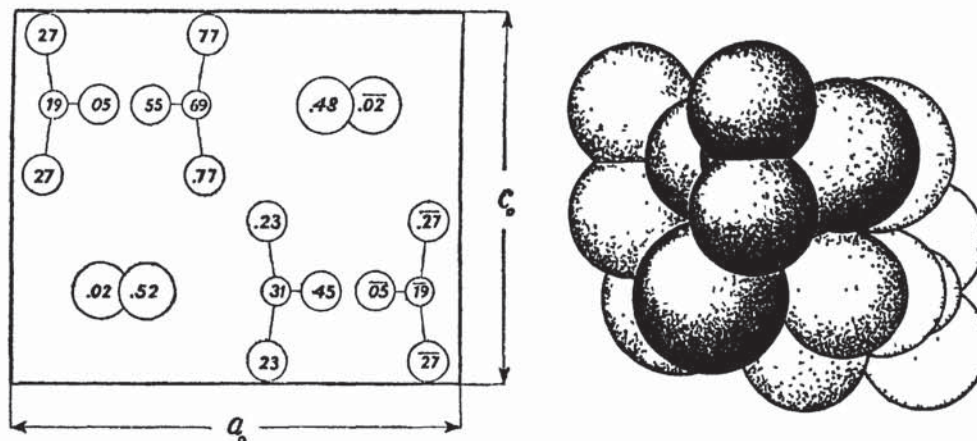
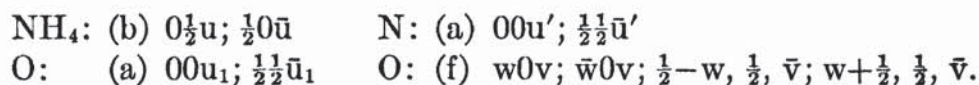


FIG. 299a.—(left) The atoms of the third modification of  $\text{NH}_4\text{NO}_3$  projected on the b-face of its orthorhombic unit. Atoms of the  $\text{NO}_3$  groups are joined by light lines.

FIG. 299b.—(right) A packing drawing of  $a$ .



The origin used in 1932, 470 is displaced one half along the  $c$ -axis; therefore though the parameters as listed in Table V are different, the atomic arrangements found in these two investigations are nearly identical. This can be seen from Figure 300a, wherein the unit of 1932, 204 is outlined by heavy lines, that of 1932, 470 using dotted lines.

TABLE V. PARAMETERS OF THE ATOMS IN  $\text{NH}_4\text{NO}_3$   
(Room Temperature Form IV)

Determination	$u(\text{NH}_4)$	$u'(N)$	$u_1(O)$	$w$	$v$
1932, 204	0.57	0.03	0.28	0.19	-0.095
1932, 470	.097	.500	.75	.183	.375

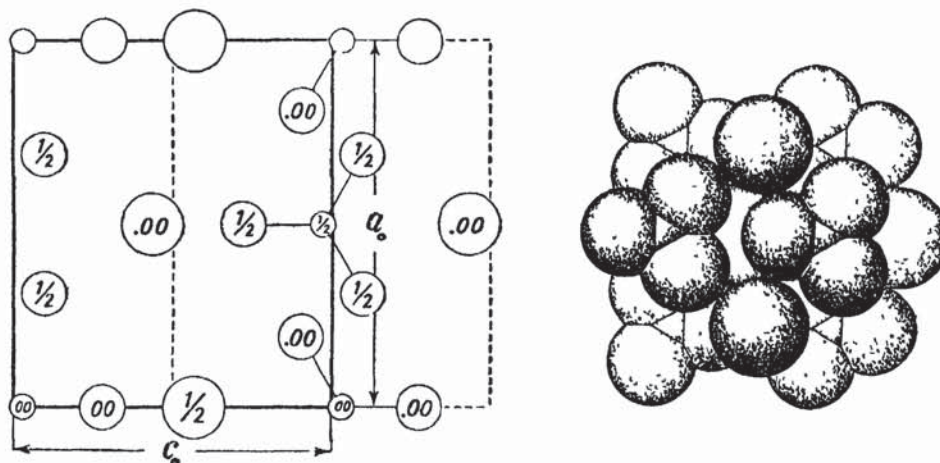


FIG. 300a.—(left) The structure of the fourth, room temperature, form of  $\text{NH}_4\text{NO}_3$  projected on its b-face. The unit cells of the two determinations are indicated by full and by dotted lines. The largest circles are  $\text{NH}_4$  ions, the smallest are N atoms.

FIG. 300b.—(right) A packing drawing of  $a$ .

(an) The fifth modification of  $\text{NH}_4\text{NO}_3$  (stable below  $-18^\circ\text{C}$ ) has been given a hexagonal, or pseudo-hexagonal, unit containing six molecules. No X-ray evidence was found which indicated the gradual transition at  $-60^\circ\text{C}$ .

(ao) The monoclinic unit chosen for  $\text{NaHCO}_3$  contains four molecules. Using photographic spectral data it has been given an atomic arrangement with all atoms in general positions of  $C_{2h}^5$  (Figure 301):  $(e) \pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2})$ . The selected parameters are listed in Table VI. In this structure the distance between oxygen atoms in adjacent  $\text{CO}_3$  groups is 2.55 Å. Such a close approach has been thought to mean that these atoms are bound by an intermediate hydrogen atom which then would be at  $x=0.319, y=0.250, z=0.064$ . The atomic parameters of Table VI and with them this evidence for the existence of a hydrogen bond should be confirmed by more quantitative intensity data.

The coordinates used in this description refer to axes so chosen that the gliding component is along the diagonal to two of them. In the conventional description it is along one axis.

TABLE VI. PARAMETERS OF THE ATOMS IN  $\text{NaHCO}_3$

Atom	$x$	$y$	$z$
Na	0.278	0.0	0.708
C	.069	.236	.314
O(1)	.069	.367	.314
O(2)	.200	.169	.183
O(3)	.939	.169	.444

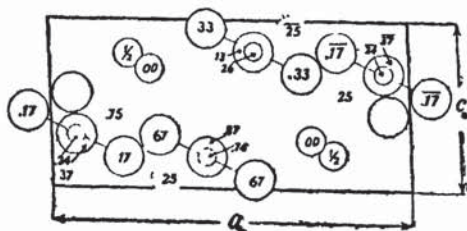


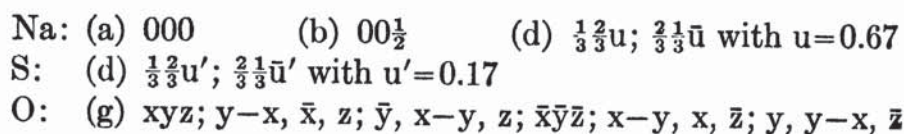
FIG. 301a.—(left) Atoms in the proposed structure for  $\text{NaHCO}_3$  projected on the  $b$ -face of its monoclinic unit. The large O and small C atoms of the  $\text{CO}_3$  groups are joined by light lines; positions thought probable for the hydrogen atoms are indicated by the dotted circles.

FIG. 301b.—(right) A packing drawing showing the small  $\text{Na}^+$  and the larger  $\text{CO}_3$  groups of  $a$ .

(ap) X-ray photographs of  $\text{NaNO}_3$  made at various temperatures up to  $280^\circ\text{C}$  have been interpreted as showing that the  $\text{NO}_3$  group is rotating at high temperature. At  $280^\circ\text{C}$  the unit rhombohedron has the dimensions  $a_0=6.56\text{Å}$ ,  $\alpha=45^\circ35'$ .

Similar measurements of unit cell size at higher temperatures, as well as Laue photographs up to  $600^\circ\text{C}$ , have been made of calcite ( $\text{CaCO}_3$ ).

(aq) Using data derived from twinned crystals the atoms in the two-molecule unit of  $\text{Na}_2\text{SO}_3$  have been placed in the following positions of  $C_{3i}^1$  (Figure 302):



with  $x=0.14, y=0.40, z=0.25$ .

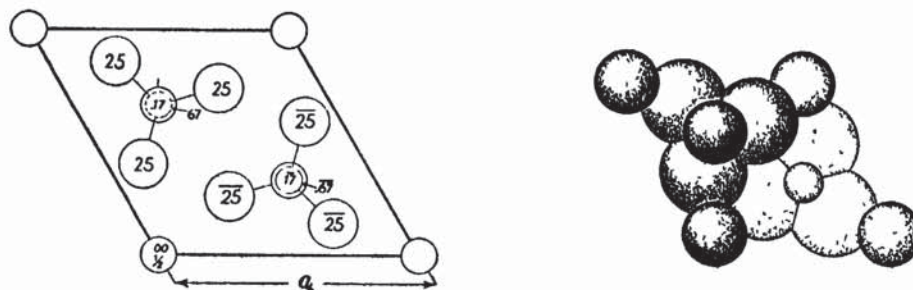


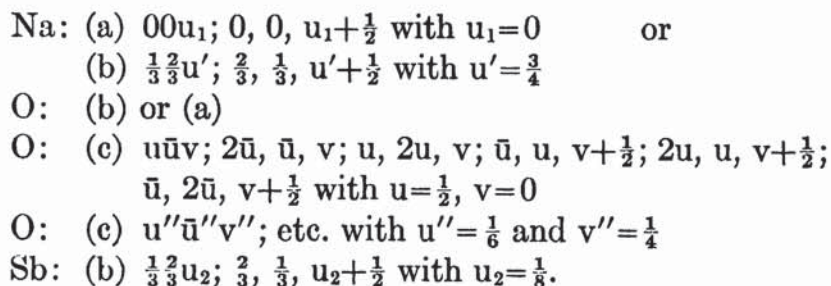
FIG. 302a.—(left) A basal projection of atoms in the hexagonal unit of  $\text{Na}_2\text{SO}_3$ . The smallest circles are the S and the largest the O atoms.

FIG. 302b.—(right) A packing drawing of  $a$ . One S atom (at  $z=0.17$ ) is shown lying above the plane of its three O atoms.

(ar) The analyses of cubic **Na-W bronzes** run from  $\text{Na}_2\text{W}_2\text{O}_8$  to apparently  $\text{Na}_2\text{W}_7\text{O}_{21}$ . This variation in composition is thought due to the gradual replacement of sodium by hydrogen.

Blue Na-W bronzes are made by the weak reduction of  $\text{NaWO}_3$  by zinc or hydrogen. The composition approaches that stated in Table IV.

(as) The formula previously given to the mineral **swedenborgite** is wrong due to the interpretation of its beryllium as aluminum. Its unit contains two of the new molecules  $\text{NaSbO}_3 \cdot 4\text{BeO}$ . One or the other of the following two structures developed from  $C_{6v}^4$  has been considered to be correct:



(at) A reexamination of **RbNO<sub>3</sub>** has led to a different structure. The large orthorhombic unit of Table IV contains 18 molecules. The crystal, however, is pseudo-hexagonal; if its slight departure from this higher symmetry is neglected, the data are those to be expected from a structure with

$a_o = 10.45$ ,  $c_o = 7.38$ , having nine molecules in the unit and  $C_{3v}^2$  as space group. The previously chosen unit was rhombohedral (or pseudo-rhombohedral) with an arrangement developed from  $C_{3v}^5$  [see (n), p. 279 of book].

(au) The unit prism of  $Cs_2S_2O_6$  contains two molecules; the space group is given as either  $D_6^6$  or  $D_{3h}^4$ . Cesium and sulfur atoms are assigned to the special positions:

$$\begin{array}{ll} \text{Cs: (a) } 000; 00\frac{1}{2} & \text{Cs: (c) } \frac{1}{3}\frac{2}{3}\frac{1}{4}; \frac{2}{3}\frac{1}{3}\frac{3}{4} \\ \text{S: (f) } \frac{1}{3}\frac{2}{3}u; \frac{2}{3}\frac{1}{3}\bar{u}; \frac{2}{3}, \frac{1}{3}, u+\frac{1}{2}; \frac{1}{3}, \frac{2}{3}, \frac{1}{2}-u & \text{with } u=0.70. \end{array}$$

No X-ray selection could be made between the two sets of oxygen positions that were considered possible.

(av) Four studies have been made of the structure of **potassium dithionate**,  $K_2S_2O_6$ . From them it is clear that the hexagonal unit contains three molecules and that atoms are in the following special positions of  $D_2^2$ :

$$\begin{array}{ll} 2 \text{ S: (c) } 00u; 00\bar{u} & 4 \text{ S in 2 sets of: (d) } \frac{1}{3}\frac{2}{3}u; \frac{2}{3}\frac{1}{3}\bar{u} \\ 3 \text{ K: (e) } uu0; 0\bar{u}0; \bar{u}00 & 3 \text{ K: (f) } uu\frac{1}{2}; 0\bar{u}\frac{1}{2}; \bar{u}0\frac{1}{2} \\ 18 \text{ O in 3 sets of: (g) } xyz; y-x, \bar{x}, z; \bar{y}, x-y, z; & \\ & yx\bar{z}; \bar{x}, y-x, \bar{z}; x-y, \bar{y}, \bar{z}. \end{array}$$

This crystal provides an instructive example of two very different arrangements (see oxygen parameters) that agree with the qualitative data from a group of spectral photographs. It has been shown that these data are about equally well explained by the two sets of parameters of Table VII. The second set (according to 1932, 201) gives so short a K-O separation, ca 2.2 Å, that it cannot be right. The satisfactory K-O distances, of ca 2.80 Å, yielded by the parameters of 1933, 210 suggest that they may be near the true values (Figure 303a and b).

(aw) The hexagonal unit which has been ascribed to  $CsNO_3$  contains nine molecules. No atomic arrangement has been deduced but the curious observation has been made that its powder pattern is nearly identical with that of the cubic (or pseudo-cubic)  $KIO_3$  and very similar to that of the cubic  $CsI$ . No change in pattern occurs on heating  $CsNO_3$  up to 200° C.

TABLE VII. PARAMETERS FOR THE ATOMS OF  $K_2S_2O_6$

Atom	Parameters according to					
	1933, 210			1932, 201		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>(x and y interchanged)</i>		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>
S(1)	0	0	0.16	0	0	0.16
S(2)	$\frac{1}{3}$	$\frac{2}{3}$	.59	$\frac{1}{3}$	$\frac{2}{3}$	.59
S(3)	$\frac{1}{3}$	$\frac{2}{3}$	.27	$\frac{1}{3}$	$\frac{2}{3}$	.27
K(1)	0.375	0.375	0	0.39	0.39	0
K(2)	.69	.69	$\frac{1}{2}$	.69	.69	$\frac{1}{2}$
O(1)	.165	.11	.23	.09	.18	.22
O(2)	.615	.17	.34	.48	.24	.35
O(3)	.505	.21	.80	.58	.42	.79

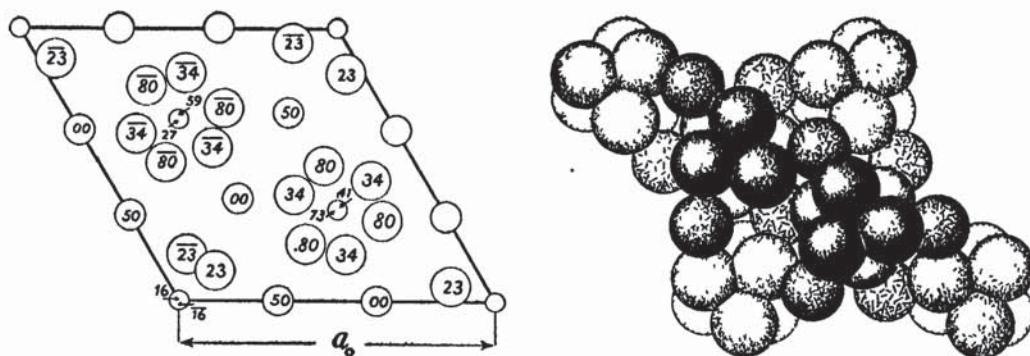
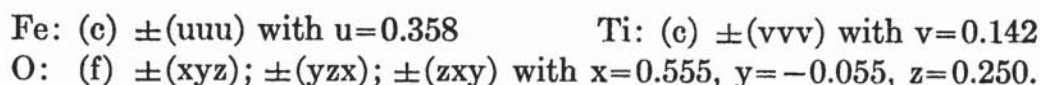


FIG. 303a.—(left) A basal projection of the atoms in the hexagonal unit of  $K_2S_2O_6$ . The largest circles are O, the smallest are S atoms.

FIG. 303b.—(right) A packing drawing of  $a$ . The K ions are indicated by line-shading.

(ax) The mineral ilmenite,  $FeTiO_3$ , has a rhombohedral structure similar to the  $Fe_2O_3$  arrangement [XIII, (a)]. Its corresponding space group,  $C_{3i}^2$ , is of lower symmetry because of the non-identity of its metal atoms but the two-molecule rhombohedra are of nearly the same size and shape. In  $FeTiO_3$ , atoms have been given the positions:



As might be expected from the close similarity in their cell sizes, it has been found that ilmenite and  $Fe_2O_3$  form a continuous series of solid solutions (1934, 216).

Nickel titanate,  $NiTiO_3$ , has the ilmenite structure. The parameters assigned to its atoms are identical, within the limit of experimental error, with those of  $FeTiO_3$ .

Cadmium titanate,  $CdTiO_3$ , occurs in two forms. The previously described structure, isomorphous with  $CaTiO_3$  (d), is found in material prepared by quenching from above  $1000^\circ C$ . Crystals made below this temperature are like  $FeTiO_3$ . The parameters given their atoms,  $u(Cd)=0.342$ ,  $v(Ti)=0.156$ ,  $x=0.54$ ,  $y=-0.03$ ,  $z=0.26$ , yield the short Cd-O distance of 2.24 Å but it is said that other values would make it shorter still.

(ay) The triclinic cell chosen for crystals of boric acid,  $H_3BO_3$ , includes four molecules. If the space group is  $C_1^1$ , as is undoubtedly the case, all atoms are in general positions  $\pm(xyz)$ . Boron and oxygen atoms have been assigned parameters (Table VIII) which yield a thoroughly platy structure. The resulting interatomic distances are B-O=1.36 Å and, between adjacent groups, O-O=2.71 Å. It is stated that this O-O separation is sufficiently below the normal 2.80 Å to show that hydrogen atoms are situated between them. Inasmuch as the entire determination of structure, involving many variable parameters, has been based on

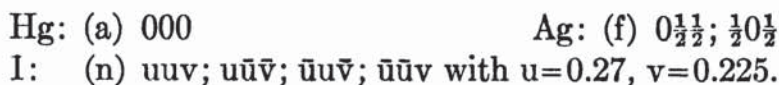
TABLE VIII. PARAMETERS FOR THE ATOMS OF  $\text{H}_3\text{BO}_3$ 

<i>Atom</i>	<i>x</i>	<i>y</i>	<i>z</i>
B(1)	0.653	0.430	0.25
B(2)	.319	.764	.25
O(1)	.430	.319	.25
O(2)	.764	.319	.25
O(3)	.764	.653	.25
O(4)	.208	.542	.25
O(5)	.208	.875	.25
O(6)	.542	.875	.25

qualitative visual estimates of photographic intensities, it is hard to attach much significance to this argument. In several instances unexpectedly short interatomic distances have been ascribed to hydrogen bonds rather than to errors or inaccuracies in parameter determinations. It should be pointed out that, except with certain especially favorable crystals (such as the alkali acid fluorides), intensity data better than the usual qualitative estimates on simple reflections are needed to fix parameters with enough certainty to provide real evidence for such bonds.

## Chapter XVIA. Structures of the Type $R_x(MX_4)_y$

(*ac*) The unimolecular tetragonal cell of  $\beta$ - $\text{Ag}_2\text{HgI}_4$  has atoms in the following special positions of  $V_d^1$  (Figure 304a and b):



The form of  $\text{Cu}_2\text{HgI}_4$  stable at room temperature has the same structure with  $u=0.255$ ,  $v=0.275$ .

(*ad*) The  $\alpha$ -modification of  $\text{Ag}_2\text{HgI}_4$ , stable above  $50^\circ\text{C}$ , is said to be truly cubic. The arrangement in the low temperature form (*ac*) is a distortion of the ZnS structure; this  $\alpha$ -structure is described as an exact ZnS grouping [XI, (c)] with three-fourths of the positions (4b)  $000$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$  occupied by an irregular distribution of Hg+2 Ag.

The  $\alpha$ -form of  $\text{Cu}_2\text{HgI}_4$ , stable above  $70^\circ\text{C}$ , is like the silver salt.

(*ae*) Three studies have been made of the structure of anhydrous sodium sulfate,  $\text{Na}_2\text{SO}_4$ . They agree in choosing an eight-molecule unit and in selecting  $V_h^{24}$  as corresponding space group. The atomic arrange-

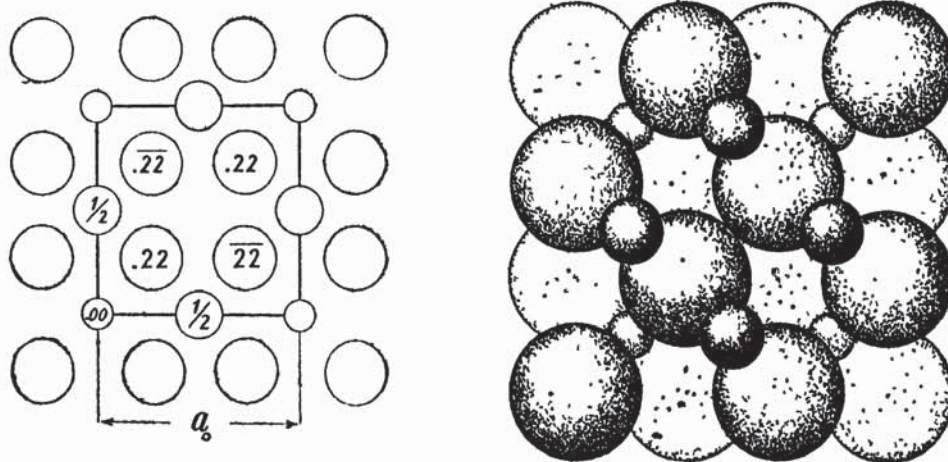


FIG. 304a.—(left) A c-face projection of atoms of the room temperature ( $\beta$ ) modification of  $\text{Ag}_2\text{HgI}_4$ . Atoms of I are represented by the largest, of Hg by the smallest circles.

FIG. 304b.—(right) A packing drawing of  $a$  with the atoms given their usual ionic sizes.

TABLE I. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $R_x(MX_4)_y$ 

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
$\beta$ -Ag <sub>2</sub> HgI <sub>4</sub>	Tetragonal	Ag <sub>2</sub> HgI <sub>4</sub> ( <i>ac</i> )	6.340	6.340	1931, 257.
$\alpha$ -Ag <sub>2</sub> HgI <sub>4</sub> (stable above 50° C)	Cubic	( <i>ad</i> )	6.383		1934, 133.
AgIO <sub>4</sub>	Tetragonal	CaWO <sub>4</sub> ( <i>d</i> )	5.368	12.013	1932, 51.
AgReO <sub>4</sub>	Tetragonal	CaWO <sub>4</sub> ( <i>d</i> )	5.349	11.916	1933, 81.
Ag <sub>2</sub> SO <sub>4</sub>	Ortho- rhombic	Na <sub>2</sub> SO <sub>4</sub> ( <i>ae</i> )	5.847	10.251 $b_o=12.659$	1931, 179; 1932, 492.
Ag <sub>3</sub> SbS <sub>4</sub> (Stephanite)	Ortho- rhombic	( <i>af</i> )	7.85	8.58 $b_o=12.48$	1932, 394.
Ag <sub>2</sub> SeO <sub>4</sub>	Ortho- rhombic	Na <sub>2</sub> SO <sub>4</sub> ( <i>ae</i> )	6.069	10.211 $b_o=12.815$	1931, 179.
BAsO <sub>4</sub>	Tetragonal	BPO <sub>4</sub> ( <i>ag</i> )	4.459	6.796	1933, 421; 1934, 240.
BPO <sub>4</sub>	Tetragonal	BPO <sub>4</sub> ( <i>ag</i> )	4.334	6.636	1933, 421; 1934, 240.
BaWO <sub>4</sub>	Tetragonal	CaWO <sub>4</sub> ( <i>d</i> ), ( <i>ah</i> )	5.64	12.70	1931, 344; 1932, 247.
BeNaPO <sub>4</sub> (Beryllonite)	Monoclinic	( <i>ba</i> )	8.13	14.17 $b_o=7.76, \beta=90^\circ$	1934, 86.
CaCrO <sub>4</sub>	Tetragonal	ZrSiO <sub>4</sub> ( <i>f</i> ), ( <i>ai</i> )	7.25	6.34	1930, 381; 1932, 106.
Ca(F, Cl)Ca <sub>4</sub> (PO <sub>4</sub> ) <sub>3</sub> (Apatite)	Hexagonal	( <i>aj</i> )			1930, 426; 1931, 298, 380; 1932, 203.
CaMg(OH)AsO <sub>4</sub> (Adelite)	Ortho- rhombic	( <i>ay</i> )	5.88	7.43 $b_o=8.85$	1933, 7.
CaMg(OH)AsO <sub>4</sub> (Tilasite)	Monoclinic	( <i>ay</i> )	5.68	7.57 $b_o=8.63, \beta=91^\circ 28'$	1933, 7.
CdCr <sub>2</sub> S <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	10.190		1931, 347.
CdFe <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.45		1931, 116.
CoAl <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.101		1931, 269; 1932, 30.
(Co, Ni) <sub>3</sub> S <sub>4</sub> (Linneite)	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	9.41	(for several minerals)	1931, 318.
CoSO <sub>4</sub>	Ortho- rhombic*		4.65	8.45 $b_o=6.71$	1931, 209.
Co <sub>2</sub> TiO <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.420		1930, 246c; 1931, 212.
CsOsNO <sub>3</sub>	Ortho- rhombic	( <i>al</i> )	8.08	7.22 $b_o=8.35$	1932, 239, 241.
CsReO <sub>4</sub>	Ortho- rhombic	( <i>am</i> )	5.73	14.26 $b_o=5.98$	1933, 222.
Cs <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	Monoclinic	( <i>bb</i> )	8.13	6.46 $b_o=8.33, \beta=95^\circ 19'$	1934, 306.
CuAl <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.064 †		1931, 269; 1932, 223.
Cu <sub>3</sub> AsS <sub>4</sub> (Enargite)	Ortho- rhombic	( <i>an</i> )	6.46	6.18 $b_o=7.43$	1933, 463; 1934, 208.
CuFe <sub>2</sub> O <sub>4</sub> (quenched)	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )			1934, 281.

\* This unit contains four molecules.

† The other determination (1931, 269) gives  $a_o=8.074$  A.



Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
CuFe <sub>2</sub> O <sub>4</sub> (annealed)	Tetragonal	(ak)	8.28	8.68	1934, 281.
Cu <sub>2</sub> FeSnS <sub>4</sub> (Stannite)	Tetragonal	(bd)	5.46	10.725	1923, 64; 1934, 318.
$\beta$ -Cu <sub>2</sub> HgI <sub>4</sub>	Tetragonal	Ag <sub>2</sub> HgI <sub>4</sub> (ac)	6.08	6.135	1931, 257.
$\alpha$ -Cu <sub>2</sub> HgI <sub>4</sub> (stable above 70° C)	Cubic	(ad)	6.103		1934, 133.
Cu <sub>3</sub> VS <sub>4</sub> (Sulvanite)	Cubic	(u), (ao)	5.370		1933, 350.
FeAl <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.119*		1931, 80, 269; 1932, 30.
FeCr <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k)	8.344		1931, 80.
(Fe, Mg)Cr <sub>2</sub> O <sub>4</sub> (Chromite)	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ap)			1932, 104.
Fe <sub>3</sub> O <sub>4</sub> (Magnetite)	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (aq)	8.374		1931, 80; 1932, 345; 1934, 77.
Fe <sub>2</sub> TiO <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.50		1932, 30.
FeV <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k)			1932, 302.
Ga <sub>2</sub> ZnO <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k)	8.323		1931, 75.
KBF <sub>4</sub>	Ortho- rhombic	BaSO <sub>4</sub> (a)	7.84	7.38	1930, 436.
KClO <sub>4</sub> (low)	Ortho- rhombic	BaSO <sub>4</sub> (a), (ar)	8.834	$b_o=5.68$ 7.240	1931, 404; 1932, 177.
K <sub>2</sub> CrO <sub>4</sub>	Ortho- rhombic	K <sub>2</sub> SO <sub>4</sub> (m), (as)	5.92	$b_o=5.650$ 7.61	1931, 88, 501.
K <sub>2</sub> Mg <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> (Langbeinite)	Cubic	(at)	9.96	$b_o=10.40$	1931, 134.
KMnO <sub>4</sub>	Ortho- rhombic	BaSO <sub>4</sub> (a), (ar)	9.09	7.41 $b_o=5.72$	1931, 306.
KO <sub>3</sub> NO <sub>3</sub>	Tetragonal	CaWO <sub>4</sub> (d), (ah)	5.65	13.08	1932, 240, 241.
Li(Fe, Mn)PO <sub>4</sub> (Triphylite)	Ortho- rhombic	(au)	4.67	6.00 $b_o=10.34$	1932, 175.
Li <sub>3</sub> PO <sub>4</sub>	Ortho- rhombic	(au)	4.86	6.07 $b_o=10.26$	1932, 495.
Li <sub>2</sub> SO <sub>4</sub>	Monoclinic	(av)	8.25	8.44 $b_o=4.95, \beta=107^{\circ}54'$	1932, 5a.
MgAl <sub>2</sub> O <sub>4</sub> (Spinel)	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.059 ± 0.004		1931, 80, 286; 1932, 101, 165, 286; 1934, 51.
MgCr <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k)	8.305		1931, 80.
MgFe <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.366		1931, 33, 80, 270; 1932, 30.
MgGa <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.279		1931, 33; 1932, 30, 198, 295.

\* In 1931, 269,  $a_o=8.084$  A.

Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References
MgIn <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.81	1932, 30.
Mg <sub>2</sub> TiO <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.44	1931, 212; 1932, 30.
MnAl <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.271	1931, 80, 269; 1932, 30.
MnCr <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.436	1931, 80.
MnCr <sub>2</sub> S <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	10.045	1931, 347.
MnFe <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.457	1931, 80.
Mn <sub>2</sub> TiO <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.67	1931, 212.
(NH <sub>4</sub> ) <sub>2</sub> BeF <sub>4</sub>	Ortho- rhombic	K <sub>2</sub> SO <sub>4</sub> ( <i>m</i> ), ( <i>as</i> )	5.8 b <sub>o</sub> =10.2	7.5 1934, 114.
NH <sub>4</sub> ClO <sub>4</sub> (low)	Ortho- rhombic	BaSO <sub>4</sub> ( <i>a</i> ), ( <i>ar</i> )	9.202 b <sub>o</sub> =5.816	7.449 1931, 404; 1932, 177.
(NH <sub>4</sub> ) <sub>2</sub> CrO <sub>4</sub>	Monoclinic	( <i>aw</i> )	6.15 b <sub>o</sub> =6.27, $\beta=115^\circ 13'$	7.66 1931, 73.
NH <sub>4</sub> OsNO <sub>3</sub>	Ortho- rhombic	( <i>ax</i> )	5.53 b <sub>o</sub> =5.86	13.54 1932, 238, 241.
(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	Monoclinic	( <i>bb</i> )	7.83 b <sub>o</sub> =8.04, $\beta=95^\circ 9'$	6.13 1934, 306.
Na <sub>2</sub> SO <sub>4</sub>	Ortho- rhombic	Na <sub>2</sub> SO <sub>4</sub> ( <i>r</i> ), ( <i>ae</i> )	5.85 b <sub>o</sub> =12.29	9.75 1931, 87; 1932, 493.
NiAl <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.050	1931, 269; 1932, 30.
NiCr <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.30	1932, 224.
Pb <sub>10</sub> Cl <sub>2</sub> (AsO <sub>4</sub> ) <sub>6</sub> (Mimetite)	Hexagonal	( <i>aj</i> )		1932, 203.
Pb <sub>10</sub> Cl <sub>2</sub> (PO <sub>4</sub> ) <sub>6</sub> (Pyromorphite)	Hexagonal	( <i>aj</i> )		1932, 203.
Pb <sub>10</sub> Cl <sub>2</sub> (VO <sub>4</sub> ) <sub>6</sub> (Vanadinite)	Hexagonal	( <i>aj</i> )		1932, 203.
PbCrO <sub>4</sub> (Krokoite)	Monoclinic	( <i>i</i> )	7.10 b <sub>o</sub> =7.40, $\beta=102^\circ 27'$	6.80 1931, 63.
PbZn(OH)VO <sub>4</sub> (Descloizite)	Ortho- rhombic	( <i>az</i> )	6.05 b <sub>o</sub> =9.39	7.56 1933, 24.
RbOsNO <sub>3</sub>	Ortho- rhombic	( <i>ax</i> )	5.57 b <sub>o</sub> =5.84	13.64 1932, 238, 241.
RbReO <sub>4</sub>	Tetragonal	CaWO <sub>4</sub> ( <i>d</i> )	5.80	13.17 1933, 222.
TlOsNO <sub>3</sub>	Ortho- rhombic	( <i>ax</i> )	5.42 b <sub>o</sub> =5.68	13.45 1932, 238, 241.
TlRcO <sub>4</sub>	Ortho- rhombic	( <i>am</i> )	5.63 b <sub>o</sub> =5.80	13.33 1932, 222.
YVO <sub>4</sub>	Tetragonal	ZrSiO <sub>4</sub> ( <i>f</i> )	7.126	6.197 1933, 75.
ZnAl <sub>2</sub> O <sub>4</sub>	Cubic*	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> ), ( <i>ak</i> )	8.062	1931, 80, 269; 1932, 30, 165, 198.
ZnCr <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.296	1931, 80.
ZnCr <sub>2</sub> S <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	9.92	1931, 318.
ZnFe <sub>2</sub> O <sub>4</sub>	Cubic	MgAl <sub>2</sub> O <sub>4</sub> ( <i>k</i> )	8.423	1931, 80.

\* In 1931, 269 and 1932, 198,  $a_o=8.093$  A.

Substance, symmetry and structure type	$a_o$	$c_o$ or $\alpha$	References
ZnSO <sub>4</sub> Orthorhombic (bc)	8.58	4.76	1934, 237.
		$b_o = 6.73$	
Zn <sub>2</sub> SnO <sub>4</sub> Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k), (ak)	8.61	1932, 30.
Zn <sub>2</sub> TiO <sub>4</sub> Cubic	MgAl <sub>2</sub> O <sub>4</sub> (k)	8.410	1930, 246c; 1931, 212.

ments proposed in the first two investigations are obviously wrong since they are chemically unreasonable, give unsatisfactory interatomic distances and fail to agree with observed intensities of reflection. The most recent structure (1932, 493), illustrated in Figure 305a and b, meets these requirements, the data being drawn from spectral photographs. Transferring the origin of 1930, 352, p. 69 to a center of symmetry at  $\frac{1}{8}\frac{1}{8}\frac{1}{8}$ , atoms are in the following positions:

- 8 S: (a)  $\pm(\frac{1}{8}\frac{1}{8}\frac{1}{8})$  and 6 similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}\frac{1}{2}0$  and  $0\frac{1}{2}0$   
 16 Na: (g)  $\pm(\frac{1}{8}\frac{1}{8}u)$ ;  $\pm(\frac{1}{8}, \frac{1}{8}, \frac{1}{4}-u)$  and 12 similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}\frac{1}{2}0$ ,  $\frac{1}{2}0\frac{1}{2}$  with  $u=0.436$   
 32 O: (h)  $\pm(xyz)$ ;  $\pm(x, \frac{1}{4}-y, \frac{1}{4}-z)$ ;  $\pm(\frac{1}{4}-x, y, \frac{1}{4}-z)$ ;  $\pm(\frac{1}{4}-x, \frac{1}{4}-y, z)$  and 24 similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}\frac{1}{2}0$ ,  $\frac{1}{2}0\frac{1}{2}$  with  $x=-0.022$ ,  $y=0.056$ ,  $z=0.214$ .

Silver sulfate, Ag<sub>2</sub>SO<sub>4</sub>, and the corresponding selenate, Ag<sub>2</sub>SeO<sub>4</sub>, are isomorphous with Na<sub>2</sub>SO<sub>4</sub>. The structure given them from a study of their photographic reflections is not like that outlined above and yields improbable interatomic distances. It has since been shown that these data are explicable in terms of the Na<sub>2</sub>SO<sub>4</sub> structure with the following slightly different parameters for Ag<sub>2</sub>SO<sub>4</sub>:  $u(\text{Ag})=0.450$ ,  $x=0.022$ ,  $y=0.058$ ,  $z=0.208$ .

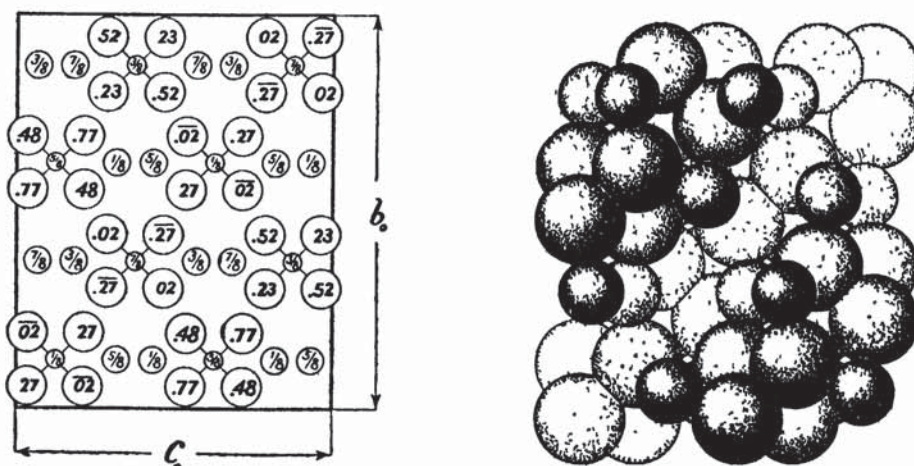


FIG. 305a.—(left) Atoms in the orthorhombic arrangement found for Na<sub>2</sub>SO<sub>4</sub> projected on an a-face. The atoms forming the SO<sub>4</sub> ions are joined by light lines.

FIG. 305b.—(right) A packing drawing of the Na<sup>+</sup> and SO<sub>4</sub><sup>2-</sup> ions in Na<sub>2</sub>SO<sub>4</sub>.

(af) The cell of **stephanite**,  $\text{Ag}_5\text{SbS}_4$ , contains four molecules; its space group is said to be  $V_h^{17}$ .

(ag) The tetragonal cells of  $\text{BPO}_4$  and  $\text{BaSO}_4$  are bimolecular. According to a structure developed from  $S_4^2$  they have atoms in the following positions:

B: (c)  $0\frac{1}{2}\frac{1}{4}; \frac{1}{2}0\frac{3}{4}$

P (or As): (a)  $000; \frac{1}{2}\frac{1}{2}\frac{1}{2}$

O: (g)  $xyz; \bar{y}x\bar{z}; \bar{x}\bar{y}z; y\bar{x}\bar{z}$ ;

$x+\frac{1}{2}, y+\frac{1}{2}, z+\frac{1}{2}; \frac{1}{2}-y, x+\frac{1}{2}, \frac{1}{2}-z; \frac{1}{2}-x, \frac{1}{2}-y, z+\frac{1}{2}; y+\frac{1}{2}, \frac{1}{2}-x, \frac{1}{2}-z$ .

For  $\text{BPO}_4$ ,  $x=0.138$ ,  $y=0.260$ ,  $z=0.131$ ; for  $\text{BaSO}_4$ ,  $x=0.160$ ,  $y=0.260$ ,  $z=0.140$ . This arrangement, as a distortion of the high cristobalite grouping [XII, (ae), (bd)], consists of linked  $\text{BO}_4$  and P (or As)  $\text{O}_4$  tetrahedra (Figure 306a and b).

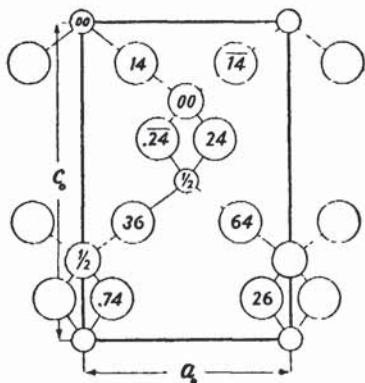


FIG. 306a.—(left) Atoms of the structure chosen for  $\text{BPO}_4$  projected on one of the  $a$ -faces of its tetragonal cell. The smallest circles are P, the intermediate circles B atoms.

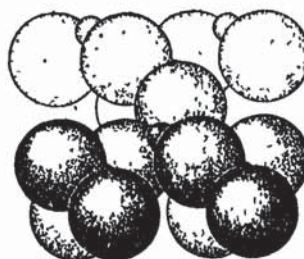


FIG. 306b.—(right) A packing drawing of  $a$ . The O atoms have their ionic radius; the size of the B atom is without significance.

(ah) Every study of crystals with the  $\text{CaWO}_4$  (d) arrangement has resulted in different oxygen parameters. A new set, for  $\text{BaWO}_4$ , is  $x=0.20$ ,  $y=0.46$ ,  $z=0.32$ .

Potassium osmiumate,  $\text{KOsNO}_3$ , is reported to have this structure with N and O atoms indistinguishable from one another. The parameters chosen for these atoms are  $x=0.23$ ,  $y=0.05$ ,  $z=-0.065$ .

(ai) The positions found for the oxygen atoms in  $\text{CaCrO}_4$  are those established in other crystals having the zircon grouping (f):  $u=0.17$ ,  $v=0.34$ .

(aj) X-ray measurements have been made upon a number of substances with structures like **apatite**,  $\text{Ca}(\text{F}, \text{Cl})\text{Ca}_4(\text{PO}_4)_3$ , (z). The hexagonal unit prisms found in this way are recorded in Table II.

In the apatite arrangement (z) fluorine atoms are in (a)  $00\frac{1}{4}; 00\frac{3}{4}$ . Another possible pair of positions, which could not be rigorously excluded by the observed intensities, would place them in the larger holes (b)  $000; 00\frac{1}{2}$ . It has been shown that in the lead compounds,  $\text{Pb}_{10}\text{Cl}_2(\text{MO}_4)_6$ , where  $\text{M}=\text{P}$  or  $\text{As}$ , packing requires that the chlorine atoms must be in

these alternative positions (b). Parameters chosen to give suitable packing throughout the structure for these two crystals and for the chlor-X-apatite of Table II are listed in Table III. The previously found values for apatite itself are included for comparison.

TABLE II. UNIT CELLS OF APATITE-LIKE SUBSTANCES

Name	Formula	$a_0$	$c_0$
Apatite	$Ca_{10}(F, Cl)_2(PO_4)_6$	9.36	6.85
Chlor-X-Apatite	$Ca_{10}(Cl, X)_2(PO_4)_6$	9.52	6.85
Pyromorphite	$Pb_{10}Cl_2(PO_4)_6$	9.95	7.31
		9.95	7.32
Mimetite	$Pb_{10}Cl_2(AsO_4)_6$	10.24	7.43
		10.36	7.52
Vanadinite	$Pb_{10}Cl_2(VO_4)_6$	10.31	7.34
		10.47	7.43
Hydroxy-Apatite	$Ca_{10}(OH)_2(PO_4)_6$	9.40	6.93
Tricalcium Phosphate Hydrate	$Ca_9(H_2O)_2(PO_4)_6$	9.25	6.88
Oxy-Apatite	$Ca_{10}O(PO_4)_6$	9.38	6.93
Bone (Naphtha extracted)		9.27	6.95

TABLE III. PARAMETERS IN APATITE AND RELATED CRYSTALS

Atom	Apatite			Pyromorphite			Mimetite			Chlor-X-Apatite		
	$x$	$y$	$z$	$x$	$y$	$z$	$x$	$y$	$z$	$x$	$y$	$z$
F, Cl	0	0	$\frac{1}{2}$	0	0	0	0	0	0	0	0	0
Ca, Pb(1) (f)	$\frac{1}{2}$	$\frac{1}{2}$	0	$\frac{1}{2}$	$\frac{1}{2}$	0	$\frac{1}{2}$	$\frac{1}{2}$	0	$\frac{1}{2}$	$\frac{1}{2}$	0
Ca, Pb(2) (h)	$\frac{1}{2}$	0	$\frac{1}{2}$	$\frac{1}{2}$	0.003	$\frac{1}{2}$	$\frac{1}{2}$	0	$\frac{1}{2}$	$\frac{1}{2}$	0	$\frac{1}{2}$
P, As (h)	0.416	0.361	$\frac{1}{2}$	0.417	.369	$\frac{1}{2}$	0.411	0.392	$\frac{1}{2}$	0.417	0.361	$\frac{1}{2}$
O(1) (h)	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	.344	.480	$\frac{1}{2}$	.317	.458	$\frac{1}{2}$	.333	.500	$\frac{1}{2}$
O(2) (h)	.60	.466	$\frac{1}{2}$	.600	.464	$\frac{1}{2}$	.644	.503	$\frac{1}{2}$	.600	.467	$\frac{1}{2}$
O(3)	$\frac{1}{2}$	$\frac{1}{2}$	0.062	.350	.250	0.063	.336	.272	0.061	.333	.250	0.063

(ak) Unexpected intensities are observed from a number of compounds which obviously have the spinel,  $MgAl_2O_4$ , structure (k). Two explanations have been offered: one is that the metal atoms are distributed haphazardly among all the metal positions, both (8f) and (16c); the other considers that half of the sixteen chemically alike atoms are in (8f) and that the rest together with the eight chemically alike metal atoms are irregularly distributed throughout (16c). The latter has been called an "equipoint" structure. Qualitative estimates of intensity do not seem to conflict with the second interpretation but more quantitative observations and calculations are needed for final confirmation. Accurate parameters have been found for the oxygen atoms in several compounds. These additional data are collected in Table IV.

Cupric ferrite,  $CuFe_2O_4$ , when quenched, is cubic with the spinel structure; if it is slowly cooled or annealed at 350° C for some time its pattern is said to be that of the tetragonal cell of Table I.

## THE STRUCTURE OF CRYSTALS

TABLE IV. TYPE AND PARAMETER FOUND FOR SEVERAL SPINEL STRUCTURES

<i>Normal</i>	<i>Type</i>	<i>Parameter</i>
	"Equipoint"	<i>u</i>
CoAl <sub>2</sub> O <sub>4</sub>		0.390
FeAl <sub>2</sub> O <sub>4</sub>		.390
	FeTiFeO <sub>4</sub>	.390
MgAl <sub>2</sub> O <sub>4</sub>		.390
	FeMgFeO <sub>4</sub>	.390
	GaMgGaO <sub>4</sub>	.392
	InMgInO <sub>4</sub>	.372
	MgTiMgO <sub>4</sub>	.390
MnAl <sub>2</sub> O <sub>4</sub>		.390
NiAl <sub>2</sub> O <sub>4</sub>		.390
ZnAl <sub>2</sub> O <sub>4</sub>		.390
	ZnSnZnO <sub>4</sub>	.390

(al) The unit prism of CsOsNO<sub>3</sub> contains four molecules. Its Cs and Os atoms are said to be in the following positions of V<sup>2</sup>:

$$\begin{aligned} \text{Os: (c) } & 0u\frac{1}{4}; 0\bar{u}\frac{3}{4} \quad \text{and} \quad \text{(d) } \frac{1}{2}u'\frac{1}{4}; \frac{1}{2}\bar{u}'\frac{3}{4} \quad \text{with } u=u'=\frac{1}{8} \\ \text{Cs: (e) } & xyz; x\bar{y}\bar{z}; \bar{x}, y, \frac{1}{2}-z; \bar{x}, \bar{y}, z+\frac{1}{2} \quad \text{with } x=\frac{1}{4}, y=\frac{5}{8}, z=\frac{1}{4}. \end{aligned}$$

(am) Four molecules are included in the pseudo-tetragonal orthorhombic cells of CsReO<sub>4</sub> and TlReO<sub>4</sub>. The space group has been given as V<sub>h</sub><sup>16</sup>.

(an) Two differing determinations have been made of the structure of enargite, Cu<sub>3</sub>AsS<sub>4</sub>. According to one the atoms of its single molecule cell are all in positions (g) of the orthorhombic space group V<sub>h</sub><sup>12</sup>. Sulfur atoms are in one set of these special positions, copper and arsenic atoms, grouped together, in another.

The unit prism of the other and presumably correct arrangement (see Table I) is twice as high in the direction of the b-axis, i.e. b<sub>0</sub>=7.43 Å. The atoms in the bimolecular unit are distributed according to the following cases of C<sub>2v</sub><sup>7</sup>:

$$\text{(a) } u0v; \bar{u}, \frac{1}{2}, v+\frac{1}{2} \quad \text{(b) } xyz; \bar{x}, \frac{1}{2}-y, z+\frac{1}{2}; \bar{x}, y+\frac{1}{2}, z+\frac{1}{2}; x\bar{y}z$$

with the parameters listed in Table V. The axes of this description differ from those of 1930, 352 by an interchange of X' and Y'. Like so many

TABLE V. PARAMETERS OF THE ATOMS IN Cu<sub>3</sub>AsS<sub>4</sub>

<i>Atom</i>	<i>No. per cell</i>	<i>Positions</i>	<i>x</i>	<i>y</i>	<i>z</i>
As	2	(a)	0.820	0	0
Cu(1)	2	(a)	.165	0	0.500
Cu(2)	4	(b)	.333	0.245	.990
S(1)	2	(a)	.830	0	.360
S(2)	2	(a)	.140	0	.875
S(3)	4	(b)	.330	.255	.367

other sulfides this grouping is a system of sulfur tetrahedra linked by sharing corners and having metal atoms at their centers. As Figure 307b shows, the packing is excellent if the crystal is assumed to be made up of neutral atoms (As-S=2.21 Å, Cu-S=2.31 Å).

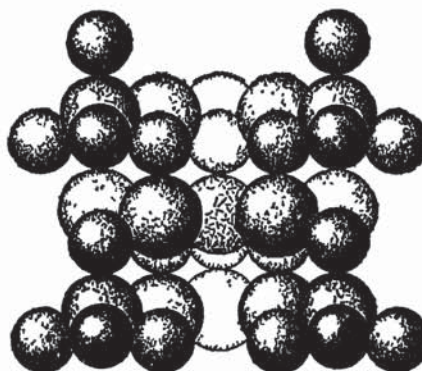
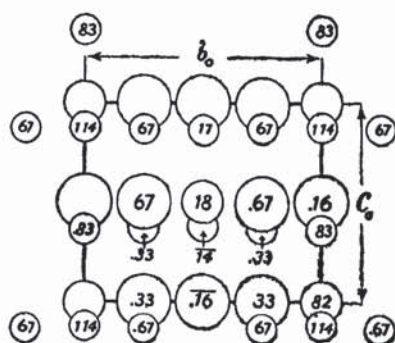
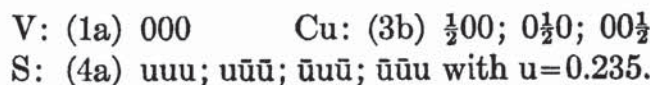


FIG. 307a.—(left) The orthorhombic unit of enargite,  $\text{Cu}_3\text{AsS}_4$ , projected on its  $a$ -face. The small circles are S, the largest circles are Cu atoms.

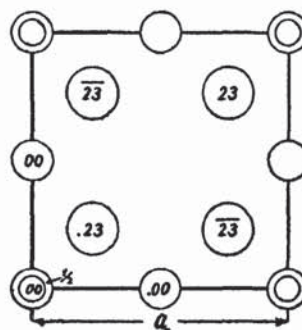
FIG. 307b.—(right) A packing drawing of  $\text{Cu}_3\text{AsS}_4$  giving the atoms their neutral radii. Atoms of As are line-shaded.

(ao) A new and simpler structure has been found for sylvanite,  $\text{Cu}_3\text{VS}_4$ . With a cube edge half that previously chosen, the unimolecular cell has atoms in the following positions of  $T_d^1$  (Figure 308):



This leads to a V-S separation of 2.18 Å; the Cu-S distance is 2.28 Å.

FIG. 308.—A cube face projection of the atoms in the new grouping established for sylvanite,  $\text{Cu}_3\text{VS}_4$ . The smallest circles are V, the largest S atoms.



(ap) The edge lengths of the unit cube of several chromites, (Fe, Mg).  $(\text{Cr, Al})_2\text{O}_4$ , have been measured. These lengths increase with the  $\text{Cr}_2\text{O}_3$  content.

(aq) Powder photographs of magnetite,  $\text{Fe}_3\text{O}_4$ , made at various temperatures down to ca  $-170^\circ\text{C}$  prove that the anomalous heat effect found

at  $-160^{\circ}$  C is not due to a change in structure. Like  $\text{MnO}$ ,  $\text{Fe}_3\text{O}_4$  is, however, reported to have a region in which it shrinks on being warmed (see Table II, Chapter XIA).

(ar) Positions have been assigned to all the atoms in three substances,  $\text{KMnO}_4$ ,  $\text{KClO}_4$  and  $\text{NH}_4\text{ClO}_4$ , with the barite,  $\text{BaSO}_4$ , structure (a). These crystals have units which are almost identical in size and it is probable that their real atomic positions are practically the same. Nevertheless the structures proposed for the permanganate and for the perchlorates show important differences (Figures 309 and 310). The  $\text{KMnO}_4$  determination rests on photographic spectral data; the observations on  $\text{KClO}_4$  and  $\text{NH}_4\text{ClO}_4$  are more quantitative spectrometric measurements. It is, however, difficult to be sure of the deductions from the latter results. The published parameters are obviously wrong: they correspond to an utterly impossible grouping. If the drawing of the perchlorate paper (1932, 177) is assumed to be correct and the parameters are altered to fit it, a structure is obtained which yields the interatomic distances stated in the paper and which therefore is probably the intended one. These parameters, and the values for  $\text{KMnO}_4$  expressed in terms of a unit with

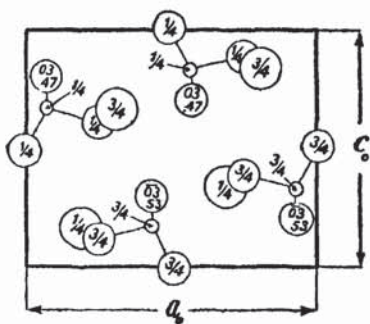


FIG. 309.—(left) The unit cell of the orthorhombic structure found for  $\text{KMnO}_4$  projected on its b-face. Atoms of the  $\text{MnO}_4$  ions are connected by light lines.

FIG. 310.—(right) The arrangement selected for  $\text{KClO}_4$  projected upon its b-face. Atoms of the  $\text{ClO}_4$  tetrahedra are united by light lines.

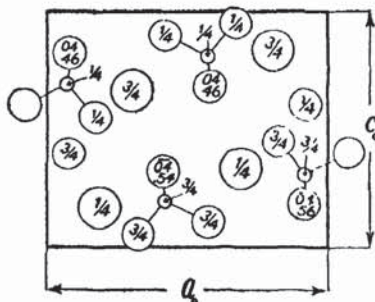


TABLE VI. PARAMETERS OF THE ATOMS IN  $\text{KMnO}_4$ ,  $\text{KClO}_4$  AND  $\text{NH}_4\text{ClO}_4$

Atom	Positions	$\text{KMnO}_4$			$\text{KClO}_4^*$			$\text{NH}_4\text{ClO}_4^*$		
		$x$	$y$	$z$	$x$	$y$	$z$	$x$	$y$	$z$
K or $\text{NH}_4$	(c)	0.19	$\frac{1}{2}$	0.16	0.192	$\frac{1}{2}$	0.167	0.197	$\frac{1}{2}$	0.172
Mn or Cl	(c)	.07	$\frac{1}{2}$	.67	.075	$\frac{1}{2}$	.689	.067	$\frac{1}{2}$	.694
O(1)	(c)	.99	$\frac{1}{2}$	.49	.175	$\frac{1}{2}$	.550	.169	$\frac{1}{2}$	.550
O(2)	(c)	.25	$\frac{1}{2}$	.61	-.078	$\frac{1}{2}$	.606	-.078	$\frac{1}{2}$	.600
O(3)	(d)	.07	0.03	.80	.083	0.042	.819	.075	0.042	.819

\* These values are obtained from the parameters of 1932, 177 by adding  $\frac{1}{2}$  to the  $z$  coordinates of Cl, O(1) and O(2), and by changing the sign of  $x$  of O(2) and  $z$  of O(3).



the same origin, are listed in Table VI. The necessary coordinates (as stated on p. 283 of book) are:

$$(c) \pm(u\frac{1}{4}v); \pm(u+\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-v)$$

$$(d) \pm(xyz); \pm(x, \frac{1}{2}-y, z); \pm(x+\frac{1}{2}, y, \frac{1}{2}-z); \pm(x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z).$$

It is interesting that in spite of the very different positions of atoms O(1) and O(2), neither of these arrangements gives unreasonable atomic separations and each is supposed to be required by the observed data. Additional work will undoubtedly provide another demonstration of the fact that acceptable interatomic distances and qualitative agreement with a limited number of intensity estimations are insufficient to establish most structures with many parameters.

(as) Atomic positions have been found in two crystals isomorphous with  $K_2SO_4$  (*m*)— $K_2CrO_4$  and  $(NH_4)_2BeF_4$ . Their parameters (Table VII) are essentially those previously chosen for the alkali sulfates. The values recorded for  $K_2CrO_4$  in Table VII have been derived from the conclusions of 1931, 501 by reversing the signs along *c* and adding one half.

TABLE VII. PARAMETERS OF THE ATOMS IN  $K_2CrO_4$  AND  $(NH_4)_2BeF_4$

Atom	No. per cell	For $K_2CrO_4$			Atom	For $(NH_4)_2BeF_4$		
		<i>x</i>	<i>y</i>	<i>z</i>		<i>x</i>	<i>y</i>	<i>z</i>
K(1)	4	$\frac{1}{4}$	0.417	0.644	NH <sub>4</sub> (1)	$\frac{1}{4}$	0.393	0.675
K(2)	4	$\frac{1}{4}$	-.305	0	NH <sub>4</sub> (2)	$\frac{1}{4}$	-.325	-.046
Cr	4	$\frac{1}{4}$	.417	.230	Be	$\frac{1}{4}$	.417	.263
O(1)	4	$\frac{1}{4}$	.417	.019	F(1)	$\frac{1}{4}$	.390	.051
O(2)	4	$\frac{1}{4}$	.561	.300	F(2)	$\frac{1}{4}$	.573	.300
O(3)	8	0.028	.345	.300	F(3)	0.024	.353	.350

(at) The unit cube of  $K_2Mg_2(SO_4)_3$  contains four molecules. Its space group has been found to be  $T^4$ .

(au) The mineral triphylite,  $Li(Fe,Mn)PO_4$ , and the compound  $Li_3PO_4$  have orthorhombic cells similar in size and shape to the unit of chrysoberyl,  $BeAl_2O_4$ , (*l*). It has been inferred that their structures too are similar.

(av) The monoclinic unit of  $Li_2SO_4$  contains four molecules. An arrangement, based on spectral photographs, places all its atoms in the general positions (e)  $\pm(xyz); \pm(\frac{1}{2}-x, y+\frac{1}{2}, z)$  of  $C_{2h}^5$ . The chosen parameters, recorded in Table VIII, give the grouping illustrated in Figure 311.

(aw) The monoclinic cell of  $(NH_4)_2CrO_4$  is bimolecular. The space group is reported to be  $C_s^1$ .

(ax) Ammonium osmiumate,  $NH_4OsNO_3$ , like  $CsReO_4$  (*am*), has a four-molecule pseudo-tetragonal orthorhombic unit suggesting the tetragonal  $CaWO_4$  (*d*) arrangement. The space group assigned to  $NH_4OsNO_3$ ,  $V^4$ , is different from that proposed for  $CsReO_4$ .

The rubidium and thallium salts,  $\text{RbOsNO}_3$  and  $\text{TlOsNO}_3$ , are structurally isomorphous with  $\text{NH}_4\text{OsNO}_3$ . It is thought that the rubidium and osmium atoms in the general positions of  $V^4$ ,  $xyz$ ;  $x+\frac{1}{2}$ ,  $\frac{1}{2}-y$ ,  $\bar{z}$ ;  $\bar{x}$ ,  $y+\frac{1}{2}$ ,  $\frac{1}{2}-z$ ;  $\frac{1}{2}-x$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ , have as approximate parameters, for Rb:  $x=0.03$ ,  $y=\frac{1}{4}$ ,  $z=\frac{5}{8}$ ; for Os:  $x=0.03$ ,  $y=\frac{1}{4}$ ,  $z=\frac{1}{8}$ .

TABLE VIII. PARAMETERS OF THE ATOMS IN  $\text{Li}_2\text{SO}_4$

Atom	$x$	$y$	$z$
Li(1)	0.205	0.582	0.375
Li(2)	.455	.582	.125
S	.319	.061	.250
O(1)	.492	-.042	.250
O(2)	.186	-.042	.099
O(3)	.280	-.042	.401
O(4)	.319	.367	.250

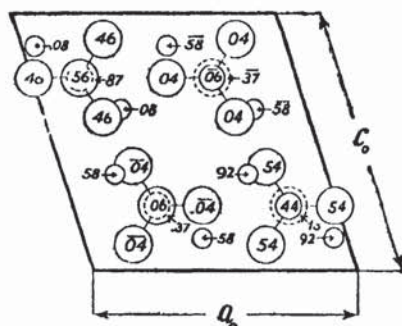


FIG. 311a.—(left) The monoclinic structure determined for  $\text{Li}_2\text{SO}_4$  projected on its  $b$ -face. The largest circles are O, the smallest are Li atoms.

FIG. 311b.—(right) A packing drawing of  $a$  showing Li ions and  $\text{SO}_4$  tetrahedra.

(ay) Two minerals, **adelite** and **tilasite**, each of which is essentially  $\text{CaMg}(\text{OH})\text{AsO}_4$ , have units similar in shape and size though the first is orthorhombic, the latter monoclinic. Both cells contain four molecules.

(az) The space group of **descloizite**,  $\text{PbZn}(\text{OH})\text{VO}_4$ , has been fixed as  $V_h^{16}$ ; its cell includes four molecules. The following minerals are said to be isomorphous, with Cu sometimes replacing some Zn: **cuprodescloizite**, **mottramite**, **psittacinite**, **chileite**, **eusynchite** and **dechinite**.

(ba) The mineral **beryllonite** is orthorhombic both in its crystallography and its X-ray data. The optical anomalies it shows have, however, been considered to be sufficiently marked to prove its monoclinic symmetry. For such a crystal the 12-molecule cell of the table is without doubt too large to be the true unit.

(bb) The monoclinic cell of ammonium persulfate,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , is bimolecular. From Laue and spectral photographs it has been concluded that the space group is  $C_{2h}^6$  with all atoms in the general positions  $(e) \pm(xyz); \pm(x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2})$  [see p. 58]. The chosen atomic parameters are listed in Table IX. As can be seen from Figure 312a and b, this determination yields an  $\text{S}_2\text{O}_8$  ion which consists of two  $\text{SO}_4$  tetrahedra joined through an oxygen-to-oxygen bond (O-O=1.46 Å).

The cesium analogue,  $\text{Cs}_2\text{S}_2\text{O}_8$ , is isomorphous. Cesium parameters have been taken as  $x=0.144$ ,  $y=0.125$ ,  $z=0.228$ ; the parameters for the other atoms have the same values as in the ammonium salt.

TABLE IX. PARAMETERS OF THE ATOMS IN  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ 

Atom	$x$	$y$	$z$
$\text{NH}_4$	0.144	0.125	0.250
S	.136	.350	.708
O(1)	.042	.500	.611
O(2)	.028	.194	.680
O(3)	.208	.417	.930
O(4)	.292	.347	.597

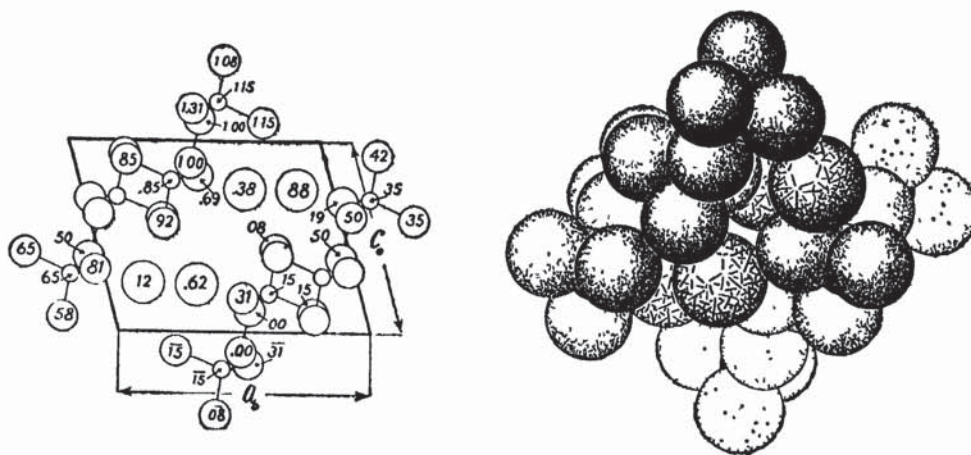


FIG. 312a.—(left) The monoclinic arrangement found for  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  projected on its b-face. The largest circles are  $\text{NH}_4$  ions; the atoms of  $\text{S}_2\text{O}_8$  ions are connected by light lines.

FIG. 312b.—(right) A packing drawing of *a*. The  $\text{NH}_4$  ions are line-shaded.

(bc) Four molecules are to be found in the unit prism of anhydrous  $\text{ZnSO}_4$ . Its space group has not yet been established.

(bd) The sulfide mineral stannite,  $\text{Cu}_2\text{FeSnS}_4$ , has a tetragonal two-molecule unit. Photographic data have placed its atoms in the following special positions of  $V_d^{11}$ :

Fe: (a)  $000$ ;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ Sn: (b)  $00\frac{1}{2}$ ;  $\frac{1}{2}\frac{1}{2}0$ Cu: (d)  $\frac{1}{2}0\frac{1}{4}$ ;  $\frac{1}{2}0\frac{3}{4}$ ;  $0\frac{1}{2}\frac{1}{4}$ ;  $0\frac{1}{2}\frac{3}{4}$ S: (i)  $uuv$ ;  $u\bar{u}\bar{v}$ ;  $\bar{u}\bar{u}v$ ;  $\bar{u}u\bar{v}$ 

and four similar positions about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ .

Like most other sulfides this structure for stannite (Figure 312c and d) can be considered as an assemblage of tetrahedra with sulfur at their centers. The interatomic distances that prevail are Cu-S=2.31 Å, Sn-S=2.43 Å, Fe-S=2.36 Å. Of these the iron-sulfur separation is exceptionally large.

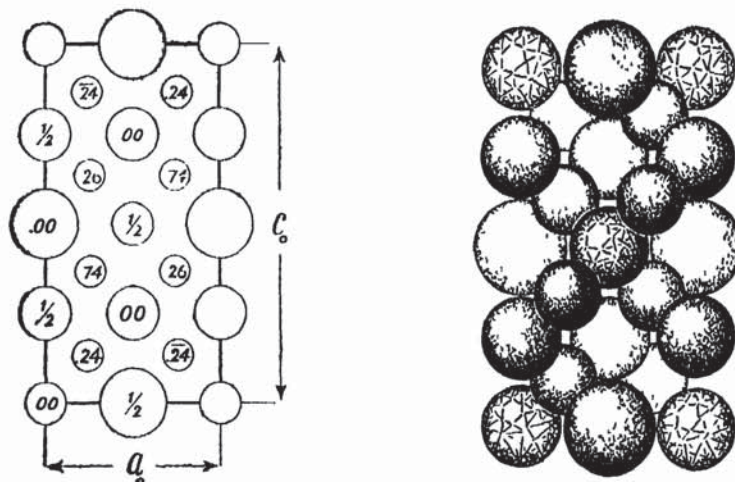


FIG. 312c.—(left) The tetragonal structure found for stannite,  $\text{Cu}_2\text{FeSnS}_4$ , projected on an  $a$ -face. Atoms of Sn, Cu, Fe and S are represented by circles of decreasing size.

FIG. 312d.—(right) A packing drawing of  $c$  in which atoms have their neutral radii. Atoms of Fe are line-shaded.

## Chapter XVIIIA. Structures of the Type $R_x(MX_6)_y$

(j) A number of compounds isomorphous with  $Ba_2Ni(NO_2)_6$  are reported to be cubic and to have the  $(NH_4)_2PtCl_6$  structure (a). A more detailed study of one or more of these crystals is needed to insure that the symmetry really is cubic and to establish the positions of the nitrogen and oxygen atoms.

(k) Several complex nitrites isomorphous with  $Cs_3Rh(NO_2)_6$  are said to be cubic. Their atomic arrangements are considered to be like that of  $(NH_4)_3FeF_6$  (g) with nitrogen in place of fluorine and oxygen in positions (48f)  $u\bar{u}0$ ; etc. (1930, 352, p. 113). For several of these crystals  $u(N)$  has been chosen as 0.26,  $u'(O)$  as 0.13. Such a distribution is improbable since it would cause the oxygen atoms to be shared between neighboring nitrogen atoms instead of forming distinct  $NO_2$  groups.

(l) The unit cubes of  $Ca_3[Al(OH)_6]_2$  and of  $Sr_3[Al(OH)_6]_2$  have been described as containing eight molecules; their space groups are given as  $O_h^{10}$ .

(m) The unit cubes of the **alkali fluophosphates** isomorphous with  $KPF_6$  contain four molecules. Their space group is thought to be  $T^2$  and it is asserted that the observed intensities conflict with the idea of  $PF_6$  radicals. Further work is obviously needed before anything is known about the structures of these crystals.

(n) The bromine parameter in  $K_2SeBr_6$  has been determined as 0.245; in  $(NH_4)_2SeBr_6$  it lies between 0.24 and 0.25. It has been stated that for all similar compounds listed in Table I,  $u(Cl)$  is greater than 0.23 and less than 0.25.

(o) The compound  $NaK_2AlF_6$  has been given a structure which is a slight distortion of the  $(NH_4)_3FeF_6$  arrangement (g). Aluminum atoms are at (4b) 000; etc., Na at (4c)  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ ; etc. The potassium atoms are in (8h) with a parameter  $u=0.25$  thus making their positions identical with (8e)  $\frac{1}{4}\frac{1}{4}\frac{1}{4}$ ; etc. The fluorine atoms are put in general positions (d)  $xyz$ ; etc. of  $T_h^6$  (see p. 268 of book) with  $x=0.03$ ,  $y=0.01$ ,  $z=0.22$ . No data have been published which allow an estimate of the accuracy of this determination.

TABLE I. THE CRYSTAL STRUCTURES OF THE COMPOUNDS  $R_x(MX_6)_y$ 

Substance, symmetry and structure type		$a_0$	$c_0$ or $\alpha$	References
$Ba_2Ni(NO_2)_6$	Cubic (j)	10.67		1933, 135.
$Ba_3[Rh(NO_2)_6]_2$	Cubic or Pseudo-cubic (r)	10.70		1933, 134.
$Ca_3[Al(OH)_6]_2$	Cubic (l)	12.56		1933, 70.
$Cs_2AgAuCl_6$	Cubic (s)	5.33		1934, 78.
$Cs_2Au^+Au^{+++}Cl_6$	Cubic (s)	5.33		1934, 78.
$Cs_3Co(NO_2)_6$	Cubic (k)	11.15		1933, 133.
$Cs_3Fe(CN)_6$	(e)			1931, 314.
$Cs_3Ir(NO_2)_6$	Cubic (k)	11.17		1933, 132.
$CsPF_6$	Cubic (m)	8.19		1931, 407, 408.
$Cs_2PbCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.415		1933, 126; 1934, 325.
		(a), (n)		
$Cs_2PtCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.185*		1932, 325; 1933, 54, 126; 1934, 325.
		(a), (n)		
$Cs_3Rh(NO_2)_6$	Cubic (k)	11.30		1933, 134.
$Cs_2ScCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.260		1934, 325.
		(a), (n)		
$Cs_2SnCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.348		1933, 126; 1934, 325.
		(a), (n)		
$Cs_2TeCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.449		1932, 326; 1933, 126; 1934, 325.
		(a), (n)		
$Cs_2TiCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.219		1934, 325.
		(a), (n)		
$Cs_2ZrCl_6$	Cubic	$(NH_4)_2PtCl_6$ 10.407		1934, 325.
		(a), (n)		
$K_2BaCo(NO_2)_6$	Cubic (f)	10.45		1931, 111.
$K_2BaNi(NO_2)_6$	Cubic (f)	10.67		1931, 111.
$K_2CaCo(NO_2)_6$	Cubic (f)	10.17		1931, 111.
$K_2CaNi(NO_2)_6$	Cubic (f)	10.29		1931, 111.
$K_3Co(NO_2)_6$	Cubic (?) (k)	10.44		1931, 111a; 1933, 133.
$K_3Cr(CN)_6$	(d)			1931, 314.
$K_3Fe(CN)_6$	(d)			1931, 314; 1933, 83.
$K_3Ir(CN)_6$	(d)			1931, 314.
$K_3Ir(NO_2)_6$	Cubic (k)	10.57		1933, 132.
$K_3Mn(CN)_6$	(d)			1931, 314.
$K_2NaAlF_6$	Cubic (o)	8.69		1932, 311.
$K_4Ni(NO_2)_6$	Cubic (?) (p)	10.49		1931, 111a.
$K_2OsNCl_6$ †				1933, 483.
$K_2OsO_2Cl_4$	Tetragonal (q)	6.99	8.75	1934, 109.
$KPF_6$	Cubic (m)	7.76		1931, 407, 408.
$K_2PtCl_6$	Cubic	$(NH_4)_2PtCl_6$ 9.725		1934, 325.
		(a), (n)		
$K_3Rh(NO_2)_6$	Cubic (k)	10.63		1933, 134.
$K_2SeBr_6$	Cubic	$(NH_4)_2PtCl_6$ 10.363		1933, 200.
		(a), (n)		

\* According to 1933, 54,  $a_0=10.120$  A.

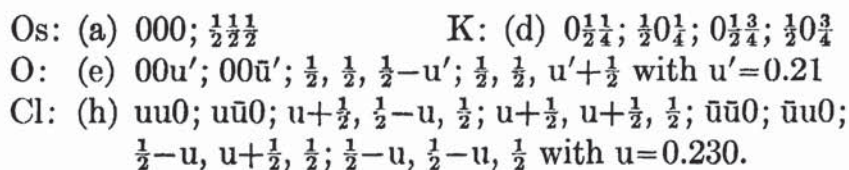
† Journal not available.

Substance, symmetry and structure type		$a_0$	$c_0$ or $\alpha$	References
$K_2SnCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.983	1934, 325.
$K_2SrCo(NO_2)_6$	Cubic	(f)	10.23	1931, 111.
$K_2SrNi(NO_2)_6$	Cubic	(f)	10.49	1931, 111.
$K_2TeCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.143	1934, 325.
$(NH_4)_3Co(NO_2)_6$	Cubic	(k)	10.81	1933, 133.
$(NH_4)_3CrF_6$	Cubic	(g)	9.01	1932, 356.
$(NH_4)_3Ir(NO_2)_6$	Cubic	(k)	10.73	1933, 132.
$NH_4PF_6$	Cubic	(m)	7.92	1931, 407, 408.
$(NH_4)_2PbCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.135	1934, 325.
$(NH_4)_2PtCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.834	1934, 325.
$(NH_4)_3Rh(NO_2)_6$	Cubic	(k)	10.91	1933, 134.
$(NH_4)_2SeBr_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.46	1932, 425.
$(NH_4)_2SeCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.935	1934, 325.
$(NH_4)_2SiF_6$	Hexagonal	(t)	5.76	4.77 1934, 87.
$(NH_4)_2SnCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.038	1934, 325.
$(NH_4)_2TeCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.178	1934, 325.
$(NH_4)_3VF_6$	Cubic	(g)	9.04	1932, 355.
$N_3AlF_6$		(h)		1932, 311.
$Pb_2Ni(NO_2)_6$	Cubic	(j)	10.55	1933, 135.
$Pb_3[Rh(NO_2)_6]_2$	Cubic or Pseudo-cubic	(r)	10.53	1933, 134.
$Rb_3Co(NO_2)_6$	Cubic	(k)	10.73	1933, 133.
$Rb_3Fe(CN)_6$	Monoclinic	$K_3Fe(CN)_6$ (d)	13.74 $b_0=10.66, \beta=90^\circ 3'$	8.63 1933, 83.
$Rb_3Ir(NO_2)_6$	Cubic	(k)	10.77	1933, 132.
$Rb_2PbCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.198	1933, 126; 1934, 325.
$Rb_2PtCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.882	1932, 325; 1933, 54, 126; 1934, 325.
$Rb_3Rh(NO_2)_6$	Cubic	(k)	10.83	1933, 134.
$Rb_2SeCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.978	1934, 325.
$Rb_2SnCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.100	1933, 126; 1934, 325.
$Rb_2TeCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.233	1933, 126; 1934, 325.
$Rb_2TiCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	9.922	1934, 325.
$Rb_2ZrCl_6$	Cubic	$(NH_4)_2PtCl_6$ (a), (n)	10.178	1934, 325.
$Sr_3[Al(OH)_6]_2$	Cubic	(l)	13.02	1933, 70.
$Sr_2Ni(NO_2)_6$	Cubic	(j)	10.54	1933, 135.
$Tl_3Co(NO_2)_6$	Cubic	(k)	10.72	1933, 133.

Substance, symmetry and structure type			$a_0$	$c_0$ or $\alpha$	References
$\text{Ti}_3\text{Ir}(\text{NO}_2)_6$	Cubic	( <i>k</i> )	10.73		1933, 132.
$\text{Ti}_2\text{PtCl}_6$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( <i>a</i> ), ( <i>n</i> )	9.755		1934, 325.
$\text{Ti}_3\text{Rh}(\text{NO}_2)_6$	Cubic	( <i>k</i> )	10.91		1933, 134.
$\text{Ti}_2\text{SiF}_6$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( <i>a</i> )	8.60		1933, 460.
$\text{Ti}_2\text{SnCl}_6$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( <i>a</i> ), ( <i>n</i> )	9.970		1934, 325.
$\text{Ti}_2\text{TeCl}_6$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( <i>a</i> ), ( <i>n</i> )	10.107		1934, 325.

(*p*) The compound  $\text{K}_4\text{Ni}(\text{NO}_2)_6$ , which probably has less than cubic symmetry, has been given a cubic or pseudo-cubic unit containing four molecules.

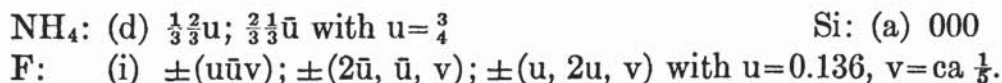
(*q*) The tetragonal unit of potassium osmyl chloride,  $\text{K}_2\text{OsO}_2\text{Cl}_4$ , is bimolecular. Its atoms, with an arrangement which is a slight distortion of the familiar  $(\text{NH}_4)_2\text{PtCl}_6$  grouping (*a*), are in the following special positions of  $D_{4h}^{17}$ :



(*r*) Unit cubes or pseudo-cubes of  $\text{Ba}_3[\text{Rh}(\text{NO}_2)_6]_2$  and  $\text{Pb}_3[\text{Rh}(\text{NO}_2)_6]_2$  are supposed to contain two molecules.

(*s*) Powder patterns obtained from the triple halides  $\text{Cs}_2\text{AgAuCl}_6$  and  $\text{Cs}_2\text{Au}^+\text{Au}^{+++}\text{Cl}_6$  have only the lines required by a perovskite,  $\text{CaTiO}_3$  [XV, (*d*)], arrangement. Such a unit would contain only half a molecule; hence it is suggested that the atoms have as coordinates: Cs: 000, Cl:  $0\frac{1}{2}\frac{1}{2}; \frac{1}{2}0\frac{1}{2}; \frac{1}{2}\frac{1}{2}0$ , Ag (or  $\text{Au}^+$ ) and  $\text{Au}^{+++}$  at  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  in different cells. Though the structure probably approaches such a simple atomic distribution, the true unit undoubtedly is a larger one.

(*t*) The unit prism established for the hexagonal modification of  $(\text{NH}_4)_2\text{SiF}_6$  is unimolecular. A structure has been proposed which places atoms in the following special positions of  $D_{3d}^3$ :



Practically no data have been published in support of this arrangement.



## Chapter XVIII A. Structures of Hydrates and Ammoniates and of Miscellaneous Inorganic Compounds

### Hydrates and Ammoniates

The hydrates thus far analyzed by X-ray methods fall into three types. Most of them, and all of the ammoniates, are coordination compounds in which the  $\text{H}_2\text{O}$  or  $\text{NH}_3$  molecules are closely bound to the metal atoms present. In a few of the crystals described below water molecules are not thus associated with cations but occupy holes in the lattice. Such a water molecule is present in  $\text{Pd}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O}$  (*ba*); the fifth  $\text{H}_2\text{O}$  in  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  is similarly held. Though these  $\text{H}_2\text{O}$  molecules cannot be driven off without destroying the rest of the atomic arrangement, in many other ways their bonding resembles that seen in the zeolites. To the third type belong those compounds, like  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$  or  $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ , with water so firmly held that it may be present as hydroxyl groups. Many minerals contain such "water of constitution."

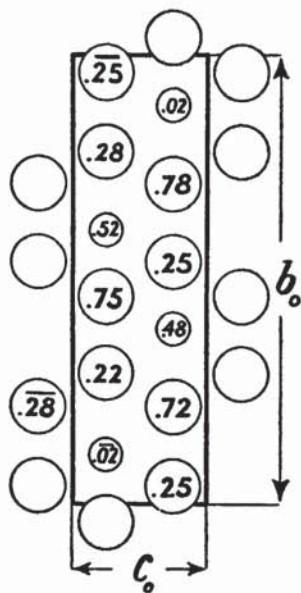


FIG. 313a.—(*left*) A portion of the diaspore,  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ , structure projected on an *a*-face. Small circles are Al atoms. Water molecules, as such, do not exist in this grouping.

FIG. 313b.—(*right*) A packing drawing of *a*.



TABLE I. THE CRYSTAL STRUCTURES OF HYDRATES AND AMMONIATES

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ (Diaspore)	Ortho- rhombic	$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ( $a$ ), ( $ae$ )	4.43	2.80 $b_o = 9.36$	1932, 121; 1933, 462.
$\text{CaCrO}_4 \cdot \text{H}_2\text{O}$	Ortho- rhombic	( $af$ )	7.99	8.11 $b_o = 12.77$	1932, 106.
$2(\text{CaSO}_4) \cdot \text{ca. H}_2\text{O}$	Hexagonal	( $ag$ )	6.76	6.24	1934, 46
$\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ (Goethite)	Ortho- rhombic	$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ( $a$ ), ( $ae$ )	4.64	3.03 $b_o = 10.0$	1931, 132; 1932, 168.
$\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ (Lepidocrocite)	Ortho- rhombic	( $ae$ )	3.87	3.06 $b_o = 12.4$	1931, 132.
$3(\text{KPbCl}_3) \cdot \text{H}_2\text{O}$	Triclinic		14.35	14.50* $b_o = 9.05$	1934, 172.
$\text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$	Monoclinic	( $br$ )	5.43	8.14 $b_o = 4.83, \beta = 107^\circ 35'$	1934, 361.
$\text{Mn}_2\text{O}_3 \cdot \text{H}_2\text{O}$ (Manganite)	Ortho- rhombic	( $ah$ )	4.41	2.83 $b_o = 5.19$	1931, 112.
$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Ortho- rhombic	( $bs$ )	10.72	5.24 $b_o = 6.44$	1934, 321.
$\text{Rb}_2(\text{CrF}_6) \cdot \text{H}_2\text{O}$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( $m$ )	8.38		1932, 356.
$\text{Sb}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (Stibiconite)	Cubic	( $ai$ )	10.24		1933, 316.
$\text{Ti}_2(\text{VF}_6) \cdot \text{H}_2\text{O}$	Cubic	$(\text{NH}_4)_2\text{PtCl}_6$ ( $m$ )	8.45		1932, 355.
$\text{AgNO}_3 \cdot 2\text{NH}_3$	Ortho- rhombic	( $bt$ )	8.00	6.29 $b_o = 10.58$	1934, 58.
$\text{CaCrO}_4 \cdot 2\text{H}_2\text{O}$	Ortho- rhombic	( $aj$ )	16.02	5.60 $b_o = 11.39$	1932, 106.
$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ (Brushite)	Monoclinic	( $ak$ )	10.47	6.28 $b_o = 15.15, \beta = 98^\circ 58'$	1931, 149.
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (Gypsum)	Monoclinic	( $d$ ), ( $ak$ )			1931, 149; 1934, 347a.
$\text{Cd}(\text{ClO}_2)_2 \cdot 2\text{H}_2\text{O}$	Monoclinic	( $al$ )	8.86	9.76 $b_o = 7.12, \beta = 90^\circ 18'$	1931, 282.
$\text{K}_2\text{CuCl}_4 \cdot 2\text{H}_2\text{O}$	Tetragonal	( $e$ ), ( $am$ )			1934, 48.
$(\text{NH}_4)_2\text{CuBr}_4 \cdot 2\text{H}_2\text{O}$	Tetragonal	$(\text{NH}_4)_2\text{CuCl}_4 \cdot 2\text{H}_2\text{O}$ ( $e$ ), ( $am$ )	7.83	8.14	1933, 370.
$(\text{NH}_4)_2\text{CuCl}_4 \cdot 2\text{H}_2\text{O}$	Tetragonal	( $e$ ), ( $am$ )			1934, 48.
$\text{NaBr} \cdot 2\text{H}_2\text{O}$	Monoclinic	( $an$ )	6.59	6.51 $b_o = 10.20, \beta = 112^\circ 30'$	1932, 482.
$\text{NaI} \cdot 2\text{H}_2\text{O}$	Triclinic	( $ao$ )	6.85	7.16 $b_o = 5.76, \alpha = 98^\circ, \beta = 119^\circ, \gamma = 68\frac{1}{2}^\circ$	1932, 482
$\alpha\text{-Pt}(\text{NH}_3)_2\text{Cl}_4$	Tetragonal	( $ap$ )	5.72	10.37	1933, 109.
$\beta\text{-Pt}(\text{NH}_3)_2\text{Cl}_4$	Ortho- rhombic	( $ap$ )	10.0	6.0 $b_o = 11.2$	1933, 109.

\* For  $3(\text{KPbCl}_3) \cdot \text{H}_2\text{O}$ ,  $\alpha = \gamma = \text{ca } 90^\circ$ ,  $\beta = 113^\circ$ .

Substance, symmetry and structure type			$a_o$	$c_o$ or $\alpha$	References
Zn(ClO <sub>2</sub> ) <sub>2</sub> ·2H <sub>2</sub> O	Monoclinic	(al)	8.67	9.38	1931, 282.
			$b_o=6.88$ , $\beta=90^{\circ}20'$		
Al <sub>2</sub> O <sub>3</sub> ·3H <sub>2</sub> O (Gibbsite)	Monoclinic	(aq)	8.6236	9.699	1934, 170.
			$b_o=5.0602$ , $\beta=85^{\circ}26'$		
Ba(ClO <sub>4</sub> ) <sub>2</sub> ·3H <sub>2</sub> O	Hexagonal	(bq)	7.28	9.64	1934, 290.
LiClO <sub>4</sub> ·3H <sub>2</sub> O	Hexagonal	(bp)	7.71	5.42	1934, 290
LiI·3H <sub>2</sub> O	Hexagonal	(f), (bp)			1934, 290.
Ag <sub>2</sub> SO <sub>4</sub> ·4NH <sub>3</sub>	Tetragonal	(ar)	8.43	6.35	1934, 59.
BaNi(CN) <sub>4</sub> ·4H <sub>2</sub> O	Monoclinic	(as)	11.71	6.63	1934, 32, 33.
			$b_o=13.48$ , $\beta=104^{\circ}50'$		
BaPt(CN) <sub>4</sub> ·4H <sub>2</sub> O	Monoclinic	(as)	11.89	6.54	1933, 72.
			$b_o=14.08$ , $\beta=103^{\circ}42'$		
BeSO <sub>4</sub> ·4H <sub>2</sub> O	Tetragonal	(h), (at)	8.02	10.75	1931, 401; 1932, 31.
[Co <sup>(O)</sup> <sub>(NH<sub>3</sub>)<sub>4</sub>]</sub> ClO <sub>4</sub>	Ortho- rhombic	(au)	18.05	6.95	1933, 455.
			$b_o=8.10$		
1, 6[Co <sup>(NO<sub>2</sub>)<sub>2</sub></sup> <sub>(NH<sub>3</sub>)<sub>4</sub>]</sub> Cl	Ortho- rhombic	(av)	14.36	13.98	1933, 456.
			$b_o=17.97$		
Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·4H <sub>2</sub> O (Kernite)	Monoclinic	(aw)	15.65	7.01	1932, 156, 157.
			$b_o=9.07$ , $\beta=108^{\circ}52'$		
[Pt(NH <sub>3</sub> ) <sub>4</sub> ]PtCl <sub>4</sub> (Magnus' Green Salt)	Tetragonal	(ax)	6.297	6.42 †	1931, 193; 1932, 119.
[Pt(NH <sub>3</sub> ) <sub>4</sub> ]PtCl <sub>4</sub> (Magnus' Red Salt)		(ax)			1931, 193; 1932, 119.
Zn(ClO <sub>4</sub> ) <sub>2</sub> ·4NH <sub>3</sub>	Cubic	(ay)	10.250		1933, 53.
CuSO <sub>4</sub> ·5H <sub>2</sub> O	Triclinic	(v), (az)	5.12	5.97	1933, 32; 1934, 15, 15a.
			$b_o=10.7$ , $\alpha=82^{\circ}16'$ , $\beta=107^{\circ}26'$ , $\gamma=102^{\circ}40'$		
H <sub>3</sub> PW <sub>12</sub> O <sub>40</sub> ·5H <sub>2</sub> O	Cubic	(bk)	12.14		1934, 131.
Pd(NH <sub>3</sub> ) <sub>4</sub> Cl <sub>2</sub> ·H <sub>2</sub> O	Tetragonal	(ba)	10.302	4.34	1933, 109; 1934, 71.
Pt(NH <sub>3</sub> ) <sub>4</sub> Cl <sub>2</sub> ·H <sub>2</sub> O	Tetragonal	(ba)	10.44	4.21	1932, 114; 1933, 109.
AlCl <sub>3</sub> ·6H <sub>2</sub> O	Hexagonal	(bb)	7.85	$97^{\circ}\pm 20'$	1934, 3.
BaI <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	SrCl <sub>2</sub> ·6H <sub>2</sub> O (p)	8.90	4.60	1931, 185.
CaBr <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	SrCl <sub>2</sub> ·6H <sub>2</sub> O (p)	7.97	3.97	1931, 184.
CaBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.706		1933, 56.
CaCl <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	SrCl <sub>2</sub> ·6H <sub>2</sub> O (p)	7.86	3.905	1931, 184.
CaI <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	SrCl <sub>2</sub> ·6H <sub>2</sub> O (p)	8.4	4.25	1931, 185.
CaI <sub>2</sub> ·6NH <sub>3</sub>	Cubic	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.24		1933, 56.
Cd(BF <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.380		1933, 54.
CdBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic	(NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.540		1933, 56.

† In 1931, 193,  $c_o=5.15$  A.

Substance, symmetry and structure type		$a_0$	$c_0$ or $\alpha$	References
$\text{Cd}(\text{ClO}_4)_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.588		1933, 53.
$\text{CdI}_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.046		1933, 53.
$\text{Cd}(\text{SO}_3\text{F})_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.619		1933, 54.
$\text{CdSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	Monoclinic ( <i>n</i> )	9.35	6.27	1931, 211.
$[\text{Co} \cdot 6\text{NH}_3](\text{BF}_4)_2$	Cubic ( <i>m</i> )	11.265	$b_0=12.705, \beta=106^\circ 41'$	1932, 195; 1933, 54.
$[\text{Co} \cdot 6\text{NH}_3](\text{BF}_4)_3$	Cubic ( <i>l</i> )	11.211		1933, 53.
$[\text{Co} \cdot 6\text{NH}_3]\text{Br}_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.389		1933, 55.
$[\text{Co} \cdot 6\text{NH}_3]\text{Cl}_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.10		1933, 55.
$[\text{Co} \cdot 6\text{NH}_3](\text{ClO}_4)_2$	Cubic ( <i>m</i> )	11.449		1932, 195.
$[\text{Co} \cdot 6\text{NH}_3](\text{ClO}_4)_3$	Cubic ( <i>l</i> )	11.384		1932, 195.
$[\text{Co} \cdot 6\text{NH}_3]\text{I}_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.914		1933, 55.
$[\text{Co} \cdot 6(\text{NH}_2 \cdot \text{CH}_3)]\text{I}_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	12.05		1933, 55.
$[\text{Co} \cdot 6\text{NH}_3](\text{PF}_6)_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.942		1933, 53.
$[\text{Co} \cdot 6\text{NH}_3](\text{PF}_6)_3$	Cubic ( $\text{NH}_4$ ) <sub>3</sub> FeF <sub>6</sub> ( <i>l</i> )	11.670		1933, 53.
$[\text{Co} \cdot 6\text{NH}_3](\text{SO}_3\text{F})_2$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.490		1933, 54.
$\text{CoSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	Monoclinic ( <i>n</i> )	9.23	6.23	1931, 150.
$\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$	Hexagonal ( <i>bb</i> )	7.95	$97^\circ \pm 20'$	1934, 3.
$\text{Fe}(\text{BF}_4)_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.340		1933, 54.
$\text{FeBr}_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.468		1933, 53.
$\text{FeCl}_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.148		1933, 53.
$\text{Fe}(\text{ClO}_4)_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.517		1933, 53.
$\text{FeI}_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	10.965		1933, 53.
$\text{Fe}(\text{SO}_3\text{F})_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.544		1933, 54.
$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	Monoclinic ( <i>n</i> ), ( <i>bc</i> )	9.28	6.22	1931, 211.
$\text{Mg}(\text{BF}_4)_2 \cdot 6\text{NH}_3$	Cubic ( $\text{NH}_4$ ) <sub>2</sub> PtCl <sub>6</sub> ( <i>m</i> )	11.337	$b_0=12.57, \beta=106^\circ 50'$	1933, 54.

Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References
MgBr <sub>2</sub> ·6H <sub>2</sub> O	Monoclinic (bd)	10.25	6.30	1934, 4.
		$b_o=7.40, \beta=93^\circ30'$		
MgBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.468		1933, 53.
MgCl <sub>2</sub> ·6H <sub>2</sub> O	Monoclinic (bd)	9.90	6.10	1934, 4.
		$b_o=7.15, \beta=94^\circ\pm20'$		
MgCl <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.158		1933, 53.
Mg(ClO <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.531		1933, 53.
MgI <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.978		1933, 53.
MgSO <sub>4</sub> ·K <sub>2</sub> SO <sub>4</sub> ·6H <sub>2</sub> O	(n)	9.04	6.095	1931, 211.
	Monoclinic	$b_o=12.24, \beta=104^\circ48'$		
MgNH <sub>4</sub> AsO <sub>4</sub> ·6H <sub>2</sub> O	(bo)	7.00	11.14	1933, 397.
	Orthorhombic	$b_o=6.14$		
MgSO <sub>4</sub> ·(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ·6H <sub>2</sub> O	(n), (bc)	9.28	6.20	1931, 211; 1932, 220.
	Monoclinic	$b_o=12.57, \beta=107^\circ6'$		
MgSeO <sub>4</sub> ·(NH <sub>4</sub> ) <sub>2</sub> SeO <sub>4</sub> ·6H <sub>2</sub> O	(n)	9.42	6.30	1931, 211.
	Monoclinic	$b_o=12.72, \beta=106^\circ27'$		
MgSO <sub>4</sub> ·Ti <sub>2</sub> SO <sub>4</sub> ·6H <sub>2</sub> O	(n)	9.22	6.185	1931, 211.
	Monoclinic	$b_o=12.42, \beta=106^\circ30'$		
Mn(BF <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.374		1933, 54.
MnBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.519		1933, 53.
MnCl <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.198		1933, 53.
Mn(ClO <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.578		1933, 53.
MnI <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.037		1933, 53.
Mn(SO <sub>3</sub> F) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.593		1933, 54.
Ni(BF <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.219		1932, 195; 1933, 54.
NiBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.34		1933, 55.
NiCl <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.064		1933, 55.
Ni(ClO <sub>4</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.410		1932, 195.
NiI <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	10.875		1933, 55.
NiI <sub>2</sub> ·6(NH <sub>2</sub> CH <sub>3</sub> )	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	12.027		1933, 55.
Ni(PF <sub>6</sub> ) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.912		1933, 53.
Ni(SO <sub>3</sub> F) <sub>2</sub> ·6NH <sub>3</sub>	Cubic (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> (m)	11.445		1932, 195; 1933, 54.

Substance, symmetry and structure type		$a_o$	$c_o$ or $\alpha$	References
NiSO <sub>4</sub> ·6H <sub>2</sub> O	Tetragonal (be)	6.80	18.3	1932, 32; 1933, 102.
SrBr <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	8.212	4.146	1931, 183.
SrCl <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	7.906	4.07	1931, 185.
	(p)			
SrI <sub>2</sub> ·6H <sub>2</sub> O	Hexagonal	8.51	4.29	1931, 184.
	(p), (bf)			
ZnBr <sub>2</sub> ·6NH <sub>3</sub>	Cubic	10.46		1933, 56.
ZnI <sub>2</sub> ·6NH <sub>3</sub>	Cubic	10.964		1933, 53.
	(m)			
ZnSO <sub>4</sub> ·(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ·6H <sub>2</sub> O	Monoclinic	9.205	6.225	1931, 211.
	(n)	$b_o = 12.475, \beta = 106^\circ 52'$		
[Co <sup>CO<sub>3</sub></sup> (NH <sub>4</sub> ) <sub>4</sub> ] <sub>2</sub> SO <sub>4</sub> ·3H <sub>2</sub> O	Monoclinic (bn)	11.80	7.42	1933, 454.
MgPt(CN) <sub>4</sub> ·7H <sub>2</sub> O	Monoclinic	14.6	6.26	1932, 60.
	(t), (bg)			
MgSO <sub>4</sub> ·7H <sub>2</sub> O	Tetragonal			1930, 378; 1932, 19.
	Orthorhombic			
3CdSO <sub>4</sub> ·8H <sub>2</sub> O	Monoclinic (bh)	14.65	16.35	1932, 138.
		$b_o = 11.84, \beta = 34^\circ 48'$		
SrO <sub>2</sub> ·8H <sub>2</sub> O	Tetragonal (v), (bi)			1932, 322.
Na <sub>3</sub> SbS <sub>4</sub> ·9H <sub>2</sub> O	Cubic*			1933, 483.
4(PO <sub>4</sub> )Al·2Al(OH) <sub>3</sub> ·9H <sub>2</sub> O (Wavellite)	Orthorhombic (bl)	7.27	10.80	1933, 225.
		$b_o = 14.41$		
Zn <sub>14</sub> (AsO <sub>4</sub> ) <sub>9</sub> OH·12H <sub>2</sub> O (Legrandite)	Monoclinic (bm)	12.70	10.18	1932, 130.
		$b_o = 7.90, \beta = 75^\circ 35'$		
2Na <sub>3</sub> PO <sub>4</sub> ·NaF·19H <sub>2</sub> O	Cubic (bj)	27.86		1933, 322.
GdPMo <sub>12</sub> O <sub>40</sub> ·30H <sub>2</sub> O	Cubic (bk)	23.1		1933, 198.
	Cubic			
H <sub>3</sub> PMo <sub>12</sub> O <sub>40</sub> ·30H <sub>2</sub> O	Cubic (bk)	23.281		1933, 198, 248, 249.
	Cubic			
NdPMo <sub>12</sub> O <sub>40</sub> ·30H <sub>2</sub> O	Cubic (bk)	23.10		1933, 198.
	Cubic			
SaPMo <sub>12</sub> O <sub>40</sub> ·30H <sub>2</sub> O	Cubic (bk)	23.1		1933, 198.
	Cubic			
Be <sub>2</sub> SiW <sub>12</sub> O <sub>40</sub> ·31H <sub>2</sub> O	Cubic (bk)	23.3		1933, 198.
	Cubic			
Mg <sub>2</sub> SiMo <sub>12</sub> O <sub>40</sub> ·31H <sub>2</sub> O	Cubic (bk)	23.04		1933, 198.
	Cubic			
Ni <sub>2</sub> SiMo <sub>12</sub> O <sub>40</sub> ·31H <sub>2</sub> O	Cubic (bk)	23.0		1933, 198.
	Cubic			

\* The journal describing this work was not available.

(ae) The orthorhombic units of **diaspore**,  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$  or  $\text{H}_2\text{Al}_2\text{O}_4$ , and of **goethite**,  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ , are bimolecular. Studied independently, they have been given identical arrangements. All atoms are in special positions (c) of  $V_h^{16}$ :  $uv\frac{1}{4}$ ;  $\bar{u}\bar{v}\frac{3}{4}$ ;  $u+\frac{1}{2}$ ,  $\frac{1}{2}-v$ ,  $\frac{3}{4}$ ;  $\frac{1}{2}-u$ ,  $v+\frac{1}{2}$ ,  $\frac{1}{4}$  with the values of  $u$  and  $v$  listed in Table II. Except for  $c_0$  which is half as long, the units of these oxides and of chrysoberyl,  $\text{BeAl}_2\text{O}_4$ , are similar in size and shape. As is readily seen by comparing Figure 313 with Figure 219 (book, p. 293) the two groupings are similar, both being packings of oxygen atoms with the small metal atoms lying in interstices.

The other monohydrate of  $\text{Fe}_2\text{O}_3$ , **lepidocrocite**, also has a bimolecular orthorhombic unit but its space group is  $V_h^1$ . It is said that lepidocrocite, unlike goethite, gives magnetic  $\text{Fe}_2\text{O}_3$  on dehydration.

(af) The unit cell of **CaCrO<sub>4</sub>·H<sub>2</sub>O** contains eight molecules. Its probable space group is  $V_h^{15}$ .

TABLE II. PARAMETERS OF THE ATOMS IN DIASPORE AND GOETHITE

Atom	For $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$		For $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$	
	$u$	$v$	$u$	$v$
Al or Fe	-0.02	0.11	-0.05	0.103
O(1)	.75	-.04	.75	-.04
O(2)	.22	.29	.25	.29

(ag) Recent work on **plaster of Paris** revives the question of whether a hemihydrate,  $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ , or anhydrous  $\text{CaSO}_4$  provides the proper formula. It has been shown that the water remaining in burnt plaster can be nearly all removed without destroying the crystal form and without altering the diffraction pattern. From this it has been concluded that the water is present in a zeolitic condition—within holes existing in the crystalline structure. Assuming that plaster of Paris is built only of  $\text{CaSO}_4$  molecules, three of them are to be found within the prism of Table I.

(ah) The unit cell selected for **manganite**,  $\text{Mn}_2\text{O}_3 \cdot \text{H}_2\text{O}$ , contains one molecule. Except for the fact that the length of the b-axis is halved, this cell is practically the same in size and shape as those of diaspore and goethite.

(ai) **Stibiconite**,  $\text{Sb}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , gives a powder pattern identical with that of the anhydrous tetroxide  $\text{Sb}_2\text{O}_4$ . It is therefore not surprising that no change of pattern occurs on dehydration.

(aj) The unit prism of **CaCrO<sub>4</sub>·2H<sub>2</sub>O** contains eight molecules; its space group is described as  $V_h^{11}$ .

(ak) **Gypsum**,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , and **brushite**,  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ , are said to have indistinguishable X-ray patterns and hence to possess unit cells of the same size and shape. It is reported that **ardealite** is a 1:1 double salt of these compounds with practically the same cell.

In a very recent study it is concluded that the correct space group of gypsum is  $C_{2h}^6$  and not  $C_{2h}^3$  upon which the structure described in (d) (book, p. 314) was based.

(al) Four molecules are found within the monoclinic units assigned to  $Cd(ClO_2)_2 \cdot 2H_2O$  and  $Zn(ClO_2)_2 \cdot 2H_2O$ .

(am) Previous studies of crystals isomorphous with  $(NH_4)_2CuCl_4 \cdot 2H_2O$  gave two different atomic arrangements. A reinvestigation, based on quantitative intensity data and Fourier plots of electron distributions, leads to a structure that differs from the first one described under (e) (book, p. 314) only in making equal the parameters  $u$  and  $v$  for the chlorine atoms in (f) and (g) [ $u=v=0.220$ ] and in reducing the water parameters in (e) to  $w=0.25$ . This arrangement for  $K_2CuCl_4 \cdot 2H_2O$  gives definitely better agreement with quantitative data than does the second one having chlorine atoms in (j). It is interesting that no choice between these arrangements could be made from the  $(NH_4)_2CuCl_4 \cdot 2H_2O$  reflections.

(an) The monoclinic unit found for  $NaBr \cdot 2H_2O$  contains four molecules; its space group is  $C_{2h}^5$ .

(ao) The compound  $NaI \cdot 2H_2O$  is described as triclinic with a two-molecule unit and with  $C_1^1$  as space group.

(ap) The  $\alpha$ -diamminoplatinic chloride is reported to be tetragonal with a two-molecule unit; its space group has been given as  $D_{4h}^{14}$ .

The  $\beta$ -form, of a lower symmetry, is said to have a four-molecule orthorhombic unit.

(aq) Making use of photographic data and some spectrometric measurements a pronouncedly layer-like structure has been assigned to gibbsite (hydrargillite),  $Al_2O_3 \cdot 3H_2O$ . Atoms of the 8  $Al(OH)_3$  molecules within the large monoclinic unit that has been used are in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;  $\pm(\frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z)$ . The chosen atomic parameters are stated in Table III.

TABLE III. PARAMETERS OF THE ATOMS IN GIBBSITE,  $Al_2O_3 \cdot 3H_2O$

Atom	$x$	$y$	$z$
Al(1)	0.177	0.520	-0.005
Al(2)	.333	.020	-.005
O(1)	.182	.202	-.110
O(2)	.682	.672	-.110
O(3)	.515	.132	-.110
O(4)	-.015	.632	-.110
O(5)	.298	.702	-.100
O(6)	.838	.172	-.100

(ar) The tetragonal unit of  $Ag_2SO_4 \cdot 4NH_3$  contains two molecules. Its atoms are in the following special and general positions of  $V_d^4$ :



S: (a)  $000; \frac{1}{2}\frac{1}{2}\frac{1}{2}$       Ag: (d)  $0\frac{1}{2}u; \frac{1}{2}0\bar{u}; \frac{1}{2}, 0, \frac{1}{2}-u; 0, \frac{1}{2}, u+\frac{1}{2}$  with  $u=0.50$   
 O: (e)  $xyz; y\bar{x}\bar{z}; \bar{x}\bar{y}z; \bar{y}x\bar{z}; \frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z; \frac{1}{2}-y, \frac{1}{2}-x, z+\frac{1}{2}; x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z;$   
 $y+\frac{1}{2}, x+\frac{1}{2}, z+\frac{1}{2}$  with  $x=0.14, y=0.07, z=0.15$   
 NH<sub>4</sub>: (e)  $x'y'z'$ ; etc. with  $x'=0.10, y'=0.30, z'=0.50$ .

As Figure 314 shows, this arrangement is a packing of SO<sub>4</sub> tetrahedra and linear NH<sub>3</sub>-Ag-NH<sub>3</sub> groups.

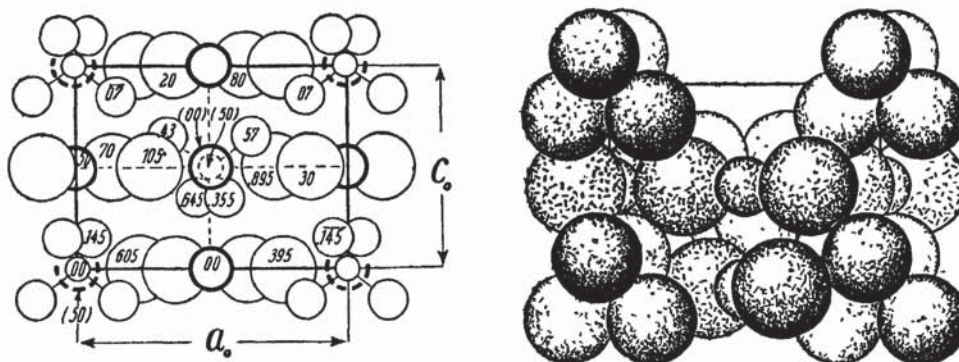


FIG. 314a.—(left) The structure of  $\text{Ag}_2\text{SO}_4 \cdot 4\text{NH}_3$  projected on one of the  $a$ -faces of its tetragonal unit. The largest circles are NH<sub>3</sub> groups, the smallest are S atoms; Ag atoms are shown as thick rings.

FIG. 314b.—(right) A packing drawing of  $a$ . The small Ag atoms and the large NH<sub>3</sub> groups are line-shaded.

(as) The monoclinic units assigned to  $\text{BaNi}(\text{CN})_4 \cdot 4\text{H}_2\text{O}$  and to the isomorphous  $\text{BaPt}(\text{CN})_4 \cdot 4\text{H}_2\text{O}$  contain four molecules. The space group is given as  $C_{2h}^6$  with Pt (or Ni) atoms at  $000; 00\frac{1}{2}; \frac{1}{2}\frac{1}{2}0; \frac{1}{2}\frac{1}{2}\frac{1}{2}$  and Ba atoms at  $0\frac{3}{8}\frac{1}{4}; 0\frac{5}{8}\frac{3}{4}; \frac{1}{2}\frac{1}{8}\frac{1}{4}; \frac{1}{2}\frac{7}{8}\frac{3}{4}$ . Other atomic positions have not been fixed.

(at) Two determinations of the structure of  $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$  have been made from photographic data. The first (1931, 401), based on  $D_{4h}^{18}$ , is undoubtedly wrong. The other places atoms in the following positions of  $V_d^{10}$  (written not as the face-centered grouping of 1930, 352, p. 77 but in terms of a diagonal body-centered unit):

S: (c)  $\frac{1}{2}00; 0\frac{1}{2}0; \frac{1}{2}0\frac{1}{2}; 0\frac{1}{2}\frac{1}{2}$       Be: (b)  $00\frac{1}{4}; 0u\frac{3}{4}; \frac{1}{2}\frac{1}{2}\frac{1}{4}; \frac{1}{2}\frac{1}{2}\frac{3}{4}$   
 O: (i)  $xyz; yx\bar{z}; \bar{x}\bar{y}z; \bar{y}\bar{x}\bar{z}; x+\frac{1}{2}, \frac{1}{2}-y, z; y+\frac{1}{2}, \frac{1}{2}-x, \bar{z};$   
 $\frac{1}{2}-y, x+\frac{1}{2}, \bar{z}; \frac{1}{2}-x, y+\frac{1}{2}, z$  and 8 similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ .

For the sulfate oxygen atoms  $x=0.40, y=0.13, z=0.08$ . The water molecules, likewise in (i), are given the parameters  $x'=0.10, y'=0.14, z'=0.16$ .

In this arrangement the beryllium atoms, which of course cannot be located with certainty by the X-ray data, have been placed at the centers of the groups of water molecules. The structure as a whole thus is a packing together of SO<sub>4</sub> and Be(H<sub>2</sub>O)<sub>4</sub> tetrahedra (Figure 315).

(au) The orthorhombic cell which has been given to  $[\text{Co}(\text{NH}_3)_4]\text{ClO}_4$  contains four molecules. Its space group is said to be  $C_{2v}^7$ .

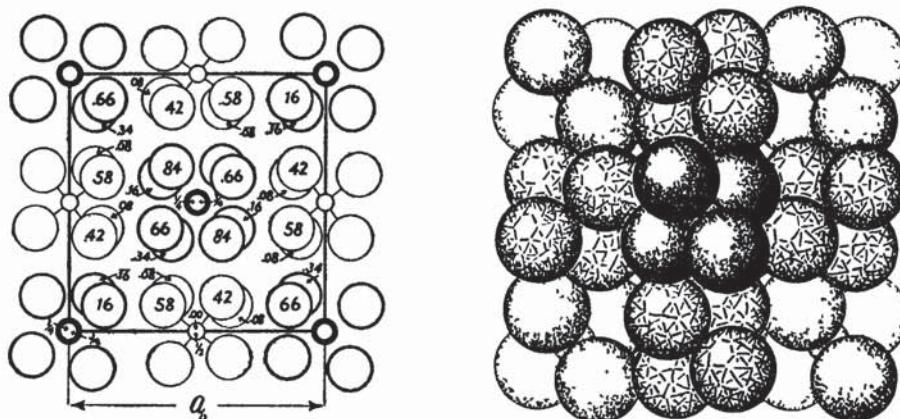


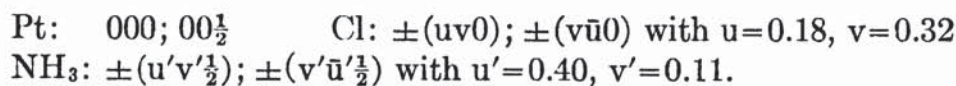
FIG. 315a.—(left) The atomic arrangement in tetragonal  $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$  projected on its  $c$ -face. Be atoms are shown as thick rings, S atoms as the smallest circles. The  $\text{H}_2\text{O}$  molecules are thickened large circles.

FIG. 315b.—(right) A packing drawing of  $a$ . Oxygens of the  $\text{SO}_4$  tetrahedra are line-shaded; Be atoms show within their water tetrahedra.

(av) The orthorhombic unit of  $1,6[\text{Co}(\text{NO}_2)_2(\text{NH}_3)_4]\text{Cl}$  is reported to contain 16 molecules.

(aw) The unit found for  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 4\text{H}_2\text{O}$  possesses four molecules; its space group is thought to be  $C_{2h}^4$ .

(ax) The two studies that have been made of Magnus' green salt,  $[\text{Pt}(\text{NH}_3)_4]\text{PtCl}_4$ , do not agree in the height of its one-molecule tetragonal unit. The following atomic positions, suggested in 1932, 119, are not adequately established by the published data:



In 1931, 193 it is said that Magnus' red salt has not only the same composition as the green salt but a cell of similar size and shape ( $a_0=6.293$  A,  $c_0=5.25$  A). Another determination (1932, 119) concludes that it is orthorhombic with  $a_0=7.9$  A,  $b_0=8.2$  A,  $c_0=7.9$  A.

(ay) The unit cube of  $\text{Zn}(\text{ClO}_4)_2 \cdot 4\text{NH}_3$  is said to contain four molecules. It would be interesting to establish, by further chemical analyses, that this salt is really a tetrammoniate and not a hexammoniate.

(az) A recent study of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , based on photographic data and a Fourier analysis of some spectrometric measurements, has led to an arrangement which places the copper atoms in its bimolecular triclinic unit in the symmetry centers  $000$  and  $\frac{1}{2}\frac{1}{2}0$ . The other atoms are in general positions  $\pm(xyz)$  of  $C_1^1$  with the parameters of Table IV.

As can be seen from Figure 316 the water molecules are of two kinds. Four of them are coordinated with the copper atoms. The fifth, like zeolitic water, occupies a hole in the structure, its neighbors being two

TABLE IV. PARAMETERS OF THE ATOMS IN  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 

Atom	No. per cell	$x$	$y$	$z$
Cu(1)	1	0	0	0
Cu(2)	1	$\frac{1}{2}$	$\frac{1}{2}$	0
S	2	0	0.28	0.64
O(1)	2	0.89	.15	.69
O(2)	2	.24	.31	.82
O(3)	2	.86	.38	.64
O(4)	2	.02	.30	.38
H <sub>2</sub> O(1)	2	.83	.08	.16
H <sub>2</sub> O(2)	2	.29	.11	.17
H <sub>2</sub> O(3)	2	.48	.41	.32
H <sub>2</sub> O(4)	2	.76	.42	.01
H <sub>2</sub> O(5)	2	.43	.12	.65

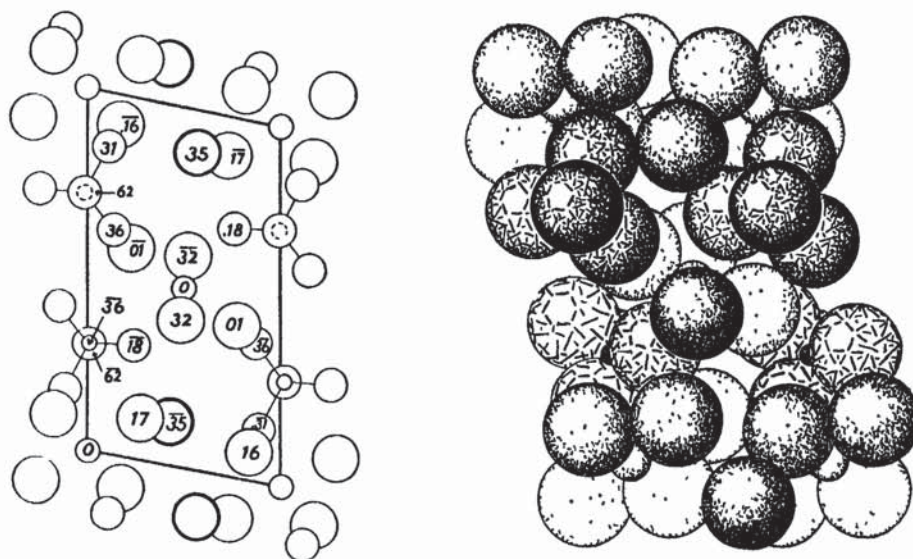


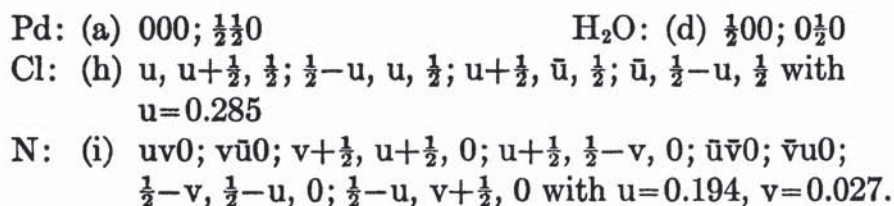
FIG. 316a.—(left) A projection of the triclinic  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  arrangement viewed down the  $c$ -axis (after 1934, 15a). The smallest circles are S atoms, the largest are  $\text{H}_2\text{O}$  molecules. Non-coordinated  $\text{H}_2\text{O}$  molecules are thickened. Atoms of the  $\text{SO}_4$  ions are connected by light lines.

FIG. 316b.—(right) A packing drawing of *a*. The sulfate oxygens are line-shaded; small black S atoms show in two of the  $\text{SO}_4$  groups. All  $\text{H}_2\text{O}$  molecules, whether coordinated with the Cu atoms or not, are dot-shaded.

water molecules and two sulfate oxygen atoms. Each copper atom is surrounded by an octahedron of atoms. Four of these are its coordinated water molecules situated approximately at the corners of a square, the other two, farther away, are oxygens from different sulfate groups.

(*ba*) Three unit cells have been suggested for  $[\text{Pd}(\text{NH}_3)_4]\text{Cl}_2 \cdot \text{H}_2\text{O}$  and its platinum analogue. The small one-molecule and the large four-molecule prisms are presumably wrong. The most recent study (1934, 71),

using Laue and spectral photographic data, places the atoms of the two-molecule cells (Table I) in the following special positions of  $D_{4h}^5$ :



Four coordinated  $\text{NH}_3$  molecules are arranged about a central palladium atom at the corners of a square ( $\text{NH}_3\text{-Pd}=2.02 \text{ \AA}$ ). The extent to which packing prevails in basal planes of this structure and the way the  $\text{H}_2\text{O}$  molecules lie in holes present in these planes can best be seen from Figure 317.

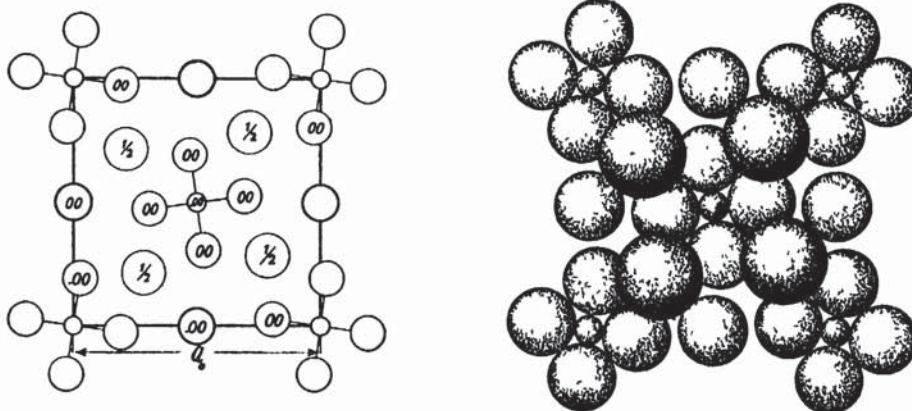
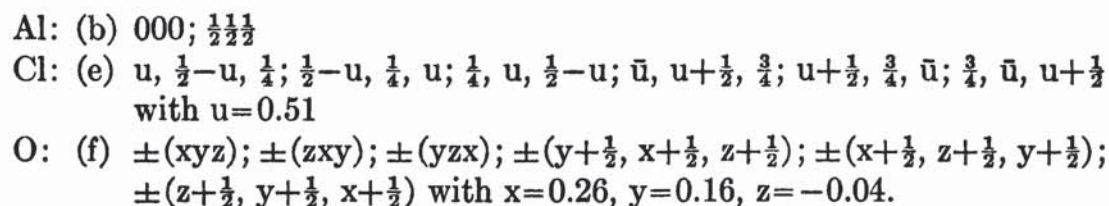


FIG. 317a.—(left) The atomic arrangement assigned to tetragonal  $\text{Pd}(\text{NH}_3)_4\text{Cl}_2\cdot\text{H}_2\text{O}$  as projected on its  $c$ -face. The largest circles are Cl ions, the smallest are Pd atoms. Intermediate circles are  $\text{NH}_3$  if light in outline,  $\text{H}_2\text{O}$  if heavy.

FIG. 317b.—(right) A packing drawing of *a*. The  $\text{H}_2\text{O}$  and  $\text{NH}_3$  molecules are not distinguished.

(bb) The rhombohedral unit found for  $\text{AlCl}_3\cdot 6\text{H}_2\text{O}$ , and for the isomorphous  $\text{CrCl}_3\cdot 6\text{H}_2\text{O}$ , contains two molecules. An atomic arrangement of both salts, based on  $D_{3d}^6$ , has been determined as:



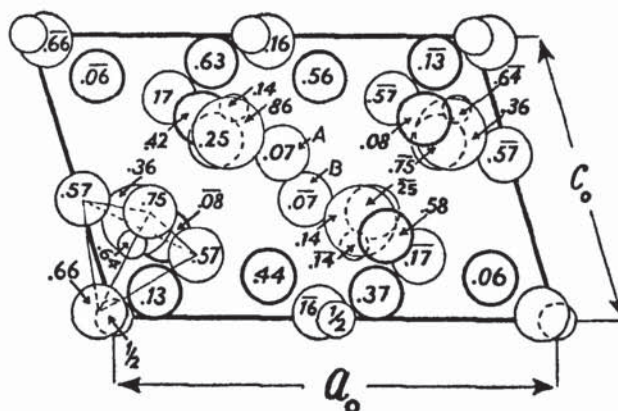
(bc) The two-molecule cells of  $\text{MgSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$  and of the many crystals isomorphous with it have arrangements developed from

$C_{2h}^5$ . A structure deduced from an elaborate consideration of photographic data has all atoms except magnesium in the general positions: (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$ , with the parameters listed in Table V. Mg atoms are at (a)  $000; \frac{1}{2}\frac{1}{2}0$ . This arrangement can scarcely be correct because while the  $NH_4$ -O and Mg- $H_2O$  separations are satisfactory, sulfate oxygens belonging to different  $SO_4$  groups (such as A and B in Figure 318) are much too near together (O-O=ca 2.10 Å).

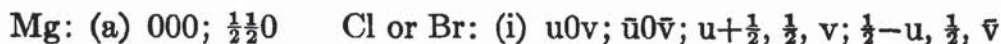
TABLE V. PARAMETERS OF THE ATOMS IN  $MgSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$

Atom	$x$	$y$	$z$
$NH_4$	0.120	0.357	0.345
S	.090	.635	.260
O(1)	.153	.746	.376
O(2)	-.032	.656	.020
O(3)	.227	.566	.225
O(4)	.012	.571	.419
$H_2O(1)$	.108	.127	.094
$H_2O(2)$	.168	-.084	.290
$H_2O(3)$	.123	-.062	-.150

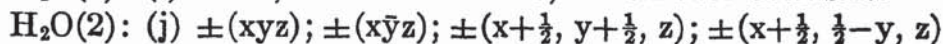
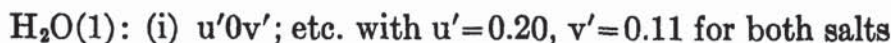
FIG. 318.—The unit cell of the structure proposed for  $MgSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$  as projected on its b-face. The smallest circles are S atoms, the largest  $NH_4$  ions. Circles slightly larger than S are Mg ions. Molecules of  $H_2O$  are distinguished from sulfate O atoms by being heavily ringed.



(bd) The monoclinic units of  $MgBr_2 \cdot 6H_2O$  and  $MgCl_2 \cdot 6H_2O$  contain two molecules. Photographic data have been used in choosing the following arrangement, based on  $C_{2h}^3$ :



For Cl,  $u=0.320$ ,  $v=0.615$ ; for Br,  $u=0.318$ ,  $v=0.615$ .



with  $x=0.96$ ,  $y=0.20$  for both salts,  $z=0.230$  for the chloride and 0.225 for the bromide.

(be) The four molecules in the elongated tetragonal unit of  $NiSO_4 \cdot 6H_2O$  are arranged according to the demands of the enantiomorphic pair

of space groups  $D_4^4$  and  $D_4^8$ . Expressed in terms of  $D_4^4$  the atomic positions found for this crystal are:

Ni: (a)  $uu0$ ;  $\bar{u}\bar{u}\frac{1}{2}$ ;  $u+\frac{1}{2}$ ,  $\frac{1}{2}-u$ ,  $\frac{3}{4}$ ;  $\frac{1}{2}-u$ ,  $u+\frac{1}{2}$ ,  $\frac{1}{4}$  with  $u=0.71$

S: (a)  $u'u'0$ ; etc. with  $u'=0.21$

H<sub>2</sub>O and O: (b)  $xyz$ ;  $y+\frac{1}{2}$ ,  $\frac{1}{2}-x$ ,  $z+\frac{3}{4}$ ;  $\bar{x}$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ ;  $\frac{1}{2}-y$ ,  $x+\frac{1}{2}$ ,  $z+\frac{1}{4}$ ;  
 $\frac{1}{2}-x$ ,  $y+\frac{1}{2}$ ,  $\frac{1}{4}-z$ ;  $\bar{y}$ ,  $\bar{x}$ ,  $\frac{1}{2}-z$ ;  $x+\frac{1}{2}$ ,  $\frac{1}{2}-y$ ,  $\frac{3}{4}-z$ ;  $yx\bar{z}$

with the parameters of Table VI.

TABLE VI. PARAMETERS OF THE OXYGEN ATOMS IN  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$

Atom	$x$	$y$	$z$
O(1)	0.12	0.12	0.068
O(2)	.43	.17	.000
H <sub>2</sub> O(1)	.67	.45	.054
H <sub>2</sub> O(2)	.97	.75	.054
H <sub>2</sub> O(3)	.56	.86	.077

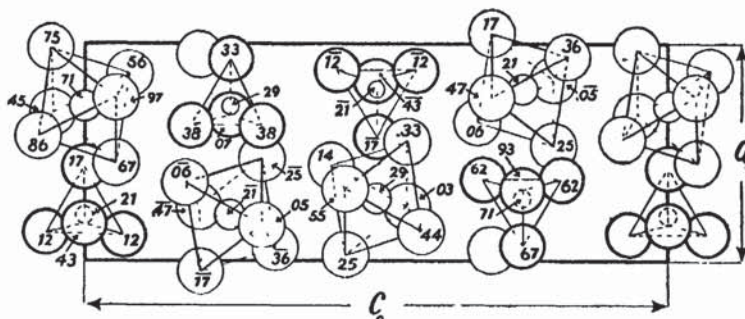


FIG. 319a.—A projection of the tetragonal  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  grouping upon one of its  $a$ -faces. In this figure the sulfate oxygens are heavily ringed, the H<sub>2</sub>O molecules lightly outlined. Medium sized circles, at the centers of the water octahedra, are the Ni atoms.

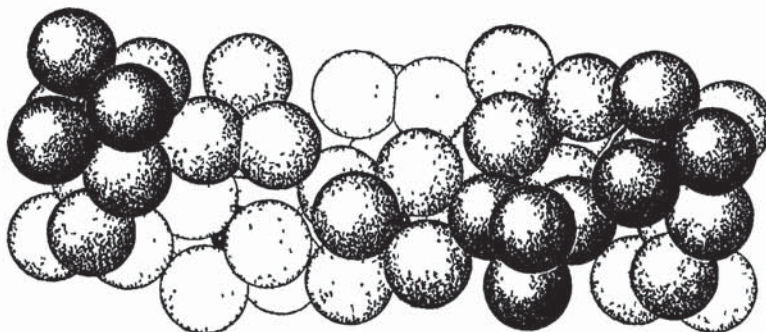


FIG. 319b.—A packing drawing of  $a$ . The Ni atoms appear as black spheres within their H<sub>2</sub>O octahedra; S atoms do not show.

The structure as a whole is a packing of  $\text{SO}_4$  tetrahedra and  $(\text{Ni} \cdot 6\text{H}_2\text{O})$  octahedra (Figure 319).

(bf) In a recent study a set of atomic positions, developed from  $C_{3i}^1$ , has been proposed for  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ . They are:

Sr: (a) 000 Cl: (d)  $\frac{1}{3}\frac{2}{3}u$ ;  $\frac{2}{3}\frac{1}{3}\bar{u}$  with  $u=\frac{1}{2}$   
 H<sub>2</sub>O: (g)  $\pm(xyz)$ ;  $\pm(y-x, \bar{x}, z)$ ;  $\pm(\bar{y}, x-y, z)$  with  $x=y=0.387$ ,  $z=0.0$ .

The published data, which are scanty, are insufficient to establish this structure.

(bg) The previously chosen unit for **MgPt(CN)<sub>4</sub>·7H<sub>2</sub>O** ( $a_0=14.6$  Å,  $c_0=3.13$  Å) was too small. The true cell is twice as high,  $c_0=6.26$  Å, and contains four molecules.

(bh) The space group of the four-molecule cell of **3CdSO<sub>4</sub>·8H<sub>2</sub>O** is said to be  $C_{2h}^6$ .

(bi) Estimates of the intensities of powder lines have been used to suggest the following atomic arrangement, based on  $D_{4h}^1$ , for **SrO<sub>2</sub>·8H<sub>2</sub>O**:

Sr: (a) 000 O: (h)  $\frac{1}{2}\frac{1}{2}w$ ;  $\frac{1}{2}\frac{1}{2}\bar{w}$  with  $w=0.10$   
 H<sub>2</sub>O: (r)  $\pm(uuv)$ ;  $\pm(u\bar{u}v)$ ;  $\pm(\bar{u}\bar{u}v)$ ;  $\pm(\bar{u}uv)$  with  $u=0.20$ ,  $v=0.25$ .

In this arrangement strontium atoms are surrounded by cubes of H<sub>2</sub>O molecules with the somewhat short Sr-H<sub>2</sub>O distance of 2.26 Å; inside the O<sub>2</sub> group the atomic separation is 1.11 Å.

(bj) The large unit found for **2Na<sub>3</sub>PO<sub>4</sub>·NaF·19H<sub>2</sub>O** holds 40 molecules. The data are those demanded by  $O_h^8$ .

(bk) The unit cubes of several **phosphotungstic** and **phosphomolybdic acids** and their salts crystallizing with  $30\pm 1$  molecules of H<sub>2</sub>O have been found to contain eight molecules. Their space group is said to be  $O_h^7$ .

Powder photographic data have been used to determine the shape and size of the  $PW_{12}O_{40}$  anion in the partially dehydrated **H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>·5**, or **7**, H<sub>2</sub>O and to suggest probable positions for the H<sub>2</sub>O molecules. The unit cube of this lower hydrate contains two molecules; the atoms of its anions are in the following special positions of  $O_h^4$ :

P: (2a) 000;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$   
 W: (24u)  $uuv$ ;  $u\bar{u}\bar{v}$ ;  $\bar{u}u\bar{v}$ ;  $\bar{u}\bar{u}v$ ;  $vuu$ ;  $\bar{v}u\bar{u}$ ;  $\bar{v}\bar{u}u$ ;  $v\bar{u}\bar{u}$ ;  $uvu$ ;  $\bar{u}\bar{v}u$ ;  $u\bar{v}\bar{u}$ ;  $\bar{u}v\bar{u}$ ;  
 $\frac{1}{2}-u, \frac{1}{2}-u, \frac{1}{2}-v$ ;  $\frac{1}{2}-u, u+\frac{1}{2}, v+\frac{1}{2}$ ;  $u+\frac{1}{2}, \frac{1}{2}-u, v+\frac{1}{2}$ ;  
 $u+\frac{1}{2}, u+\frac{1}{2}, \frac{1}{2}-v$ ;  $\frac{1}{2}-v, \frac{1}{2}-v, \frac{1}{2}-u$ ;  $v+\frac{1}{2}, \frac{1}{2}-u, u+\frac{1}{2}$ ;  
 $v+\frac{1}{2}, u+\frac{1}{2}, \frac{1}{2}-u$ ;  $\frac{1}{2}-v, u+\frac{1}{2}, u+\frac{1}{2}$ ;  $\frac{1}{2}-u, \frac{1}{2}-v, \frac{1}{2}-u$ ;  
 $u+\frac{1}{2}, v+\frac{1}{2}, \frac{1}{2}-u$ ;  $\frac{1}{2}-u, v+\frac{1}{2}, u+\frac{1}{2}$ ;  $u+\frac{1}{2}, \frac{1}{2}-v, u+\frac{1}{2}$

with  $u=0.205$ ,  $v=0.006$

O(1): (8d)  $u'u'u'$ ;  $\bar{u}'\bar{u}'\bar{u}'$ ;  $\frac{1}{2}-u', \frac{1}{2}-u', \frac{1}{2}-u'$ ;  $u'+\frac{1}{2}, \frac{1}{2}-u', u'+\frac{1}{2}$ ;  
 $u'\bar{u}'\bar{u}'$ ;  $\bar{u}'\bar{u}'u'$ ;  $\frac{1}{2}-u', u'+\frac{1}{2}, u'+\frac{1}{2}$ ;  $u'+\frac{1}{2}, u'+\frac{1}{2}, \frac{1}{2}-u'$

with  $u=0.081$

O(2): (24u)  $u_1u_1v_1$ ; etc. with  $u_1=-0.080$ ,  $v_1=-0.234$

O(3): (24u)  $u_2u_2v_2$ ; etc. with  $u_2=0.123$ ,  $v_2=0.292$

O(4): (24u)  $u_3u_3v_3$ ; etc. with  $u_3=-0.312$ ,  $v_3=-0.008$ .

The shape of one of these  $\text{PW}_{12}\text{O}_{40}$  ions is shown in Figure 320. It consists of a central  $\text{PO}_4$  tetrahedron and 12 surrounding  $\text{WO}_6$  octahedra that share oxygen atoms with it. The  $\text{H}_2\text{O}$  molecules are considered to be of two sorts: three in the positions:  $(6e) 00\frac{1}{2}; \frac{1}{2}00; 0\frac{1}{2}0; \frac{1}{2}\frac{1}{2}0; \frac{1}{2}0\frac{1}{2}; 0\frac{1}{2}\frac{1}{2}$  and four at  $(4e) \frac{3}{4}\frac{3}{4}\frac{3}{4}; \frac{3}{4}\frac{1}{4}\frac{1}{4}; \frac{1}{4}\frac{3}{4}\frac{1}{4}; \frac{1}{4}\frac{1}{4}\frac{3}{4}$ , if the crystal is a heptahydrate.

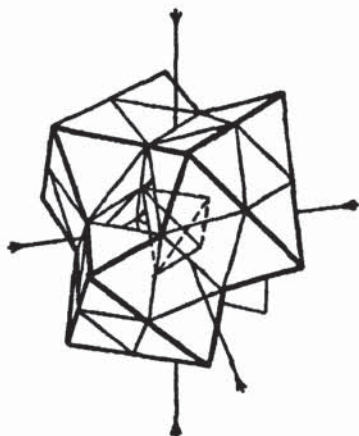


FIG. 320.—The  $\text{PW}_{12}\text{O}_{40}$  ion is considered to have a shape suggested by this diagram. The central P atom is surrounded by four O atoms at the corners of the dotted tetrahedron. About it are 12 oxygen octahedra which share atoms with one another and with the  $\text{PO}_4$  group and which have W atoms at their centers (after 1934, 131).

(bl) The unit assigned to the complex phosphate wavellite,  $4(\text{PO}_4)\text{Al}\cdot 2\text{Al}(\text{OH})_3\cdot 9\text{H}_2\text{O}$ , is bimolecular.

(bm) The unit cell of legrandite has one molecule of the composition  $\text{Zn}_{14}(\text{AsO}_4)_9\text{OH}\cdot 12\text{H}_2\text{O}$ .

(bn) The monoclinic unit given to  $[\text{Co}_{(\text{NH}_3)_4}^{\text{CO}}]_2\text{SO}_4\cdot 3\text{H}_2\text{O}$  contains two molecules; the space group is thought to be  $C_2^1$ .

(bo) Two molecules are found in the unit of  $\text{MgNH}_4\text{AsO}_4\cdot 6\text{H}_2\text{O}$ ; its space group is  $C_{2v}^7$ .

(bp) The structure recently developed for  $\text{LiClO}_4\cdot 3\text{H}_2\text{O}$  differs from that previously found for the isomorphous  $\text{LiI}\cdot 3\text{H}_2\text{O}$  (f) in the positions of the lithium atoms. In the perchlorate, atoms are in the following special positions of  $C_{6v}^4$ :

Li: (a)  $00u_1; 0, 0, u_1+\frac{1}{2}$  with  $u_1=0.25$

Cl: (b)  $\frac{1}{3}\frac{2}{3}u'; \frac{2}{3}, \frac{1}{3}, u'+\frac{1}{2}$  with  $u'=0$

O(1): (b)  $\frac{1}{3}\frac{2}{3}w; \frac{2}{3}, \frac{1}{3}, w+\frac{1}{2}$  with  $w=0.278$

O(2): (c)  $u\bar{u}v; 2\bar{u}, \bar{u}, v; u, 2u, v; \bar{u}, u, v+\frac{1}{2}; 2u, u, v+\frac{1}{2}; \bar{u}, 2\bar{u}, v+\frac{1}{2}$

with  $u=0.439, v=-0.092$

$\text{H}_2\text{O}$ : (c)  $u_2\bar{u}_2v_2$ ; etc. with  $u_2=0.125, v_2=0.50$ .

This arrangement, which coordinates six  $\text{H}_2\text{O}$  molecules about each lithium atom (Figure 321), is practically identical with one found from quantitative spectrometric data.\*

\* Unpublished measurements of R. B. Corey in this laboratory. The parameters derived from this study are  $u_1(\text{Li})=0.25, u'(\text{Cl})=0, w(\text{O})=0.265, u(\text{O})=0.435, v(\text{O})=-0.09, u_2(\text{H}_2\text{O})=0.125, v_2(\text{H}_2\text{O})=0.50$ .



It is probable that the lithium atoms in  $\text{LiI} \cdot 3\text{H}_2\text{O}$  should be in (a)  $00u_1; 0, 0, u_1 + \frac{1}{2}$  instead of the earlier (b)  $\frac{1}{3}\frac{2}{3}u_1; \frac{2}{3}, \frac{1}{3}, u_1 + \frac{1}{2}$ . Photographic data lead to the parameters:  $u_1(\text{Li})=0.25$ ,  $u'(I)=0$ ,  $u_2(\text{H}_2\text{O})=0.142$ ,  $v_2(\text{H}_2\text{O})=0.50$ .

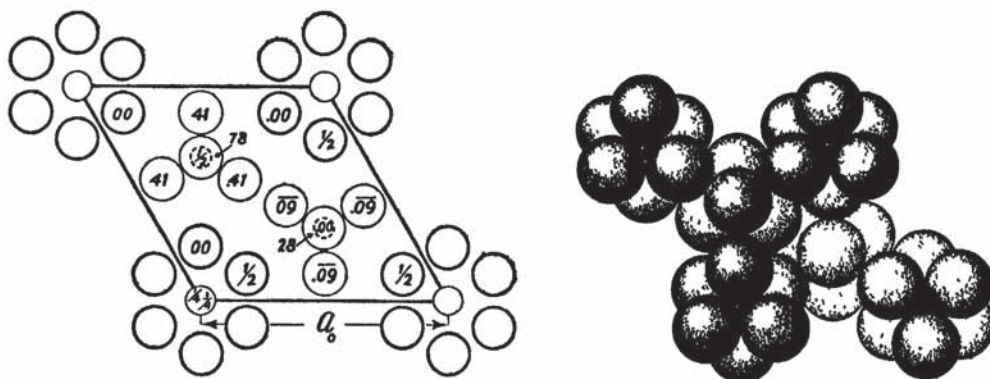


FIG. 321a.—(left) A basal projection of the structure found for  $\text{LiClO}_4 \cdot 3\text{H}_2\text{O}$ . Li atoms are at the corners of the diagram surrounded by the heavily outlined water molecules.

FIG. 321b.—(right) A packing drawing of *a* showing the tetrahedral  $\text{ClO}_4$  ions and (black) Li ions equidistant from six  $\text{H}_2\text{O}$  molecules. The O atoms and  $\text{H}_2\text{O}$  molecules, of equal size, are not distinguished by different shadings.

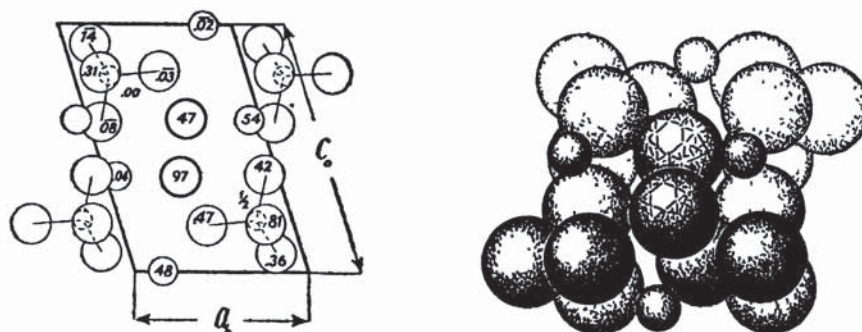


FIG. 321c.—(left) A projection upon its *b*-face of the monoclinic structure deduced for  $\text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ . Atoms of the  $\text{SO}_4$  ions are connected by light lines; the  $\text{H}_2\text{O}$  molecules are heavily ringed.

FIG. 321d.—(right) A packing drawing of *c* with the  $\text{H}_2\text{O}$  molecules distinguished by line-shading. The S atoms within their tetrahedra cannot be seen.

(*bq*) The hexagonal unit prism of  $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$  is bimolecular. The space group is reported to be  $C_6^6$  or  $C_{6h}^2$ .

(*br*) The atoms of the two  $\text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$  molecules contained within the unit cell are in general positions of  $C_2^2$  with the coordinates (a)  $xyz$ ;  $\bar{x}$ ,  $y + \frac{1}{2}$ ,  $\bar{z}$ . Parameters, determined from photographic data, are those of Table VII. In this structure (Figure 321c and d) the lithium atoms are

TABLE VII. PARAMETERS OF THE ATOMS IN  $\text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ 

Atom	$x$	$y$	$z$
S	0.208	0.000	-0.211
O(1)	.153	-.139	-.083
O(2)	.500	-.014	-.192
O(3)	.078	-.078	-.397
O(4)	.161	.305	-.197
H <sub>2</sub> O	.569	.472	-.386
Li(1)	-.069	.542	-.386
Li(2)	.167	.478	.003

surrounded by tetrahedra consisting of three sulfate oxygen atoms and one H<sub>2</sub>O molecule.

(bs) The unit prism of  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$  contains four molecules; its space group is said to be  $V_h^{16}$ .

(bt) Four molecules are within the cell of  $\text{AgNO}_3 \cdot 2\text{NH}_3$ . The space group is  $C_{2v}^{10}$ .

### Miscellaneous Inorganic Compounds

(ca) The cube lengths found for a large number of **atopites** and atopite-like minerals, and for ochers of calcium and of lead vary between ca 10.25 Å and ca 10.43 Å.

(cb) A partial atomic arrangement for **azurite**,  $2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$ , has been based on some rotation photographs and spectrometer data. Copper atoms have been placed in (a)  $000$ ;  $0\frac{1}{2}\frac{1}{2}$  and in general positions of  $C_{2h}^5$ : (c)  $\pm(xyz)$ ;  $\pm(\bar{x}, y + \frac{1}{2}, \frac{1}{2} - z)$  with  $x=0.25$ ,  $y=0.486$ ,  $z=0.083$ . Suggested parameters for the other atoms are not proved by the existing data.

(cc) The mineral **hambergite**,  $\text{Be}_2\text{BO}_3(\text{OH})$ , has been given a structure which explains both a series of spectrometric intensities and the qualita-

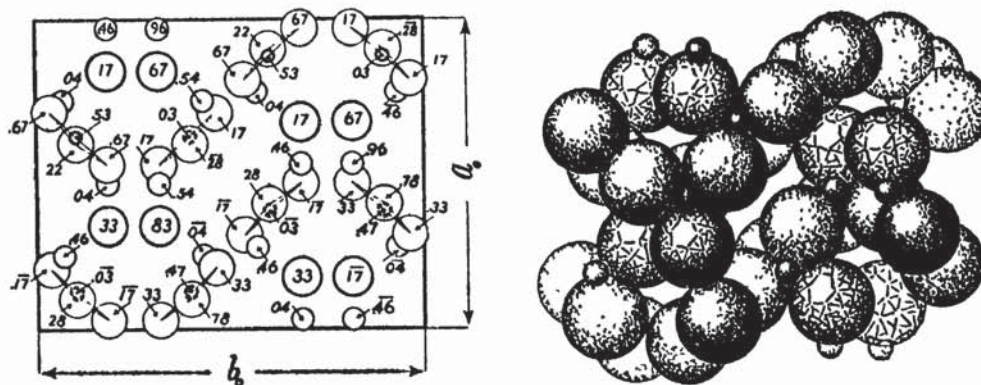


FIG. 322a.—(left) A projection upon the  $c$ -face of the orthorhombic structure assigned to hambergite,  $\text{Be}_2\text{BO}_3(\text{OH})$ . Atoms of the  $\text{BO}_3$  groups are connected by light lines. Intermediate circles represent Be atoms; the  $(\text{OH})$  groups are heavily ringed.

FIG. 322b.—(right) A packing drawing of  $a$ . Small spheres are the Be atoms; the B atoms do not show. Hydroxyl groups are line-shaded.

TABLE VIII. THE CRYSTAL STRUCTURES OF MISCELLANEOUS INORGANIC COMPOUNDS

Substance, symmetry and type	$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Arsenoklasite	9.19	18.01	5.79		4	1931, 10.
$\text{Mn}_3(\text{AsO}_4)_2 \cdot 2\text{Mn}(\text{OH})_2$						
$\text{AsI}_3 \cdot 3\text{S}_8$	14.2		4.48	$\text{C}_{3v}^1$	1	1931, 186. 1932, 323, 324; 1933, 4.
Atopites (Ca, Mn, Na) $_2\text{Sb}_2(\text{O}, \text{OH}, \text{F})_7$	(ca)					
Azurite	(cb)					
$2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$	4.96	5.83	10.27	$\text{C}_{2h}^6$	2	1932, 72; 1933, 71.
Bindheimite	(ca)				8	1933, 316.
$\text{Pb}_2\text{Sb}_2\text{O}_7$	10.44					
Binnite (Cu, Fe) $_{12}\text{As}_4\text{S}_{13}$	(cj)			$\text{T}_d^3$	2	1934, 205.
Boleite	10.19					
Boracite	(aa'), (ck)	17.07	12.07		8	1930, 105b. 1932, 307; 1933, 201; 1934, 171.
$\text{Mg}_5\text{ClB}_7\text{O}_{13}$ (low temp. form)						
Boracite	(ck)	12.1			4	1932, 307; 1934, 171.
$\text{Mg}_5\text{ClB}_7\text{O}_{13}$ (high temp. form, > 265°C)						
Bourbonite	8.10	8.65	7.75	$\text{V}_h^{18}$	4	1932, 343.
$\text{CuPbSbS}_3$						
$\text{CHI}_3 \cdot 3\text{S}_8$	14.1		4.44	$\text{C}_{3v}^1$	1	1931, 186. 1930, 105b.
Cumengeite						
Hambergite	(cc)	9.73	4.42	$\text{V}_h^{18}$	8	1931, 495.
$\text{Bc}_2\text{BO}_2(\text{OH})$						
Hanksite	10.52		21.25	$\text{C}_{6h}^2$	2	1931, 134.
$9\text{Na}_2\text{SO}_4 \cdot 2\text{Na}_2\text{CO}_3 \cdot \text{KCl}$						
Koppite	(ca)	10.37			8	1931, 57.
(Ca, Ce, Na, K) $_2(\text{Cb}, \text{Fe})_2\text{O}_6(\text{O}, \text{OH}, \text{F})$						
Lewisite	(ca)	10.269			8	1932, 298.
(Ca, Fe, Na) $_2(\text{Sb}, \text{Ti})_2(\text{O}, \text{OH})_7$						
Malachite	(cd)	9.38	3.18	$\text{C}_{2h}^2$	4	1932, 71; 1933, 71.
$\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$		11.95				
		$\beta = 91^\circ 3'$				

Substance, symmetry and type	$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Microelite (Ca, Na) <sub>2</sub> (Ta, Cb) <sub>2</sub> (O, F) <sub>7</sub>	10.381			(ca)	8	1933, 374.
Northupite Na <sub>2</sub> Mg(CO <sub>3</sub> ) <sub>2</sub> ·NaCl	13.98			T <sub>h</sub> <sup>1</sup>	16	1931, 134, 413; 1933, 491.
Brominated Northupite Na <sub>2</sub> Mg(CO <sub>3</sub> ) <sub>2</sub> ·NaBr	14.17			T <sub>h</sub> <sup>1</sup>	16	1931, 413.
Triphosphonitrile (PNCl <sub>2</sub> ) <sub>3</sub>	14.00	6.16	12.94	V <sub>h</sub> <sup>16</sup>	4	1932, 235.
Tetraphosphonitrile (PNCl <sub>2</sub> ) <sub>4</sub>	10.79		5.93	C <sub>4h</sub>	2	1932, 235.
Potassium Trithionate K <sub>2</sub> S <sub>3</sub> O <sub>6</sub>	9.77	13.63	5.76	V <sub>h</sub> <sup>16</sup>	4	1934, 305, 305b.
Potassium Pyrosulfite K <sub>2</sub> S <sub>2</sub> O <sub>5</sub>	6.95	6.19	7.55	C <sub>2h</sub>	2	1932, 487, 490.
Pesudoboleite				(ac)		1930, 105b.
Pyrrhite	10.40			(ca)	8	1932, 296.
Pyroaurite Fe(OH) <sub>3</sub> ·3Mg(OH) <sub>2</sub> ·3H <sub>2</sub> O	3.089		23.23	(ch)	4	1931, 11.
Pyrochlore (Na, Ca) <sub>2</sub> (Cb, Ti) <sub>2</sub> (O, F) <sub>7</sub>	10.376			(ca)	8	1930, 391.
Romeite (Ca, Na, Mn) <sub>2</sub> Sb <sub>2</sub> (O, OH, F) <sub>7</sub>	10.261			(ca)	8	1932, 324, 496; 1933, 316.
Schneebergite (Ca, Na, Fe) <sub>3</sub> Sb <sub>2</sub> O <sub>6</sub> (OH)	10.296			(ca)	8	1932, 496.
Sulphohalite 2Na <sub>2</sub> SO <sub>4</sub> ·NaCl·NaF	10.08			O <sub>h</sub> <sup>1</sup>	4	1934, 347.
Tetradymite	10.31		$\alpha=24^\circ 10'$	(cm)	1	1934, 332.
Tychite 2MgCO <sub>3</sub> ·2Na <sub>2</sub> CO <sub>3</sub> ·Na <sub>2</sub> SO <sub>4</sub>	13.90			T <sub>h</sub> <sup>1</sup>	8	1931, 413; 1933, 491.
Voltaites				(ad), (ci)		1932, 171; 1933, 150, 151.
Basic Zinc Chloride ZnCl <sub>2</sub> ·4Zn(OH) <sub>2</sub>	12.56		15.84	Hex.	8	1933, 131.

tive data from oscillation photographs. Atoms in its eight-molecule unit are in general positions (c)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, \bar{z})$ ;  $\pm(\bar{x}, y+\frac{1}{2}, \frac{1}{2}-z)$ ;  $\pm(\frac{1}{2}-x, \bar{y}, z+\frac{1}{2})$  of  $V_h^{15}$  with the coordinates listed in Table IX. The axes of this description are the same as those of 1930, 352, p. 64.

As can be seen from Figure 322a and b the grouping consists of linked  $BO_3$  triangles and  $Be(O_3OH)$  tetrahedra. Each oxygen atom is shared by one B and two Be atoms; OH groups join two Be atoms.

TABLE IX. PARAMETERS OF THE ATOMS IN HAMBERGITE

Atom	No. per cell	x	y	z
Be(1)	8	-0.031	0.183	0.458
Be(2)	8	.236	.069	.458
B	8	.117	.103	-.028
O(1)	8	.031	.183	-.167
O(2)	8	.097	.103	.278
O(3)	8	.194	.037	-.167
OH	8	-.167	.183	.167

(cd) The copper atoms of the four molecules of  $CuCO_3 \cdot Cu(OH)_2$  lying in the unit cell of malachite are reported to be in two sets of general positions of  $C_{2h}^6$ : (e)  $\pm(xyz)$ ;  $\pm(\frac{1}{2}-x, y+\frac{1}{2}, \bar{z})$  with the parameters  $x=0$ ,  $y=0.208$ ,  $z=0.125$ ,  $x'=0.264$ ,  $y'=0.104$ ,  $z'=0.625$ . The other atomic parameters have not been found.

(ce) Some photographic data have been used in assigning a structure to northupite,  $Na_2Mg(CO_3)_2 \cdot NaCl$ . According to this arrangement the 16 molecules of its unit cube are in the following special and general positions of  $T_h^4$  (1930, 352, p. 125):

Mg: (16c)  $\frac{5}{8}\frac{5}{8}\frac{5}{8}$ ;  $\frac{5}{8}\frac{7}{8}\frac{7}{8}$ ;  $\frac{7}{8}\frac{5}{8}\frac{7}{8}$ ;  $\frac{7}{8}\frac{7}{8}\frac{5}{8}$  and 3 sets of similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}0\frac{1}{2}$  and  $\frac{1}{2}\frac{1}{2}0$

Cl: (16b)  $\frac{1}{8}\frac{1}{8}\frac{1}{8}$ ;  $\frac{1}{8}\frac{3}{8}\frac{3}{8}$ ;  $\frac{3}{8}\frac{1}{8}\frac{3}{8}$ ;  $\frac{3}{8}\frac{3}{8}\frac{1}{8}$  and 3 sets of similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}0\frac{1}{2}$  and  $\frac{1}{2}\frac{1}{2}0$

C: (32b)  $uu\bar{u}$ ;  $u\bar{u}u$ ;  $\bar{u}u\bar{u}$ ;  $\bar{u}\bar{u}u$ ;  $\frac{1}{4}-u$ ,  $\frac{1}{4}-u$ ,  $\frac{1}{4}-u$ ;  $\frac{1}{4}-u$ ,  $u+\frac{1}{4}$ ,  $u+\frac{1}{4}$ ;  $u+\frac{1}{4}$ ,  $\frac{1}{4}-u$ ,  $u+\frac{1}{4}$ ;  $u+\frac{1}{4}$ ,  $u+\frac{1}{4}$ ,  $\frac{1}{4}-u$  and similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}0\frac{1}{2}$  and  $\frac{1}{2}\frac{1}{2}0$  with  $u(C)=0.405$

Na: (48c)  $u00$ ;  $\bar{u}00$ ;  $0u0$ ;  $0\bar{u}0$ ;  $00u$ ;  $00\bar{u}$ ;  $\frac{1}{4}-u$ ,  $\frac{1}{4}$ ,  $\frac{1}{4}$ ;  $u+\frac{1}{4}$ ,  $\frac{1}{4}$ ,  $\frac{1}{4}$ ;  $\frac{1}{4}$ ,  $\frac{1}{4}-u$ ,  $\frac{1}{4}$ ;  $\frac{1}{4}$ ,  $u+\frac{1}{4}$ ,  $\frac{1}{4}$ ;  $\frac{1}{4}$ ,  $\frac{1}{4}$ ,  $\frac{1}{4}-u$ ;  $\frac{1}{4}$ ,  $\frac{1}{4}$ ,  $u+\frac{1}{4}$  and similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}0\frac{1}{2}$ ,  $\frac{1}{2}\frac{1}{2}0$  with  $u(Na)=0.225$

O: (g)  $xyz$ ;  $x\bar{y}\bar{z}$ ;  $\bar{x}y\bar{z}$ ;  $\bar{x}\bar{y}z$ ;  $zxy$ ;  $\bar{z}x\bar{y}$ ;  $\bar{z}\bar{x}y$ ;  $z\bar{x}\bar{y}$ ;  $yzx$ ;  $\bar{y}\bar{z}x$ ;  $y\bar{z}\bar{x}$ ;  $\bar{y}z\bar{x}$ ;  $\frac{1}{4}-x$ ,  $\frac{1}{4}-y$ ,  $\frac{1}{4}-z$ ;  $\frac{1}{4}-x$ ,  $y+\frac{1}{4}$ ,  $z+\frac{1}{4}$ ;  $x+\frac{1}{4}$ ,  $\frac{1}{4}-y$ ,  $z+\frac{1}{4}$ ;  $x+\frac{1}{4}$ ,  $y+\frac{1}{4}$ ,  $\frac{1}{4}-z$ ;  $\frac{1}{4}-z$ ,  $\frac{1}{4}-x$ ,  $\frac{1}{4}-y$ ;  $z+\frac{1}{4}$ ,  $\frac{1}{4}-x$ ,  $y+\frac{1}{4}$ ;  $z+\frac{1}{4}$ ,  $x+\frac{1}{4}$ ,  $\frac{1}{4}-y$ ;  $\frac{1}{4}-z$ ,  $x+\frac{1}{4}$ ,  $y+\frac{1}{4}$ ;  $\frac{1}{4}-y$ ,  $\frac{1}{4}-z$ ,  $\frac{1}{4}-x$ ;  $y+\frac{1}{4}$ ,  $z+\frac{1}{4}$ ,  $\frac{1}{4}-x$ ;  $\frac{1}{4}-y$ ,  $z+\frac{1}{4}$ ,  $x+\frac{1}{4}$ ;  $y+\frac{1}{4}$ ,  $\frac{1}{4}-z$ ,  $x+\frac{1}{4}$  and similar points about  $0\frac{1}{2}\frac{1}{2}$ ,  $\frac{1}{2}0\frac{1}{2}$ ,  $\frac{1}{2}\frac{1}{2}0$ . The chosen parameters are  $x=0.392$ ,  $y=0.348$ ,  $z=0.475$ .

The arrangement given to tychite,  $\text{Na}_4\text{Mg}_2(\text{CO}_3)_4 \cdot \text{Na}_2\text{SO}_4$ , is like this northupite grouping with  $\text{Na}_2\text{SO}_4$  in place of  $2\text{NaCl}$ . The eight sulfur atoms in the unit cube are at  $(8f) 000; \frac{1}{2}\frac{1}{2}0; \frac{1}{2}0\frac{1}{2}; 0\frac{1}{2}\frac{1}{2}; \frac{1}{4}\frac{1}{4}\frac{1}{4}; \frac{1}{4}\frac{3}{4}\frac{3}{4}; \frac{3}{4}\frac{1}{4}\frac{3}{4}; \frac{3}{4}\frac{3}{4}\frac{1}{4}$ . Sulfate oxygen atoms are in  $(32b) u'u'u'$ ; etc. with  $u'(O)=0.062$ . The other atoms are placed as in northupite: Mg in  $(16c)$ ; C in  $(32b)$  with  $u=0.400$ ; Na in  $(48c)$  with  $u=0.225$  and carbonate oxygens in  $(g)$  of  $T_h^4$  with  $x=0.375, y=0.352, z=0.473$ .

(*cf*) From spectral photographic data it has been found that the atoms in potassium trithionate,  $\text{K}_2\text{S}_3\text{O}_6$ , are in the following positions of  $V_h^{16}$ :

$$(c) \pm(uv\frac{1}{4}); \pm(\frac{1}{2}-u, v+\frac{1}{2}, \frac{3}{4}) \quad (d) \pm(xyz); \pm(x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z); \\ \pm(x, y, \frac{1}{2}-z); \pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$$

with the parameters of Table X. In this structure (Figure 322c) S-O within the same ion is ca 1.50 Å, S-S=2.19 Å and the angle joining the three sulfur atoms is  $103^\circ$ .

TABLE X. PARAMETERS OF THE ATOMS IN  $\text{K}_2\text{S}_3\text{O}_6$

Atom	No. per cell	Position	$x$	$y$	$z$
K(1)	4	(c)	0.131	0.089	0.25
K(2)	4	(c)	.180	-.236	.25
S(1)	4	(c)	.033	.319	.25
S(2)	4	(c)	.083	.472	.25
S(3)	4	(c)	.305	.472	.25
O(1)	4	(c)	-.125	.305	.25
O(2)	4	(c)	.347	.583	.25
O(3)	8	(d)	.083	.278	.042
O(4)	8	(d)	.361	.417	.042

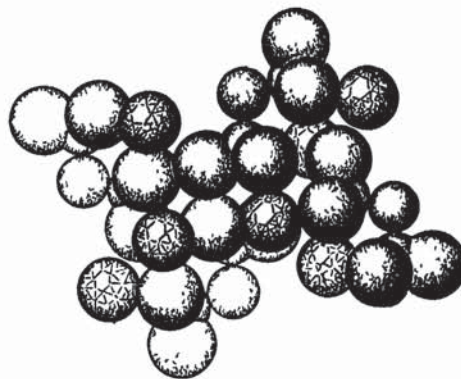
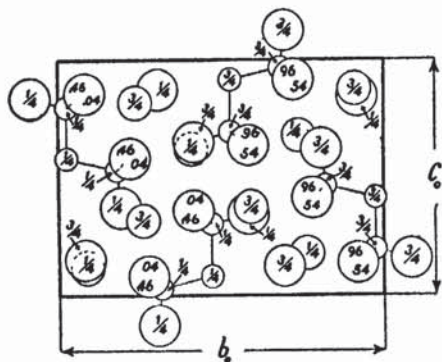


FIG. 322c.—(*left*) The structure found for  $\text{K}_2\text{S}_3\text{O}_6$  projected on the  $a$ -face of its orthorhombic cell. Largest circles are O atoms, intermediate circles K ions. Atoms of the  $\text{S}_3\text{O}_6$  ions are connected by light lines.

FIG. 322d.—(*right*) A packing drawing of  $c$ . The K ions are line-shaded. No significance is to be attached to the radii chosen for sulfur.

(*cg*) Estimated intensities on oscillation photographs have been used in choosing a structure for **potassium pyrosulfite**,  $K_2S_2O_5$ . The atoms in its two-molecule cell are placed in the following special and general positions of  $C_{2h}^2$ : (e)  $\pm(u\frac{1}{4}v)$ , (f)  $\pm(xyz)$ ;  $\pm(x, \frac{1}{2}-y, z)$  with the parameters of Table XI. The pyrosulfite ion resulting from this arrangement has the structure  $O_3S-SO_2$  (Figure 323) with a S-S distance of 2.18 Å.

(*ch*) It is natural to infer from the fractional number of molecules found in the unit of **pyroaurite** that either the formula, the cell size or

TABLE XI. PARAMETERS OF THE ATOMS IN  $K_2S_2O_5$ 

Atom	No. per cell	Position	$x$	$y$	$z$
K(1)	2	(e)	0.22	0.25	0.95
K(2)	2	(e)	.65	.25	.67
S(1)	2	(e)	.70	.25	.22
S(2)	2	(e)	.01	.25	.32
O(1)	4	(f)	.07	.06	.24
O(2)	4	(f)	.63	.06	.31
O(3)	2	(e)	.67	.25	.03

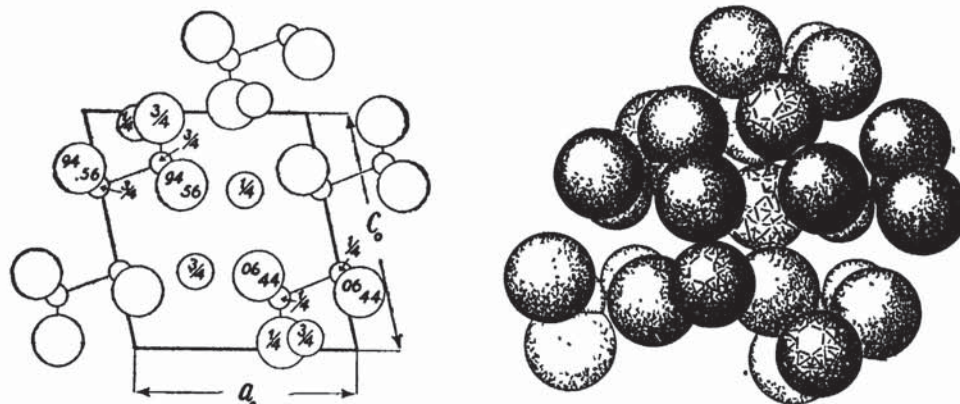


FIG. 323a.—(*left*) The monoclinic structure of  $K_2S_2O_5$  projected on its  $b$ -face. Oxygen atoms are represented by the largest, S by the smallest circles. The light lines joining them outline the  $S_2O_5$  ions.

FIG. 323b.—(*right*) A packing drawing of  $a$ . The size given the S atoms was determined by convenience only. K ions are line-shaded.

the density is in error. Existing data contain no evidence for a larger cell and the formula is said to be substantially correct.

(*ci*) A large number of the cubic or pseudo-cubic **voltaites** have been prepared and their unit cells measured (see Table XII). The composition of these sulfates is still uncertain though a recent study (1932, 171) places it as approximately  $(SO_4)_{12}Fe_4^{++}R_5^{++}R_2^+ \cdot 16-18H_2O$ .

(*cj*) A structure has been found for the complex sulfide **binnite**,  $(Cu, Fe)_{12}As_4S_{13}$ , which, except for the addition of two extra sulfur atoms and

TABLE XII. UNIT CUBE SIZE OF VOLTAITES

Voltaite	$a_0$	Voltaite	$a_0$
K-Cd compound	27.54	Rb-Cd compound	27.80
K-Fe <sup>++</sup>	27.33	Rb-Fe <sup>++</sup>	27.60
K-Mn <sup>++</sup>	27.25	Rb-Mn <sup>++</sup>	27.60
K-Zn	27.10	Rb-Zn	27.15
NH <sub>4</sub> -Cd	27.85	Tl-Cd	27.69
NH <sub>4</sub> -Fe <sup>++</sup>	27.35	Tl-Fe <sup>++</sup>	27.43
NH <sub>4</sub> -Mg	27.42	Tl-Mn <sup>++</sup>	27.71
NH <sub>4</sub> -Mn <sup>++</sup>	27.55	Tl-Zn	27.01

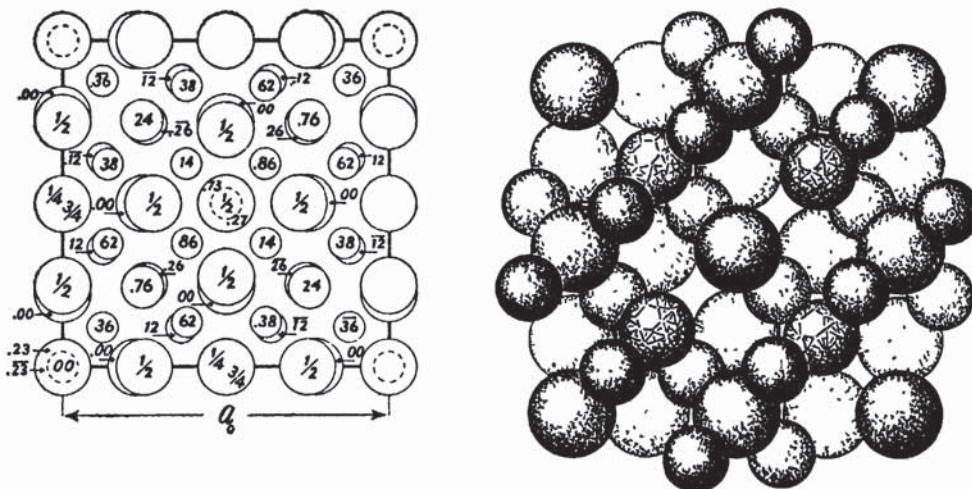
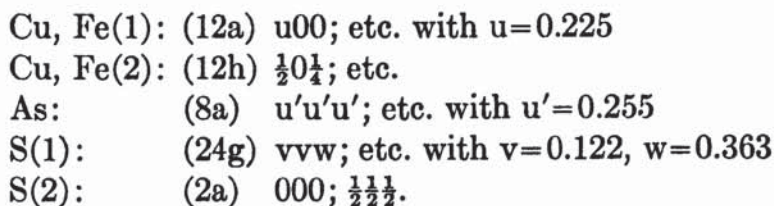


FIG. 324a.—(left) A cube face projection of the structure of binnite,  $(\text{Cu}, \text{Fe})_{12}\text{As}_4\text{S}_{12}$ . The largest circles are  $(\text{Cu}, \text{Fe})$ , the smallest are S atoms.

FIG. 324b.—(right) A packing drawing showing the bottom half of the unit cube of binnite. The atoms have their neutral radii; As atoms are line-shaded.

somewhat altered parameters, is identical with that previously determined for tetrahedrite,  $(\text{Cu}, \text{Ag})_3(\text{Sb}, \text{As})\text{S}_3$  [XV, (p), p. 280]. The binnite arrangement, as developed from photographic data, places the atoms of its two-molecule cube in positions (of  $T_d^3$ ):



The packing is that to be expected from neutral atoms. It is instructive to compare the atomic environments in this arrangement (Figure 324a and b) with those prevailing in enargite,  $\text{Cu}_3\text{AsS}_4$  [XVIA, (an)]. All the sulfurs in enargite and those [in (24g)] which are common to both binnite



and tetrahedrite have four metal atoms tetrahedrally placed about them. The two binnite sulfurs at 000 and  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  are enclosed by six octahedrally grouped (Cu, Fe) atoms. In enargite the copper and arsenic atoms are tetrahedrally surrounded but in binnite and tetrahedrite the metal environments are more complex. Arsenic atoms and the Cu, Fe(1) atoms in (12a) have only three neighboring sulfurs; four sulfurs, however, are to be found about Cu, Fe(2) in (12h).

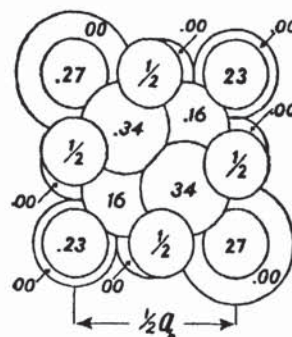
(ck) The mineral **boracite**, with eight molecules of the composition  $\text{Mg}_6\text{Cl}_2\text{B}_{14}\text{O}_{26}$  in its orthorhombic unit, has recently been assigned one of the space groups  $C_{2v}^{12}$  or  $C_{2v}^{14}$ . Previous studies had preferred  $C_{2v}^{11}$ .

Above  $265^\circ\text{C}$  this mineral is cubic with a four-molecule cell. Possible space groups and structures have been discussed but no definite conclusions have been reached.

(cl) It has been shown that the photographically observed powder lines of **sulphohalite**,  $2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$ , can be explained in terms of the following arrangement based on  $O_h^5$  (Figure 324c):

- F: (4b)  $000; \frac{1}{2}\frac{1}{2}0; \frac{1}{2}0\frac{1}{2}; 0\frac{1}{2}\frac{1}{2}$       Cl: (4c)  $\frac{1}{2}\frac{1}{2}\frac{1}{2}; 00\frac{1}{2}; 0\frac{1}{2}0; \frac{1}{2}00$   
 S: (8e)  $\pm(\frac{1}{4}\frac{1}{4}\frac{1}{4}); \pm(\frac{1}{4}\frac{3}{4}\frac{3}{4}); \pm(\frac{3}{4}\frac{1}{4}\frac{3}{4}); \pm(\frac{3}{4}\frac{3}{4}\frac{1}{4})$   
 Na: (24a)  $u'00$ ; etc. (see book, p. 303) with  $u'=0.226$   
 O: (32a)  $\pm(uuu); \pm(u\bar{u}\bar{u}); \pm(\bar{u}u\bar{u}); \pm(\bar{u}\bar{u}u)$  and 3 sets of similar points about  $0\frac{1}{2}\frac{1}{2}, \frac{1}{2}0\frac{1}{2}$  and  $\frac{1}{2}\frac{1}{2}0$  (1930, 352, p. 110) with  $u=0.164$ .

FIG. 324c.—A cube face projection of one eighth of the unit of sulphohalite,  $2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$ . The circles have the radii of their corresponding atoms; in order of decreasing size they refer to  $\text{Cl}^-$ , O,  $\text{F}^-$  and  $\text{Na}^+$ .



(cm) The sulfide mineral **tetradymite**,  $\text{Bi}_2\text{Te}_2\text{S}$ , has an elongated rhombohedral unit containing one molecule. From photographic data it has been found that atoms are in the following positions:

- S: 000      Bi:  $\pm(uuu)$  with  $u=0.392$       Te:  $\pm(vvv)$  with  $v=0.788$ .

This yields a structure which is a succession of layers of atoms of a kind normal to the three-fold axis. The shortest interatomic distances are: Bi-S=3.05 Å, Bi-Te=3.12 Å, Te-Te=3.69 Å.

## Chapter XIXA. Structures of the Silicates

Some of the work of the last three years has been devoted to simple silicates not yet analyzed, some to a revision of previous studies, but for the most part it has been concerned with more complex mineral types whose structures are still imperfectly understood. Most of these are either sheet structures, like the micas, or network groupings, such as the sodalite minerals and the zeolites. The zeolites are of especial interest because of the way they can be dehydrated and can reabsorb water without destruction of their atomic frameworks; approximate arrangements have been deduced for several which aid in understanding how this can occur. Real progress is also being made towards unraveling the crystal structures of the feldspars, one of the few important groups of the silicate minerals for which reasonable atomic arrangements have not been proposed.

### A, I. Simple Orthosilicates

(*ak*) A reinvestigation of the structure of **andalusite**,  $\text{Al}_2\text{SiO}_5$ , has led to slightly changed parameters in the X direction for the atoms O(3), O(4) and Al(2) (see Table IX, book, p. 333). The new arrangement, with  $x(\text{O}, 3)=0.11$ ,  $x(\text{O}, 4)=0.25$ ,  $x(\text{Al}, 2)=-0.125$ , agrees with the earlier one in giving aluminum atoms an oxygen coordination of five.

(*al*) A considerably altered grouping has been proposed for **sillimanite**,  $\text{Al}_2\text{SiO}_5$ . This structure yielding good agreement with the lines observed on a powder photograph differs from the previous one in having regular  $\text{SiO}_4$  tetrahedra. Atoms are in the same special positions of  $V_h^{16}$  as before (book, pp. 331 and 293) with the new parameters listed in Table II.

(*am*) A structure has been assigned to the **high** temperature cubic form of **carnegieite**,  $\text{NaAlSiO}_4$ , which, if its silicate and aluminate tetrahedra are considered equivalent, is closely related to that of high cristobalite. This arrangement gives calculated intensities agreeing with a powder photograph made at ca 760° C. The atoms in its four-molecule cube are in the following positions of  $T^4$  (book, p. 267):

TABLE I. THE CRYSTAL STRUCTURES OF THE SILICATES

Substance, symmetry and type	$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Acmite $\text{NaFe}(\text{SiO}_3)_2$	9.71	8.89	5.24	$C_{2h}^6$	2	1931, 464.
Afwillite $3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$	11.39	5.47 $\beta = 74^\circ 10'$	13.09	$C_2^2$	4	1932, 172.
Albite $\text{NaAlSi}_3\text{O}_8$	8.14	12.86 $\beta = 98^\circ 26'$	7.17	$C_1^1$	4	1934, 262.
Amphibole (var. Pargasite)	9.88	17.70 $\alpha = 94^\circ 3', \beta = 116^\circ 29', \gamma = 88^\circ 9'$ $\beta = \text{ca } 102^\circ$	10.92			1931, 139.
Analcite $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$	13.684					1931, 158, 242.
Andalusite $\text{Al}_2\text{SiO}_5$						1931, 204.
Apophyllite $\text{KF} \cdot \text{Ca}_4\text{Si}_8\text{O}_{20} \cdot 8\text{H}_2\text{O}$	9.00		15.8	$D_{4h}^6$	2	1931, 443.
Ardenneite $(\text{SiO}_4)_5\text{AsO}_4\text{Al}_3\text{Mn}(\text{AlOH})_2\text{Mn}_4 \cdot 2\text{H}_2\text{O} (?)$	18.56	5.83	8.72	$V_h^{13}$	2	1932, 175.
Ashcroftine $\text{NaK}(\text{Ca}, \text{Mg}, \text{Mn})\text{Al}_4\text{Si}_5\text{O}_{18} \cdot 8\text{H}_2\text{O}$	34.04		17.49		40	1933, 195.
Astrophyllite $[\text{Si}_2\text{O}_7]_2[(\text{Ti}, \text{Zr})(\text{OH}, \text{F})_2](\text{Fe}, \text{Mn})_4(\text{K}, \text{Na})_2$	11.70	5.40	21.10		4	1934, 91.
Axinite $\text{MgHCa}_2\text{BAI}_2(\text{SiO}_4)_4$	12.87	7.15	8.91		2	1932, 172.
Bavenite $9\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{BeO} \cdot 4\text{CaO} \cdot \text{H}_2\text{O}$	9.67	11.53	4.95	$V_h^1 (?)$	1	1933, 276.
Bertrandite $\text{H}_2\text{Be}_2\text{Si}_2\text{O}_9$	15.19	8.67	4.53	$C_{2v}^{12}$	4	1932, 232.

Substance, symmetry and type	$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Braunite $3\text{Mn}_2\text{O}_3 \cdot \text{MnSiO}_3$	9.50		18.98	$D_{4h}^{20}$	8	1931, 9.
Cancrinite $3(\text{Na}_2\text{Al}_2\text{O}_4 \cdot 2\text{SiO}_2) \cdot 2\text{CaCO}_3$	12.72	( <i>ab</i> , ( <i>bl</i> ))	5.18	$C_6^1$ (?)	1	1930, 457; 1932, 275; 1933, 269, 270.
$\alpha$ -Carnegieite $\text{NaAlSiO}_4$	7.37 at ca. 760° C	( <i>am</i> )		$T^4$	4	1932, 27.
Chabazite $\text{CaAl}_2\text{Si}_4\text{O}_{12} \cdot 6\text{H}_2\text{O}$	13.75	( <i>bf</i> )	14.94	$D_{3d}^5$	2	1931, 488; 1932, 286; 1933, 514.
Chlorites		( <i>v</i> ), ( <i>ax</i> )				
Cyanite $\text{Al}_2\text{SiO}_5$		( <i>h</i> )				1930, 423; 1934, 161. 1931, 204.
Dickite $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	5.14	8.94	14.42	$C_6^1$	4	1932, 184.
Edingtonite $\text{Be}_2\text{Al}_4\text{Si}_5\text{O}_{20} \cdot 8\text{H}_2\text{O}$	9.7	$\beta = 96^\circ 50'$	6.6	$V_3^d$	2	1933, 468; 1934, 108.
Enstatite (var. Bronzite)	18.16	8.84	5.19	$V_6^{15}$	16	1932, 436.
Epididymite $\text{NaBeSi}_3\text{O}_7(\text{OH})$	12.63	7.32	13.58	$V_6^{16}$	8	1934, 119.
Epidote $(\text{SiO}_4)_3\text{Al}_2\text{Ca}_2(\text{Al,Fe})\text{OH}$	16.39	5.63	10.20		4	1930, 111d; 1931, 74; 1932, 172.
Euclase $\text{HBeAlSiO}_5$	4.62	$\beta = 98^\circ 57'$ 14.24	4.75	$C_{2h}^5$	4	1933, 46.
Eudialyte		$\beta = 79^\circ 44'$				
Eulytite		( <i>af</i> )				1930, 105a, 193b.
$\text{B}_4\text{Si}_3\text{O}_{12}$	10.272	( <i>ao</i> )		$T_6^d$	4	1928, 253a; 1931, 299.
Feldspars		( <i>ac</i> ), ( <i>bm</i> )				
Tri.						1929, 289a, 381; 1931, 391; 1933, 467; 1934, 262.

Fuller's Earth $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$	Hex.	10.20	10.00	4	1932, 486.
Garnet	Cub.	(c), (bs)			1933, 182, 521, 522.
Gehlenite (synthetic)	Tet.	(n)	5.10	$V_3^d$	1930, 437, 438.
Haüynite $\text{Na}_3\text{Si}_6\text{Al}_6\text{O}_{24}(1-2, \text{SO}_4)$	Cub.	(aa), (bj)	9.11		1932, 23, 26; 1933, 30; 1934, 162.
Hemimorphite	Ort.	(ag), (ar)	8.38	$C_{2v}^{20}$	1932, 231.
$\text{Zn}_4(\text{OH})_2\text{Si}_2\text{O}_7 \cdot \text{H}_2\text{O}$	Mon.	(aj), (bi)	7.5438	$C_{2h}^4$	1932, 503; 1933, 514.
Heulandite	Mon.	(t)	17.85 $\beta = 88^\circ 34'$		1931, 139, 227; 1934, 128.
Hornblendes*	Mon.	(t)	9.88		1932, 351.
Joaquinite $\text{NaBa}(\text{Ti}, \text{Fe})_3\text{Si}_4\text{O}_{15}$	Ort.	(aj)	10.45	4	1931, 28.
Kaliophilite $\text{KAlSiO}_4$	Hex.	(aj)	27.0	54	1932, 183.
Kaolin $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	Mon.	(az)	5.14	4	1932, 183.
Lawsonite $\text{Ca}(\text{SiO}_3)_2 \cdot (\text{AlO}_2\text{H}_2)_2$	Ort.	(az)	8.90	$C_4^4$	1931, 135a.
Leucophanite $(\text{Ca}, \text{Na})_2\text{BeSi}_2(\text{O}, \text{OH}, \text{F})_7$	Ort.	(n)	7.38	4	1931, 493.
Lievrite $\text{CaFe}_2^{++}(\text{Fe}^{+++}\text{OH})(\text{SiO}_4)_2$	Ort.	(n)	8.85	$V_h^{17}$	1932, 172.
			13.22		
			9.96		
			5.86		
			13.07		
			8.82		
			13.07		
			5.86		

\*A Korean hornblende of the composition  $(\text{O}, \text{OH}, \text{F})_2(\text{Ca}, \text{Na}, \text{K})_2(\text{Mg}, \text{Fe}, \text{Mn}, \text{Fe}, \text{Ti})_4[\text{Si}, \text{Al}, \text{O}]_{11}$  has  $a_0 = 9.77 \text{ \AA}$ ,  $b_0 = 17.85 \text{ \AA}$ ,  $c_0 = 5.32 \text{ \AA}$ ,  $\beta = 73^\circ 58'$ .

Substance, symmetry and type		$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Lusakite	Ort.	7.86	16.62	5.65	$V_h^{17}$	1	1934, 246.
$H_2O \cdot 4(Fe, Co, Ni, Mg)O \cdot 9(Al, Fe)_2O_3 \cdot 8H_2O$							
Meliphanite	Tet. (n)	10.58		9.88		8	1931, 493.
Mesolite	Mon. (be)	56.7	6.54	18.44	$C_2^3$	8	1933, 194, 469.
$N_2C_2Al_6Si_9O_{30} \cdot 8H_2O$			$\beta = 90^\circ$				
Milarite	Hex.	10.46		13.90	$D_{6h}^2$	2	1930, 111b.
$HKC_{32}Al_2(Si_2O_5)_6$							
Montmorillonite	Ort. (az)	5.095	8.83	15.2			1933, 205.
$H_2O \cdot Al_2O_3 \cdot 4SiO_2$							
Muscovite	Mon. (v), (aw)	5.18	9.02	20.04	$C_{2h}^6$	4	1930, 153.
$KAl_2(AlSi_3)O_{10}(OH)_2$			$\beta = 95^\circ 30'$				
Nacrite	Mon. (az)	5.16	8.93	28.66	$C_6^4$	8	1933, 160.
$Al_2Si_2O_5(OH)_4$			$\beta = 91^\circ 43'$				
Narsarsukite	Tet.	10.78		7.99	$C_{4h}^4$	4	1932, 174.
$(Si_4O_{11})(Ti, FeF)Na_2$							
Natrolite	Ort. (y), (bc)						1931, 489; 1932, 189, 217; 1933, 469, 514.
$N_2Al_2Si_3O_{10} \cdot 2H_2O$							
Nephelite	Hex. (ad), (bp)						1931, 28.
$NaAlSiO_4$							
Noselite	Cub. (aa), (bj)	9.03				1	1932, 23, 26; 1934, 162.
$Na_8Al_6Si_6O_{24} \cdot SO_4$							
Pectolite	Tri. (s), (bn)	7.91	7.08	7.05		2	1931, 464.
$NaHC_2(SiO_3)_2$		$\alpha = 90^\circ$ , $\beta = 95^\circ 10'$ , $\gamma = 103^\circ 0'$					
Pollucite	Cub.	13.66				16	1932, 173.
$(CsAl, H_4)Si_2O_6$							
Prehnite	Ort.	4.65	5.52	18.53	$V_h^{17}$ or $C_{2v}^4$	2	1931, 135a.
$C_2(SiO_3)_2(AlOH)AlO_2H$							

Pyrophyllite $\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$	Mon.	( <i>ay</i> )	5.14	8.90 $\beta=99^\circ55'$	18.55	4	1934, 96, 200.
Pyrosmalite $\text{Si}_3\text{O}_7(\text{Mn}, \text{Fe})_3(\text{Mn}, \text{Fe})(\text{OH}, \text{Cl})_2$	Hex.		13.44		7.20	4	1931, 135.
Rinkite $(\text{SiO}_4)_4[\text{Ti}, \text{Ce}]\text{F}[\text{Ca}_2\text{Na}]$	Ort.	( <i>br</i> )	18.37	5.63	7.42	4	1933, 153; 1934, 90.
Sanidine $\text{KAlSi}_3\text{O}_8$	Mon.	( <i>bm</i> )	8.45	12.90 $\beta=116^\circ6'$	7.15	4	1934, 262.
Scapolite	Tet.	( <i>z</i> ), ( <i>bt</i> )					1931, 228; 1932, 402.
Scolecite $\text{CaAl}_2\text{Si}_3\text{O}_{10}\cdot3\text{H}_2\text{O}$	Mon. (?)	( <i>y</i> ), ( <i>bd</i> )	18.44	18.90 $\beta=\text{ca. } 90^\circ$	6.53		1930, 258; 1933, 469, 514.
Sillimanite $\text{Al}_2\text{O}_3\cdot\text{SiO}_2$	Ort.	( <i>f</i> ), ( <i>al</i> )	7.43	7.58	5.74	4	1931, 204.
Sodalite $\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}\text{Cl}$	Cub.	( <i>x</i> ), ( <i>bj</i> )	8.89			2	1932, 26.
Disodium Calcium Orthosilicate, $\text{Na}_2\text{CaSiO}_4$	Cub.	( <i>l</i> ), ( <i>an</i> )	7.49			4	1932, 28, 29.
Spodumene $\text{LiAl}(\text{SiO}_3)_2$	Mon.	( <i>r</i> ), ( <i>at</i> )	9.50	8.30 $\beta=69^\circ40'$	5.24	4	1931, 464.
Talc $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$	Mon.	( <i>ay</i> )	5.26	9.10 $\beta=100^\circ$	18.81	4	1934, 96.
Thaumasite $\text{CaCO}_3\cdot\text{CaSO}_4\cdot\text{CaSiO}_3\cdot15\text{H}_2\text{O}$	Hex.		10.90		10.29	2	1933, 6.
Thomsonite $\text{NaCa}_2\text{Al}_3\text{Si}_5\text{O}_{20}\cdot6\text{H}_2\text{O}$	Ort.	( <i>bh</i> )	13.04	13.06	13.22	4	1931, 489; 1932, 216; 1933, 469, 514.

Substance, symmetry and type	$a_0$	$b_0$	$c_0$	Space group	No. mol. per cell	References
Ultramarines Cub. ( <i>aa</i> ), ( <i>bj</i> )	15.63	11.83		$D_{4h}^1$	4	1930, 154a.
Vesuvianite Tet. ( <i>ah</i> ), ( <i>aq</i> )	10.80	10.26	7.26	$C_{2h}^2$ or $C_2^2$	4	1930, 193a; 1931, 465; 1932, 294; 1933, 461.
Wöhlerite ( $SiO_4$ ) <sub>2</sub> (ZrF,CbO)Ca <sub>2</sub> Na Mon.	7.88	7.27	7.03		6	1933, 152; 1934, 90.
Wollastonite Tri. ( <i>s</i> ), ( <i>bn</i> )	16.21	5.57	10.08		4	1931, 464.
CaSiO <sub>3</sub> Mon.	13.820	$\beta = 90^\circ$		$T_d^2$	4	1930, 111d; 1932, 172, 175.
Zoisite ( $SiO_4$ ) <sub>3</sub> · Al <sub>2</sub> Ca <sub>2</sub> (AlOH) Cub.						
Zunyite Al <sub>10</sub> Si <sub>5</sub> O <sub>30</sub> (OH,F) <sub>18</sub> Cl						

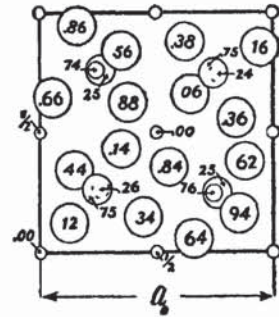


FIG. 325.—A cube face projection of the unit of the structure given to the high temperature ( $\alpha$ ) modification of carnegieite, NaAlSiO<sub>4</sub>. In order of decreasing size the circles refer to O, Na, Al and Si atoms.

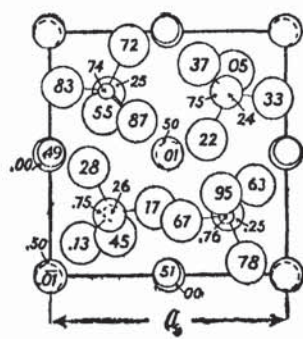


FIG. 326a.—A cube face projection of the atomic arrangement found for Na<sub>2</sub>CaSiO<sub>4</sub>. Oxygen atoms are shown as the largest circles, Na by slightly smaller ones. Light lines connecting smallest Si with O atoms outline SiO<sub>4</sub> tetrahedra.



FIG. 326b.—A packing drawing of *a*. Si atoms cannot be seen; the Ca ions are line-shaded.



TABLE II. PARAMETERS OF THE ATOMS IN SILLIMANITE

Atom	No. per cell	Position	x	y	z
O(1)	4	(c)	0.15	0.07	0.25
O(2)	4	(c)	-.15	-.07	.25
O(3)	4	(c)	.03	.47	.25
O(4)	8	(d)	-.11	.22	0
Si	4	(c)	.14	-.35	.25
Al(1)	4	(a)	0	0	0
Al(2)	4	(c)	-.14	.35	.25

Al: (4f)  $uuu$ ; etc. with  $u=0.258$       Si: (4f) with  $u=0$   
 Na: (4f) with  $u=0.744$       O(1): (4f) with  $u=0.125$   
 O(2):  $xyz$ ; etc. with  $x=0.658$ ,  $y=0.644$ ,  $z=0.055$ .

It will be noticed that this structure (Figure 325) would more closely resemble that of  $\text{Na}_2\text{CaSiO}_4$  (*an*) if the Si and Al positions were interchanged; such an alternative grouping would give indistinguishable X-ray effects.

(*an*) A further study of the cubic  $\text{Na}_2\text{CaSiO}_4$  has led to a structure that accounts well for powder photographic data and resembles  $\beta$ -cristobalite and  $\alpha$ -carnegieite (*am*). It differs from the previously suggested arrangement (book, p. 338) in an interchange of calcium and half the sodium atoms and in the parameters for oxygen lying in general positions. Its atoms (Figure 326) are in the following special positions of  $T^4$  (book, p. 267):

Si: (4f)  $uuu$ ; with  $u=0.258$       Ca: (4f) with  $u=-0.007$   
 Na(1): (4f) with  $u=0.750$       Na(2): (4f) with  $u=0.500$   
 O(1): (4f) with  $u=0.134$       O(2):  $xyz$ ; etc. with  $x=0.555$ ,  $y=0.667$ ,  
 $z=0.222$ .

(*ao*) The structure proposed for the cubic mineral **eulytite** yields discrete  $\text{SiO}_4$  tetrahedra. Its space group is  $T_d^2$  with bismuth atoms in positions:

(16f)  $uuu$ ;  $u, \bar{u}, \frac{1}{2}-u; \frac{1}{2}-u, u, \bar{u}; \bar{u}, \frac{1}{2}-u, u;$   
 $u+\frac{1}{4}, u+\frac{1}{4}, u+\frac{1}{4}; \frac{1}{4}-u, u+\frac{1}{4}, \frac{3}{4}-u; u+\frac{1}{4}, \frac{3}{4}-u, \frac{1}{4}-u; \frac{3}{4}-u, \frac{1}{4}-u, u+\frac{1}{4},$

and 8 similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ , with  $u=0.083$ .

If the other atoms are arranged as follows, a reasonable grouping is obtained that does not conflict with the powder data:

Si: (12k)  $\frac{3}{8}0\frac{1}{4}; \frac{1}{8}0\frac{3}{4}; \frac{3}{8}\frac{7}{8}; \frac{3}{8}0; 0\frac{1}{4}\frac{3}{8}; 0\frac{3}{8}\frac{1}{4}; \frac{7}{8}\frac{3}{4}; \frac{5}{8}\frac{1}{4}; \frac{1}{4}\frac{3}{8}; \frac{1}{8}\frac{5}{8}; \frac{1}{4}\frac{3}{8}; \frac{1}{4}\frac{5}{8}$

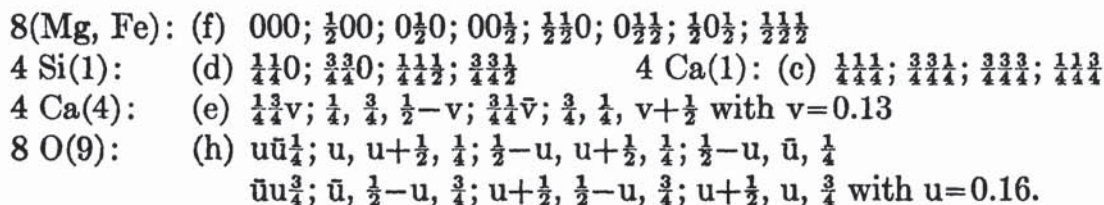
O:  $xyz$ ; etc. (1930, 352, p. 131) with the parameters  $x=-0.035$ ,  $y=0.125$ ,  $z=0.284$ . Other values,  $x=0.055$ ,  $y=0.11$ ,  $z=0.284$ , which cannot be excluded, are thought improbable.

(ap) A structure has been proposed for euclase,  $\text{HBeAlSiO}_5$ , which explains spectrometric measurements of the first twelve orders of reflection from the (010) face. This arrangement based on  $C_{2h}^6$ , instead of the previously chosen  $C_{2h}^2$ , has all its atoms in general positions: (e)  $\pm(xyz)$ ;  $\pm(x, \frac{1}{2}-y, z+\frac{1}{2})$  with the parameters of Table III. It is considered that the  $y$  parameters are essentially correct but that those along  $X$  and  $Z$  are only approximate.

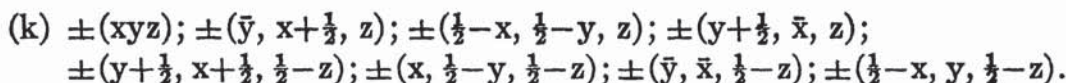
TABLE III. PARAMETERS CHOSEN FOR THE ATOMS OF EUCLASE

Atom	No. per cell	$x$	$y$	$z$
Be	4	0.50	-0.20	0.02
Al	4	.03	-.06	.25
Si	4	.47	.10	.15
O(1)	4	.22	.05	.39
O(2)	4	.26	-.03	-.17
O(3)	4	.54	.19	.37
O(4)	4	-.28	-.15	.11
OH	4	.22	-.17	.31

(aq) A series of spectrometric measurements have been used to give the mineral vesuvianite a grouping with four  $\text{Ca}_{10}\text{Al}_4(\text{Mg, Fe})_2\text{Si}_9\text{O}_{34}(\text{OH})_4$  molecules in its tetragonal unit. This arrangement, which somewhat resembles that found for the cubic garnets, has atoms in the following general and special positions of  $D_{4h}^4$ :



The remaining atoms are in the following general positions with parameters as listed in Table IV:



The coordinates used in this description are derived from those of 1930, 352, p. 91 by transferring the origin to such a point as  $(-\frac{3}{4}, -\frac{1}{4}, \frac{1}{4})$ .

It is uncertain how closely this structure describes the atomic arrangement that prevails in vesuvianite crystals. The chosen chemical formula differs from that previously given to the mineral and subsequent studies of the available chemical analyses are said to favor formulas departing appreciably from  $\text{Ca}_{10}\text{Al}_4(\text{Mg, Fe})_2\text{Si}_9\text{O}_{34}(\text{OH})_4$ . One of these (1932, 294) is  $\text{X}_{19}\text{Y}_{13}\text{Si}_{18}(\text{O, OH, F})_{36}$  where  $\text{X}=\text{Ca}(\text{Na, etc.})$  and  $\text{Y}=(\text{Al, Fe, Mg, etc.})$ ; another (1933, 461) is  $\text{Ca}_8\text{Al}_4(\text{Si, Al})_9(\text{Fe, Mg, etc.})_4(\text{O, OH, F})_{38}$ .

TABLE IV. PARAMETERS FOR ATOMS OF VESUVIANITE LYING IN GENERAL POSITIONS

Atom	No. per cell	$x$	$y$	$z$
Si(2)	16	0.19	0.05	0.87
Si(3)	16	.09	-.17	.37
Ca(2)	16	.19	.05	.36
Ca(3)	16	.09	-.17	.88
Al	16	.11	.11	.13
O(1)	16	.22	.17	.08
O(2)	16	.13	.16	.28
O(3)	16	.06	.22	.08
O(4)	16	.07	.13	.48
O(5)	16	.17	.01	.18
O(6)	16	.01	.06	.17
O(7)	16	-.05	.18	.32
O(8)	16	.10	-.08	.07
OH	16	.13	-.25	.06

## A, II. Complex Silicate Groups

(ar) The structure found for **hemimorphite**,  $\text{H}_2\text{Zn}_2\text{SiO}_5$ , indicates that it is a pyrosilicate with a formula best written as  $\text{Zn}_4(\text{OH})_2\text{Si}_2\text{O}_7 \cdot \text{H}_2\text{O}$ . Atoms are in the following positions of  $C_{2v}^{20}$ :

- 2 O(1): (a)  $00u; \frac{1}{2}, \frac{1}{2}, u + \frac{1}{2}$  with  $u=0$   
 2  $\text{H}_2\text{O}$ : (b)  $\frac{1}{2}0u; 0, \frac{1}{2}, u + \frac{1}{2}$  with  $u=-0.150$   
 4 OH: (c)  $u0v; \bar{u}0v; u + \frac{1}{2}, \frac{1}{2}, v + \frac{1}{2}; \frac{1}{2} - u, \frac{1}{2}, v + \frac{1}{2}$  with  $u=0.75, v=0.350$   
 4 O(3): (d)  $0uv; 0\bar{u}v; \frac{1}{2}, u + \frac{1}{2}, v + \frac{1}{2}; \frac{1}{2}, \frac{1}{2} - u, v + \frac{1}{2}$  with  $u=0.187, v=0.305$   
 4 Si: (d)  $0u'v'$ ; etc. with  $u'=0.160, v'=0$   
 8 Zn: (e)  $xyz; \bar{x}\bar{y}z; x\bar{y}z; \bar{x}yz$ , and four similar points about  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  (1930, 352, p. 56) with  $x=0.300, y=0.342, z=0.010$ .  
 8 O(2): (e)  $x'y'z'$ ; etc. with  $x'=0.161, y'=0.187, z'=0.850$ .

In this arrangement a zinc atom is surrounded by three oxygen atoms and one OH group (Figure 327); the water molecules, bounded by OH groups and oxygen atoms lie loosely in big holes that exist in the structure (minimum  $\text{H}_2\text{O}$  to OH or O = ca 3.3 Å).

(as) An arrangement has been described for the mineral **bertrandite**,  $\text{Be}_2(\text{BeOH})_2\text{SiO}_3\text{SiO}_4$ , using spectrometric and photographic data. Based on a hexagonal close packing of oxygen atoms it is said to be intermediate between a silicate chain structure and one having discrete silicate groups. Half the silicon atoms are centers of isolated  $\text{SiO}_4$  tetrahedra; the other half form parts of tetrahedra which are linked together in endless  $\text{SiO}_3$  chains such as those of diopside. Atoms are placed in special and general positions of  $C_{2v}^{12}$ :

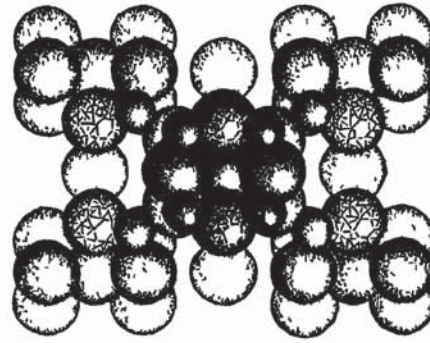
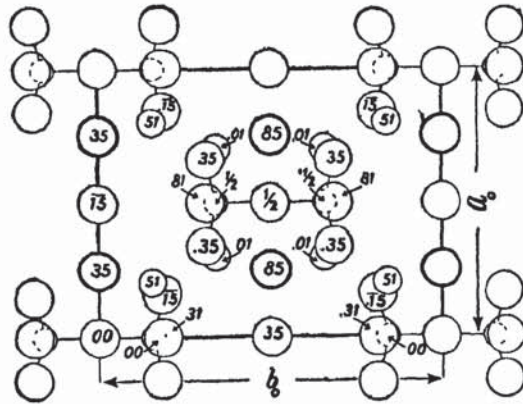


FIG. 327a.—(left) The structure assigned to hemimorphite,  $H_2Zn_2SiO_5$ , as projected upon the  $c$ -face of its orthorhombic unit. The largest circles represent O atoms,  $H_2O$  groups and (OH) radicals. The silicate O atoms are joined by light lines with Si (the smallest circles) to form  $Si_2O_7$  groups; the heavily ringed circles are (OH).

FIG. 327b.—(right) A packing drawing of  $a$ . The (OH) radicals are line-shaded; the Si atoms cannot be seen.

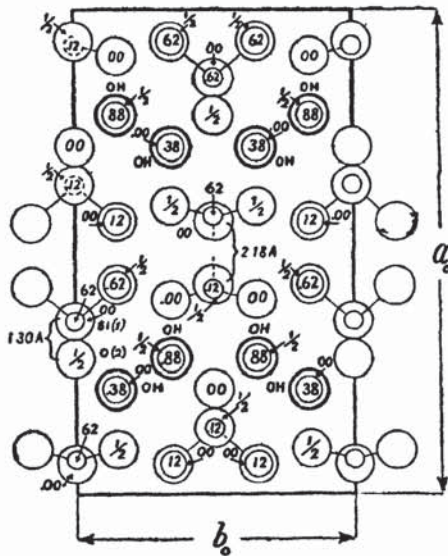


FIG. 328.—The structure proposed for bertrandite,  $Be_2(BeOH)_2Si_2O_7$ , as projected on the  $c$ -face of its orthorhombic unit. The smallest circles are Si, the largest are O atoms [or (OH) if heavily ringed].

TABLE V. PARAMETERS OF THE ATOMS IN BERTRANDITE

Atom	No. per cell	Position	$x$	$y$	$z$
Be(1)	8	(b)	0.215	0.155	0.375
Be(2)	8	(b)	.435	.155	.625
Si(1)	4	(a)	.074	0	.625
Si(2)	4	(a)	.360	0	.625
OH	8	(b)	.215	.155	0
O(1)	4	(a)	.065	0	0
O(2)	4	(a)	.283	0	.50
O(3)	4	(a)	.360	0	0
O(4)	8	(b)	.095	.155	.50
O(5)	8	(b)	.435	.155	.50

- (a)  $u0v$ ;  $\bar{u}$ , 0,  $v+\frac{1}{2}$ ;  $u+\frac{1}{2}$ ,  $\frac{1}{2}$ ,  $v$ ;  $\frac{1}{2}-u$ ,  $\frac{1}{2}$ ,  $v+\frac{1}{2}$   
 (b)  $xyz$ ;  $\bar{x}$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ ;  $x\bar{y}z$ ;  $\bar{x}$ ,  $y$ ,  $z+\frac{1}{2}$  and four similar points about  $\frac{1}{2}\frac{1}{2}0$ .

The atomic parameters are reproduced in Table V.

As can be seen from Figure 328 this grouping yields several improbable interatomic distances. Atoms Be(2) and O(5) are only 0.57 Å apart. Both kinds of silicate groups are distorted. Thus Si(2)-O(2) is 1.30 Å while the distance from Si(1) to one of its four surrounding oxygen atoms is 2.18 Å.

### B. Silicate Chain Structures

(*at*) The structure found for **acmite**,  $\text{NaFe}(\text{SiO}_3)_2$ , is identical with that of diopside (*r*) both in cell dimensions and in atomic parameters, with sodium in place of calcium and ferric iron replacing magnesium.

Spodumene,  $\text{LiAl}(\text{SiO}_3)_2$ , has the same arrangement but in a cell of appreciably different size and shape. The atomic parameters, with lithium and aluminum in place of calcium and magnesium, have been given approximate values close to those for diopside (see Table VI).

TABLE VI. PARAMETERS OF THE ATOMS IN SPODUMENE

Atom	No. per cell	Position	<i>x</i>	<i>y</i>	<i>z</i>
Li	4	(e)	0	-0.31	0.25
Al	4	(e)	0	.09	.25
Si	8	(f)	0.21	.41	.25
O(1)	8	(f)	.39	.41	.14
O(2)	8	(f)	.13	.25	.32
O(3)	8	(f)	.14	.49	0

(*au*) Photographic spectral data have been used to assign to a **bronzite**,  $(\text{Fe, Mg})\text{SiO}_3$ , parameters that agree well with those previously found for enstatite (book, p. 344).

(*av*) A structure for **epididymite**,  $\text{HNaBeSi}_3\text{O}_8$ , has been deduced from spectrometric measurements. It places atoms in special and general positions of  $V_h^{16}$  with the parameters listed in Table VII:

- (a)  $000$ ;  $00\frac{1}{2}$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$   
 (b)  $0\frac{1}{2}0$ ;  $0\frac{1}{2}\frac{1}{2}$ ;  $\frac{1}{2}00$ ;  $\frac{1}{2}0\frac{1}{2}$   
 (c)  $\pm(uv\frac{1}{4})$ ;  $\pm(u+\frac{1}{2}, \frac{1}{2}-v, \frac{1}{4})$   
 (d)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}-z)$ ;  
 $\pm(\bar{x}, \bar{y}, z+\frac{1}{2})$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$ .

The axes of this description differ from those used for chrysoberyl (book, p. 293) by an interchange of a and b.

This structure must be revised since some of its interatomic distances are impossible; for example Be-Si(1)=0.54 Å, Be-O(3)=0.72 Å, Si(1)-O(3)=1.13 Å (see Table VII).

TABLE VII. PARAMETERS OF THE ATOMS IN EPIDIDYMIT

Atom	No. per cell	Position	$x$	$y$	$z$
Na(1)	4	(a)	0	0	0
Na(2)	4	(b)	0	$\frac{1}{2}$	0
Be	8	(d)	0.035	0.250	0.052
Si(1)	8	(d)	.065	.200	.060
Si(2)	8	(d)	.435	.200	-.060
Si(3)	8	(d)	.335	.250	.000
OH(1)	4	(c)	-.155	.150	$\frac{1}{4}$
OH(2)	4	(c)	.041	.250	$\frac{1}{4}$
O(1)	8	(d)	.040	.000	.145
O(2)	8	(d)	.040	.500	.145
O(3)	8	(d)	.000	.250	.010
O(4)	8	(d)	.167	.000	-.040
O(5)	8	(d)	.167	.500	-.040
O(6)	8	(d)	.182	.250	.130
O(7)	8	(d)	.318	.250	-.130

### C. Two-dimensional Silicate Nets

(aw) The atomic arrangement which has been given to the mica **muscovite**,  $\text{KAl}_2(\text{AlSi}_3)\text{O}_{10}(\text{OH})_2$ , through a study of photographic and spectrometric intensities is based on  $C_{2h}^6$  (book, p. 343). Approximate parameters of its atoms, all of which except potassium are in general positions, are listed in Table VIII. The central atoms of one quarter of the tetrahedra linked together to produce sheets are aluminum instead of silicon.

(ax) Six **chlorites** with the approximate composition  $\text{Al}_2\text{Mg}_5\text{Si}_3\text{O}_{10}(\text{OH})_8$  possess four-molecule monoclinic units having dimensions within the limits  $a_0 = 5.304\text{--}5.352$  A,  $b_0 = 9.187\text{--}9.270$  A,  $c_0 = 28.306\text{--}28.582$  A,  $\beta = 97^\circ 9'$ . Their space group is considered to be either  $C_{2h}^3$  or  $C_{2h}^6$ . Adopting earlier suggestions that the brittle micas are built up of alternate mica- and brucite-like layers, structures have been assumed and compared with intensities on powder photographs. A grouping with the symmetry of  $C_{2h}^6$  has been preferred but it is obvious that at least until the arrangements based on  $C_{2h}^3$  are definitely eliminated the chlorite structure has not been established.

(ay) Taking the data from powder photographs practically identical structures have been proposed for a **talc**,  $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ , and for **pyrophyllite**,  $\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$ . The distribution within layers is that previously suggested but the layers are said to be stacked according to the requirements of  $C_{2h}^6$ , not of  $C_{2h}^3$ . Parameters have been proposed but additional confirmation and a more precise description of the arrangement is desirable.

(az) The **clay minerals** with their pronounced micaceous cleavage should have silicon-oxygen tetrahedra united to form sheets. Structures built up of such sheets have been proposed for kaolin, dickite and nacrite,

TABLE VIII. PARAMETERS OF THE ATOMS IN MUSCOVITE

Atom	No. per cell	Position	<i>x</i>	<i>y</i>	<i>z</i>
OH	8	(f)	0.062	0.083	0.055
O(1)	8	(f)	.062	.417	.055
O(2)	8	(f)	.062	.250	.055
O(3)	8	(f)	.478	.083	.164
O(4)	8	(f)	.228	.166	.164
O(5)	8	(f)	.228	.332	.164
(Si+Al)(1)	8	(f)	.033	.417	.135
(Si+Al)(2)	8	(f)	.033	.250	.135
Al	8	(f)	.250	.083	0
K	4	(e)	0	.083	.250

each with the composition  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ . These groupings, based of necessity on powder photographic data and not conclusively proved, are similar; they differ mainly in the orientation of their silicate layers. Each is developed from  $\text{C}_s^4$  with all atoms in general positions: (a)  $xyz$ ;  $x+\frac{1}{2}$ ,  $y+\frac{1}{2}$ ,  $z$ ;  $x$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ ;  $x+\frac{1}{2}$ ,  $\frac{1}{2}-y$ ,  $z+\frac{1}{2}$ . Kaolin and dickite have four-molecule cells; the unit of nacrite is twice as big. The atomic parameters are listed in Tables IX and X. Other arrangements for both kaolin and dickite are in almost equally good agreement with the data. It is suggested that some samples may have these alternative groupings—or one which combines both. The mineral anauxite resembles kaolin but contains a greater amount of silicon. If this silicon replaces aluminum, as has been proposed, it would have a hitherto unknown six-fold coordination.

Some measurements have indicated that the substance called montmorillonite has an orthorhombic cell similar in size and shape to the monoclinic cell of kaolin (1933, 205).

(*ba*) Apophyllite is a mineral which is sometimes considered a zeolite, sometimes more nearly a mica. Some water can be driven from it without

TABLE IX. ATOMIC PARAMETERS FOR KAOLIN AND DICKITE

Atom	Kaolin			Dickite		
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>
Al(1)	0.25	-0.17	0	0.25	0.41	0
Al(2)	.25	.17	0	.25	.08	0
Si(1)	.01	.50	0.19	.14	.25	0.19
Si(2)	.01	.17	.19	-.36	.08	.19
O(1)	-.05	.50	.08	.11	.25	.08
O(2)	-.05	.17	.08	-.39	.08	.08
O(3)	.03	.33	.23	.16	.42	.23
O(4)	.28	-.41	.23	.41	.17	.23
O(5)	.28	.08	.23	-.09	.17	.23
OH(1)	-.05	-.17	.08	-.39	.42	.08
OH(2)	-.05	0	.42	-.11	.25	.42
OH(3)	-.45	.17	.42	.39	.08	.42
OH(4)	.05	.33	.42	.39	.42	.42

TABLE X. ATOMIC PARAMETERS FOR NACRITE

Atom	x	y	z	Atom	x	y	z
Al(1)	0.75	0.33	0	O(6)	-0.08	0.50	0.29
Al(2)	.75	0	0	O(7)	-.08	.17	.29
Al(3)	.08	0	0.25	O(8)	.44	-.17	.36
Al(4)	.08	-.33	.25	O(9)	.19	-.42	.36
Si(1)	.43	.33	.09	O(10)	.19	.08	.36
Si(2)	.43	0	.09	OH(1)	-.08	.17	.04
Si(3)	.43	-.33	.34	OH(2)	-.26	-.33	.21
Si(4)	.43	0	.34	OH(3)	-.26	0	.21
O(1)	-.08	.50	.04	OH(4)	-.26	.33	.21
O(2)	-.08	-.17	.04	OH(5)	-.08	-.17	.29
O(3)	.44	.17	.11	OH(6)	.08	-.33	.46
O(4)	.19	.42	.11	OH(7)	.08	0	.46
O(5)	.19	-.08	.11	OH(8)	.08	.33	.46

loss of structure but this is more difficult to do than with the true zeolites and the lost water is not readily reabsorbed. The arrangement given it from a study of rotation photographs and some spectrometric measurements is mica-like and contains hydroxyl groups rather than H<sub>2</sub>O molecules. Its two molecules of composition Ca<sub>4</sub>Si<sub>8</sub>O<sub>20</sub>·8H<sub>2</sub>O·KF are in the following positions (1930, 352, p. 92) of D<sub>4h</sub><sup>6</sup>:

2 F: (a) 000;  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$                                       2 K: (b)  $00\frac{1}{2}; \frac{1}{2}\frac{1}{2}0$   
 8 Ca: (h)  $uv0; v\bar{u}0; v+\frac{1}{2}, u+\frac{1}{2}, \frac{1}{2}; u+\frac{1}{2}, \frac{1}{2}-v, \frac{1}{2}; \bar{u}\bar{v}0; \bar{v}u0; \frac{1}{2}-v, \frac{1}{2}-u, \frac{1}{2};$   
 $\frac{1}{2}-u, v+\frac{1}{2}, \frac{1}{2}$  with  $u=0.120, v=0.243$   
 8 O(1): (g)  $u, u+\frac{1}{2}, \frac{1}{4}; u+\frac{1}{2}, \bar{u}, \frac{1}{4}; \bar{u}, \frac{1}{2}-u, \frac{1}{4}; \frac{1}{2}-u, u, \frac{1}{4}; u, u+\frac{1}{2}, \frac{3}{4};$   
 $u+\frac{1}{2}, \bar{u}, \frac{3}{4}; \bar{u}, \frac{1}{2}-u, \frac{3}{4}; \frac{1}{2}-u, u, \frac{3}{4}$  with  $u=0.362$   
 16 O(2): (i)  $\pm(xyz); \pm(y\bar{x}z); \pm(\bar{x}\bar{y}z); \pm(\bar{y}xz); \pm(\frac{1}{2}-x, y+\frac{1}{2}, z+\frac{1}{2});$   
 $\pm(\frac{1}{2}-y, \frac{1}{2}-x, z+\frac{1}{2}); \pm(x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}); \pm(y+\frac{1}{2}, x+\frac{1}{2}, z+\frac{1}{2})$   
 with  $x=0.089, y=0.184, z=0.217$   
 16 O(3): (i)  $x_1y_1z_1$ ; etc. with  $x_1=0.287, y_1=0.117, z_1=0.094$   
 16 O(4): (i)  $x_2y_2z_2$ ; etc. with  $x_2=0.237, y_2=0.445, z_2=0.094$   
 16 Si: (i)  $x'y'z'$ ; etc. with  $x'=0.237, y'=0.091, z'=0.188$ .

#### D. Three-dimensional Silicate Nets

The zeolites have the surprising property of being able to lose and to reabsorb water without destruction of their underlying atomic arrangements. Previous studies [see (w)] of analcite, NaAlSi<sub>2</sub>O<sub>6</sub>·H<sub>2</sub>O, have indicated that this zeolite consists of a three-dimensional network of interlocking AlO<sub>4</sub> and SiO<sub>4</sub> tetrahedra with metal atoms and water molecules occupying holes in the framework thus formed. Measurements on several other zeolites have now yielded similar results.

(bb) A recent investigation of analcite indicates that it is not cubic but tetragonal with D<sub>4h</sub><sup>20</sup> as the correct space group. It has also been



shown that dehydration does not seriously alter the observed powder pattern of this zeolite.

(bc) The structure found for natrolite from photographic spectral data has the symmetry of  $C_{2v}^{19}$  and contains eight molecules of the composition  $Na_2Al_2Si_3O_{10} \cdot 2H_2O$ . All atoms except one set of silicon are in general positions (1930, 352, p. 55):

(b)  $xyz$ ;  $\bar{x}\bar{y}z$ ;  $x+\frac{1}{4}$ ,  $\frac{1}{4}-y$ ,  $z+\frac{1}{4}$ ;  $\frac{1}{4}-x$ ,  $y+\frac{1}{4}$ ,  $z+\frac{1}{4}$  and three sets of similar points about  $\frac{1}{2}\frac{1}{2}0$ ,  $\frac{1}{2}0\frac{1}{2}$ ,  $0\frac{1}{2}\frac{1}{2}$ .

The parameters of these atoms are listed in Table XI. Si(1) atoms are in special positions (a)  $00u$ ; etc. with  $u=0$ ; i.e. at the points  $000$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$ ;  $\frac{1}{4}\frac{1}{4}\frac{1}{4}$ ;  $\frac{3}{4}\frac{3}{4}\frac{1}{4}$ ;  $\frac{3}{4}\frac{1}{4}\frac{3}{4}$ ;  $\frac{1}{4}\frac{3}{4}\frac{3}{4}$ . The nature of the tetrahedral network and the positions occupied by water molecules can be seen from Figure 329.

Besides closely agreeing determinations of cell dimensions on natural natrolites from several sources, cell data exist on four synthetic compounds of this type (Table XII).

TABLE XI. PARAMETERS FOR ATOMS OF NATROLITE LYING IN GENERAL POSITIONS

Atom	No. per cell	$x$	$y$	$z$
Na	16	0.222	0.028	0.625
Al	16	.036	.089	.625
Si(2)	16	.153	.208	.625
O(1)	16	.069	.180	.625
O(2)	16	.014	.067	.875
O(3)	16	.183	.236	.375
O(4)	16	.097	.042	.500
O(5)	16	.208	.153	.750
H <sub>2</sub> O	16	.069	.180	.125

FIG. 329.—A projection upon its  $c$ -face of one eighth of the unit of natrolite,  $Na_2Al_2Si_3O_{10} \cdot 2H_2O$ . The small light circles are Si, the ringed small circles are Al atoms. Na atoms are shown by circles of intermediate size; the largest ringed circles are H<sub>2</sub>O molecules.

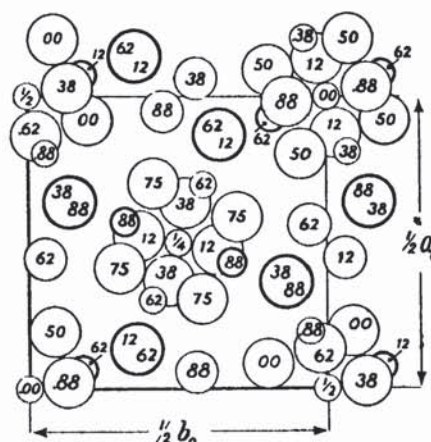


TABLE XII. CELL DIMENSIONS ON SUBSTITUTED NATROLITES

Compound	$a_0$	$b_0$	$c_0$
$Na_2Al_2Si_3O_{10} \cdot 2H_2O$	18.3	18.6	6.57
$Li_2Al_2Si_3O_{10} \cdot 2H_2O$	18.0	18.6	6.5
$Ag_2Al_2Si_3O_{10} \cdot 2H_2O$	18.6	18.9	6.6
$(NH_4)_2Al_2Si_3O_{10}$ anhydrous	17.9	18.4	6.6

(bd) The X-ray patterns of **scolecite**,  $\text{CaAl}_2\text{Si}_3\text{O}_{10}\cdot 3\text{H}_2\text{O}$ , are indistinguishable from those of natrolite so that it must have a unit of practically the same size and shape. Earlier it was suggested (1930, 258) that  $\text{Ca}+3\text{H}_2\text{O}$  of scolecite replace  $2\text{Na}+2\text{H}_2\text{O}$  of natrolite. A recent study proposes instead that Ca atoms in the former occupy half the Na positions in natrolite leaving the other half vacant and that the eight new  $\text{H}_2\text{O}$  molecules go into half the 16-fold unoccupied holes in the natrolite structure with the approximate parameters  $x=0.22$ ,  $y=0.028$ ,  $z=0.125$ . No data are available for distinguishing between these possibilities.

(be) **Mesolite**,  $\text{Na}_2\text{Ca}_2\text{Al}_6\text{Si}_9\text{O}_{30}\cdot 8\text{H}_2\text{O}$ , is another zeolite which gives a pattern nearly identical with that of natrolite. The large cell of Table I is indicated by certain faint lines seen on some rotation photographs.

(bf) Data from spectral photographs have been used to assign an atomic arrangement to the rhombohedral zeolite **chabazite**. The atoms of the two  $\text{CaAl}_2\text{Si}_4\text{O}_{12}\cdot 6\text{H}_2\text{O}$  molecules are in the following positions of  $D_{3d}^6$ :

- 2 Ca: (c)  $\pm(\text{uuu})$  with  $u=0.17$   
 6 O(1): (f)  $\pm(\text{u}\bar{u}0)$ ;  $\pm(\bar{u}0\text{u})$ ;  $\pm(0\text{u}\bar{u})$  with  $u=0.34$   
 6 O(2): (g)  $\pm(\text{u}\bar{u}\frac{1}{2})$ ;  $\pm(\bar{u}\frac{1}{2}\text{u})$ ;  $\pm(\frac{1}{2}\text{u}\bar{u})$  with  $u=0.14$   
 6 O(3): (h)  $\pm(\text{uuv})$ ;  $\pm(\text{uvu})$ ;  $\pm(\text{vuu})$  with  $u=0.35$ ,  $v=-0.02$   
 6 O(4): (h)  $\text{u}'\text{u}'\text{v}'$  with  $u'=0.13$ ,  $v'=0.50$   
 $\text{H}_2\text{O}(1)$ : (h)  $\text{u}_1\text{u}_1\text{v}_1$  with  $u_1=0.31$ ,  $v_1=0.70$   
 $\text{H}_2\text{O}(2)$ : (h)  $\text{u}_2\text{u}_2\text{v}_2$  with  $u_2=0.14$ ,  $v_2=-0.08$   
 (Si+Al): (i)  $\pm(\text{xyz})$ ;  $\pm(\text{yzx})$ ;  $\pm(\text{zxy})$ ;  $\pm(\text{yxz})$ ;  $\pm(\text{xzy})$ ;  $\pm(\text{zyx})$

with  $x=0.23$ ,  $y=0.44$ ,  $z=-0.01$ .

X-ray studies have been made of the dehydration of chabazite. It has also been shown that mercury can replace the water in this zeolite without destroying its diffraction pattern; by one investigator (1932, 286) this has been taken to mean that the water molecules do not occupy definite positions within the chabazite structure.

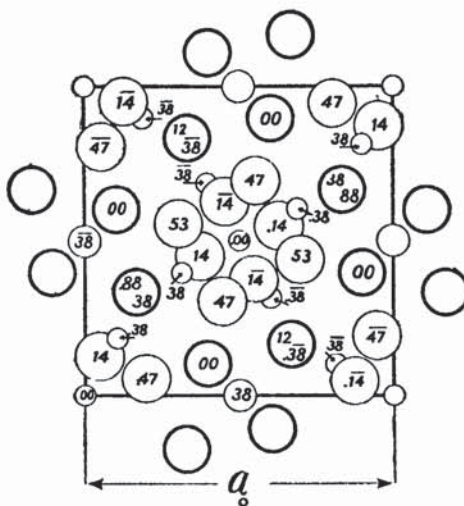
(bg) Another zeolite to which an atomic arrangement has been assigned is **edingtonite**,  $\text{Ba}_2\text{Al}_4\text{Si}_6\text{O}_{20}\cdot 8\text{H}_2\text{O}$ . There is some uncertainty as to the true symmetry of this mineral. Samples of Swedish origin have been found to be orthorhombic with  $a_o=9.56$  A,  $b_o=9.68$  A,  $c_o=6.53$  A; their space group has been reported as  $V^3$ . Material from Old Kilpatrick, Scotland with the cell dimensions of Table I is said to be completely tetragonal.

The Laue photographs of edingtonite from Böhlet, Sweden are apparently tetragonal. Proceeding on the assumption that its atomic arrangement does not depart significantly from this higher symmetry, photographic spectral data have been used to place its two molecules in the following special and general positions of  $V_d^3$  (1930, 352, p. 74):

Ba:	(c) $0\frac{1}{2}u$ ; $\frac{1}{2}0\bar{u}$ with $u=0.375$	(Al+Si)(1): (a) $000$ ; $\frac{1}{2}\frac{1}{2}0$
H <sub>2</sub> O(1):	(e) $u$ , $\frac{1}{2}-u$ , $v$ ; $\frac{1}{2}-u$ , $\bar{u}$ , $\bar{v}$ ; $\bar{u}$ , $u+\frac{1}{2}$ , $v$ ; $u+\frac{1}{2}$ , $u$ , $\bar{v}$ with $u=0.333$ , $v=0.875$	
H <sub>2</sub> O(2):	(e) $u'$ , $\frac{1}{2}-u'$ , $v'$ with $u'=0.103$ , $v'=0$	
O(1):	(e) $u_1$ , $\frac{1}{2}-u_1$ , $v_1$ with $u_1=0.333$ , $v_1=0.375$	
O(2):	(f) $xyz$ ; $y\bar{x}\bar{z}$ ; $\bar{x}\bar{y}z$ ; $\bar{y}x\bar{z}$ ; $\frac{1}{2}-x$ , $y+\frac{1}{2}$ , $\bar{z}$ ; $\frac{1}{2}-y$ , $\frac{1}{2}-x$ , $z$ ; $x+\frac{1}{2}$ , $\frac{1}{2}-y$ , $\bar{z}$ ; $y+\frac{1}{2}$ , $x+\frac{1}{2}$ , $z$ with $x=0.055$ , $y=0.194$ , $z=0.472$	
O(3):	(f) $x_1y_1z_1$ with $x_1=0.128$ , $y_1=0.047$ , $z_1=0.139$	
(Al+Si)(2):	(f) $x'y'z'$ with $x'=0.186$ , $y'=0.103$ , $z'=0.375$ .	

The similarity between the tetrahedral strings in this mineral and in other zeolites can be seen by comparing Figure 330 with Figure 329 for natrolite. In this edingtonite arrangement four H<sub>2</sub>O molecules are coordinated about each barium atom. The positions chosen for water are considered most probable but because of the heavy barium the available X-ray data cannot locate them uniquely.

FIG. 330.—A basal projection of the tetragonal structure given to edingtonite, Ba<sub>2</sub>Al<sub>4</sub>Si<sub>6</sub>O<sub>20</sub>·8H<sub>2</sub>O. Al and Si atoms, which are not separately determined, are the smallest circles; intermediate circles are Ba. Molecules of H<sub>2</sub>O are heavily ringed.



(bh) The true unit prism of thomsonite, NaCa<sub>2</sub>Al<sub>5</sub>Si<sub>5</sub>O<sub>20</sub>·6H<sub>2</sub>O, contains four molecules (Table I). There is, however, an approximate halving along the *c* direction that suggests a two-molecule pseudo-unit. A structure based on this pseudo-cell with its atoms in the following positions of  $V_h^7$  is thought to be approximately correct.

Ca:	(c) $\frac{1}{2}0\frac{1}{2}$ ; $0\frac{1}{2}\frac{1}{2}$
(2 Ca+2 Na):	(h) $\pm(u0v)$ ; $\pm(\frac{1}{2}-u, \frac{1}{2}, v)$ with $u=0.069$ , $v=0.25$
(2 Si+2 Al):	(g) $\pm(\frac{1}{4}\frac{1}{4}u)$ ; $\pm(\frac{3}{4}\frac{1}{4}\bar{u})$ with $u=0.875$
H <sub>2</sub> O(1):	(e) $\pm(0u0)$ ; $\pm(\frac{1}{2}, \frac{1}{2}-u, 0)$ with $u=0.139$
H <sub>2</sub> O(2):	(h) $u_10v_1$ with $u_1=0.403$ , $v_1=0.75$
H <sub>2</sub> O(3):	(h) $u_20v_2$ with $u_2=0.111$ , $v_2=0.75$
O(1):	(f) $\pm(\frac{1}{2}u\frac{1}{2})$ ; $\pm(0, u+\frac{1}{2}, \frac{1}{2})$ with $u=0.361$
O(2):	(h) $u'0v'$ with $u'=0.402$ , $v'=0.25$ .

TABLE XIII. PARAMETERS FOR ATOMS OF THOMSONITE LYING IN GENERAL POSITIONS

Atom	No. per cell	$x$	$y$	$z$
O(3)	8	0.167	0.194	0.75
O(4)	8	.180	.119	.375
O(5)	8	.305	.139	0
O(6)	8	.375	.194	.375
(Si+Al)(2)	8	.125	.194	.500
(Al+Si)(3)	8	.305	.125	.250

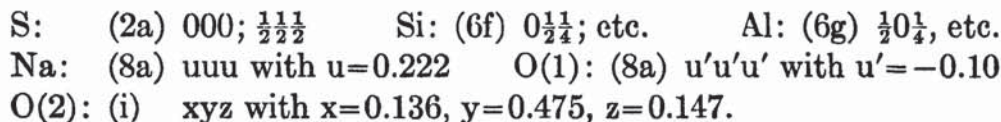
The rest of the atoms are in general positions: (i)  $\pm(xyz)$ ;  $\pm(x\bar{y}z)$ ;  $\pm(\frac{1}{2}-x, \frac{1}{2}-y, z)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, \bar{z})$ , with the parameters of Table XIII. It has been suggested that this simplified structure departs from the true one mainly in the distribution of Al and Si atoms within the positions assigned to them as centers of oxygen tetrahedra. The existing data are inadequate to show whether this is the case.

The axes of this description (abc) are connected with those of 1930, 352, p. 61 (X'Y'Z') by the relations:  $a=Z'$ ,  $b=X'$ ,  $c=Y'$ .

(bi) X-ray patterns have been made of partially dehydrated heulandite and of the  $\beta$ -heulandite obtained by dehydrating above 210° C. The former gives a heulandite-like pattern and takes up water reversibly. The  $\beta$ -form cannot be reversed. Digestion of heulandite in HCl results in a silica which, though pseudomorphic after the original crystal, yields an amorphous X-ray pattern.

(bj) Other minerals which have been found to have three-dimensional linked  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedral networks are members of the sodalite group.

A structure for sodalite itself,  $\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}\text{Cl}$ , has already been proposed [(x) book, p. 352]. This was developed from the space group  $T_d^4$ . Similar arrangements have been suggested for h a ynite and noselite. Writing the formula for h a ynite as essentially  $\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}\text{SO}_4$  with some substitution of calcium for sodium, the necessary atomic positions, already listed for sodalite, [(x) book, p. 352], are:



The coordinates for noselite are nearly identical, the assumption being made that only some of the sulfate positions centering about 000 and  $\frac{1}{2}\frac{1}{2}\frac{1}{2}$  are occupied. For Na,  $u=0.217$ ; for O(1),  $u'=-0.100$ ; for O(2),  $x=0.136$ ,  $y=0.475$ ,  $z=0.147$ .

A debate over the true formula of h a ynite has led to another, but closely related, structure based on  $T_d^4$ . Considering this mineral to be a solid solution of the composition  $(\text{Na}, \text{Ca})_{4-8}\text{Al}_6\text{Si}_6\text{O}_{24} \cdot (\text{SO}_4)_{1-2}$ , sodalite

TABLE XIV. THE  $T_d^1$  STRUCTURES FOR SODALITE AND NOSELITE

Arrangement	Sodalite		Noselite	
	Atom	Parameters	Atom	Parameters
(1a) 000	Cl	—	S	—
(1b) $\frac{1}{2}\frac{1}{2}\frac{1}{2}$	Cl	—	—	—
(12f) $u0\frac{1}{2}$ ; etc.	(6Al+6Si)	$u=0.25$	(6Al+6Si)	$u=0.25$
(4a) $uuu$ ; etc.	Na(1)	$u=0.175$	Na(1)	$u=0.150$
(4a) $u'u'u'$ ; etc.	Na(2)	$u'=0.675$	Na(2)	$u'=0.750$
(4a) $u_1u_1u_1$ ; etc.	—	—	O(1)	$u_1=0.897$
(12g) $uuv$ ; etc.	O(1)	$u=0.147$	O(2)	$u=0.144$
		$v=0.445$		$v=0.473$
(12g) $u'u'v'$ ; etc.	O(2)	$u'=0.660$	O(3)	$u'=0.645$
		$v'=-0.056$		$v'=-0.028$

(4a):  $uuu$ ;  $u\bar{u}\bar{u}$ ;  $\bar{u}u\bar{u}$ ;  $\bar{u}\bar{u}u$   
(12f):  $u0\frac{1}{2}$ ;  $\bar{u}0\frac{1}{2}$ ;  $u\frac{1}{2}0$ ;  $\bar{u}\frac{1}{2}0$ ;  $\frac{1}{2}u0$ ;  $\frac{1}{2}\bar{u}0$ ;  $0u\frac{1}{2}$ ;  $0\bar{u}\frac{1}{2}$ ;  $0\frac{1}{2}u$ ;  $0\frac{1}{2}\bar{u}$ ;  $\frac{1}{2}0u$ ;  $\frac{1}{2}0\bar{u}$   
(12g):  $uuv$ ;  $u\bar{u}\bar{v}$ ;  $\bar{u}u\bar{v}$ ;  $\bar{u}\bar{u}v$ ;  $vuu$ ;  $\bar{v}u\bar{u}$ ;  $\bar{v}\bar{u}u$ ;  $v\bar{u}\bar{u}$ ;  $uvu$ ;  $\bar{u}\bar{v}u$ ;  $u\bar{v}\bar{u}$ ;  $\bar{u}v\bar{u}$ .

and noselite have been assigned the groupings of Table XIV. Upon this basis some but not all cells of haüynite would contain  $SO_4$  groups in approximately the positions (1b) and (4a) [for O(1)] of noselite; Ca would replace Na(1) or Na(2). A convincing choice between these alternative arrangements cannot be made from the existing X-ray data.

It has been found that the two minerals ittnerite and skolopside (1934, 162) give weak haüynite patterns and presumably are alteration products.

None of these more recent studies of substances related to the ultramarines (*aa*) gives support to the suggestion earlier advanced that their alkali atoms are "wandering" without fixed positions in the structure.

(*bk*) The following structure, which gives qualitative agreement with the data from two oscillation photographs, has been proposed for zunyite,  $Al_{13}Si_5O_{20}(OH, F)_{18}Cl$ . Based on  $T_d^2$  it places four molecules within the unit cube. Atoms are in points having the coordinates listed below and in three similar sets of points (1930, 352, p. 128) about  $\frac{1}{2}\frac{1}{2}0$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $0\frac{1}{2}\frac{1}{2}$ .

Cl:	(4c) $\frac{1}{2}\frac{1}{2}\frac{1}{2}$	Si: (4d) $\frac{1}{4}\frac{1}{4}\frac{1}{4}$	Al(1): (4e) $\frac{3}{4}\frac{3}{4}\frac{3}{4}$
Si:	(16a) $uuu$ ; $u\bar{u}\bar{u}$ ; $\bar{u}u\bar{u}$ ; $\bar{u}\bar{u}u$ with $u=0.117$		
O(1):	(16a) $u_1u_1u_1$ with $u_1=-0.177$		
O(2):	(16a) $u_2u_2u_2$ with $u_2=0.184$		
(OH, F)(1):	(24a) $u00$ ; $\bar{u}00$ ; $0u0$ ; $0\bar{u}0$ ; $00u$ ; $00\bar{u}$ with $u=0.273$		
(OH, F)(2):	(48d) $uuv$ ; $u\bar{u}\bar{v}$ ; $\bar{u}u\bar{v}$ ; $\bar{u}\bar{u}v$ ; $vuu$ ; $\bar{v}u\bar{u}$ ; $\bar{v}\bar{u}u$ ; $v\bar{u}\bar{u}$ ; $uvu$ ; $\bar{u}\bar{v}u$ ; $u\bar{v}\bar{u}$ ; $\bar{u}v\bar{u}$ with $u=0.181$ , $v=0.545$		
O(3):	(48d) $u'u'v'$ with $u'=0.139$ , $v'=0.006$		
Al(2):	(48d) $u_1u_1v_1$ with $u_1=0.089$ , $v_1=-0.228$ .		

(*bl*) The hexagonal unit of cancrinite contains one molecule whose ideal composition is said to be  $Ca_2Na_6Al_6Si_6O_{24}\cdot 2CO_3$ . An arrangement which is compared with some estimated intensity data from rotation photographs has been developed from  $C_6^6$ . It is as follows:

- 2 Ca: (b)  $\frac{1}{3}\frac{2}{3}u$ ;  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $u+\frac{1}{2}$  with  $u=0.36$   
 2 C: (b)  $\frac{1}{3}\frac{2}{3}u'$ ;  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $u'+\frac{1}{2}$  with  $u'=-0.14$ .

The rest of the atoms are in general positions:

- (c):  $xyz$ ;  $y-x$ ,  $\bar{x}$ ,  $z$ ;  $\bar{y}$ ,  $x-y$ ,  $z$ ;  $\bar{x}$ ,  $\bar{y}$ ,  $z+\frac{1}{2}$ ;  $x-y$ ,  $x$ ,  $z+\frac{1}{2}$ ;  $y$ ,  $y-x$ ,  $z+\frac{1}{2}$

with the parameters of Table XV.

TABLE XV. PARAMETERS FOR ATOMS OF CANCRINITE  
 LYING IN GENERAL POSITIONS

Atom	No. per cell	$x$	$y$	$z$
Na	6	0.50	0.50	0.22
Si	6	.03	.26	.26
Al	6	.26	.23	.24
O(1)	6	.05	.36	.01
O(2)	6	.36	.32	-.03
O(3)	6	.17	.27	.26
O(4)	6	-.13	.16	.24
O(5)	6	.20	.64	.36

(*bm*) The feldspars have a grouping which thus far has resisted complete analysis. A few years ago (1929, 289a; 1931, 391) a type of structure was proposed which did not provide reasonable interatomic distances. Recently a different arrangement has been suggested for a sanidine ( $\text{KAlSi}_3\text{O}_8$ , with some Na replacing K). This gives approximately the right atomic separations and is in fairly good agreement with the reflections on several rotation photographs. It has atoms in the following special and general positions of  $C_{2h}^3$  with the parameters of Table XVI:

- (g)  $\pm(0u0)$ ;  $\pm(\frac{1}{2}, u+\frac{1}{2}, 0)$       (i)  $\pm(u0v)$ ;  $\pm(u+\frac{1}{2}, \frac{1}{2}, v)$   
 (j)  $\pm(xyz)$ ;  $\pm(x\bar{y}z)$ ;  $\pm(x+\frac{1}{2}, y+\frac{1}{2}, z)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$ .

A projection of the unit cell on its *ac*-plane is reproduced in Figure 331. Its interlocking network of (Al, Si) $\text{O}_4$  tetrahedra is seen to be very different from those deduced for the sodalite and zeolitic minerals.

Measurements upon celsian,  $\text{BaAl}_2\text{Si}_2\text{O}_8$ , have indicated that it is triclinic but that it probably does not depart far from the monoclinic arrange-

TABLE XVI. PARAMETERS OF THE ATOMS IN SANIDINE

Atom	No. per cell	Position	$x$	$y$	$z$
O(1)	4	(g)	0	0.139	0
O(2)	4	(i)	0.658	0	0.236
O(3)	8	(j)	.819	.153	.236
O(4)	8	(j)	.000	.319	.250
O(5)	8	(j)	.153	.125	.417
Si+Al(1)	8	(j)	.000	.186	.217
Si+Al(2)	8	(j)	.703	.111	.347
K	4	(i)	.294	0	.139

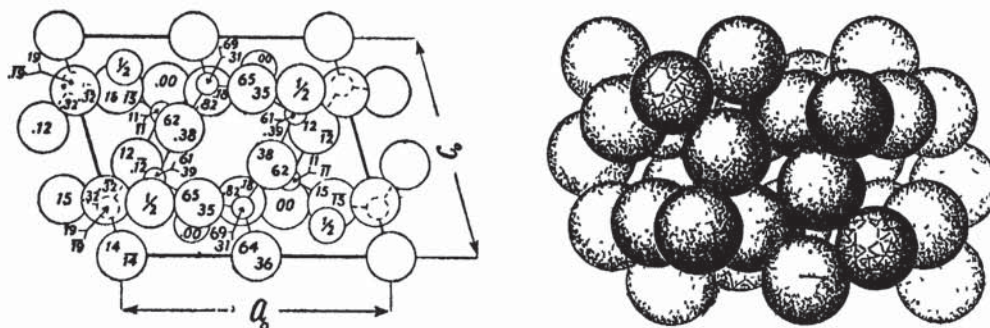


FIG. 331a.—(left) A portion of the monoclinic structure found for sanidine,  $\text{KAlSi}_3\text{O}_8$ , as projected on its  $b$ -face. The smallest circles are Al or Si (not distinguished in the structure), the largest are O atoms.

FIG. 331b.—(right) A packing drawing of  $a$ . The K ions are line-shaded; two of the (Si, Al) atoms are visible as black spheres.

ment of sanidine. The K-Ba feldspars, adularia and hyalophane, containing up to ca 15% BaO are truly monoclinic. In these minerals there is thought to be a haphazard replacement of K and Si by Ba and Al.

The soda feldspar albite,  $\text{NaAlSi}_3\text{O}_8$ , though definitely triclinic can be described in terms of a sanidine-like cell. It has been found that an arrangement with parameters modified from those of  $\text{KAlSi}_3\text{O}_8$  yields fairly satisfactory agreement with photographic data. The cell for this description, being base-centered and hence not the simplest one possible has for its general positions the coordinates: (i)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, y+\frac{1}{2}, z)$ . Its parameters are recorded in Table XVII. The similarity between this structure and the sanidine grouping is best seen by comparing this table with Table XVI.

TABLE XVII. PARAMETERS OF THE ATOMS IN ALBITE

Atom	$x$	$y$	$z$
O(1)	0.014	0.125	-0.014
O(2)	.611	-.014	.278
O(3, a)	.833	.125	.214
O(3, b)	.311	.361	.250
O(4, a)	.014	.305	.264
O(4, b)	.536	.194	.230
O(5, a)	.194	.139	.389
O(5, b)	.658	.389	.411
Si+Al(1, a)	.000	.175	.222
Si+Al(1, b)	.472	.328	.233
Si+Al(2, a)	.714	.105	.333
Si+Al(2, b)	.214	.383	.361
Na	.278	-.167	.172

All of the plagioclase feldspars do not have albite-like cells. The units of two andesites with albite-anorthite ratios of 3:1 and 2:1 are like albite, but anorthite itself,  $\text{CaAl}_2\text{Si}_2\text{O}_8$ , and a labradorite with the ratio 1:1 both have  $c_0$  axes that are twice as long.

New data bearing on the cell dimensions of the feldspars are assembled in Table XVIII (cf. Table XVIII, p. 354 of book).

TABLE XVIII. NEW CELL DIMENSIONS ON FELDSPARS

<i>Mineral</i>	<i>Symmetry</i>	$a_0$	$b_0$	$c_0$	$\alpha$	$\beta$	$\gamma$
Sanidine	Monoclinic	8.45	12.90	7.15	—	116°6'	—
Adularia	Monoclinic	8.45	12.90	7.15	—	116°3'	—
Hyalophane (A, B)	Monoclinic	8.45	12.90	7.15	—	115°35'	—
Hyalophane (C)	Monoclinic	8.52	12.95	7.14	—	115°35'	—
Celsian	Triclinic	8.63	13.10	7.29	ca 90°	116°	ca 90°
Albite	Triclinic	8.14	12.86	7.17	94°3'	116°29'	88°9'
Andesite	Triclinic	8.14	12.86	7.17	93°23'	116°28'	89°59'
Labradorite	Triclinic	8.21	12.95	14.16	93°31'	116°3'	89°55'
Anorthite	Triclinic	8.21	12.95	14.16	93°13'	115°56'	91°12'

(bn) An earlier (1929, 115) study of wollastonite,  $\text{CaSiO}_3$ , resulted in a monoclinic unit. More recently it has been shown to be triclinic. Similar cells can be given to pectolite,  $\text{NaHCa}_2(\text{SiO}_3)_3$ , and probably to schizolite,  $\text{HNa}(\text{Ca}, \text{Mn})_2(\text{SiO}_3)_3$ .

(bo) The cell dimensions for epidote quoted in Table I are those of 1932, 172. The two earlier studies gave it a two-molecule unit with  $a_0$  half as long. Clinzoisite, an epidote without iron, is structurally like the ordinary variety.

(bp) The structure of nephelite is yet to be determined. A few suggestions have been made but, unsupported by the necessary X-ray data, they are of little value.

(bq) Unit cells have been assigned to two wöhlerite-like minerals besides the one quoted in Table I. Lavenite with 20% of its Zr replaced by  $\text{Ca}_2$  has  $a_0=10.93$  A,  $b_0=9.99$  A,  $c_0=7.18$  A,  $\beta=110^\circ 28'$ ; hiortdahlite with  $\text{Ca}_2$  substituting for 25% of its (Zr, F)Na is reported to be triclinic but with very similar cell dimensions:  $a_0=10.91$  A,  $b_0=10.29$  A,  $c_0=7.32$  A,  $\alpha=90^\circ 29'$ ,  $\beta=108^\circ 50'$ ,  $\gamma=90^\circ 8'$ .

(br) Mosandrite is a rinkite in which H and OH replace Na and F atoms. Though monoclinic the cell dimensions found for it are nearly the same as those given to the orthorhombic rinkite. They are  $a_0=18.47$  A,  $b_0=5.67$  A,  $c_0=7.46$  A,  $\beta=91^\circ 13'$ .

(bs) The unit cubes of several garnets have been measured. One, a 48% grossularite-andradite, 52% almandite-pyrope, has  $a_0=11.668$  A (1933, 182). Spessartite gives 11.603 A; partschinite, a spessartite with



some iron in place of manganese has  $a_0 = 11.613 \text{ \AA}$  (1933, 522). It has also been found that  $a_0$  for Ca-Fe garnets increases from 11.93  $\text{\AA}$  to 12.14  $\text{\AA}$  as the titanium content mounts from zero.

(*bt*) It is suggested that though they seemingly are tetragonal, the **scapolites** really are complex twinings of monoclinic and triclinic individuals.

## Chapter XXA. Structures of Organic Compounds

Nearly all the X-ray studies of organic crystals being published are limited to unit cell and space group determinations. Some, as indicated in the large tables, prove molecular symmetry for suitably constituted compounds. Others point to the existence of associated molecules in the crystalline state. Though a few of these associations, marked A in the tables, may be real most of them are to be explained by the choice of too large a unit cell.

Some progress has been made towards an understanding of the atomic arrangements in aliphatic structures but most of the increase in our knowledge of atomic positions in organic crystals has come through the investigation of several aromatic hydrocarbons.

### A. The Structures of Organic Salts and of Metallo-organic Compounds

(*bd*) A previous study of  $\text{Be}_4\text{O}(\text{C}_2\text{H}_3\text{O}_2)_6$  has indicated that the eight molecules in its unit cube are arranged according to the requirements of  $T_h^4$ . This has been confirmed and it has been shown that by placing atoms in the following positions of this space group an arrangement is obtained which yields plausible interatomic distances and does not conflict with data from Laue and oscillation photographs.

Be: (32b)  $uuu$ ; etc. [see XVIII A, (*ce*)] with  $u = -0.060$   
O(1): (8f)  $000$ ;  $\frac{1}{2}0\frac{1}{2}$ ;  $\frac{1}{2}\frac{1}{2}0$ ;  $0\frac{1}{2}\frac{1}{2}$ ;  $\frac{1}{4}\frac{1}{4}\frac{1}{4}$ ;  $\frac{3}{4}\frac{1}{4}\frac{3}{4}$ ;  $\frac{3}{4}\frac{3}{4}\frac{1}{4}$ ;  $\frac{1}{4}\frac{3}{4}\frac{3}{4}$   
O(2): (g)  $xyz$ ; etc. with  $x = -0.163$ ,  $y = -0.064$ ,  $z = -0.038$   
C(1): (48c)  $u_100$ ; etc. with  $u_1 = 0.197$   
C(2): (48c)  $u'00$ ; etc. with  $u' = 0.295$ .

As was earlier pointed out this choice of space group implies that the two oxygen atoms of the acetate group are geometrically equivalent.

(*be*) Two studies of the dihydrate of copper formate,  $\text{Cu}(\text{HCOO})_2 \cdot 2\text{H}_2\text{O}$ , differ both in the size of the unit and in the chosen space group.

(*bf*) It is said that the lead atoms in  $\text{Pb}(\text{HCOO})_2$  have the coordinates  $\frac{1}{2}, \frac{1}{2}, \frac{1}{4}$ ;  $\frac{1}{2}, \frac{1}{2}, \frac{3}{4}$ ;  $\frac{5}{2}, \frac{7}{2}, \frac{1}{4}$ ;  $\frac{5}{2}, \frac{7}{2}, \frac{3}{4}$ .

TABLE I. THE CRYSTAL STRUCTURES OF ORGANIC SALTS AND METALLO-ORGANIC COMPOUNDS

Substance, symmetry and type	Space group	No. mol. per cell	Mon. mol. symmetry	$a_0$	$b_0$	$c_0$	References
Silver Nitrate Urea $\text{AgNO}_3 \cdot \text{CO}(\text{NH}_2)_2$	$\text{C}_{2h}^2$	8	A	10.23	16.84 $\beta = 77^\circ$	6.25	1934, 55.
Mellite $\text{Al}_2\text{C}_{12}\text{O}_{12} \cdot 18\text{H}_2\text{O}$		16		22.0		23.3	1933, 31.
Barium Chloride Glycine $\text{BaCl}_2 \cdot 2\text{CH}_2\text{NH}_2\text{COOH}$	$\text{V}_h^{16}$	4	$\text{C}_1$ or $\text{C}_s$	7.96	14.7	9.21	1933, 41.
Beryllium Oxyacetate $\text{Be}_4\text{O}(\text{C}_2\text{H}_3\text{O}_2)_6$			(j), (bd)				1934, 206.
Triphenyl Bismuthine Dichloride	$\text{V}^2$	8	A	17.31	22.39	9.20	1931, 138.
Calcium Sulfate Urea $\text{CaSO}_4 \cdot \text{CO}(\text{NH}_2)_2$	Ort.						
Cesium Acid Tartrate $\text{CsHC}_4\text{H}_4\text{O}_6$	Tri.	4	A	14.74	14.95	6.47	1933, 179.
Copper Formate Dihydrate $\text{Cu}(\text{HCO}_2)_2 \cdot 2\text{H}_2\text{O}$	Ort.	4		$\alpha = 91^\circ 26'$ , $\beta = 90^\circ 22'$ , $\gamma = 86^\circ 42'$		8.03	1931, 169.
Copper Formate Tetrahydrate $\text{Cu}(\text{HCO}_2)_2 \cdot 4\text{H}_2\text{O}$	Mon.						1931, 241, 364.
Cuprous Glutathione $\text{KHC}_4\text{H}_4\text{O}_6$	Ort.	4		28.0	8.74	5.57	1932, 366.
Potassium Acid Tartrate $\text{NaOOC}(\text{CHOH})_2\text{COOK} \cdot 4\text{H}_2\text{O}$	Ort.	4		7.614	10.70	7.80	1932, 147.
Lead Formate $\text{Pb}(\text{HCO}_2)_2$	Ort.	4		11.913	14.324	6.153	1933, 444.
			(b), (bf)				1932, 190.

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
Bisethylene Diamino Platinous Chloride Tri. $\text{Pt}(\text{C}_2\text{H}_3\text{N}_2)_2\text{Cl}_2$	$C_1^1$	1		8.37	4.95	6.86	1933, 109.
$\alpha$ -Bisdiethyl Sulfine Platinous Chloride Mon. $\text{Pt}[\text{S}(\text{C}_2\text{H}_5)_2]_2\text{Cl}_2$		2		$12.0$ $\alpha=100^\circ 46'$ , $\beta=111^\circ 40'$ , $\gamma=81^\circ 56'$	7.9	7.7	1934, 61.
$\alpha$ -Bisdimethyl Sulfine Platinous Chloride Mon. $\text{Pt}[\text{S}(\text{CH}_3)_2]_2\text{Cl}_2$	$C_{2h}^2$ or $C_{2h}^6$	2		10.16	6.01	8.48	1934, 61.
$\beta$ -Bisdimethyl Sulfine Platinous Chloride Mon. $\text{Pt}[\text{S}(\text{CH}_3)_2]_2\text{Cl}_2$		4		9.3	13.2	8.7	1934, 61.
Methyl Silicate $(\text{CH}_3)_4\text{SiO}_4$	$T^1$	4	$C_3$	9.85 at $-80^\circ\text{C}$ (?)	$\beta=105^\circ$		1931, 106.
Dimethyl Thallium Bromide Tet. $\text{Tl}(\text{CH}_3)_2\text{Br}$	$D_{4h}^{17}$	2	$D_4^h$	4.47		13.78	1934, 217.
Dimethyl Thallium Chloride Tet. $\text{Tl}(\text{CH}_3)_2\text{Cl}$	$D_{4h}^{17}$	2	$D_4^h$	4.29		14.01	1934, 217.
Dimethyl Thallium Iodide Tet. $\text{Tl}(\text{CH}_3)_2\text{I}$	$D_{4h}^{17}$	2	$D_4^h$	4.78		13.43	1934, 217.

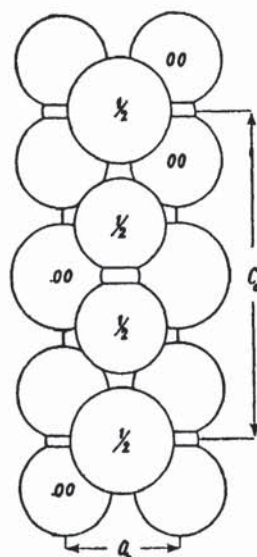
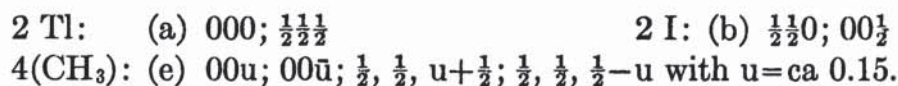


FIG. 332.—A projection upon one of its  $a$ -faces of atoms in the tetragonal arrangement deduced for  $\text{Tl}(\text{CH}_3)_2\text{I}$ . The largest circles are I ions, the slightly smaller ones are  $\text{CH}_3$  groups. The sizes used in this drawing are determined by the results on the methyl substituted ammonium salts.

(bg) An elaborate discussion, based on powder photographs, has been given of possible arrangements for the atoms in **methyl silicate**,  $(\text{CH}_3)_4\text{SiO}_4$ . It was shown that the structure is based on  $T^1$  but the correct grouping was not definitely established.

(bh) Spectral photographs have been used to find an atomic arrangement for the atoms in **dimethyl thallium iodide**,  $\text{Tl}(\text{CH}_3)_2\text{I}$ . They are in the following special positions of  $D_{4h}^{17}$ :



The  $\text{Tl}-\text{CH}_3$  separation is 2.01 Å; the distance between  $\text{CH}_3$  groups through which contact is made along  $c$ , is 4.17 Å (Figure 332). This large  $\text{CH}_3-\text{CH}_3$  separation may mean that  $u$  should be greater than 0.15.

The bromide and chloride are structurally isomorphous with the iodide but the methyl parameter could not be found for them.

### B. The Structures of Substituted Ammonium Salts

(bi) Quantitative spectrometer measurements have been used to find the structure of **dimethyl ammonium chlorostannate**,  $[\text{NH}_2(\text{CH}_3)_2]_2\text{SnCl}_6$ . Atoms are in special and general positions of  $C_{2v}^7$ :

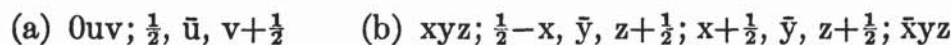


TABLE II. THE CRYSTAL STRUCTURES OF SUBSTITUTED AMMONIUM SALTS

Substance, symmetry and structure type	$a_0$	$b_0$	$c_0$	No. mol. per cell	References
Monomethyl Ammonium Cupric Chloride $(\text{NH}_3\text{CH}_3)_2\text{CuCl}_4$ Ort.	7.30	7.535	18.55	4	1933, 157.
Dimethyl Ammonium Chlorostannate $[\text{NH}_2(\text{CH}_3)_2]_2\text{SnCl}_6$ Ort. (bi)	7.26	14.28	7.38	2	1933, 157; 1934, 60.
Tetramethyl Ammonium Fluosilicate $[\text{N}(\text{CH}_3)_4]_2\text{SiF}_6$ Tet. (bj)	7.88		11.19	2	1934, 57.
n-Monoamyl Ammonium Chloride $n\text{-C}_8\text{H}_{17}\text{NH}_3\text{Cl}$ Tet. (bk)	7.03 at -80° C		16.70	4	1933, 438.
Octadecyl Ammonium Chloride $\text{C}_{18}\text{H}_{37}\text{NH}_3\text{Cl}$ Ort. (bl)	5.45	5.40	69.4	4	1932, 41, 42.

with the parameters of Table III. The axes of this description,  $XYZ=abc$ , are connected with those used for an earlier crystallographic description,  $a'b'c'$ , by the relations  $X=a=a'$ ,  $Y=b=c'$ ,  $Z=c=b'$ .

TABLE III. PARAMETERS OF THE ATOMS IN  $[(\text{CH}_3)_2\text{NH}_2]_2\text{SnCl}_6$ 

Atom	No. per cell	Position	$x$	$y$	$z$
Sn	2	(a)	0	0.250	0
Cl(1)	2	(a)	0	.390	0.180
Cl(2)	2	(a)	0	.110	-.180
Cl(3)	4	(b)	0.235	.185	.190
Cl(4)	4	(b)	.235	.315	-.190
N(1)	2	(a)	0	.620	.690
N(2)	2	(a)	0	.880	.310
CH <sub>3</sub> (1)	2	(a)	0	.605	.875
CH <sub>3</sub> (2)	2	(a)	0	.895	.125
CH <sub>3</sub> (3)	2	(a)	0	.530	.625
CH <sub>3</sub> (4)	2	(a)	0	.970	.375

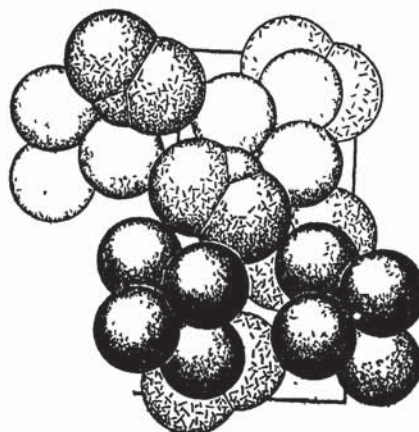
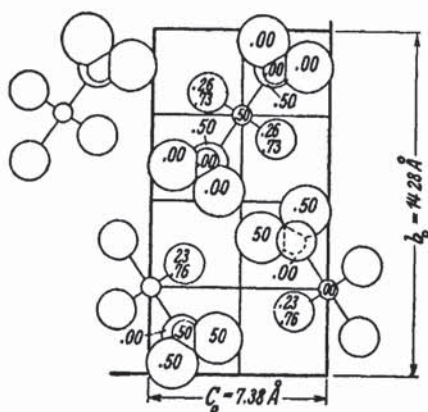


FIG. 333a.—(left) A portion of the structure of  $[\text{NH}_2(\text{CH}_3)_2]_2\text{SnCl}_6$  projected on the  $a$ -face of its orthorhombic cell. The segments of circles, representing  $\text{NH}_2$ , join  $\text{CH}_3$  groups (largest circles). The Cl and Sn (smallest circles) of  $\text{SnCl}_6$  ions are connected by light lines.

FIG. 333b.—(right) A packing drawing of  $a$ . The  $\text{NH}_2(\text{CH}_3)_2$  ions are line-shaded. Atoms of Sn cannot be seen.

The substituted ammonium chlorostannates thus far analyzed have been relatively simple distortions of the  $(\text{NH}_4)_2\text{PtCl}_6$  grouping [XVII, (a)]. This arrangement (Figure 333) can be similarly viewed but the distortion is great. The two C-N bonds of a  $(\text{CH}_3)_2\text{NH}_2^+$  ion make the tetrahedral angle with one another; the  $\text{CH}_3$ -Cl separation, ca 3.83 Å, is the same as that found in other substituted ammonium chlorostannates.

(b) The tetragonal packing found for the atoms in tetramethyl ammonium fluosilicate,  $[\text{N}(\text{CH}_3)_4]_2\text{SiF}_6$ , is a distortion of that prevailing in the cubic  $[\text{N}(\text{CH}_3)_4]_2\text{SnCl}_6$  ( $u$ ). Atoms are in the following general and special positions (1930, 352, p. 82) of  $C_{4h}^5$  with parameters fixed by a series of spectrometric measurements (Figure 334):

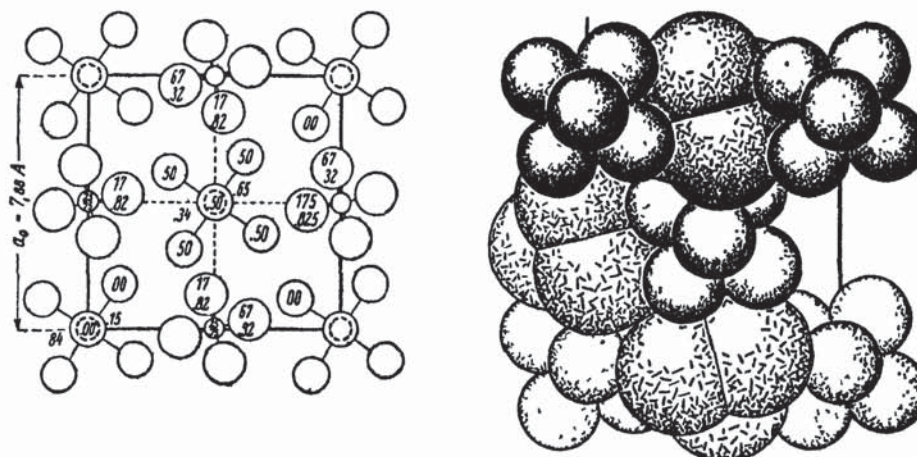


FIG. 334a.—(left) A basal projection of the tetragonal arrangement found for  $[\text{N}(\text{CH}_3)_4]_2\text{SiF}_6$ . The smallest circles, as N atoms, are each joined to their four surrounding  $\text{CH}_3$  groups by light lines. The heavy dotted circles are the Si atoms at the centers of their surrounding F octahedra.

FIG. 334b.—(right) A packing drawing of *a*. The  $\text{CH}_3$  groups of the  $\text{N}(\text{CH}_3)_4$  tetrahedra are line-shaded.

- |                      |  |  |
|----------------------|--|--|
| 2 Si:                | (a) 000; $\frac{1}{2}\frac{1}{2}\frac{1}{2}$   | 4 N: (d) $0\frac{1}{2}\frac{1}{4}$ ; $\frac{1}{2}0\frac{1}{4}$ ; $0\frac{1}{2}\frac{3}{4}$ ; $\frac{1}{2}0\frac{3}{4}$ |
| 4 F:                 | (e) 00u; 00 $\bar{u}$ ; $\frac{1}{2}$ , $\frac{1}{2}$ , $u+\frac{1}{2}$ ; $\frac{1}{2}$ , $\frac{1}{2}$ , $\frac{1}{2}-u$ with $u=0.155$   |  |
| 8 F:                 | (h) $\pm(uv0)$ ; $\pm(\bar{v}u0)$ ; $\pm(\frac{1}{2}-v, u+\frac{1}{2}, \frac{1}{2})$ ; $\pm(u+\frac{1}{2}, v+\frac{1}{2}, \frac{1}{2})$ with $u=0.18, v=0.12$                        |  |
| 16( $\text{CH}_3$ ): | (i) $\pm(xyz)$ ; $\pm(\bar{y}xz)$ ; $\pm(\bar{x}\bar{y}z)$ ; $\pm(y\bar{x}z)$ and eight similar points around $\frac{1}{2}\frac{1}{2}\frac{1}{2}$ , with $x=0.14, y=0.47, z=0.175$ . |  |

(*bk*) In contrast with the two-molecule tetragonal cell found at room temperature, a four-molecule tetragonal unit is observed for **n-amyl ammonium chloride** both at  $\text{CO}_2$ -snow and at liquid air temperatures. The observed data are said to indicate that at low temperatures molecular rotation is arrested but not necessarily with the carbon chains in equilibrium positions.

(*bl*) The orthorhombic arrangement found for **octadecyl ammonium chloride** is said to be evidence that in this long chain compound the molecules are not rotating.

### C. The Structures of Aliphatic and Aromatic Compounds

#### Aliphatic Compounds

(*bm*) A redetermination of  $a_0$  for **methane** yields a value much lower than that previously found and in better agreement with the experimental density. The attempt to establish hydrogen positions from these photographs is undoubtedly of little real significance.

TABLE IV. THE CRYSTAL STRUCTURES OF ALIPHATIC ORGANIC COMPOUNDS

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_o$	$b_o$	$c_o$	References
<i>Methane and Ethane Derivatives</i>							
Methane $CH_4$	Cub.	4	$T_d$	5.89			1931, 307, 308, 309.
Iodoform $CHI_3$	Hex.	2	$C_3$	6.818		7.524	1931, 221.
Pentaerythritol Tetrabromide $C(CH_2Br)_4$	Mon.	1	$C_2^h$	7.199	6.325 $\beta = 112^\circ 52'$	5.719	1932, 457.
Pentaerythritol Tetrachloride $C(CH_2Cl)_4$	Mon.	1	$C_2^h$	6.912	6.289 $\beta = 112^\circ 54'$	5.492	1932, 457.
Pentaerythritol Tetraiodide $C(CH_2I)_4$	Mon.	1		7.552	6.432 $\beta = ca. 113^\circ$	6.075	1932, 457.
Urea $CO(NH_2)_2$	Tet.	2	$C_2^v$				1932, 484; 1934, 303.
Thiourea $CS(NH_2)_2$	Ort.	4	$C_s$	5.50	7.68	8.57	1932, 485.
Methyl Urea $CONH_2(NHCH_3)$	Ort.	4	$V^4$	6.89	6.96	8.45	1925, 281; 1933, 103.
Guanidinium Chloride $(NH_2)_2CNH \cdot HCl$	Ort.	8	$V_h^{15}$	7.76	9.22	13.06	1931, 444.
Hexabromobutylene $CHBr_2BrC = CBrCHBr_2$	Mon.	2	$C_1$	11.5	6.40 $\beta = 44^\circ 27'$	10.06	1931, 104; 1932, 142.
Hexamethylethane $C_2(CH_3)_6$	Cub.	2		7.69			1934, 289.
Hexachlorethane (above $71^\circ C$ ) $C_2Cl_6$	Cub.	2		7.43 at $80^\circ C$			1934, 289.



## Amino Acids

$\alpha$ -Glycine $\text{CH}_2\text{NH}_2\text{COOH}$	Mon.	(bs)	$\text{C}_{2h}^6$	4	5.04	12.1 $\beta = 111^\circ 38'$	5.41	1931, 41, 173.
$\beta$ -Glycine $\text{CH}_2\text{NH}_2\text{COOH}$	Mon.	(bs)	$\text{C}_2^6$	2	5.18	6.18 $\beta = 114^\circ 20'$	5.29	1931, 41.
d-Alanine $\text{CH}_3\text{CH}(\text{NH}_2)\text{COOH}$	Ort.		$\text{V}^4$	4	6.0	12.1	5.75	1931, 41.
dl-Alanine $\text{CH}_3\text{CH}(\text{NH}_2)\text{COOH}$	Ort.		$\text{C}_{2v}^6$	4	6.0	12.0	5.8	1931, 41.
$\alpha$ -Glycylglycine $\text{CH}_2\text{NH}_2\text{CONHCH}_2\text{COOH}$	Mon.	(bt)	$\text{C}_{2h}^6$	4	7.76	9.46 $\beta = 99^\circ 30'$	7.67	1931, 41; 1932, 283.
$\beta$ -Glycylglycine $\text{CH}_2\text{NH}_2\text{CONHCH}_2\text{COOH}$	Mon.		$\text{C}_{2h}^6$	4	17.3	4.65 $\beta = 125^\circ 20'$	8.4	1931, 41.
$\gamma$ -Glycylglycine $\text{CH}_2\text{NH}_2\text{CONHCH}_2\text{COOH}$	Ort.		$\text{C}_{2v}^6$	4	8.1	9.36	7.7	1931, 41.
Asparagine Monohydrate $(\text{CONH}_2)\text{CHNH}_2\text{CH}_2\text{COOH} \cdot \text{H}_2\text{O}$	Ort.		$\text{V}^4$	4	5.6	11.8	9.86	1931, 41.
d-Alanylglycine $\text{NH}_2 \cdot \text{CH}(\text{CH}_3)\text{CONHCH}_2\text{COOH}$	Mon.			2	5.29	11.67 $\beta = 101^\circ 30'$	5.47	1932, 283.
$\alpha$ -Diglycylglycine $\text{NH}_2\text{CH}_2\text{CONHCH}_2\text{CONHCH}_2\text{COOH}$	Mon.			2	8.53	4.3 $\beta = 105^\circ 30'$	11.4	1932, 283.
$\beta$ -Diglycylglycine $\text{NH}_2\text{CH}_2\text{CONHCH}_2\text{CONHCH}_2\text{COOH}$	Mon.			4	14.6	4.79 $\beta = 105^\circ 30'$	11.67	1932, 283.
Diglycylglycine Dihydrate $\text{NH}_2\text{CH}_2\text{CONHCH}_2\text{CONHCH}_2\text{COOH} \cdot 2\text{H}_2\text{O}$	Ort.		$\text{C}_{2v}^6$	4	22.0	9.8	4.7	1931, 41.
l-Aspartic Acid $\text{COOH} \cdot \text{CH}_2 \cdot \text{CH}(\text{NH}_2)\text{COOH}$	Mon.		$\text{C}_2^2$	4	5.1	6.9 $\beta = 96^\circ$	15.1	1931, 41.
l-Glutamic Acid $\text{COOH} \cdot \text{CH}_2 \cdot \text{CH}_2\text{CH}(\text{NH}_2)\text{COOH}$	Ort.		$\text{V}^4$	4	7.06	10.3	8.75	1931, 41.
l-Cystine $\text{COOHCH}(\text{NH}_2)\text{CH}_2\text{S}=\text{SCH}_2\text{CH}(\text{NH}_2)\text{COOH}$	Hex.	(bu)						1931, 22, 41.

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
<i>Other Dicarboxylic Acid Derivatives</i>							
Oxalic Acid Dihydrate (COOH) <sub>2</sub> ·2H <sub>2</sub> O	C <sub>2h</sub> <sup>5</sup>	2	C <sub>1</sub>	6.12	3.61	12.03	1934, 305a.
α-Malonic Acid COOHCH <sub>2</sub> COOH (form stable above 80° C)	Ort.	16		8.70	11.53	17.05	1931, 98; 1932, 131.
β-Succinic Acid COH(CH <sub>2</sub> ) <sub>2</sub> COOH (room temp. form)	Mon. (bv)						1930, 383; 1931, 98; 1932, 131.
α-Succinic Acid COOH(CH <sub>2</sub> ) <sub>2</sub> COOH (second modification)	Mon.	8		5.70	26.28	7.57	1931, 98; 1932, 131.
α-Glutaric Acid COOH(CH <sub>2</sub> ) <sub>3</sub> COOH	Mon.	8		10.34	5.08	32.9	1932, 131, 132.
β-Glutaric Acid COOH(CH <sub>2</sub> ) <sub>3</sub> COOH	Mon.			10.06	4.87	17.4	1932, 131, 132.
Guanidine d-Tartrate Hydrate [C(NH <sub>2</sub> ) <sub>2</sub> NH] <sub>2</sub> (C <sub>4</sub> H <sub>6</sub> O <sub>6</sub> )·1½H <sub>2</sub> O	Mon.	2		9.88	14.77	4.78	1933, 458.
					β=104°57'		
<i>Long Chain Derivatives</i>							
Dodecanol C <sub>12</sub> H <sub>26</sub> OH	Hex. (bw)	1	D <sub>3d</sub> <sup>2</sup>	4.76		34.0	1932, 41, 42.
Hexadecanol C <sub>16</sub> H <sub>33</sub> OH	Mon.	4		8.80	4.90	44.2	1932, 41.
α-Palmitic Acid CH <sub>3</sub> (CH <sub>2</sub> ) <sub>14</sub> COOH	Mon.	4		9.41	5.00	46.1	1932, 131, 133, 441.
α-Stearic Acid CH <sub>3</sub> (CH <sub>2</sub> ) <sub>16</sub> COOH	Mon.	4		9.41	5.00	45.9	1932, 131, 440.
					β=50°33'		
					β=50°50'		

$\beta$ -Stearic Acid $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$	Mon.	(ao)	$\text{C}_{2h}^4$ or $\text{C}_{2h}^5$	4	5.54	7.38 $\beta=63^\circ 38'$	48.84	1932, 131, 133, 440.	
<i>Saturated Cyclohydrocarbons and their Derivatives</i>									
Cyclohexane $\text{C}_6\text{H}_{12}$	Cub.		$\text{T}_h^2$ or $\text{O}_h^4$	4	8.41 at $\text{CO}_2$ -acetone temp.			1930, 122a.	
1, 4 Dibromocyclohexane $\text{C}_6\text{H}_{10}\text{Br}_2$	Mon.	(bx)	$\text{C}_2h^5$	2	11.92	5.56 $\beta=101^\circ 49'$	6.02	1931, 161; 1932, 191.	
1, 4 Diiodocyclohexane $\text{C}_6\text{H}_{10}\text{I}_2$	Mon.	(bx)	$\text{C}_2h^5$	2	12.50	5.72 $\beta=98^\circ 0'$	6.20	1931, 161; 1932, 191.	
$\alpha$ -Cyclohexandiol 1, 2 $\text{C}_6\text{H}_{10}(\text{OH})_2$	Ort.		$\text{V}_h^{16}$	8	7.62	8.55	19.57	1931, 473.	
$\beta$ -Cyclohexandiol 1, 4 $\text{C}_6\text{H}_{10}(\text{OH})_2$	Mon.		$\text{C}_2h^5$	6	6.32	21.2 $\beta=96^\circ$	7.27	1931, 473; 1932, 192.	
$\gamma$ -Cyclohexandiol 1, 2 $\text{C}_6\text{H}_{10}(\text{OH})_2$	Mon.		$\text{C}_2h^6$	8	19.13	9.92 $\beta=103^\circ 54'$	7.23	1931, 473.	
$\beta$ -Cyclohexandiol Diacetate 1, 4 $\text{C}_6\text{H}_{10}(\text{CH}_3\text{CO}_2)_2$	Mon.		$\text{C}_2h^6$	2	13.56	5.83 $\beta=107^\circ 24'$	6.72	1931, 473.	
Quercitol $\text{C}_6\text{H}_7(\text{OH})_5$	Mon.		$\text{C}_2^2$	2	6.83	8.53 $\beta=110^\circ 57'$	6.45	1931, 471.	
l-Inositol $\text{C}_6\text{H}_6(\text{OH})_6$	Mon.		$\text{C}_2^2$	2	6.17	9.11 $\beta=106^\circ 36'$	6.83	1931, 471.	
i-Inositol $\text{C}_6\text{H}_6(\text{OH})_6$	Mon.		$\text{C}_2h^6$	8	6.64	12.0 $\beta=105^\circ 48'$	19.7	1931, 471.	
i-Inositol Dihydrate $\text{C}_6\text{H}_6(\text{OH})_6 \cdot 2\text{H}_2\text{O}$	Mon.		$\text{C}_2h^6$	4	8.98	16.59 $\beta=109^\circ 48'$	6.49	1931, 472.	
Methyl-l-Inositol $\text{C}_6\text{H}_6(\text{OH})_5(\text{OCH}_3)$ (Quebrachitol)	Mon.		$\text{C}_2^2$	2	6.60	7.15 $\beta=90^\circ$	8.65	1931, 350.	
Cyclododecane $\text{C}_{12}\text{H}_{24}$	Tri.			1	7.84	5.44 $\alpha=81^\circ 42', \beta=64^\circ, \gamma=81^\circ$	7.82	1933, 307.	

Substance, symmetry and type	( <i>as</i> ), ( <i>cq</i> )	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
Hexamethylenetetramine $C_6H_{12}N_4$	Cub.	$T_d^2$	2	T				1934, 303.
Cyclotetracosane 1, 13 dion $C_{24}H_{44}O_2$	Mon.		4		9.91	8.13 $\beta=68^\circ30'$	30.79	1933, 307.
Cyclooctacosane 1, 15 dion $C_{28}H_{52}O_2$	Mon.		4		9.96	8.09 $\beta=68^\circ30'$	35.78	1933, 307.
<i>Sugars, etc.</i>								
l-Arabinose $C_5H_{10}O_5$	Ort.	$V^4$	4		6.48	19.30	4.81	1931, 14, 90; 1932, 113a.
l-Xylose $C_5H_{10}O_5$	Ort.	$V^4$	4		9.21	12.48	5.56	1931, 14, 90.
d-Glucose $C_6H_{12}O_6$	Ort.	( <i>ap</i> )						1931, 423.
d-Mannose $C_6H_{12}O_6$	Ort.	$V^4$	4		5.53	17.66	7.59	1931, 291, 293; 1932, 293, 300.
$\beta$ - $\alpha$ -Galactose $C_6H_{12}O_6$	Ort.	$V^4$	4		12.50	7.67	7.75	1934, 27.
l-Rhamnose Monohydrate $C_6H_{12}O_6 \cdot H_2O$	Mon.	$C_2^2$	2		7.84	7.84 $\beta=95^\circ$	6.61	1931, 14, 90.
d-Mannitol $C_6H_{14}O_6$	Ort.	$V^4$	4	( <i>ap</i> )	8.66	16.58	5.50	1931, 284, 291, 293; 1932, 293, 300.
Dulcitol $C_6H_{14}O_6$	Mon.	$C_{2h}^5$	4		8.61	11.60 $\beta=113^\circ45'$	9.05	1931, 291, 293; 1932, 300.
$\alpha$ -Methyl-l-Arabinoside $C_6H_{12}O_5$	Ort.	$V^4$	4		9.32	16.92	4.68	1933, 108; 1934, 27.

$\beta$ -Methyl-L-Arabinoside $C_6H_{12}O_5$	Ort. Form	4	16.56	7.74	5.89	1933, 108.
$\beta$ -Methyl-L-Arabinoside $C_6H_{12}O_5$	Mon. Form	2	8.96	7.73 $\beta=115^\circ54'$	5.94	1934, 27.
2, 3, 4 Trimethyl- $\delta$ -l-Arabinolactone Ort.		4	10.8	12.2	7.30	1931, 492.
$\alpha$ -Methylxyloside $C_6H_{12}O_5$	Mon.	4	11.28	6.72 $\beta=112^\circ12'$	11.02	1932, 115; 1934, 27.
$\beta$ -d-Methylxyloside $C_6H_{12}O_5$	Mon.	2	7.82	6.89 $\beta=113^\circ10'$	7.74	1932, 113.
2, 3, 4 Trimethyl- $\alpha$ -d-Xylopyranose Mon.		2	8.68	8.31 $\beta=91^\circ0'$	6.65	1931, 492.
$\beta$ -d-Glucosan $C_6H_{10}O_5$	Ort.	4	6.65	13.14	7.46	1933, 108.
Methyl-l-Rhamnoside $C_7H_{14}O_5$	Ort.	4	8.26	13.31	7.54	1934, 27.
$\alpha$ -Methyl-d-Glucoside $C_7H_{14}O_5$	Ort. (asp)	4	11.21	14.57	5.29	1933, 108.
$\beta$ -Methyl-d-Glucoside Hemihydrate $C_7H_{14}O_6 \cdot \frac{1}{2}H_2O$	Tet.	8	7.32		33.6	1933, 108.
1, 3, 4, 5 Tetramethyl- $\beta$ -d-Fructopyranose Ort.		4	9.22	8.97	14.8	1931, 492.
1, 3, 4, 5 Tetraacetyl- $\beta$ -d-Fructopyranose Mon.		2	10.7	7.98 $\beta=144^\circ30'$	17.0	1931, 492.
$\alpha$ -Methylmannoside (m. p. $193^\circ C$ ) $C_7H_{14}O_6$ (pyranose form)	Ort.	4	9.38	9.99	9.23	1932, 118.
$\alpha$ -Methylmannoside (m. p. $118^\circ C$ ) $C_7H_{14}O_6$ (furanose form)	Ort.	4	15.87	11.73	4.64	1932, 118.
$\gamma$ -d-Mannonolactone Ort.		4	14.0	11.1	4.73	1931, 492.

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
2, 3, 5, 6 Tetramethyl- $\gamma$ -d-Mannonolactone Mon.	$C_2^2$	2		9.79	13.8 $\beta=93^\circ 18'$	4.50	1931, 492.
$\gamma$ -Monoacetylmethyl-l-Rhamnoside $C_9H_{16}O_6$ Ort.	$V^4$	4		7.98	18.25	7.08	1933, 108.
Glucose Pentaacetate Ort.		4		24.3	14.9	5.65	1934, 151.
2, 3, 5 Trimethyl- $\gamma$ -l-Rhamnonolactone Ort.	$V^1$	4		12.2	18.3	4.65	1931, 492.
d-Chitosamine Hydrochloride $C_6H_{14}O_5NCl$ Mon.	$C_2^2$	2		7.68	9.18 $\beta=112^\circ 29'$	7.11	1933, 108.
Cellulose $(C_6H_{10}O_5)_n$ (by)							1930, 448; 1931, 21, 82, 491.
Cellulose Hydrate Mon. (by)				8.14	10.3 $\beta=62^\circ$	9.14	1929, 46; 1933, 402.
Cellulose Perchlorate $2C_6H_{10}O_5 \cdot HClO_4$ Mon. (by)				16.5	10.3 $\beta=93^\circ$	10.7	1930, 372.

(bn) A further study of the structure of iodoform,  $\text{CHI}_3$ , using photographic data, has confirmed the previous choice of space group and iodine positions. Carbon atoms are thought to be in  $\frac{1}{3}\frac{2}{3}u$ ;  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $u+\frac{1}{2}$  with  $u$  between 0.50 and 0.60.

(bo) If the space group assigned to these pentaerythritol tetrahalides is correct, their molecules cannot have tetrahedral symmetry.

(bp) Additional spectrometric measurements on urea and their Fourier analyses have led to the following more accurate parameters (see book, p. 373):  $u(\text{C})=0.335$ ,  $v(\text{O})=0.60$ ,  $w(\text{N})=0.145$ ,  $t(\text{N})=0.18$ .

(bq) Spectrometric measurements of intensity and Fourier analyses have been used in a reexamination of the structure of thiourea,  $\text{CS}(\text{NH}_2)_2$ . Atoms are in the positions of  $V_h^{16}$  previously chosen, (ab), with the parameters (the origin in a center of symmetry):

C: (c)  $\pm(uv\frac{1}{4})$ ;  $\pm(\frac{1}{2}-u, v+\frac{1}{2}, \frac{1}{4})$  with  $u=-0.14$ ,  $v=0.10$

S: (c)  $\pm(u_1v_1\frac{1}{4})$ ; etc. with  $u_1=0.120$ ,  $v_1=-0.007$

N: (d)  $\pm(xyz)$ ;  $\pm(x, y, \frac{1}{2}-z)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2})$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, \bar{z})$   
with  $x=0.278$ ,  $y=-0.130$ ,  $z=-0.125$ .

The way the molecules pack together is illustrated by Figure 335. In contrast with the earlier arrangement all the atoms in a molecule lie in one plane. The C-S separation is 1.64 Å;  $\text{NH}_2$  and S of different molecules are 3.45 Å apart. Other interatomic distances are practically the same as in urea.

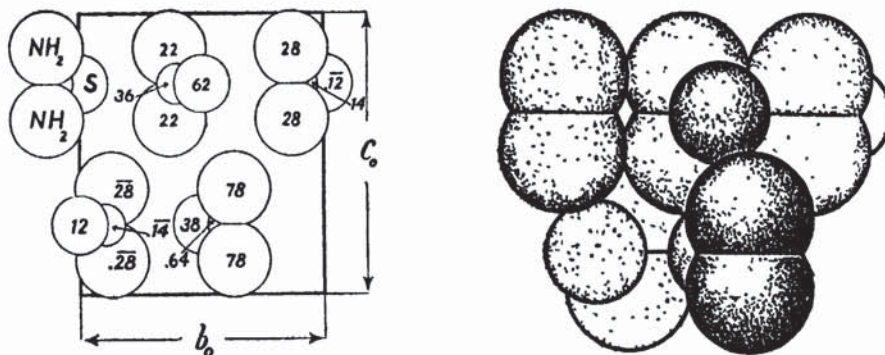


FIG. 335a.—(left) The orthorhombic structure of thiourea projected on the a-face.

FIG. 335b.—(right) A packing drawing of a. The C atoms do not appear.

(br) Spectrometric measurements of crystals of methyl urea,  $\text{CONH}_2 \cdot (\text{NHCH}_3)$ , have been made in an attempt to find its complete structure. All atoms are in general positions of  $V^4$ . Values of  $x$  and  $y$  atomic parameters have been determined from structure factor calculations and Fourier analysis. They account for all the (hk0) reflections but a satisfactory

structure using them in an explanation of the more complicated (h0l) data was not found.

(bs) A structure for the ordinary ( $\alpha$ ) form of **glycine**,  $\text{CH}_2\text{NH}_2\text{COOH}$ , has been deduced which is in good agreement with spectrometric measurements of the simplest reflections but which like that mentioned for methyl urea, (*br*), is unable to explain the more complex intensities. In this proposed arrangement all atoms are in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;  $\pm(\frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z)$  with the parameters of Table V.

It has been reported that X-ray results indicate the reality of the supposed  $\beta$ -modification of glycine but the published evidence for this conclusion is not convincing.

TABLE V. PARAMETERS OF THE ATOMS IN GLYCINE

Atom	No. per cell	$x$	$y$	$z$
O(1)	4	0.42	0.35	0.74
O(2)	4	.44	.47	.63
N	4	.88	.33	.15
C(1)	4	.22	.41	.58
C(2)	4	.12	.40	.26

(bt) By choosing axes in the  $ac$ -plane different from those of Table IV,  $\alpha$ -**glycylglycine** has been given a four-molecule cell with the dimensions  $a_o=7.7$ ,  $b_o=9.56$ ,  $c_o=9.5$ ,  $\beta=125^\circ 20'$  (1931, 41).

(bu) Conflicting cell dimensions have been published for the hexagonal crystals of **l-cystine**. One determination (1931, 41), choosing  $D_6^2$  as space group, finds a six-molecule cell with  $a_o=5.40$  A,  $c_o=57.8$  A. The other with three molecules in its unit has  $a_o=9.40$  A,  $c_o=9.42$  A (1931, 22).

(bv) Recent measurements of the unit cell of the room temperature form of **succinic acid** (1931, 98; 1932, 131) confirm a previous assignment of unit cell.

(bw) The simple hexagonal unit found for **dodecanol** at room temperature is considered to show that its molecules are rotating.

(bx) Positions have been found for the iodine atoms in **1,4 diiodocyclohexane**. They are in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$  with  $x=0.150$ ,  $y=0.135$  or  $0.365$ ,  $z=0.385$ . An earlier space group assignment, of  $C_{2h}^4$  (1931, 161), was wrong for this compound and for the isomorphous dibromide.

(by) Debate over the unit cell and structure of cellulose continues, the symmetry being sometimes treated as tetragonal, sometimes as orthorhombic and sometimes as monoclinic. The last is most probably correct. It is obvious that from the standpoint of sound crystal analysis the arrangements frequently described for cellulose and its derivatives must be considered as speculations, more or less compatible with chemical information, and perhaps not conflicting with the very limited X-ray data at



hand. Assignments of positions to the atoms in rubber, in silk fibroin and in most other macromolecular substances are equally uncertain.

### Aromatic Compounds

Greater progress has been made in determining atomic positions in crystals of aromatic compounds. This is largely due to the fact that their benzene rings provide large building blocks that always have the same size and shape and can enter into the known units in only a limited number of ways.

(bz) It has been found that the iodine atoms in **o-iodobenzoic acid**,  $C_6H_4ICOOH$ , are in general positions of  $C_{2h}^6$ : (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$  with the parameters  $x=0.14$ ,  $y=0.08$ ,  $z=0.02$ . The positions of the other atoms are not known.

(ca) **p-Dibromobenzene**,  $C_6H_4Br_2$ , has its bromine atoms in general positions of  $C_{2h}^6$ : (e)  $\pm(xyz)$ ;  $\pm(\bar{x}, y+\frac{1}{2}, \frac{1}{2}-z)$  with  $x=0.03$ ,  $y=0.167$ ,  $z=0.170$ . For the isomorphous chloride  $x_1=0.04$ ,  $y_1=0.16$ ,  $z_1=0.16$ . Parameters compatible with atomic packing and with observed optical properties have been suggested for the carbon atoms. The available X-ray data are not able to show whether these carbon positions are right.

The diffraction effects of p-bromochlorobenzene,  $C_6H_4BrCl$ , are intermediate between those of the chlorine and bromine derivatives. This presumably means that the molecules go into the structure, with half the bromine atoms pointing one way and with the other half pointing in the opposite direction.

(cb) The **p-diiodobenzene**,  $C_6H_4I_2$ , is not isomorphous with its chloro- and bromo-analogues. Iodine atoms in the orthorhombic unit are in general positions of  $V_h^{15}$ : (c)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, \bar{z})$ ;  $\pm(\bar{x}, y+\frac{1}{2}, \frac{1}{2}-z)$ ;  $\pm(\frac{1}{2}-x, \bar{y}, z+\frac{1}{2})$  with  $x=0.172$ ,  $y=0.40$ ,  $z=0.22$ .

m-Diiodobenzene also is orthorhombic. Iodine atoms are at  $\pm(uv\frac{1}{4})$ ;  $\pm(u\bar{v}\frac{3}{4})$ ;  $\pm(u+\frac{1}{2}, v+\frac{1}{2}, \frac{1}{4})$ ;  $\pm(\frac{1}{2}-u, v+\frac{1}{2}, \frac{1}{4})$  with  $u=0.172$ ,  $v=0.200$ . The space group may be  $C_{2v}^{12}$ .

(cc) A thorough study, including spectrometric measurements and a Fourier analysis, has been made of the crystal structure of **drene**, 1, 2, 4, 5  $C_6H_2(CH_3)_4$ . All atoms are in general positions of  $C_{2h}^6$ : (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$  with the parameters listed in Table VII. This arrangement (Figure 336) gives a molecule that, like  $C_6(CH_3)_6$ , is planar. The packing is, however, a totally different one. In this crystal the nearest approach of atoms belonging to adjacent molecules is relatively large—3.90 Å.

(cd) The x and z parameters of both the chlorine and the carbon atoms in  $C_6Cl_6$  have been selected from a Fourier analysis of the spectrometrically determined (h0l) intensities. Data needed to establish the y parameters could not be obtained so that the structure remains only partly known.

TABLE VI. THE CRYSTAL STRUCTURES OF AROMATIC ORGANIC COMPOUNDS

<i>Substance, symmetry and type</i>	<i>Ort.</i>	<i>(au)</i>	<i>Space group</i>	<i>No. mol. per cell</i>	<i>Min. mol. symmetry</i>	<i>a<sub>0</sub></i>	<i>b<sub>0</sub></i>	<i>c<sub>0</sub></i>	<i>References</i>
Benzene C <sub>6</sub> H <sub>6</sub>	Ort.	(au)							1930, 45a; 1932, 117.
Phenylaminoacetic Acid C <sub>6</sub> H <sub>5</sub> CHNH <sub>2</sub> COOH	Ort.								1931, 354.
d-Phenyl Alanine C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> ·CH(NH <sub>2</sub> )COOH	Ort.		V <sup>4</sup>	8	A	30.8	11.0	4.8	1931, 41.
o-Iodobenzoic Acid C <sub>6</sub> H <sub>4</sub> ICOOH	Mon.	(bz)	C <sub>2h</sub> <sup>5</sup>	4		11.30	15.17 β=90°44'	4.336	1933, 262.
p-Dibromobenzene C <sub>6</sub> H <sub>4</sub> Br <sub>2</sub>	Mon.	(ca)	C <sub>2h</sub> <sup>5</sup>	2	C <sub>i</sub>	4.11	5.80 β=112°38'	15.46	1932, 202.
p-Dichlorobenzene C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>	Mon.	(ca)	C <sub>2h</sub> <sup>5</sup>	2	C <sub>i</sub>	4.10	5.88 β=112°30'	14.83	1932, 202.
p-Bromochlorobenzene C <sub>6</sub> H <sub>4</sub> BrCl	Mon.	(ca)	C <sub>2h</sub> <sup>5</sup>	2	C <sub>i</sub>	4.13	5.81 β=113°0'	15.15	1932, 202.
o-Diiodobenzene C <sub>6</sub> H <sub>4</sub> I <sub>2</sub>	Mon.			4		8.29	12.23 β=93°56'	7.91	1933, 181.
m-Diiodobenzene C <sub>6</sub> H <sub>4</sub> I <sub>2</sub>	Ort.	(cb)	C <sub>2v</sub> <sup>12</sup>	4	C <sub>s</sub>	17.20	7.08	6.21	1933, 181.
p-Diiodobenzene C <sub>6</sub> H <sub>4</sub> I <sub>2</sub>	Ort.	(cb)	V <sub>h</sub> <sup>15</sup>	4	C <sub>i</sub>	17.00	7.38	6.21	1933, 181.
p-Quinone C <sub>6</sub> H <sub>4</sub> O <sub>2</sub>	Mon.	(co)	C <sub>2h</sub> <sup>5</sup>	2	C <sub>i</sub>	7.08	6.79 β=101°0'	5.80	1932, 96; 1934, 228.
Resorcinol m-C <sub>6</sub> H <sub>4</sub> (OH) <sub>2</sub>	Ort.		C <sub>2v</sub> <sup>9</sup>	4		10.53	9.53	5.66	1928, 451; 1934, 229a.
m-Dinitrobenzene C <sub>6</sub> H <sub>4</sub> (NO <sub>2</sub> ) <sub>2</sub>	Ort.	(av)	V <sub>h</sub> <sup>16</sup>	4	C <sub>s</sub>	13.27	14.06	3.820	1931, 171.

1, 2, 6 Dinitrophenol	Ort.	$V_h^{11}$	8	12.1	12.7	9.5	1930, 134.
$C_6H_3OH(NO_2)_2$							
2, 4, 6 Trinitrobromobenzene	Tri.		12*	15.2	15.4	15.64	1933, 185.
$C_6H_2Br(NO_2)_3$				$\alpha=74^\circ, \beta=98^\circ, \gamma=119^\circ$			
2, 4, 6 Trinitrobromobenzene	Hex.		9	14.90		22.6	1933, 185.
$C_6H_2Br(NO_2)_3$							
2, 4, 6 Trinitrochlorobenzene	Mon.	$C_{2h}^3$	8	24.9	6.8	11.0	1933, 185.
$C_6H_2Cl(NO_2)_3$					$\beta=102^\circ 51'$		
Durene	Mon.	$C_{2h}^5$	2	11.57	5.77	7.03	1933, 377, 378.
1, 2, 4, 5 $C_6H_2(CH_3)_4$	(cc)		$C_1$		$\beta=113^\circ 18'$		
2, 4, 6 Trinitroiodobenzene	Tet.	$D_4^{4s}$	4	7.03		19.80	1933, 185.
$C_6H_2I(NO_2)_3$			$C_2$				
1, 2, 4, 6 Tetranitrobenzene	Ort.	$V^4$	4	12.4	6.15	13.1	1933, 185.
$C_6H_2(NO_2)_4$							
2, 4, 6 Trinitrotoluene	Mon.	$C_{2h}^6$		40.5	6.19	15.2	1933, 186.
$C_6H_2CH_3(NO_2)_3$	(ck)				$\beta=89^\circ 29'$		
2, 4, 6 Trinitroaniline	Mon.	$C_{2h}^6$	4	15.3	9.28	6.01	1933, 185.
$C_6H_2(NH_2)(NO_2)_3$					$\beta=99^\circ 12'$		
4 Nitro-2-Methylaminotoluene	Mon.	$C_{2h}^6$	4	17.2	12.2	3.83	1932, 211.
(yellow form)					$\beta=102^\circ$		
4 Nitro-2-Methylaminotoluene	Tri.		2	7.6	8.5	7.5	1932, 211.
(red form)				$\alpha=113^\circ, \beta=98^\circ, \gamma=109^\circ$			
Styphnic Acid	Hex.	$C_{3v}^3$ or $D_{3d}^3$	6	12.7		10.0	1931, 190.
$C_6H(OH)_2(NO_2)_3$							
Hexachlorobenzene	Mon.	$C_{2h}^6$	2	8.07	3.84	16.61	1931, 283.
$C_6Cl_6$	(cd)				$\beta=116^\circ 52'$		
Hexaminobenzene	Cub.	$O_h^1$	16	14.84	at $-183^\circ C$		1931, 261.
$C_6(NH_2)_6$							

\* A triclinic unit containing as many molecules as 12 cannot possibly be the true one.

<i>Substance, symmetry and type</i>	<i>Space group</i>	<i>No. mol. per cell</i>	<i>Min. mol. symmetry</i>	<i>a<sub>0</sub></i>	<i>b<sub>0</sub></i>	<i>c<sub>0</sub></i>	<i>References</i>
2, 4, 6 Trinitrochloroglucinol $C_6(OH)_3(NO_2)_3$	(cl) $C_{3v}^4$ or $D_{3d}^4$	6		13.4		9.6	1931, 190.
Quinhydrone $C_6H_4O_2 \cdot C_6H_4(OH)_2$	(ce) $C_{2h}^5$	1		3.85	6.04 $\beta = 90^\circ$	10.9	1932, 150.
p-Aminoazobenzene $C_6H_5N = NC_6H_4(NH_2)$	Mon.	4		13.69	5.604 $\beta = 81^\circ 49'$	14.18	1933, 365.
o-Azotoluene $(CH_3)C_6H_4N = NC_6H_4(CH_3)$	Mon.	4		13.93	6.604 $\beta = 79^\circ 9'$	14.55	1932, 368.
2, 4, 6 Trinitrodiphenylamine $C_6H_5(NH)C_6H_2(NO_2)_3$	Mon.	8	A	22.0	7.8 $\beta = 107^\circ$	16.2	1931, 189.
Aniline Picrate $C_6H_5NH_2 \cdot \cdot \cdot (OH)C_6H_2(NO_2)_3$	Mon.	4		13.2	7.4 $\beta = 93^\circ$	15.2	1931, 189.
Ethyl Anisal p-Amino Cinnamate $C_{19}H_{16}NO_3$	Mon.	4		6.65	7.88 $\beta = 135^\circ 35'$	45.6	1933, 40a.
p-Azoxyanisole* $C_{14}H_{14}N_2O_3$	Mon.	4		11.0	8.10 $\beta = 107^\circ 30'$	14.95	1933, 40a; 1934, 302.
p-Azoxyphenetole $C_{18}H_{18}N_2O_3$	Mon.	4		15.4	5.41 $\beta = 94^\circ$	17.6	1933, 40a.
Dibenzylidenebenzidine	Mon.	2	$C_1$	5.9	7.7 $c \sin \beta = 21.5 \text{ \AA}$		1933, 40a.
Dibenzyl $C_6H_5CH_2 - CH_2C_6H_5$	(cm) Mon.	2	$C_1$	12.77	6.12 $\beta = 116^\circ$	7.70	1934, 70, 227, 229.
Stilbene $C_6H_5CH = CHC_6H_5$	(cm) Mon.	4		12.20	5.72 $\beta = 113^\circ 48'$	16.00	1933, 364.

Tolane	Mon.	(cm)	4	12.80	5.68	15.74	1933, 364.
$C_6H_5C \equiv CC_6H_5$					$\beta = 114^\circ 56'$		
p-Nitrostilbene	Ort.		8	7.94	28.3	10.22	1931, 188.
$C_6H_5CH = CHC_6H_4(NO_2)$							
p-Cyano-o-Nitro-p'-Methoxystilbene	$V_h^1, V_h^6$ or $C_{2v}^{II}$		8	14.2	27.8	7.6	1932, 212.
$C_6H_3(CN)(NO_2)CH = CHC_6H_4(OCH_3)$ (metastable yellow form)	Ort.						
p-Cyano-o-Nitro-p'-Methoxystilbene	2		2	8.50	7.45	13.35	1932, 212.
$C_6H_3(CN)(NO_2)CH = CHC_6H_4(OCH_3)$ (orange form)	Tri.			$\alpha = 98^\circ 6', \beta = 106^\circ 20', \gamma = 75^\circ 40'$			
Stilbene+2 mol. 1, 3, 5 Trinitrobenzene	2		2	12.7	15.4	7.7	1931, 192.
$C_6H_5CH = CHC_6H_5 \cdot 2[C_6H_3(NO_2)_3]$	Tri.			$\alpha = 102^\circ 16', \beta = 85^\circ 30', \gamma = 87^\circ 35'$			
Diphenylbutadiene	Mon.		4	7.71	11.70	13.41	1930, 401.
$C_6H_5CH = (CH)_2 = CHC_6H_5$				$C_{2h}^1$ or $C_{2h}^2$	$\beta = 97^\circ$		
Diphenylhexatriene	Mon.		2	6.63	7.43	14.43	1930, 401.
$C_6H_5CH = (CH-CH)_2 = CHC_6H_5$				$C_{2h}^5$	$\beta = 90^\circ$		
Diphenyloctatetraene	Mon.		2	6.25	7.44	16.03	1930, 401.
$C_6H_5(CH = CH)_4C_6H_5$				$C_{2h}^6$	$\beta = 90^\circ$		
Diphenyldecapentaene	Ort.		4	10.25	7.66	21.2	1930, 401.
$C_6H_5(CH = CH)_5C_6H_5$				$V_h^{18}$			
Diphenyldodecahexaene	Ort.		4	10.20	7.60	23.58	1930, 401.
$C_6H_5(CH = CH)_6C_6H_5$							
Diphenyltetradecaheptaene	Ort.		4	10.2	7.57	25.95	1930, 401.
$C_6H_5(CH = CH)_7C_6H_5$							
Diphenyl Disulfide	Ort.		4	23.5	8.21	5.63	1932, 137, 188.
$(C_6H_5S)_2$				$V^4$			
Diphenyl Diselenide	Ort.		4	23.70	8.25	5.64	1932, 137, 188.
$(C_6H_5Se)_2$				$V^4$			

\* In 1934,  $302, s_o = 16.0$  A,  $b_o = 8.08$  A,  $c_o = 20.5$  A,  $\beta = 107^\circ 30'$ ,  $C_{2h}^4$ , 8 mol.

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
Dibenzyl Disulfide (C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> S) <sub>2</sub>	C <sub>2h</sub> <sup>6</sup> or C <sub>s</sub> <sup>1</sup>	4		13.46	8.23 β=99°30'	11.29	1932, 137.
Dibenzyl Diselenide (C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Se) <sub>2</sub>	C <sub>2h</sub> <sup>6</sup> or C <sub>s</sub> <sup>1</sup>	4		13.50	8.17 β=99°30'	11.57	1932, 137.
Dibenzoyl Disulfide (C <sub>6</sub> H <sub>5</sub> COS) <sub>2</sub>	C <sub>2h</sub> <sup>6</sup>	4		12.26	12.00 β=107°40'	9.04	1932, 137.
Diphenyl Nitrosoamine C <sub>6</sub> H <sub>5</sub> N—NO—C <sub>6</sub> H <sub>5</sub>	C <sub>2h</sub> <sup>3</sup>	16	A	17.08	8.867 <sub>s</sub> β=90°58'	28.07	1933, 366.
Diphenyl C <sub>6</sub> H <sub>5</sub> ·C <sub>6</sub> H <sub>5</sub>	( <i>ax</i> '), ( <i>cf</i> )						1931, 84, 354; 1932, 129; 1933, 359.
Diphenic Acid (COOH·C <sub>6</sub> H <sub>4</sub> ) <sub>2</sub>	V <sub>h</sub> <sup>13</sup>	8		13.80	11.90	14.12	1931, 84.
Hexachlorodiphenyl (C <sub>6</sub> H <sub>2</sub> Cl <sub>3</sub> ) <sub>2</sub>	V <sub>h</sub> <sup>16</sup>	8		15.80	8.54	21.48	1931, 84.
<i>o</i> -Tolidine (C <sub>6</sub> H <sub>3</sub> CH <sub>3</sub> NH <sub>2</sub> ) <sub>2</sub>							1931, 84.
Naphthalene C <sub>10</sub> H <sub>8</sub>	( <i>bb</i> ), ( <i>cg</i> )	2	C <sub>1</sub>	8.29	5.97 β=122°42'	8.68	1933, 379.
1 Brom-2-Naphthylamine C <sub>10</sub> H <sub>6</sub> Br(NH <sub>2</sub> )		4		12.8	15.9	4.2	1931, 191.
4 Brom-1-Naphthylamine C <sub>10</sub> H <sub>6</sub> Br(NH <sub>2</sub> )		8		25.2	16	4.2	1931, 191.
4 Brom-1-Naphthylamine, 2, 6 Dinitrophenol (red form) C <sub>10</sub> H <sub>6</sub> Br(NH <sub>2</sub> ), C <sub>6</sub> H <sub>3</sub> OH(NO <sub>2</sub> ) <sub>2</sub>	C <sub>2h</sub> <sup>5</sup>	4		9.5	13.5 β=105°	13.8	1931, 191.

Mon.

4 Brom-1-Naphthylamine, 2, 6 Dinitrophenol (yellow form) $C_{10}H_6Br(NH_2)$ , $C_6H_3OH(NO_2)_2$	4	$C_{2h}^5$	14.0	8.0 $\beta=102^\circ 6'$	14.5	1931, 191.
1, 2 Naphthoquinone $C_{10}H_6O_2$	2	$C_2^5$	3.84	8.10 $\beta=118^\circ 40'$	13.40	1932, 96.
1, 4 Naphthoquinone $C_{10}H_6O_2$	4	$C_{2h}^5$	13.50	7.74 $\beta=121^\circ 10'$	8.25	1932, 96.
Naphthazarin (Alizarin Black)	2	$C_{2h}^5$	3.85	8.02 $\beta=97^\circ 6'$	14.5	1934, 201.
Acenaphthene, Styphnic Acid $C_{10}H_6(CH_2)_2$ , $C_6H(OH)_2(NO_2)_2$	2		9.05	14.8 $\beta=99^\circ$	6.8	1932, 412.
Anisal 1, 5 Diaminonaphthalene $C_{10}H_{12}O_2N_2$	4	$C_{2h}^5$	21.7	12.7 $\beta=119^\circ 30'$	7.7	1933, 40a.
Anthracene $C_{14}H_{10}$	2	$C_{2h}^5$	8.58	6.02 $\beta=125^\circ$	11.18	1933, 375, 376, 380.
Anthraquinone $C_{14}H_8O_2$	8		19.7	24.5	3.95	1930, 405.
1, 2 Anthraquinone $C_{14}H_8O_2$	4	$C_{2h}^5$	11.41	11.56 $\beta=130^\circ 30'$	9.30	1932, 96.
1, 4 Anthraquinone (tabular form) $C_{14}H_8O_2$	2	$C_2^5$	4.19	5.81 $\beta=101^\circ 30'$	19.62	1932, 96.
1, 4 Anthraquinone (acicular form) $C_{14}H_8O_2$	4	$C_{2h}^5$	13.82	9.54 $\beta=100^\circ 50'$	7.31	1932, 96.
2, 7 Dinitroanthraquinone $C_{14}H_6(NO_2)_2O_2$	4		5.7		38.4	1930, 405.
2, 7 Dinitroanthraquinone Fluorene $C_{14}H_6(NO_2)_2O_2$ , $(C_6H_4)_2CH_2$	2		8.2	7.4 $\alpha=78^\circ$ , $\beta=82^\circ$ , $\gamma=ca. 80^\circ$	19.0	1930, 405.

\* Optical examination makes this crystal monoclinic.

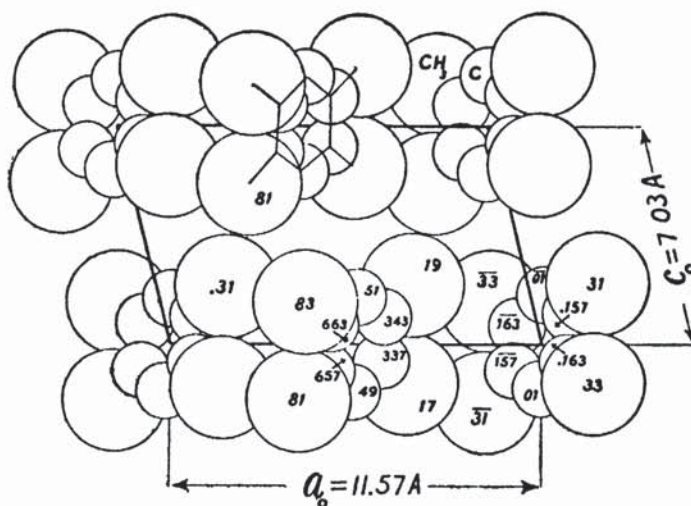
Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
Mesanthraquinone	$V_6^6$	8		19.65	24.57	4.00	1932, 96.
p-Diphenylbenzene (Terphenyl)	$C_{2h}^6$	2	$C_1$	8.08	5.60	13.59	1933, 292, 359, 360.
$C_6H_5(C_6H_4)_2C_6H_5$ Mon.					$\beta=91^\circ55'$		
Chrysenes $C_{18}H_{12}$	$C_{2h}^6$ or $C_1^4$	4		8.34	6.18	25.0	1933, 214; 1934, 117.
Quaterphenyl $C_6H_5(C_6H_4)_2C_6H_5$	$C_{2h}^5$	2	$C_1$	8.14	5.64	18.4	1933, 187, 359.
Triphenylbenzene $C_6H_5(C_6H_5)_3$	$C_{2v}^9$	4		7.55	$\beta=97^\circ$	11.22	1933, 187; 1934, 157, 194.
1, 2, 5, 6 Dibenzoanthracene	$C_{2h}^2$ or $C_2^2$	2		6.59	7.84	14.17	1933, 214.
Dianthracene $(C_{14}H_{10})_2$	$V_h^{18}$	4	$C_1$	8.18	12.15	18.75	1932, 207.
$\gamma, \gamma'$ -Dibenzocarbazole		4		14.05	12.02	8.40	1931, 113.
Dimesityl	$C_{2h}^6$	4		8.21	$\beta=114^\circ14'$	22.25	1930, 59; 1931, 84.
3, 3'-Diaminodimesityl	$C_{2h}^6$	4		8.26	$\beta=96^\circ30'$	22.62	1930, 59; 1931, 84.
					$\beta=90^\circ$		



TABLE VII. PARAMETERS OF THE CARBON ATOMS IN DURENE

Atom	No. per cell	$x$	$y$	$z$
C(1)	4	0.188	0.314	0.267
C(2)	4	.093	.157	.127
C(3)	4	.037	-.005	.212
C(4)	4	-.055	-.162	.090
C(5)	4	-.108	-.325	.194

FIG. 336.—A projection upon the  $b$ -face of molecules in the monoclinic structure of durene,  $C_6H_2(CH_3)_4$ . The sizes given C and  $CH_3$  in this drawing are without real significance.



All atoms are in general positions of  $C_{2h}^5$  (not  $C_{2h}^4$ , as previously chosen): (e)  $\pm(xyz)$ ;  $\pm(x, \frac{1}{2}-y, z+\frac{1}{2})$ . The  $x$  and  $z$  parameters are given in Table VIII.

TABLE VIII. PARAMETERS OF THE ATOMS IN  $C_6Cl_6$ 

Atom	$x$	$z$	Atom	$x$	$z$
C(1)	0.181	0.026	Cl(1)	0.412	0.070
C(2)	.118	.087	Cl(2)	.278	.205
C(3)	.048	-.062	Cl(3)	.133	-.137

(ce) A structure has been proposed for **quinhydrone**,  $C_6H_4O_2 \cdot C_6H_4(OH)_2$ , which makes the oxygen atoms and hydroxyl groups equivalent. Though parameters have been published adequate data in support of them are lacking.

(cf) A partially described and tested structure has been suggested for **diphenyl**,  $C_6H_5 \cdot C_6H_5$ . No atomic coordinates have been published. The orientation of its molecule within the unit may be reproduced by rotating a planar  $(C_6H_5)_2$  molecule having its center at a center of symmetry and its plane in  $bc$ . This rotation amounts to  $32^\circ$  about the  $c$ -axis and  $20^\circ$  about the  $b$ -axis.

(cg) A complete determination based upon quantitative intensity data and their Fourier analysis has been made of the atomic arrangement in **naphthalene**,  $C_{10}H_8$ . All atoms are in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;

$\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$  with the parameters of Table IX. The molecules of this grouping pack together as shown in Figure 337. Atoms of neighboring molecules come within 3.60 Å of one another.

TABLE IX. PARAMETERS OF THE ATOMS  
IN NAPHTHALENE

Atom	$x$	$y$	$z$
C(1)	0.087	0.014	0.328
C(2)	.114	.162	.217
C(3)	.047	.104	.035
C(4)	.074	.251	-.078
C(5)	.007	.193	-.260

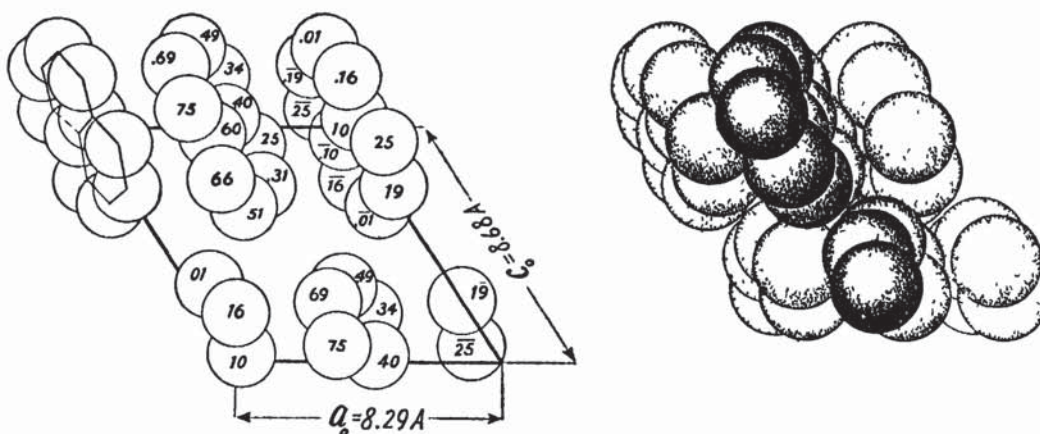


FIG. 337a.—(left) A projection of a portion of the monoclinic structure of naphthalene,  $C_{10}H_8$ , upon its b-face. The circles represent C atoms. Only parts of the molecules belonging to the bottom half of the cell are shown.

FIG. 337b.—(right) A packing drawing of *a* indicating the way the naphthalene molecules contact with one another.

(*ch*) The structure of **anthracene**,  $C_{14}H_{10}$ , also has been completely worked out from a series of spectrometer measurements and their Fourier analysis. Like naphthalene the atoms of its two-molecule monoclinic cell are in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$ . The parameters are those of Table X. The close similarity that exists between this anthracene arrangement (Figure 338) and the naphthalene grouping (Figure 337) may be most easily seen by comparing the two figures. In anthracene contacting molecules are slightly farther apart, the nearest intermolecular atomic separation being 3.77–3.80 Å.

(*ci*) Another ring structure, established from quantitative data, is that of **p-diphenylbenzene**,  $C_6H_5(C_6H_4)C_6H_5$ . With its atoms also in general positions of  $C_{2h}^5$ : (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z)$  (parameters in Table XI) this arrangement (Figure 339) is very similar to the two preceding. It

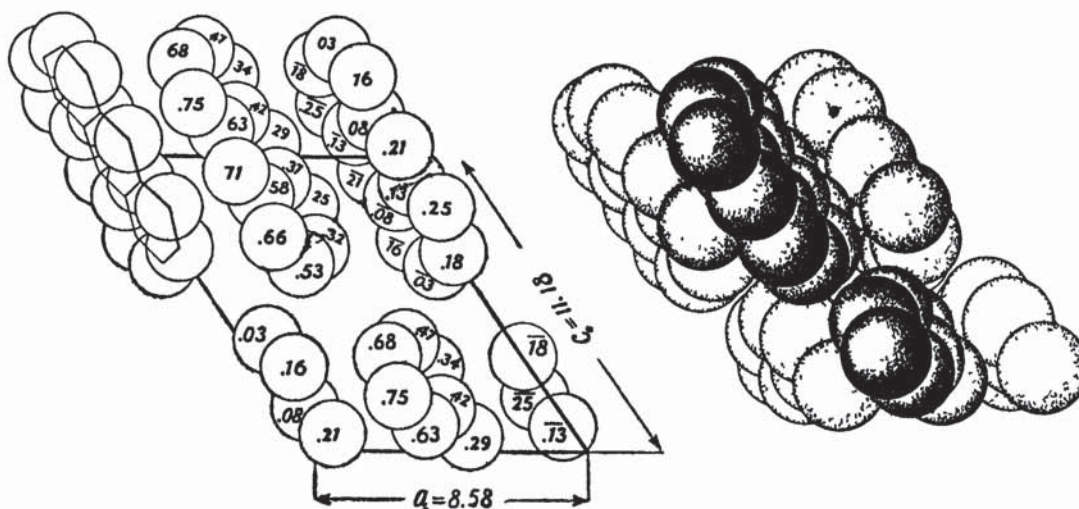


FIG. 338a.—(left) A portion of the monoclinic structure of anthracene,  $C_{14}H_{10}$ , projected on a  $b$ -face of its monoclinic unit. The close similarity between the arrangements found for naphthalene and anthracene may be seen by comparing Figures 337 and 338.

FIG. 338b.—(right) A packing drawing of  $a$ . In this and Figure 337b the radius chosen for the atomic spheres is that suggested by the closest approach of C atoms in adjacent molecules.

TABLE X. PARAMETERS OF THE ATOMS IN ANTHRACENE

Atom	$x$	$y$	$z$	Atom	$x$	$y$	$z$
C(1)	0.094	0.032	0.369	C(5)	0.033	0.130	-0.089
C(2)	.124	.157	.279	C(6)	.065	.254	-.179
C(3)	.062	.082	.140	C(7)	.002	.177	-.319
C(4)	.095	.207	.050				

TABLE XI. PARAMETERS OF THE ATOMS IN P-DIPHENYLBENZENE

Atom	$x$	$y$	$z$	Atom	$x$	$y$	$z$
C(1)	0.059	0.182	0.064	C(6)	-0.082	0.182	0.368
C(2)	-.046	0	.100	C(7)	-.187	0	.402
C(3)	-.105	-.182	.036	C(8)	-.246	-.182	.339
C(4)	-.094	0	.204	C(9)	-.200	-.182	.239
C(5)	-.036	.182	.268				

differs from them mainly in the fact that the long axes of its molecules are parallel to the  $ac$ -plane, whereas those of  $C_{10}H_8$  and  $C_{14}H_{10}$  are tilted at considerable angles.

Diphenyl,  $C_6H_5 \cdot C_6H_5$ , *p*-diphenylbenzene,  $C_6H_5(C_6H_4)C_6H_5$ , and quaterphenyl,  $C_6H_5(C_6H_4)_2C_6H_5$ , have nearly equal  $a_0$  and  $b_0$  axes and  $\beta$  angles. The molecules must therefore be similarly oriented in their crystals.

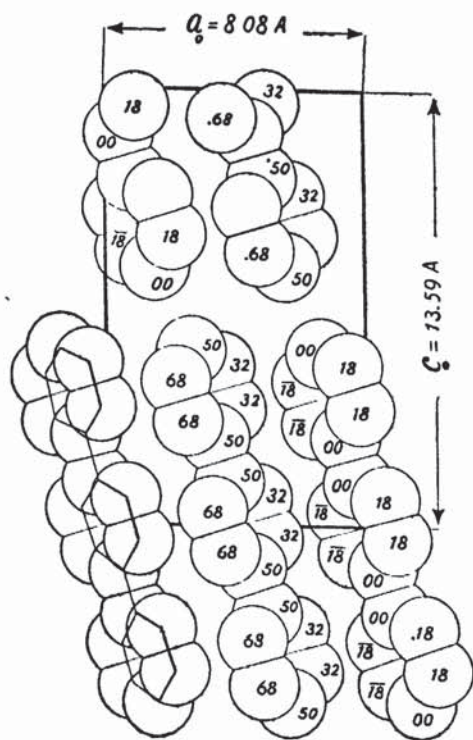


FIG. 339.—(left) A portion of the structure of p-diphenylbenzene,  $C_6H_4(C_6H_5)_2$ , projected on the b-face of its monoclinic unit. As in the figures immediately preceding circles represent C atoms. Only parts of the molecules associated with the upper half of the unit are included.

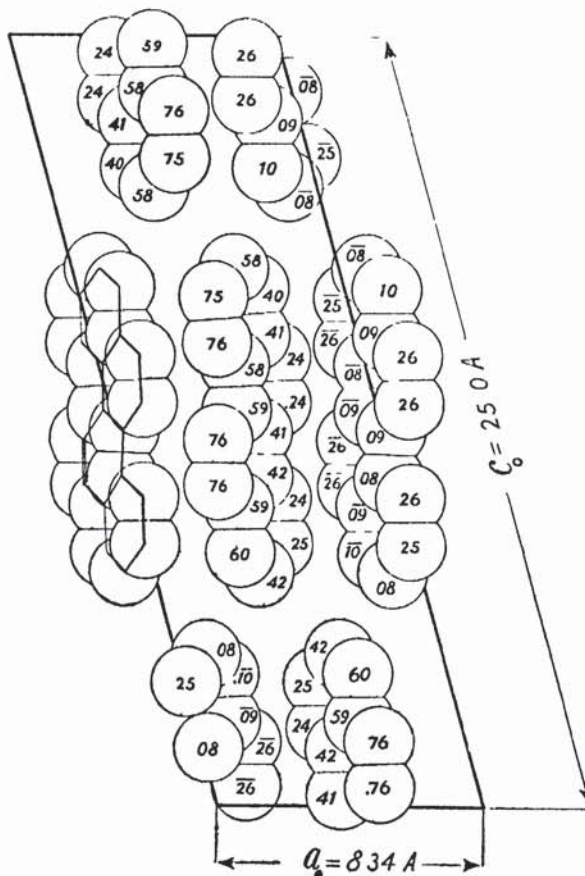


FIG. 340.—(right) A portion of the chrysene,  $C_{18}H_{14}$ , structure projected on its b-face. Only the molecules midway along the c-axis are fully shown.

TABLE XII. PARAMETERS OF THE ATOMS IN CHRYSENE

Atom	No. per cell	$x$	$y$	$z$
C(1)	8	-0.026	0.086	0.013
C(2)	8	.018	.084	.074
C(3)	8	-.036	.256	.100
C(4)	8	.010	.254	.161
C(5)	8	.108	.079	.196
C(6)	8	.161	-.095	.171
C(7)	8	.116	-.089	.110
C(8)	8	.170	-.262	.084
C(9)	8	.125	-.260	.023

(*cj*) **Chrysene**,  $\begin{array}{c} \text{C}_{10}\text{H}_6\text{---CH} \\ | \\ \text{C}_6\text{H}_4\text{---CH} \end{array}$  || , differs from the preceding aromatic hydro-

carbons in having a four-molecule arrangement developed from  $C_{2h}^6$ . All atoms are in the general positions: (f)  $\pm(xyz)$ ;  $\pm(\bar{x}, y, \frac{1}{2}-z)$ ;  $\pm(x+\frac{1}{2}, y+\frac{1}{2}, z+\frac{1}{2})$ ;  $\pm(\frac{1}{2}-x, y+\frac{1}{2}, \bar{z})$ . The parameters of Table XII, deduced from spectrometric measurements of intensity, lead to the symmetrical ring formula outlined in one of the molecules of Figure 340. Atoms of adjacent chrysene molecules come especially close to one another (ca 3.5 Å).

(*ck*) It is said that there exists a 1:1 compound of 2, 4, 6  $C_6H_2Cl(NO_2)_3$  and 2, 4, 6  $C_6H_2ClI_3(NO_2)_3$  which has cell dimensions identical with those of 2, 4, 6  $C_6H_2CH_3(NO_2)_3$ .

(*cl*) It is interesting that styphnic acid,  $C_6H(NO_2)_3(OH)_2$ , and 2, 4, 6 trinitrophenol,  $C_6H_2(NO_2)_3(OH)_3$ , which differ by an OH group, should have similar crystal structures.

(*cm*) It has been pointed out (1933, 364) that azobenzene,  $C_6H_5N=NC_6H_5$ , stilbene,  $C_6H_5CH=CHC_6H_5$ , and toluene,  $C_6H_5C\equiv CC_6H_5$  have cells of nearly the same shape and size.

The unit of dibenzyl,  $C_6H_5CH_2-CH_2C_6H_5$ , differs only in having a *c*-axis reported to be half as long. Two structures have been proposed for this crystal. One of them gives a molecule that is almost planar. The molecule of the other has its two phenyl groups stepped with respect to one another but lying in parallel planes. Unfortunately the atomic parameters for the second arrangement have not been given.

(*cn*) From a preliminary study of 1, 3, 5 triphenylbenzene,  $C_6H_3(C_6H_5)_3$ , it has been concluded that the molecular centers are in general positions of  $C_{2v}^9$ : (a)  $xyz$ ;  $\bar{x}, \bar{y}, z+\frac{1}{2}$ ;  $x+\frac{1}{2}, \frac{1}{2}-y, z$ ;  $\frac{1}{2}-x, y+\frac{1}{2}, z+\frac{1}{2}$  with  $z=0$ . Atomic parameters have not yet been found.

(*co*) A preliminary note, without a complete description of atomic positions, has recently been published on *p*-quinone,  $C_6H_4O_2$ .

### Recent Aliphatic Structures

(*cp*) Laue and spectral photographs have been used to assign positions to the atoms in oxalic acid dihydrate,  $(COOH)_2 \cdot 2H_2O$ . The space group is  $C_{2h}^6$  with two molecules per cell. Writing the coordinates of the general positions of this space group as (e)  $\pm(xyz)$ ;  $\pm(x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2})$ , the chosen atomic parameters are those of Table XIII. As can be seen from Figure 341, each water molecule is nearly equally distant (2.60–2.87 Å) from three oxygen atoms. Within a molecule C-C=1.59 Å, C-O=1.25 Å; the angle between C-O bonds is 126°.

TABLE XIII. PARAMETERS OF THE ATOMS IN  
(COOH)<sub>2</sub>·2H<sub>2</sub>O

Atom	<i>x</i>	<i>y</i>	<i>z</i>
C	-0.011	0.041	0.056
O(1)	.089	-.062	.150
O(2)	-.222	.222	.041
H <sub>2</sub> O	-.444	-.375	.174

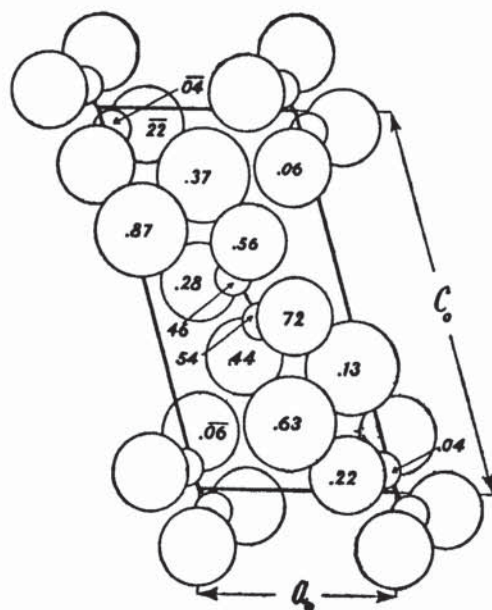



FIG. 341.—A b-face projection of the molecules of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O associated with its monoclinic unit. The large circles are H<sub>2</sub>O molecules. The O and OH of carboxyl groups, which are equivalent in this structure, are shown by the intermediate circles. The radii used in this figure were determined by convenience only.

(*cq*) A spectrometric study of hexamethylenetetramine, C<sub>6</sub>H<sub>12</sub>N<sub>4</sub>, and a Fourier analysis of the intensities thus obtained are in complete agreement with one of the earlier investigations of this crystal. Carbon atoms are in (12a) *v*00; etc. [(*as*), book, p. 389], nitrogen in (8a) *uuu*; etc. with the same parameters: *u*=0.12, *v*=0.23.\*

\* On p. 390 of the book, values of *u* and *v* have been erroneously interchanged.

TABLE XIV. THE CRYSTAL STRUCTURES OF MISCELLANEOUS ORGANIC COMPOUNDS

Substance, symmetry and type	Space group	No. mol. per cell	Min. mol. symmetry	$a_0$	$b_0$	$c_0$	References
$\beta$ -Polyoxymethylene	$C_2^2$ or $C_3^2$			4.46		17.35	1927, 126; 1932, 397, 406.
Eu-Polyethylene Oxide ( $CH_2CH_2O$ ) <sub>x</sub> (mol. wt. = ca 100,000)	$C_{2h}^4$	36 (=x)		9.5	19.5 $\beta = 101^\circ$	12.0	1933, 405.
Hexuronic Acid $C_6H_8O_6$	$C_2^2$	4	A	17.71	6.32 $\beta = 102^\circ 30'$	6.38	1932, 116.
Diketopiperazine	$C_{2h}^5$	2	$C_1$	5.19	11.5 $\beta = 83^\circ$	3.96	1931, 41.
							
$C_4H_2N_4O_3$ ON=CHCH=CN(NO)N=CO		4		7.38	4.92 $\beta = 105^\circ 34'$	17.45	1931, 113.
Cyanuric Triazide $C_3N_3(N_3)_3$		2		8.66		5.94	1933, 457; 1934, 28.
Methylbixin $C_{28}H_{32}O_4$	$C_{2h}^6$	4		10.56	13.40 $\beta = 121^\circ$	20.62	1930, 402; 1932, 458.
Choleic Acid*	Ort.			25.9	13.69	7.20	1931, 199
Thiophene $C_4H_4S$	Tet.		(ar)				1930, 45a.
Monomeric Butadiene Sulfone $C_4H_6SO_2$	$C_{2h}^5$	4		9.55	11.35 $\beta = 48^\circ 46'$	6.23	1932, 398.
Monomeric Isoprene Sulfone $C_4H_8SO_2$	$C_2^2$	2		6.60	7.62 $\beta = 69^\circ 26'$	6.67	1932, 398.

\* If the formula of choleic acid is written as  $8 C_{24}H_{40}O_4 + 1$  mol. palmitic or stearic acid (cf. Wieland and Sorge), this cell contains only half a molecule.

<i>Substance, symmetry and type</i>	<i>Space group</i>	<i>No. mol. per cell</i>	<i>Min. mol. symmetry</i>	<i>a.</i>	<i>b.</i>	<i>c.</i>	<i>References</i>
Monomeric Dimethyl Butadiene Sulfone $C_6H_{10}SO_2$ Ort.	$V_h^{18}$	4	$C_s$	7.55	12.12	7.39	1932, 398.
4 Isoxazolyl-5-Isoxazolyl Ketone $C_7H_4N_2O_4$ Mon.		16	A	15.31	14.75 $\beta=91^\circ37'$	12.77	1931, 113.
3, 4 Bis (5 Methyl-3-Isoxazolylcarbonyl) Furazan-2-Oxide $C_{12}H_8N_4O_6$ Ort.		4		12.98	11.00	9.57	1931, 113.
<hr/>							
5, 5 Diethyl Barbituric Acid (Veronal) $C_8H_{12}N_2O_3$ Ort.	$V_h^{17}$	4	$C_2^i$	7.11	14.4	9.7	1930, 404.
Veramon (Pyramidon, Veronal 1:1) Pyramidon	$C_{2v}^2$ or $C_{2v}^4$	4		27.1	12.2	7.20	1931, 187.
		4		7.4	18.1	10.8	1931, 187.
				$\alpha, \beta, \gamma$ not determined			
l-Ephedrine Hydrobromide $C_{10}H_{15}ON, HBr$ Mon.	$C_2^i$	2		12.74	6.20	7.62	1933, 155.
l-Ephedrine Hydrochloride $C_{10}H_{15}ON, HCl$ Mon.	$C_2^i$	2		12.64	6.15	7.34	1933, 155.
				$\beta=100^\circ48'$			
l-Ephedrine Hydroiodide $C_{10}H_{15}ON, HI$ Ort.	$V^3$	12	A	25.66	7.33	19.14	1933, 155.
				$\beta=102^\circ6'$			
Racemic Ephedrine Hydrobromide $C_{10}H_{15}ON, HBr$ Mon.	$C_{2h}^5$	4		13.15	7.11	14.00	1933, 154.
Racemic Ephedrine Hydrochloride $C_{10}H_{15}ON, HCl$ Mon.	$C_{2h}^5$	4		13.27	7.04	13.44	1933, 154.
				$\beta=119^\circ25'$			
Racemic Ephedrine Hydroiodide $C_{10}H_{15}ON, HI$ Mon.	$C_{2h}^5$	4		13.40	7.23	14.70	1933, 154.
d-Pseudoephedrine Hydrobromide $C_{10}H_{15}ON, HBr$ Ort.	$V^4$	4		24.68	6.93	6.78	1933, 155.
				$\beta=120^\circ24'$			



d-Pseudoephedrine Hydrochloride C <sub>10</sub> H <sub>13</sub> ON, HCl Ort.	V <sup>4</sup>	4	25.49	6.48	6.91	1933, 155.
d-Pseudoephedrine Hydroiodide C <sub>10</sub> H <sub>13</sub> ON, HI Ort.	V <sup>4</sup>	4	11.39	6.83	15.62	1933, 155.
Racemic Pseudoephedrine Hydrobromide C <sub>10</sub> H <sub>13</sub> ON, HBr Mon.	C <sub>2h</sub> <sup>5</sup>	4	13.87	6.80 β = 116°51'	14.04	1933, 154.
Racemic Pseudoephedrine Hydrochloride C <sub>10</sub> H <sub>13</sub> ON, HCl Ort.	V <sub>h</sub> <sup>1s</sup>	16	24.48	9.97	18.58	1933, 154.
Racemic Pseudoephedrine Hydroiodide C <sub>10</sub> H <sub>13</sub> ON, HI Mon.	C <sub>2h</sub> <sup>5</sup>	4	13.49	6.97 β = 114°26'	14.62	1933, 154.
d-Pseudococaine-l-Ephedrine-d-Tartrate Monohydrate C <sub>21</sub> H <sub>27</sub> O <sub>11</sub> N <sub>2</sub> ·H <sub>2</sub> O Mon.		2	18.90	9.92	9.36	1932, 82.
d-Pseudococaine-l-Methyl Ephedrine-d-Tartrate Dihydrate C <sub>32</sub> H <sub>44</sub> O <sub>11</sub> N <sub>2</sub> ·2H <sub>2</sub> O Mon.		2	19.62	9.76 β = 98°42'	9.61	1932, 82.
l-Methyl Ephedrine Hydrobromide C <sub>11</sub> H <sub>17</sub> ON, HBr Ort.	V <sup>4</sup>	4	17.30	7.36	9.64	1934, 330.
l-Methyl Ephedrine Hydrochloride C <sub>11</sub> H <sub>17</sub> ON, HCl Ort.	V <sup>4</sup>	4	16.90	7.22	9.66	1934, 330.
l-Methyl Ephedrine Hydroiodide C <sub>11</sub> H <sub>17</sub> ON, HI Ort.	V <sup>3</sup>	8	6.10	31.60	13.25	1934, 330.
Racemic Methyl Ephedrine Hydrobromide (1st mod.) C <sub>11</sub> H <sub>17</sub> ON, HBr Mon.	C <sub>2h</sub> <sup>5</sup>	4	17.28	7.30 β = 102°28'	10.16	1934, 330.
Racemic Methyl Ephedrine Hydrobromide (2nd mod.) C <sub>11</sub> H <sub>17</sub> ON, HBr Mon.	C <sub>2h</sub> <sup>6</sup>	4	5.83	30.78 β = 107°8'	7.02	1934, 330.
Racemic Methyl Ephedrine Hydrochloride C <sub>11</sub> H <sub>17</sub> ON, HCl Mon.	C <sub>2h</sub> <sup>5</sup>	4	17.15	7.08 β = 101°30'	10.01	1934, 330.
Racemic Methyl Ephedrine Hydroiodide C <sub>11</sub> H <sub>17</sub> ON, HI Tri.		2	11.2	7.67 α = 108°55', β = 95°36', γ = 84°15'	7.68	1934, 330.

<i>Substance, symmetry and type</i>	<i>Space group</i>	<i>No. mol. per cell</i>	<i>Min. mol. symmetry</i>	<i>a<sub>0</sub></i>	<i>b<sub>0</sub></i>	<i>c<sub>0</sub></i>	<i>References</i>
Cholesteryl Bromide Mon.	C <sub>2</sub> <sup>h</sup>	2		10.7	7.45 $\beta=132^\circ$	21.4	1933, 40a.
Cholesteryl Chloride Mon.	C <sub>2</sub> <sup>h</sup>	2		10.3	7.0 $\beta=131^\circ$	21.2	1933, 40a.
Cholesteryl Salicylate C <sub>34</sub> H <sub>50</sub> O <sub>2</sub> Tri.	C <sub>1</sub> <sup>i</sup>	1		9.68 $\alpha=85^\circ 53'$ , $\beta=77^\circ 41'$ , $\gamma=84^\circ 1'$	12.52	6.31	1934, 334.
Ergosterol C <sub>27</sub> H <sub>44</sub> OH Mon.	C <sub>2</sub> <sup>h</sup>	4	A	9.75	7.4 $\beta=65^\circ$	39.1	1932, 39.
Lumisterol C <sub>27</sub> H <sub>44</sub> OH Mon.	C <sub>2</sub> <sup>h</sup>	4	A	20.3	7.25 $\beta=60^\circ$	20.4	1932, 39.
Calciferol C <sub>27</sub> H <sub>44</sub> OH Mon.	C <sub>2</sub> <sup>h</sup>	8	A	20.8	7.15 $\beta=68^\circ$	38.5	1932, 39.
Calciferol Pyrocalciferol (1:1) Mon.	C <sub>2</sub> <sup>h</sup>	4		20.2	7.35 $\beta=63^\circ$	40.0	1932, 39.
Pregnandiol Ort.				10.2	7.3	24.6	1932, 38.
$\alpha$ -Dihydroergosterol, Ethyl Alcoholate C <sub>27</sub> H <sub>43</sub> OH, C <sub>2</sub> H <sub>5</sub> OH Mon.	C <sub>2</sub> <sup>h</sup>	12	A	30.8	7.4 $\beta=53^\circ$	43.1	1932, 39.
Ketohydroxyoestrin Mon.	C <sub>2</sub> <sup>h</sup>	4	A	7.5	22.1 $\beta=112^\circ$	9.06	1932, 37, 38. 1934, 343.
$\alpha$ -Follicular Hormone (unstable rhombic form) C <sub>18</sub> H <sub>26</sub> O <sub>2</sub> Ort.		4		7.84	10.0	18.2	1934, 343.
$\alpha$ -Follicular Hormone (stable rhombic form) C <sub>18</sub> H <sub>26</sub> O <sub>2</sub> Ort.		4		12.0	16.2	7.45	1934, 343.
$\alpha$ -Follicular Hormone Monohydrate C <sub>18</sub> H <sub>24</sub> O <sub>2</sub> Mon.		4		22.85	7.55 $\beta=70\frac{1}{2}^\circ$	9.15	1934, 343.
Trihydroxyoestrin Mon.	C <sub>2</sub> <sup>h</sup>	4	A	7.50	22.8 $\beta=112^\circ$	9.06	1932, 37.

(Lactone 135)	Mon.	$C_2^2$	2	10.6	7.7 $\beta = 79^\circ$	11.7	1934, 18.
$C_{23}H_{36}O_2$							
Dianhydrodigitoxigenin	Mon.	$C_2^2$	2	9.62	7.85 $\beta = 86\frac{1}{2}^\circ$	12.8	1934, 18.
$C_{23}H_{28}O_2(OH)$							
Digitoxigenin	Ort.	$V^4$	4	18.13	7.16	14.95	1934, 18.
$C_{23}H_{32}O_2(OH)_2$							
Gitoxigenin	Mon.	$C_2^2$	8	6.16	13.3 $\beta = 82^\circ$	53.3	1934, 18.
$C_{23}H_{31}O_2(OH)_3$							
Digoxigenin	Ort.	$V^4$	4	9.62	16.75	12.85	1934, 18.
$C_{23}H_{31}O_2(OH)_3$							
Vitamin B <sub>1</sub> Hydrochloride	Mon.	$C_{2h}^5$	4	12.62	20.53 $\beta = 66^\circ 5'$	6.96	1933, 39.
$C_{12}H_{18}O_2N_4S, 2HCl$ (Windaus)							
Adenine Hydrochloride*	Mon.	$C_4^4$ or $C_2^2$		8.71	4.80 $\beta = 62^\circ 0'$	20.00	1933, 39.
Insulin †	Mon.		26	130	100 $\beta = ca\ 90^\circ$	80	1932, 105.
Pepsin	Hex.			67		154	1934, 17.

\* The unit cell as stated above contains 4 hydrochloride and 2 H<sub>2</sub>O molecules. Crystals of a supposed vitamin B<sub>4</sub> have an almost identical cell; it is concluded therefore that the B<sub>4</sub> is only an impurity in this adenine hydrochloride.

† From the published data it is certain that this cell cannot be the true unit.



## Appendix

### Bibliography of Crystal Structure Data

This bibliography continues that published as an appendix to the second edition of "The Structure of Crystals." In adding items through 1930, papers by authors who already appear have been given existing numbers with an added a, b, etc.; articles by new authors have been arranged alphabetically with new numbers. The same procedure has been followed in numbering very recent additions to the lists for subsequent years.

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## Index of Substances

- Acanthite. See *Silver Sulfide*  
 Acenaphthene, Styphnic Acid,  
 $C_{10}H_6(CH_2)_2$ ,  $C_6H(OH)_2(NO_2)_3$ , 151  
 Acmite,  $NaFe(SiO_3)_2$ , 107  
 Adelite,  $CaMg(OH)AsO_4$ , 64  
 Adenine Hydrochloride, 163  
 Adularia, 128  
 Afwillite,  $3CaO \cdot 2SiO_2 \cdot 3H_2O$ , 107  
 $Ag_3Hg_4$ , 40  
 AgLi, 16  
 $Al_2Au$ , 24  
 AlCuMg, 24  
 $Al_3Fe$ , 38  
 AlLi, 16  
 $Al_2Mg_3$ , 36  
 AlNd, 16  
 Alabandite. See *Manganous Sulfide*  
 (green precipitate)  
 d-Alanine,  $CH_3CH(NH_2)COOH$ , 137  
 dl-Alanine,  $CH_3CH(NH_2)COOH$ , 137  
 d-Alanylglycine,  
 $NH_2 \cdot CH(CH_3)CONHCH_2COOH$ , 137  
 Albite,  $NaAlSi_3O_8$ , 107, 128  
 Alizarin Black. See *Naphthazarin*  
 Alstonite. See *Bromlite*  
 $\alpha$ -Alumina. See  $\alpha$ -*Aluminum Trioxide*  
 $\beta$ -Alumina. See  $\beta$ -*Aluminum Trioxide*  
 Aluminum, Al, 9  
 Aluminum Carbide,  $Al_4C_3$ , 40  
 Aluminum Chloride Hexahydrate,  
 $AlCl_3 \cdot 6H_2O$ , 83  
 Aluminum Fluoride,  $AlF_3$ , 38  
 Aluminum Trioxide,  $\alpha$ - $Al_2O_3$ , 36  
 Aluminum Trioxide,  $\beta$ - $Al_2O_3$ , 36  
 Aluminum Trioxide,  $\gamma$ - $Al_2O_3$ , 36  
 Aluminum Trioxide Monohydrate,  
 $Al_2O_3 \cdot H_2O$ , 82  
 Aluminum Trioxide Trihydrate,  
 $Al_2O_3 \cdot 3H_2O$ , 83  
 p-Aminoazobenzene,  
 $C_6H_5N=NC_6H_4(NH_2)$ , 148  
 Ammonia,  $NH_3$ , 38  
 Ammonium Acid Fluoride,  $NH_4HF_2$ , 41  
 Ammonium Beryllium Fluoride,  
 $(NH_4)_2BeF_4$ , 66  
 Ammonium Bromide,  $\gamma$ - $NH_4Br$ , 17  
 Ammonium Bromselenite,  
 $(NH_4)_2SeBr_6$ , 79  
 Ammonium Chlorite,  $NH_4ClO_2$ , 41  
 Ammonium Chloroplatinate,  
 $(NH_4)_2PtCl_6$ , 79  
 Ammonium Chloroplumbate,  
 $(NH_4)_2PbCl_6$ , 79  
 Ammonium Chloroselenite,  
 $(NH_4)_2SeCl_6$ , 79  
 Ammonium Chlorostannate,  
 $(NH_4)_2SnCl_6$ , 79  
 Ammonium Chlorotellurite,  
 $(NH_4)_2TeCl_6$ , 79  
 Ammonium Chromate,  $(NH_4)_2CrO_4$ , 66  
 Ammonium Chromfluoride,  
 $(NH_4)_3CrF_6$ , 79  
 Ammonium Cobaltinitrite,  
 $(NH_4)_3Co(NO_2)_6$ , 79  
 Ammonium Cupric Bromide Dihydrate,  
 $(NH_4)_2CuBr_4 \cdot 2H_2O$ , 82  
 Ammonium Cupric Chloride Dihydrate,  
 $(NH_4)_2CuCl_4 \cdot 2H_2O$ , 82  
 Ammonium Fluophosphate,  $NH_4PF_6$ , 79  
 Ammonium Fluosilicate,  $(NH_4)_2SiF_6$ , 79  
 Ammonium Hexafluovanadate,  
 $(NH_4)_3VF_6$ , 79  
 Ammonium Hydrosulfide,  $NH_4HS$ , 17  
 Ammonium Hypophosphite,  $NH_4H_2PO_2$ ,  
 41  
 Ammonium Iodate,  $NH_4IO_3$ , 55  
 Ammonium Iridium Nitrite,  
 $(NH_4)_3Ir(NO_2)_6$ , 79  
 Ammonium Nitrate,  $NH_4NO_3$  (I), 55  
 Ammonium Nitrate,  $NH_4NO_3$  (II), 55  
 Ammonium Nitrate,  $NH_4NO_3$  (III), 55  
 Ammonium Nitrate,  $NH_4NO_3$  (IV), 55  
 Ammonium Nitrate,  $NH_4NO_3$  (V), 55  
 Ammonium Osmiamate,  $NH_4OsNO_3$ , 66  
 Ammonium Perchlorate,  $NH_4ClO_4$  (low),  
 66  
 Ammonium Persulfate,  $(NH_4)_2S_2O_8$ , 66  
 Ammonium Rhodium Nitrite,  
 $(NH_4)_3Rh(NO_2)_6$ , 79  
 Amphibole (var. Pargasite), 107  
 Analcite,  $NaAlSi_2O_6 \cdot H_2O$ , 107  
 Anauxite, 119  
 Andalusite,  $Al_2SiO_5$ , 107  
 Andesite, 128  
 Aniline Picrate,  
 $C_6H_5NH_2 \cdot \cdot (OH)C_6H_2(NO_2)_3$ , 148  
 Anisal 1,5 Diaminonaphthalene,  
 $C_{26}H_{22}O_2N_2$ , 151  
 Anorthite, 128  
 Anthracene,  $C_{14}H_{10}$ , 151  
 Anthraquinone,  $C_{14}H_8O_2$ , 151  
 1, 2 Anthraquinone,  $C_{14}H_8O_2$ , 151  
 1, 4 Anthraquinone (acicular form),  
 $C_{14}H_8O_2$ , 151  
 1, 4 Anthraquinone (tabular form),  
 $C_{14}H_8O_2$ , 151

- Antimony, Sb, 11  
 Antimony Iodide,  $\text{SbI}_3$ , 39  
 Antimony Trisulfide,  $\text{Sb}_2\text{S}_3$ , 36  
 Apatite,  $\text{Ca}(\text{F}, \text{Cl})\text{Ca}_4(\text{PO}_4)_3$ , 64, 69  
 Apophyllite,  $\text{KF} \cdot \text{Ca}_4\text{Si}_8\text{O}_{20} \cdot 8\text{H}_2\text{O}$ , 107  
 l-Arabinose,  $\text{C}_5\text{H}_{10}\text{O}_5$ , 140  
 Ardennite,  
      $(\text{SiO}_4)_5\text{AsO}_4\text{Al}_3\text{Mn}(\text{AlOH})_2\text{Mn}_4 \cdot 2\text{H}_2\text{O}$ ,  
     107  
 Argentite. See *Silver Sulfide*  
 Armangite,  $\text{Mn}_3\text{As}_2\text{O}_6$ , 55  
 Arsenic, As, 9  
 Arsenic Iodide,  $\text{AsI}_3$ , 38  
 Arsenious Oxide,  $\text{As}_2\text{O}_3$ , 36  
 Arsenoklasite,  $\text{Mn}_3(\text{AsO}_4)_2 \cdot 2\text{Mn}(\text{OH})_2$ , 99  
 Arsine,  $\text{AsH}_3$ , 38  
 $\text{AsI}_3 \cdot 3\text{S}_8$ , 99  
 Ashcroftite,  
      $\text{NaK}(\text{Ca}, \text{Mg}, \text{Mn})\text{Al}_4\text{Si}_5\text{O}_{18} \cdot 8\text{H}_2\text{O}$ , 107  
 Asparagine Monohydrate,  
      $(\text{CONH}_2)\text{CHNH}_2\text{CH}_2\text{COOH} \cdot \text{H}_2\text{O}$ ,  
     137  
 l-Aspartic Acid,  
      $\text{COOH} \cdot \text{CH}_2 \cdot \text{CH}(\text{NH}_2)\text{COOH}$ , 137  
 Astrophyllite,  
      $[\text{Si}_2\text{O}_7]_2[(\text{Ti}, \text{Zr})(\text{OH}, \text{F})_2](\text{Fe}, \text{Mn})_4$   
      $(\text{K}, \text{Na})_2$ , 107  
 Atopites,  
      $(\text{Ca}, \text{Mn}, \text{Na})_2\text{Sb}_2(\text{O}, \text{OH}, \text{F})_7$ , 99  
 AuCd (low), 16  
 AuCd (high), 16  
 $\text{Au}_2\text{Pb}$ , 24  
 $\text{AuSb}_2$ , 24  
 $\text{AuSn}$ , 16  
 Axinite,  $\text{MgHCa}_2\text{BaI}_2(\text{SiO}_4)_4$ , 107  
 o-Azotoluene,  $(\text{CH}_3)\text{C}_6\text{H}_4\text{N}=\text{NC}_6\text{H}_4(\text{CH}_3)$   
     148  
 p-Azoxyanisole,  $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_3$ , 148  
 p-Azoxyphenetole,  $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_3$ , 148  
 Azurite,  $2\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$ , 99  
  
 $\text{B}_{10}\text{H}_{14}$ , 40  
 Barium Boride,  $\text{BaB}_6$ , 39  
 Barium Carbonate,  $\text{BaCO}_3$ , 54  
 Barium Cerate,  $\text{BaCeO}_3$ , 54  
 Barium Chloride Glycine,  
      $\text{BaCl}_2 \cdot 2\text{CH}_2\text{NH}_2\text{COOH}$ , 131  
 Barium Fluoride,  $\text{BaF}_2$ , 24  
 Barium Imide,  $\text{BaNH}$ , 16  
 Barium Iodide Hexahydrate,  
      $\text{BaI}_2 \cdot 6\text{H}_2\text{O}$ , 83  
 Barium Nickel Cyanide Tetrahydrate,  
      $\text{BaNi}(\text{CN})_4 \cdot 4\text{H}_2\text{O}$ , 83  
 Barium Nickel Nitrite,  $\text{Ba}_2\text{Ni}(\text{NO}_2)_6$ , 78  
 Barium Nitrate,  $\text{Ba}(\text{NO}_3)_2$ , 54  
 Barium Oxide,  $\text{BaO}$ , 16  
 Barium Perchlorate Trihydrate,  
      $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ , 83  
 Barium Platinum Cyanide Tetrahydrate,  
      $\text{BaPt}(\text{CN})_4 \cdot 4\text{H}_2\text{O}$ , 83  
 Barium Rhodium Nitrite,  
      $\text{Ba}_3[\text{Rh}(\text{NO}_2)_6]_2$ , 78  
 Barium Thorate,  $\text{BaThO}_3$ , 54  
 Barium Tungstate,  $\text{BaWO}_4$ , 64  
 Barium Zirconate,  $\text{BaZrO}_3$ , 54  
 Barytocalcite,  $\text{BaCa}(\text{CO}_3)_2$ , 54  
 Bastnäsite,  $(\text{Ce}, \text{La} \dots)\text{FCO}_3$ , 54  
 Bavenite,  $9\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{BeO} \cdot 4\text{CaO} \cdot \text{H}_2\text{O}$ ,  
     107  
 $\text{Be}_2\text{Cu}$ , 24  
 $\text{Be}_2\text{Fe}$ , 24  
 $\text{Be}_2\text{SiW}_{12}\text{O}_{40} \cdot 31\text{H}_2\text{O}$ , 86  
 Benzene,  $\text{C}_6\text{H}_6$ , 146  
 Bertrandite,  $\text{H}_2\text{Be}_4\text{Si}_2\text{O}_9$ , 107  
 Beryllium, Be, 9  
 Beryllium Carbide,  $\text{Be}_2\text{C}$ , 24  
 Beryllium Fluoride,  $\text{BeF}_2$ , 24  
 Beryllium Nitride,  $\text{Be}_3\text{N}_2$ , 36  
 Beryllium Oxyacetate,  $\text{Be}_4\text{O}(\text{C}_2\text{H}_3\text{O}_2)_6$ ,  
     131  
 Beryllium Phosphide,  $\text{Be}_3\text{P}_2$ , 36  
 Beryllium Sulfate Tetrahydrate,  
      $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$ , 83  
 Beryllonite,  $\text{BeNa}_3\text{PO}_4$ , 64  
 Bindheimite,  $\text{Pb}_2\text{Sb}_2\text{O}_7$ , 99  
 Binnite,  $(\text{Cu}, \text{Fe})_{12}\text{As}_4\text{S}_{13}$ , 99  
 $\alpha$ -Bisdiethyl Sulfine Platinous Chloride,  
      $\text{Pt}[\text{S}(\text{C}_2\text{H}_5)_2]_2\text{Cl}_2$ , 132  
 $\alpha$ -Bisdimethyl Sulfine Platinous Chloride,  
      $\text{Pt}[\text{S}(\text{CH}_3)_2]_2\text{Cl}_2$ , 132  
 $\beta$ -Bisdimethyl Sulfine Platinous Chloride,  
      $\text{Pt}[\text{S}(\text{CH}_3)_2]_2\text{Cl}_2$ , 132  
 Bisethylene Diamino Platinous Chloride,  
      $\text{Pt}(\text{C}_2\text{H}_8\text{N}_2)_2\text{Cl}_2$ , 132  
 3, 4 Bis(5 Methyl-3-Isoxazolylcarbonyl)  
     Furazan-2-Oxide,  $\text{C}_{12}\text{H}_8\text{N}_4\text{O}_6$ , 160  
 Bismuth, Bi, 9  
 Bismuth Iodide,  $\text{BiI}_3$ , 38  
 Bismuth Sulfide,  $\text{Bi}_2\text{S}_3$ , 36  
 Bismuthinite. See *Bismuth Sulfide*  
 Boleite, 99  
 Bone, 69  
 Boracite,  $\text{Mg}_3\text{ClB}_7\text{O}_{13}$ , (low), 99  
 Boracite,  $\text{Mg}_3\text{ClB}_7\text{O}_{13}$ , (high), 99  
 Boric Acid,  $\text{H}_3\text{BO}_3$ , 54  
 Boron Arsenate,  $\text{BAsO}_4$ , 64  
 Boron Carbide,  $\text{B}_6\text{C}$ , 39  
 Boron Phosphate,  $\text{BPO}_4$ , 64  
 Bournonite,  $\text{CuPbSbS}_3$ , 99  
 Braggite,  $(\text{Pt}, \text{Pd}, \text{Ni})\text{S}$ , 18  
 Braunite,  $3\text{Mn}_2\text{O}_3 \cdot \text{MnSiO}_3$ , 108  
 Brominated Northupite,  
      $\text{Na}_2\text{Mg}(\text{CO}_3)_2 \cdot \text{NaBr}$ , 100  
 Bromlite,  $\text{BaCa}(\text{CO}_3)_2$ , 54  
 1 Brom-2-Naphthylamine,  
      $\text{C}_{10}\text{H}_8\text{Br}(\text{NH}_2)$ , 150  
 4 Brom-1-Naphthylamine,  
      $\text{C}_{10}\text{H}_8\text{Br}(\text{NH}_2)$ , 150  
 4 Brom-1-Naphthylamine, 2, 6 Dinitro-  
     phenol (red form),  $\text{C}_{10}\text{H}_6\text{Br}(\text{NH}_2)_2$ ,  
      $\text{C}_6\text{H}_3\text{OH}(\text{NO}_2)_2$ , 150  
 4 Brom-1-Naphthylamine, 2, 6 Dinitro-  
     phenol (yellow form),  $\text{C}_{10}\text{H}_6\text{Br}(\text{NH}_2)_2$ ,  
      $\text{C}_6\text{H}_3\text{OH}(\text{NO}_2)_2$ , 151  
 p-Bromochlorobenzene,  $\text{C}_6\text{H}_4\text{BrCl}$ , 146  
 Brushite,  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ , 82  
  
 $\text{CHI}_3 \cdot 3\text{S}_8$ , 99  
 $\text{C}_4\text{H}_2\text{N}_4\text{O}_3$ , 159  
 $\text{CaPb}_3$ , 38  
 $\text{CaSn}_3$ , 38  
 $\text{CaTl}$ , 16

- CaTi<sub>3</sub>, 38  
 Cadmium, Cd, 9  
 Cadmium Ammonium Sulfate Hexahydrate, CdSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O, 84  
 Cadmium Arsenide, Cd<sub>3</sub>As<sub>2</sub>, 36  
 Cadmium Bromide, CdBr<sub>2</sub>, 24  
 Cadmium Bromide Hexammoniate, CdBr<sub>2</sub>·6NH<sub>3</sub>, 83  
 Cadmium Chlorite Dihydrate, Cd(ClO<sub>2</sub>)<sub>2</sub>·2H<sub>2</sub>O, 82  
 Cadmium Ferrite, CdFe<sub>2</sub>O<sub>4</sub>, 64  
 Cadmium Fluoborate Hexammoniate, Cd(BF<sub>4</sub>)<sub>2</sub>·6NH<sub>3</sub>, 83  
 Cadmium Fluosulfate Hexammoniate, Cd(SO<sub>3</sub>F)<sub>2</sub>·6NH<sub>3</sub>, 84  
 Cadmium Hydroxychloride, CdCl(OH), 24  
 Cadmium Iodide, CdI<sub>2</sub>, 24  
 Cadmium Iodide Hexammoniate, CdI<sub>2</sub>·6NH<sub>3</sub>, 84  
 Cadmium Oxide, CdO, 16  
 Cadmium Perchlorate Hexammoniate, Cd(ClO<sub>4</sub>)<sub>2</sub>·6NH<sub>3</sub>, 84  
 Cadmium Phosphide, Cd<sub>3</sub>P<sub>2</sub>, 36  
 Cadmium Thiochromite, CdCr<sub>2</sub>S<sub>4</sub>, 64  
 Cadmium Titanate, CdTiO<sub>3</sub>, 54  
 Calciferol, C<sub>27</sub>H<sub>41</sub>OH, 162  
 Calciferol Pyrocalciferol, 162  
 Calcite. See *Calcium Carbonate*  
 Calcium, β-Ca, 9  
 Calcium Aluminate, Ca<sub>3</sub>[Al(OH)<sub>6</sub>]<sub>2</sub>, 78  
 Calcium Boride, CaB<sub>6</sub>, 39  
 Calcium Bromide Hexahydrate, CaBr<sub>2</sub>·6H<sub>2</sub>O, 83  
 Calcium Bromide Hexammoniate, CaBr<sub>2</sub>·6NH<sub>3</sub>, 83  
 Calcium Carbonate, CaCO<sub>3</sub>, 54  
 Calcium Chloride Hexahydrate, CaCl<sub>2</sub>·6H<sub>2</sub>O, 83  
 Calcium Chlorite, Ca(ClO<sub>2</sub>)<sub>2</sub>, 40  
 Calcium Chromate, CaCrO<sub>4</sub>, 64  
 Calcium Chromate Dihydrate, CaCrO<sub>4</sub>·2H<sub>2</sub>O, 82  
 Calcium Chromate Monohydrate, CaCrO<sub>4</sub>·H<sub>2</sub>O, 82  
 Calcium Fluoride, CaF<sub>2</sub>, 24  
 Calcium Imide, CaNH, 16  
 Calcium Iodide, CaI<sub>2</sub>, 24  
 Calcium Iodide Hexahydrate, CaI<sub>2</sub>·6H<sub>2</sub>O, 83  
 Calcium Iodide Hexammoniate, CaI<sub>2</sub>·6NH<sub>3</sub>, 83  
 Calcium Magnesium Carbonate, CaMg(CO<sub>3</sub>)<sub>2</sub>, 54  
 Calcium Metaborate, CaB<sub>2</sub>O<sub>4</sub>, 40  
 Calcium Nitrate, Ca(NO<sub>3</sub>)<sub>2</sub>, 54  
 Calcium Nitride, α-Ca<sub>3</sub>N<sub>2</sub>, 36  
 Calcium Sulfate Dihydrate, CaSO<sub>4</sub>·2H<sub>2</sub>O, 82  
 Calcium Sulfate Hemihydrate, 2(CaSO<sub>4</sub>)·ca H<sub>2</sub>O, 82  
 Calcium Sulfate Urea, CaSO<sub>4</sub>·CO(NH<sub>2</sub>)<sub>2</sub>, 131  
 Cancrinite, 3(Na<sub>2</sub>Al<sub>2</sub>O<sub>4</sub>·2SiO<sub>2</sub>)·2CaCO<sub>3</sub>, 108  
 Carbon Dioxide, CO<sub>2</sub>, 24  
 Carbon Monoxide, β-CO, 16  
 Carbon Oxysulfide, COS, 24  
 Carbon Tetraiodide, CI<sub>4</sub>, 39  
 Carbonato Tetrammine Cobalti-Perchlorate, [Co<sup>CO<sub>3</sub></sup>(NH<sub>3</sub>)<sub>4</sub>]ClO<sub>4</sub>, 83  
 Carbonato Tetrammine Cobalti-Sulfate Trihydrate, [Co<sup>CO<sub>3</sub></sup>(NH<sub>3</sub>)<sub>4</sub>]<sub>2</sub>SO<sub>4</sub>·3H<sub>2</sub>O, 86  
 α-Carnegieite, NaAlSiO<sub>4</sub>, 108  
 CdLi, 16  
 3CdSO<sub>4</sub>·8H<sub>2</sub>O, 86  
 CdSb, 16  
 Cd<sub>3</sub>Sb<sub>2</sub>, 36  
 CeMg<sub>3</sub>, 38  
 CePb<sub>3</sub>, 38  
 CeSn<sub>3</sub>, 38  
 Cellulose, (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)<sub>n</sub>, 142  
 Cellulose Hydrate, 142  
 Cellulose Perchlorate, 2C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>·HClO<sub>4</sub>, 142  
 Celsius, 128  
 Cementite. See *Iron Carbide*  
 Cerium, α-Ce, 9  
 Cerium Boride, CeB<sub>6</sub>, 39  
 Cerussite. See *Lead Carbonate*  
 Cesium Acid Tartrate, CsHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>, 131  
 Cesium Aurous Auric Chloride, Cs<sub>2</sub>Au<sup>+</sup>Au<sup>+++</sup>Cl<sub>6</sub>, 78  
 Cesium Chloride, CsCl(low), 16  
 Cesium Chloride, CsCl(high), 16  
 Cesium Chloroplatinate, Cs<sub>2</sub>PtCl<sub>6</sub>, 78  
 Cesium Chloroplumbate, Cs<sub>2</sub>PbCl<sub>6</sub>, 78  
 Cesium Chloroselenite, Cs<sub>2</sub>SeCl<sub>6</sub>, 78  
 Cesium Chlorostannate, Cs<sub>2</sub>SnCl<sub>6</sub>, 78  
 Cesium Chlorotellurite, Cs<sub>2</sub>TeCl<sub>6</sub>, 78  
 Cesium Chlorotitanate, Cs<sub>2</sub>TiCl<sub>6</sub>, 78  
 Cesium Chlorozirconate, Cs<sub>2</sub>ZrCl<sub>6</sub>, 78  
 Cesium Cobaltinitrite, Cs<sub>3</sub>Co(NO<sub>2</sub>)<sub>6</sub>, 78  
 Cesium Cyanide, CsCN, 16  
 Cesium Dichloriodide, CsCl<sub>2</sub>I, 42  
 Cesium Dithionate, Cs<sub>2</sub>S<sub>2</sub>O<sub>6</sub>, 54  
 Cesium Ferricyanide, Cs<sub>3</sub>Fe(CN)<sub>6</sub>, 78  
 Cesium Fluophosphate, CsPF<sub>6</sub>, 78  
 Cesium Hydrosulfide, CsHS, 16  
 Cesium Iridium Nitrite, Cs<sub>3</sub>Ir(NO<sub>2</sub>)<sub>6</sub>, 78  
 Cesium Nitrate, CsNO<sub>3</sub>, 54  
 Cesium Osmiamate, CsOsNO<sub>3</sub>, 64  
 Cesium Perrhenate, CsReO<sub>4</sub>, 64  
 Cesium Persulfate, Cs<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, 64  
 Cesium Rhodium Nitrite, Cs<sub>3</sub>Rh(NO<sub>2</sub>)<sub>6</sub>, 78  
 Cesium Silver Auric Chloride, Cs<sub>2</sub>AgAuCl<sub>6</sub>, 78  
 Chabazite, CaAl<sub>2</sub>Si<sub>4</sub>O<sub>12</sub>·6H<sub>2</sub>O, 108  
 Chalcopyrite, CuFeS<sub>2</sub>, 41  
 Chilite, 74  
 d-Chitosamine Hydrochloride, C<sub>6</sub>H<sub>14</sub>O<sub>5</sub>NCl, 142  
 Chlorites, 108  
 Chlor-X-Apatite, Ca<sub>10</sub>(Cl,X)<sub>2</sub>(PO<sub>4</sub>)<sub>6</sub>, 69  
 Choleic Acid, 159  
 Cholesteryl Bromide, 162  
 Cholesteryl Chloride, 162  
 Cholesteryl Salicylate, C<sub>24</sub>H<sub>50</sub>O<sub>3</sub>, 162  
 Chromite, (Fe,Mg)Cr<sub>2</sub>O<sub>4</sub>, 65  
 Chromium, α-Cr, 9  
 Chromium Carbide, Cr<sub>3</sub>C<sub>2</sub>, 36

- Chromium Carbide,  $\text{Cr}_4\text{C}$ , 39  
 Chromium Chloride Hexahydrate,  
 $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ , 84  
 Chromium Oxide,  $\text{CrO}_3$ , 38  
 Chromium Tribromide,  $\text{CrBr}_3$ , 38  
 Chromium Trioxide,  $\text{Cr}_2\text{O}_3$ , 36  
 Chrysene,  $\text{C}_{18}\text{H}_{12}$ , 152  
 Clinzoisite, 128  
 $\text{Co}_4\text{S}_3$ , 40  
 Cobalt,  $\beta$ -Co, 9  
 Cobalt Sulfate,  $\text{CoSO}_4$ , 64  
 Cobalt Titanate,  $\text{Co}_2\text{TiO}_4$ , 64  
 Cobaltic Fluoride,  $\text{CoF}_3$ , 38  
 Cobalti-Fluoborate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{BF}_4)_3$ , 84  
 Cobalti-Fluophosphate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{PF}_6)_3$ , 84  
 Cobalti-Perchlorate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{ClO}_4)_3$ , 84  
 Cobaltous Aluminate,  $\text{CoAl}_2\text{O}_4$ , 64  
 Cobaltous Ammonium Sulfate Hexahydrate,  
 $\text{CoSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ , 84  
 Cobaltous Bromide Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3]\text{Br}_2$ , 84  
 Cobaltous Carbonate,  $\text{CoCO}_3$ , 54  
 Cobaltous Chloride,  $\text{CoCl}_2$ , 24  
 Cobaltous Chloride Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3]\text{Cl}_2$ , 84  
 Cobaltous Fluoborate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{BF}_4)_2$ , 84  
 Cobaltous Fluophosphate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{PF}_6)_2$ , 84  
 Cobaltous Fluosulfate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{SO}_3\text{F})_2$ , 84  
 Cobaltous Iodide Hexamethylamine,  
 $[\text{Co} \cdot 6(\text{NH}_2 \cdot \text{CH}_3)]\text{I}_2$ , 84  
 Cobaltous Iodide Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3]\text{I}_2$ , 84  
 Cobaltous Perchlorate Hexammoniate,  
 $[\text{Co} \cdot 6\text{NH}_3](\text{ClO}_4)_2$ , 84  
 Cobaltous Sulfide,  $\text{CoS}$ , 16  
 Cobaltous Titanate,  $\text{CoTiO}_3$ , 54  
 Columbium, Cb, 9  
 Colusite,  $(\text{Cu}, \text{Fe}, \text{Mo}, \text{Sn})_4(\text{S}, \text{As}, \text{Te})_{3-4}$ , 16  
 Cooperite. See *Platinum Sulfide*  
 Copper, Cu, 9  
 Copper Aluminate,  $\text{CuAl}_2\text{O}_4$ , 64  
 Copper Antimony Sulfide,  $\text{CuSbS}_2$ , 41  
 Copper Bismuth Sulfide,  $\text{CuBiS}_2$ , 40  
 Copper Ferrite,  $\text{CuFe}_2\text{O}_4$ , (quenched), 64  
 Copper Ferrite,  $\text{CuFe}_2\text{O}_4$ , (annealed), 65  
 Copper Formate Dihydrate,  
 $\text{Cu}(\text{HCO}_2)_2 \cdot 2\text{H}_2\text{O}$ , 131  
 Copper Formate Tetrahydrate,  
 $\text{Cu}(\text{HCO}_2)_2 \cdot 4\text{H}_2\text{O}$ , 131  
 Copper Glance. See *Cuprous Sulfide*  
 Copper Sulfate Pentahydrate,  
 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 83  
 Copper Vanadium Sulfide,  $\text{Cu}_3\text{VS}_4$ , 65  
 Cordylite,  $\text{BaCO}_3 \cdot 2\text{RFeCO}_3$ , 54  
 Corundum. See  *$\alpha$ -Aluminum Trioxide*  
 Covellite. See *Cupric Sulfide*  
 $\text{Cr}_7\text{C}_3$ , 40  
 $\alpha$ -Cristobalite. See *Silicon Dioxide*  
 $\beta$ -Cristobalite. See *Silicon Dioxide*  
 Cryolite,  $\text{Na}_3\text{AlF}_6$ , 79  
 $\text{Cs}_3\text{C}_8$  (brown), 40  
 $\text{CsC}_{16}$  (black), 40  
 $\text{Cu}_5\text{Cd}_3$ , 40  
 $\text{Cu}_2\text{Mg}$ , 24  
 $\text{Cu}_{16}\text{Si}_4$ , 40  
 $\text{Cu}_5\text{Zn}_8$ , 40  
 Cumengeite, 99  
 Cupric Fluoride,  $\text{CuF}_2$ , 24  
 Cupric Oxide,  $\text{CuO}$ , 16  
 Cupric Sulfide,  $\text{CuS}$ , 16  
 Cuprite. See *Cuprous Oxide*  
 Cuprodesclowitzite, 74  
 Cuprous Ferrite,  $\text{Cu}_2\text{Fe}_2\text{O}_4$ , 41  
 Cuprous Fluoride,  $\text{CuF}$ , 16  
 Cuprous Glutathione, 131  
 Cuprous Mercuric Iodide,  $\alpha$ - $\text{Cu}_2\text{HgI}_4$ , 65  
 Cuprous Mercuric Iodide,  $\beta$ - $\text{Cu}_2\text{HgI}_4$ , 65  
 Cuprous Oxide,  $\text{Cu}_2\text{O}$ , 24  
 Cuprous Sulfide,  $\text{Cu}_2\text{S}$ , 24  
 Cyanite,  $\text{Al}_2\text{SiO}_5$ , 108  
 p-Cyano-o-Nitro-p'-Methoxystilbene,  
 $\text{C}_6\text{H}_3(\text{CN})(\text{NO}_2)\text{CH}=\text{CHC}_6\text{H}_4(\text{OCH}_3)$   
 (orange form), 149  
 p-Cyano-o-Nitro-p'-Methoxystilbene,  
 $\text{C}_6\text{H}_3(\text{CN})(\text{NO}_2)\text{CH}=\text{CHC}_6\text{H}_4(\text{OCH}_3)$   
 (metastable yellow form), 149  
 Cyanuric Triazide,  $\text{C}_3\text{N}_3(\text{N}_3)_3$ , 159  
 Cyclododecane,  $\text{C}_{12}\text{H}_{24}$ , 139  
 $\alpha$ -Cyclohexandiol 1, 2,  $\text{C}_6\text{H}_{10}(\text{OH})_2$ , 139  
 $\beta$ -Cyclohexandiol 1, 4,  $\text{C}_6\text{H}_{10}(\text{OH})_2$ , 139  
 $\gamma$ -Cyclohexandiol 1, 2,  $\text{C}_6\text{H}_{10}(\text{OH})_2$ , 139  
 $\beta$ -Cyclohexandiol Diacetate 1, 4,  
 $\text{C}_6\text{H}_{10}(\text{CH}_3\text{CO}_2)_2$ , 139  
 Cyclohexane,  $\text{C}_6\text{H}_{12}$ , 139  
 Cyclooctacosane 1, 15 dion,  
 $\text{C}_{28}\text{H}_{52}\text{O}_2$ , 140  
 Cyclotetracosane 1, 13 dion,  
 $\text{C}_{24}\text{H}_{44}\text{O}_2$ , 140  
 l-Cystine,  $\text{COOHCH}(\text{NH}_2)\text{CH}_2\text{S}=\text{SCH}_2\text{CH}(\text{NH}_2)\text{COOH}$ , 137  
 Dechinite, 74  
 Desclowitzite,  $\text{PbZn}(\text{OH})\text{VO}_4$ , 66  
 3, 3'-Diaminodimesityl, 152  
 Diamminoplatinic Tetrachloride,  
 $\alpha$ - $\text{Pt}(\text{NH}_3)_2\text{Cl}_4$ , 82  
 Diamminoplatinic Tetrachloride,  
 $\beta$ - $\text{Pt}(\text{NH}_3)_2\text{Cl}_4$ , 82  
 Dianhydrogitoxigenin,  $\text{C}_{23}\text{H}_{29}\text{O}_2(\text{OH})$ , 163  
 Dianthracene,  $(\text{C}_{14}\text{H}_{10})_2$ , 152  
 Diaspore. See *Aluminum Trioxide Monohydrate*  
 1, 2, 5, 6 Dibenanthracene, 152  
 $\gamma$ ,  $\gamma'$ -Dibenzocarbazole, 152  
 Dibenzoyl Disulfide,  $(\text{C}_6\text{H}_5\text{COS})_2$ , 150  
 Dibenzyl,  $\text{C}_6\text{H}_5\text{CH}_2\text{-CH}_2\text{C}_6\text{H}_5$ , 148  
 Dibenzyl Diselenide,  $(\text{C}_6\text{H}_5\text{CH}_2\text{Se})_2$ , 150  
 Dibenzyl Disulfide,  $(\text{C}_6\text{H}_5\text{CH}_2\text{S})_2$ , 150  
 Dibenzylidenebenzidine, 148  
 p-Dibromobenzene,  $\text{C}_6\text{H}_4\text{Br}_2$ , 146  
 1, 4 Dibromocyclohexane,  $\text{C}_6\text{H}_{10}\text{Br}_2$ , 139  
 p-Dichlorobenzene,  $\text{C}_6\text{H}_4\text{Cl}_2$ , 146  
 Dickite,  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ , 108  
 5, 5 Diethyl Barbituric Acid,  
 $\text{C}_8\text{H}_{12}\text{N}_2\text{O}_3$ , 160  
 Digitoxigenin,  $\text{C}_{23}\text{H}_{32}\text{O}_2(\text{OH})_2$ , 163



- $\alpha$ -Diglycylglycine,  $\text{NH}_2\text{CH}_2\text{CONHCH}_2\text{-CONHCH}_2\text{COOH}$ , 137  
 $\beta$ -Diglycylglycine,  $\text{NH}_2\text{CH}_2\text{CONHCH}_2\text{-CONHCH}_2\text{COOH}$ , 137  
 Diglycylglycine Dihydrate,  $\text{NH}_2\text{CH}_2\text{-CONHCH}_2\text{CONHCH}_2\text{COOH}\cdot 2\text{H}_2\text{O}$ , 137  
 Digoxigenin,  $\text{C}_{23}\text{H}_{31}\text{O}_2(\text{OH})_3$ , 163  
 $\alpha$ -Dihydroergosterol, Ethyl Alcoholate,  $\text{C}_{27}\text{H}_{48}\text{OH}\cdot\text{C}_2\text{H}_5\text{OH}$ , 162  
 m-Diiodobenzene,  $\text{C}_6\text{H}_4\text{I}_2$ , 146  
 o-Diiodobenzene,  $\text{C}_6\text{H}_4\text{I}_2$ , 146  
 p-Diiodobenzene,  $\text{C}_6\text{H}_4\text{I}_2$ , 146  
 1, 4 Diiodocyclohexane,  $\text{C}_6\text{H}_{10}\text{I}_2$ , 139  
 Diketopiperazine,  

$$\text{O}=\text{C} \begin{array}{c} \text{NHCH}_2 \\ \text{CH}_2\text{NH} \end{array} \text{C}=\text{O}$$
, 159  
 Dimesityl, 152  
 Dimethyl Ammonium Chlorostannate,  $[\text{NH}_2(\text{CH}_3)_2]_2\text{SnCl}_6$ , 133  
 Dimethyl Thallium Bromide,  $\text{Tl}(\text{CH}_3)_2\text{Br}$ , 132  
 Dimethyl Thallium Chloride,  $\text{Tl}(\text{CH}_3)_2\text{Cl}$ , 132  
 Dimethyl Thallium Iodide,  $\text{Tl}(\text{CH}_3)_2\text{I}$ , 132  
 2, 7 Dinitroanthraquinone,  $\text{C}_{14}\text{H}_6(\text{NO}_2)_2\text{O}_2$ , 151  
 2, 7 Dinitroanthraquinone Fluorene,  $\text{C}_{14}\text{H}_6(\text{NO}_2)_2\text{O}_2$ ,  $(\text{C}_6\text{H}_4)_2\text{CH}_2$ , 151  
 m-Dinitrobenzene,  $\text{C}_6\text{H}_4(\text{NO}_2)_2$ , 146  
 1, 2, 6 Dinitrophenol,  $\text{C}_6\text{H}_3\text{OH}(\text{NO}_2)_2$ , 147  
 trans-Dinitrotetrammine Cobalt-Chloride, 1,  $6[\text{Co}(\text{NO}_2)_2]_4\text{Cl}$ , 83  
 Diphenic Acid,  $(\text{COOHCH}_2\text{C}_6\text{H}_4)_2$ , 150  
 Diphenyl,  $\text{C}_6\text{H}_5\cdot\text{C}_6\text{H}_5$ , 150  
 p-Diphenylbenzene,  $\text{C}_6\text{H}_5(\text{C}_6\text{H}_4)\text{C}_6\text{H}_5$ , 152  
 Diphenylbutadiene,  $\text{C}_6\text{H}_5\text{CH}=(\text{CH})_2=\text{CHC}_6\text{H}_5$ , 149  
 Diphenyldecapentaene,  $\text{C}_6\text{H}_5(\text{CH}=\text{CH})_5\text{C}_6\text{H}_5$ , 149  
 Diphenyl Diselenide,  $(\text{C}_6\text{H}_5\text{Se})_2$ , 149  
 Diphenyl Disulfide,  $(\text{C}_6\text{H}_5\text{S})_2$ , 149  
 Diphenyldodecahexaene,  $\text{C}_6\text{H}_5(\text{CH}=\text{CH})_6\text{C}_6\text{H}_5$ , 149  
 Diphenylhexatriene,  $\text{C}_6\text{H}_5\text{CH}=(\text{CH}-\text{CH})_2=\text{CHC}_6\text{H}_5$ , 149  
 Diphenyl Nitrosoamine,  $\text{C}_6\text{H}_5\text{N}-\text{NO}-\text{C}_6\text{H}_5$ , 150  
 Diphenyloctatetraene,  $\text{C}_6\text{H}_5(\text{CH}=\text{CH})_4\text{C}_6\text{H}_5$ , 149  
 Diphenyltetradecaheptaene,  $\text{C}_6\text{H}_5(\text{CH}=\text{CH})_7\text{C}_6\text{H}_5$ , 149  
 Disodium Calcium Orthosilicate,  $\text{Na}_2\text{CaSiO}_4$ , 111  
 Dodecanol,  $\text{C}_{12}\text{H}_{25}\text{OH}$ , 138  
 Dolomite. See *Calcium Magnesium Carbonate*  
 Dulcitol,  $\text{C}_6\text{H}_{14}\text{O}_6$ , 140  
 Durene, 1, 2, 4, 5  $\text{C}_6\text{H}_2(\text{CH}_3)_4$ , 147  
 Edingtonite,  $\text{Ba}_2\text{Al}_4\text{Si}_6\text{O}_{20}\cdot 8\text{H}_2\text{O}$ , 108  
 Emplectite. See *Copper Bismuth Sulfide*  
 Enargite,  $\text{Cu}_3\text{AsS}_4$ , 64  
 Enstatite (var. Bronzite), 108  
 l-Ephedrine Hydrobromide,  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HBr}$ , 160  
 Ephedrine Hydrobromide (racemic),  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HBr}$ , 160  
 l-Ephedrine Hydrochloride,  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HCl}$ , 160  
 Ephedrine Hydrochloride (racemic),  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HCl}$ , 160  
 l-Ephedrine Hydroiodide,  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HI}$ , 160  
 Ephedrine Hydroiodide (racemic),  $\text{C}_{10}\text{H}_{15}\text{ON}\cdot\text{HI}$ , 160  
 Epididymite,  $\text{NaBeSi}_4\text{O}_7(\text{OH})$ , 108  
 Epidote,  $(\text{SiO}_4)_3\text{Al}_2\text{Ca}_2(\text{Al},\text{Fe})\text{OH}$ , 108  
 Epsomite. See *Magnesium Sulfate Heptahydrate*  
 Erbium Boride,  $\text{ErB}_6$ , 39  
 Ergosterol,  $\text{C}_{27}\text{H}_{41}\text{OH}$ , 162  
 Ethyl Anisal p-Amino Cinnamate,  $\text{C}_{19}\text{H}_{18}\text{NO}_3$ , 148  
 Euclase,  $\text{HBeAlSiO}_5$ , 108  
 Eudialyte, 108  
 Eulytite,  $\text{Bi}_4\text{Si}_3\text{O}_{12}$ , 108  
 Eusynchite, 74  
 FeSi, 16  
 $\text{Fe}_3\text{W}_2$  ( $\epsilon$ -phase), 36  
 Feldspars, 108, 128  
 Ferric Chloride,  $\text{FeCl}_3$ , 38  
 Ferric Fluoride,  $\text{FeF}_3$ , 38  
 Ferric Oxide,  $\text{Fe}_2\text{O}_3$ , 36  
 Ferric Oxide,  $\text{Fe}_2\text{O}_3$ , (magnetic), 36  
 Ferric Oxide Monohydrate,  $\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$ , 82  
 Ferric Oxychloride,  $\text{FeOCl}$ , 24  
 Ferrous Aluminate,  $\text{FeAl}_2\text{O}_4$ , 65  
 Ferrous Ammonium Sulfate Hexahydrate,  $\text{FeSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , 84  
 Ferrous Bromide Hexammoniate,  $\text{FeBr}_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Carbonate,  $\text{FeCO}_3$ , 54  
 Ferrous Chloride Hexammoniate,  $\text{FeCl}_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Chromite,  $\text{FeCr}_2\text{O}_4$ , 65  
 Ferrous Ferric Oxide,  $\text{Fe}_3\text{O}_4$ , 65  
 Ferrous Fluoborate Hexammoniate,  $\text{Fe}(\text{BF}_4)_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Fluosulfate Hexammoniate,  $\text{Fe}(\text{SO}_3\text{F})_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Iodide Hexammoniate,  $\text{FeI}_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Oxide,  $\text{FeO}$ , 16  
 Ferrous Perchlorate Hexammoniate,  $\text{Fe}(\text{ClO}_4)_2\cdot 6\text{NH}_3$ , 84  
 Ferrous Sulfide,  $\text{FeS}$ , 16  
 Ferrous Titanate,  $\text{Fe}_2\text{TiO}_4$ , 65  
 Ferrous Vanadium Spinel,  $\text{FeV}_2\text{O}_4$ , 65  
 Fluorite. See *Calcium Fluoride*  
 $\alpha$ -Follicular Hormone, (unstable rhombic form),  $\text{C}_{18}\text{H}_{22}\text{O}_2$ , 162  
 $\alpha$ -Follicular Hormone, (stable rhombic form),  $\text{C}_{18}\text{H}_{22}\text{O}_2$ , 162  
 $\alpha$ -Follicular Hormone Monohydrate,  $\text{C}_{18}\text{H}_{24}\text{O}_3$ , 162  
 Fuller's Earth,  $\text{Al}_2\text{O}_3\cdot 4\text{SiO}_2\cdot \text{H}_2\text{O}$ , 109

- Gadolinium Boride,  $GdB_6$ , 39  
 Gahnite. See *Zinc Aluminate*  
 $\beta$ - $\alpha$ -Galactose,  $C_6H_{12}O_6$ , 140  
 Gallium, Ga, 10  
 Gallium Zincate,  $Ga_2ZnO_4$ , 65  
 Garnet, 109  
 $GdPMo_{12}O_{40} \cdot 30H_2O$ , 86  
 Gehlenite (synthetic), 109  
 Germanium Dioxide,  $GeO_2$ , 24  
 Germanous Sulfide,  $GeS$ , 17  
 Gibbsite. See *Aluminum Trioxide Trihydrate*  
 Gitoxigenin,  $C_{23}H_{31}O_2(OH)_3$ , 163  
 $\beta$ -d-Glucosan,  $C_6H_{10}O_6$ , 141  
 d-Glucose,  $C_6H_{12}O_6$ , 140  
 Glucose Pentaacetate, 142  
 l-Glutamic Acid,  
 $COOH \cdot CH_2 \cdot CH_2 \cdot CH(NH_2)COOH$ ,  
 137  
 $\alpha$ -Glutaric Acid,  $COOH(CH_2)_3COOH$ , 138  
 $\beta$ -Glutaric Acid,  $COOH(CH_2)_3COOH$ , 138  
 $\alpha$ -Glycine,  $CH_2NH_2COOH$ , 137  
 $\beta$ -Glycine,  $CH_2NH_2COOH$ , 137  
 $\alpha$ -Glycylglycine,  
 $CH_2NH_2CONHCH_2COOH$ , 137  
 $\beta$ -Glycylglycine,  
 $CH_2NH_2CONHCH_2COOH$ , 137  
 $\gamma$ -Glycylglycine,  
 $CH_2NH_2CONHCH_2COOH$ , 137  
 Goethite. See *Ferric Oxide Monohydrate*  
 Gold, Au, 9  
 Graphite, C, 9  
 Guanidine d-Tartrate Hydrate,  
 $[C(NH_2)_2NH]_2(C_4H_6O_6) \cdot 1\frac{1}{2}H_2O$ , 138  
 Guanidinium Chloride,  $(NH_2)_2CNH \cdot HCl$ ,  
 136  
 Gypsum. See *Calcium Sulfate Dihydrate*  
  
 Hafnium Tetrafluoride,  $HfF_4$ , 39  
 Hambergite,  $Be_2BO_3(OH)$ , 99  
 Hanksite,  $9Na_2SO_4 \cdot 2Na_2CO_3 \cdot KCl$ , 99  
 Hauerite. See *Manganese Disulfide*  
 Häuynite,  $Na_8Si_6Al_6O_{24}(1-2, SO_4)$ , 109  
 Hematite. See *Ferric Oxide*  
 Hemimorphite,  $Zn_4(OH)_2Si_2O_7 \cdot H_2O$ , 109  
 Hercynite. See *Ferrous Aluminate*  
 Hessite. See *Silver Telluride*  
 Heulandite, 109  
 Hexabromobutylene,  
 $CHBr_2BrC=CBrCHBr_2$ , 136  
 Hexachlorethane,  $C_2Cl_6$ , 136  
 Hexachlorobenzene,  $C_6Cl_6$ , 147  
 Hexachlorodiphenyl,  $(C_6H_2Cl_2)_2$ , 150  
 Hexadecanol,  $C_{16}H_{33}OH$ , 138  
 Hexamethylenetetramine,  $C_6H_{12}N_4$ , 140  
 Hexamethylethane,  $C_2(CH_3)_6$ , 136  
 Hexaminobenzene,  $C_6(NH_2)_6$ , 147  
 Hexuronic Acid,  $C_6H_8O_6$ , 159  
 HgLi, 17  
 Hiortdahlite, 128  
 Hornblendes, 109  
 Hyalophane, 128  
 Hydrargillite. See *Aluminum Trioxide Trihydrate*  
 Hydrobromic Acid,  $HBr$  (low), 17  
 Hydrobromic Acid,  $HBr$  (high), 17  
 Hydrochloric Acid,  $HCl$  (low), 17  
 Hydrochloric Acid,  $HCl$  (high), 17  
 Hydrogen Selenide,  $H_2Se$ , 25  
 Hydrogen Sulfide,  $H_2S$ , 25  
 Hydroiodic Acid,  $HI$ , 17  
 Hydroxy-Apatite,  $C_{10}(OH)_2(PO_4)_6$ , 69  
  
 Ice,  $H_2O$ , 25  
 Ilmenite,  $FeTiO_3$ , 54  
 Indium, In, 10  
 Indium Borate,  $InBO_3$ , 54  
 i-Inositol,  $C_6H_8(OH)_6$ , 139  
 l-Inositol,  $C_6H_8(OH)_6$ , 139  
 i-Inositol Dihydrate,  $C_6H_8(OH)_6 \cdot 2H_2O$ , 139  
 Insulin, 163  
 o-Iodobenzoic Acid,  $C_6H_4ICOOH$ , 146  
 Iodoform,  $CHI_3$ , 136  
 Iridium, Ir, 10  
 Iron,  $\alpha$ -Fe, 9  
 Iron Arsenide,  $FeAs_2$ , 24  
 Iron Boride,  $Fe_3B$ , 24  
 Iron Carbide,  $Fe_3C$ , 38  
 Iron Phosphide,  $FeP_2$ , 24  
 Iron Sulfide,  $FeS_2$ , 24  
 Iron Tetracarbonyl,  $Fe(CO)_4$ , 39  
 4 Isoxazolyl-5-Isloxazolyl Ketone,  
 $C_7H_4N_2O_3$ , 160  
 Ittnerite, 125  
  
 Joaquinite,  $NaBa(Ti,Fe)_2Si_4O_{16}$ , 109  
  
 KBi<sub>2</sub>, 25  
 $KC_8$  (brown), 40  
 $KC_{16}$  (black), 40  
 $KFeS_2$ , 41  
 $K_2NaAlF_6$ , 78  
 Kaliophilite,  $KAlSiO_4$ , 109  
 Kalithomsonite. See *Ashcroftine*  
 Kaolin,  $Al_2Si_2O_5(OH)_4$ , 109  
 Kernite,  $Na_2B_4O_7 \cdot 4H_2O$ , 83  
 Ketohydroxyoestrin, 162  
 Koppite,  
 $(Ca,Cc,Na,K)_2(Cb,Fe)_2O_6(O,OH,F)$ ,  
 99  
 Krokoite. See *Lead Chromate*  
 Krypton, Kr, 10  
  
 LaAl<sub>4</sub>, 39  
 LaMg<sub>3</sub>, 38  
 LaPb<sub>3</sub>, 38  
 LaSn<sub>3</sub>, 38  
 Labradorite, 128  
 (Lactone 135),  $C_{28}H_{36}O_2$ , 163  
 Langbeinite,  $K_2Mg_2(SO_4)_3$ , 65  
 Lanthanum,  $\alpha$ -La, 10  
 Lanthanum,  $\beta$ -La, 10  
 Lanthanum Borate,  $LaBO_3$ , 55  
 Lanthanum Boride,  $LaB_6$ , 39  
 Laurite. See *Ruthenium Disulfide*  
 Lavenite, 128  
 Lawsonite,  $Ca(SiO_3)_2 \cdot (AlO_2H_2)_2$ , 109  
 Lead, Pb, 10  
 Lead Bromide,  $PbBr_2$ , 25  
 Lead Carbonate,  $PbCO_3$ , 55  
 Lead Chloride,  $PbCl_2$ , 25  
 Lead Chlorite,  $Pb(ClO_2)_2$ , 41  
 Lead Chromate,  $PbCrO_4$ , 66

- Lead Dioxide,  $\text{PbO}_2$ , 26  
 Lead Ferrite,  $\text{PbFe}_2\text{O}_4$ , 41  
 Lead Fluobromide,  $\text{PbFBr}$ , 25  
 Lead Fluochloride,  $\text{PbFCl}$ , 25  
 Lead Fluoride,  $\alpha\text{-PbF}_2$ , 26  
 Lead Fluoride,  $\beta\text{-PbF}_2$ , 26  
 Lead Formate,  $\text{Pb}(\text{HCO}_2)_2$ , 131  
 Lead Nickel Nitrite,  $\text{Pb}_2\text{Ni}(\text{NO}_2)_6$ , 79  
 Lead Nitrate,  $\text{Pb}(\text{NO}_3)_2$ , 55  
 Lead Oxide,  $\text{PbO}$  (red), 18  
 Lead Oxide,  $\text{PbO}$  (yellow), 18  
 Lead Rhodium Nitrite,  $\text{Pb}_3[\text{Rh}(\text{NO}_2)_6]_2$ , 79  
 Legrandite,  $\text{Zn}_{14}(\text{AsO}_4)_9\text{OH}\cdot 12\text{H}_2\text{O}$ , 86  
 Lepidocrocite. See *Ferric Oxide Monohydrate*  
 Leucophanite,  $(\text{Ca}, \text{Na})_2\text{BeSi}_2(\text{O}, \text{OH}, \text{F})_7$ , 109  
 Lewisite,  $(\text{Ca}, \text{Fe}, \text{Na})_2(\text{Sb}, \text{Ti})_2(\text{O}, \text{OH})_7$ , 99  
 $\text{LiCd}_3$ , 38  
 $\text{LiGa}$ , 17  
 $\text{LiIn}$ , 17  
 $\text{LiTl}$ , 17  
 $\text{LiZn}$ , 17  
 Lievrite,  $\text{CaFe}_2^{++}(\text{Fe}^{+++}\text{OH})(\text{SiO}_4)_2$ , 109  
 Linneite,  $(\text{Co}, \text{Ni})_3\text{S}_4$ , 64  
 Lithium Ferrite,  $\text{Li}_2\text{Fe}_2\text{O}_4$ , 41  
 Lithium Hydride,  $\text{LiH}$ , 17  
 Lithium Hydroxide,  $\text{LiOH}$ , 17  
 Lithium Iodate,  $\text{LiIO}_3$ , 55  
 Lithium Iodide Trihydrate,  $\text{LiI}\cdot 3\text{H}_2\text{O}$ , 83  
 Lithium Orthophosphate,  $\text{Li}_3\text{PO}_4$ , 65  
 Lithium Oxide,  $\text{Li}_2\text{O}$ , 25  
 Lithium Perchlorate Trihydrate,  $\text{LiClO}_4\cdot 3\text{H}_2\text{O}$ , 83  
 Lithium Selenide,  $\text{Li}_2\text{Se}$ , 25  
 Lithium Sulfate,  $\text{Li}_2\text{SO}_4$ , 65  
 Lithium Sulfate Monohydrate,  $\text{Li}_2\text{SO}_4\cdot \text{H}_2\text{O}$ , 82  
 Lithium Sulfide,  $\text{Li}_2\text{S}$ , 25  
 Lithium Telluride,  $\text{Li}_2\text{Te}$ , 25  
 Lollingite. See *Iron Arsenide*  
 Loparite,  $(\text{Na}, \text{Ce}, \text{Ca})(\text{Ti}, \text{Cb})\text{O}_3$ , 55  
 Lorandite. See *Thallium Arsenic Sulfide*  
 Lumisterol,  $\text{C}_{27}\text{H}_{41}\text{OH}$ , 162  
 Lusakite,  $\text{H}_2\text{O}\cdot 4(\text{Fe}, \text{Co}, \text{Ni}, \text{Mg})\text{O}\cdot 9(\text{Al}, \text{Fe})_2\text{O}_3\cdot 8\text{H}_2\text{O}$ , 110
- Magnesium, Mg, 10  
 Magnesium Aluminate,  $\text{MgAl}_2\text{O}_4$ , 65  
 Magnesium Ammonium Arsenate Hexahydrate,  $\text{MgNH}_4\text{AsO}_4\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Ammonium Selenate Hexahydrate,  $\text{MgSeO}_4\cdot (\text{NH}_4)_2\text{SeO}_4\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Ammonium Sulfate Hexahydrate,  $\text{MgSO}_4\cdot (\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Bromide Hexahydrate,  $\text{MgBr}_2\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Bromide Hexammoniate,  $\text{MgBr}_2\cdot 6\text{NH}_3$ , 85  
 Magnesium Chloride Hexahydrate,  $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Chloride Hexammoniate,  $\text{MgCl}_2\cdot 6\text{NH}_3$ , 85  
 Magnesium Chromite,  $\text{MgCr}_2\text{O}_4$ , 65  
 Magnesium Ferrite,  $\text{MgFe}_2\text{O}_4$ , 65  
 Magnesium Fluoborate Hexammoniate,  $\text{Mg}(\text{BF}_4)_2\cdot 6\text{NH}_3$ , 84  
 Magnesium Gallium Spinel,  $\text{MgGa}_2\text{O}_4$ , 65  
 Magnesium Indium Spinel,  $\text{MgIn}_2\text{O}_4$ , 66  
 Magnesium Iodide,  $\text{MgI}_2$ , 25  
 Magnesium Iodide Hexammoniate,  $\text{MgI}_2\cdot 6\text{NH}_3$ , 85  
 Magnesium Nitride,  $\text{Mg}_3\text{N}_2$ , 36  
 Magnesium Perchlorate Hexammoniate,  $\text{Mg}(\text{ClO}_4)_2\cdot 6\text{NH}_3$ , 85  
 Magnesium Phosphide,  $\text{Mg}_3\text{P}_2$ , 36  
 Magnesium Platinocyanide Heptahydrate,  $\text{MgPt}(\text{CN})_4\cdot 7\text{H}_2\text{O}$ , 86  
 Magnesium Potassium Sulfate Hexahydrate,  $\text{MgSO}_4\cdot \text{K}_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Sulfate Heptahydrate,  $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$ , 86  
 Magnesium Thallous Sulfate Hexahydrate,  $\text{MgSO}_4\cdot \text{Tl}_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , 85  
 Magnesium Titanate,  $\text{MgTiO}_3$ , 55  
 Magnesium Titanate,  $\text{Mg}_2\text{TiO}_4$ , 66  
 Magnetite. See *Ferrous Ferric Oxide*  
 Magnus' Green Salt,  $[\text{Pt}(\text{NH}_3)_4]\text{PtCl}_4$ , 83  
 Magnus' Red Salt,  $[\text{Pt}(\text{NH}_3)_4]\text{PtCl}_4$ , 83  
 Malachite,  $\text{CuCO}_3\cdot \text{Cu}(\text{OH})_2$ , 99  
 $\alpha$ -Malonic Acid,  $\text{COOHCH}_2\text{COOH}$ , 138  
 Manganese, Mn, 10  
 Manganese Aluminate,  $\text{MnAl}_2\text{O}_4$ , 66  
 Manganese Chromite,  $\text{MnCr}_2\text{O}_4$ , 66  
 Manganese Disulfide,  $\text{MnS}_2$ , 25  
 Manganese Ferrite,  $\text{MnFe}_2\text{O}_4$ , 66  
 Manganese Thiochromite,  $\text{MnCr}_2\text{S}_4$ , 66  
 Manganese Trioxide Monohydrate,  $\text{Mn}_2\text{O}_3\cdot \text{H}_2\text{O}$ , 82  
 Manganite. See *Manganese Trioxide Monohydrate*  
 Manganous Bromide Hexammoniate,  $\text{MnBr}_2\cdot 6\text{NH}_3$ , 85  
 Manganous Chloride Hexammoniate,  $\text{MnCl}_2\cdot 6\text{NH}_3$ , 85  
 Manganous Fluoborate Hexammoniate,  $\text{Mn}(\text{BF}_4)_2\cdot 6\text{NH}_3$ , 85  
 Manganous Fluosulfate Hexammoniate,  $\text{Mn}(\text{SO}_3\text{F})_2\cdot 6\text{NH}_3$ , 85  
 Manganous Iodide Hexammoniate,  $\text{MnI}_2\cdot 6\text{NH}_3$ , 85  
 Manganous Oxide,  $\text{MnO}$ , 17  
 Manganous Perchlorate Hexammoniate,  $\text{Mn}(\text{ClO}_4)_2\cdot 6\text{NH}_3$ , 85  
 Manganous Sulfide,  $\text{MnS}$  (red precipitate), 17  
 Manganous Sulfide,  $\text{MnS}$  (green precipitate), 17  
 Manganous Titanate,  $\text{MnTiO}_3$ , 55  
 Manganous Titanate,  $\text{Mn}_2\text{TiO}_4$ , 66  
 d-Mannitol,  $\text{C}_6\text{H}_{14}\text{O}_6$ , 140  
 $\gamma$ -d-Mannonolactone, 141  
 d-Mannose,  $\text{C}_6\text{H}_{12}\text{O}_6$ , 140  
 Marcasite. See *Iron Sulfide*  
 Matlockite. See *Lead Fluochloride*  
 Meliphanite, 110  
 Mellite,  $\text{Al}_2\text{C}_{12}\text{O}_{12}\cdot 18\text{H}_2\text{O}$ , 131  
 Mercuric Bromide,  $\text{HgBr}_2$ , 25  
 Mercuric Chloride,  $\text{HgCl}_2$ , 25  
 Mercuric Fluoride,  $\text{HgF}_2$ , 25

- Mercuric Iodide,  $\text{HgI}_2$ , 25  
 Mercurous Fluoride,  $\text{HgF}$ , 17  
 Mercury,  $\text{Hg}$ , 10  
 Mesantraquinone, 152  
 Mesolite,  $\text{Na}_2\text{Ca}_2\text{Al}_6\text{Si}_9\text{O}_{30} \cdot 8\text{H}_2\text{O}$ , 110  
 Methane,  $\text{CH}_4$ , 136  
 $\alpha$ -Methyl-l-Arabinoside,  $\text{C}_8\text{H}_{12}\text{O}_6$ , 140  
 $\beta$ -Methyl-l-Arabinoside,  $\text{C}_8\text{H}_{12}\text{O}_6$ , 141  
 Methylbixin,  $\text{C}_{28}\text{H}_{32}\text{O}_4$ , 159  
 l-Methyl Ephedrine Hydrobromide,  
 $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HBr}$ , 161  
 Methyl Ephedrine Hydrobromide  
 (racemic, 1st mod.),  $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HBr}$ ,  
 161  
 Methyl Ephedrine Hydrobromide  
 (racemic, 2nd mod.),  $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HBr}$ ,  
 161  
 l-Methyl Ephedrine Hydrochloride,  
 $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HCl}$ , 161  
 Methyl Ephedrine Hydrochloride  
 (racemic),  $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HCl}$ , 161  
 l-Methyl Ephedrine Hydroiodide,  
 $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HI}$ , 161  
 Methyl Ephedrine Hydroiodide (racemic),  
 $\text{C}_{11}\text{H}_{17}\text{ON}, \text{HI}$ , 161  
 $\alpha$ -Methyl-d-Glucoside,  $\text{C}_7\text{H}_{14}\text{O}_6$ , 141  
 $\beta$ -Methyl-d-Glucoside Hemihydrate,  
 $\text{C}_7\text{H}_{14}\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$ , 141  
 Methyl-l-Inositol,  $\text{C}_6\text{H}_8(\text{OH})_5(\text{OCH}_3)$ , 139  
 $\alpha$ -Methylmannoside,  $\text{C}_7\text{H}_{14}\text{O}_6$  (furanose  
 form), 141  
 $\alpha$ -Methylmannoside,  $\text{C}_7\text{H}_{14}\text{O}_6$  (pyranose  
 form), 141  
 Methyl-l-Rhamnoside,  $\text{C}_7\text{H}_{14}\text{O}_6$ , 141  
 Methyl Silicate,  $(\text{CH}_3)_4\text{SiO}_4$ , 132  
 Methyl Urea,  $\text{CONH}_2(\text{NHCH}_3)$ , 136  
 $\alpha$ -Methylxyloside,  $\text{C}_6\text{H}_{12}\text{O}_5$ , 141  
 $\beta$ -d-Methylxyloside,  $\text{C}_6\text{H}_{12}\text{O}_5$ , 141  
 $\text{Mg}_3\text{As}_2$ , 36  
 $\text{Mg}_3\text{Bi}_2$ , 36  
 $\text{Mg}_2\text{Ge}$ , 25  
 $\text{MgNi}_2$ , 25  
 $\text{MgNiZn}$ , 25  
 $\text{Mg}_2\text{Pb}$ , 25  
 $\text{MgPr}$ , 17  
 $\text{Mg}_3\text{Sb}_2$ , 36  
 $\text{Mg}_2\text{SiMo}_{12}\text{O}_{40} \cdot 31\text{H}_2\text{O}$ , 86  
 $\text{Mg}_2\text{Sn}$ , 25  
 $\text{MgTl}$ , 17  
 $\text{MgZn}$ , 17  
 $\text{MgZn}_2$ , 25  
 $\text{MgZn}_3$ , 39  
 Miargyrite. See *Silver Antimony Sulfide*  
 Microlite,  $(\text{Ca}, \text{Na})_2(\text{Ta}, \text{Cb})_2(\text{O}, \text{F})_7$ , 100  
 Milarite,  $\text{HKCa}_2\text{Al}_2(\text{Si}_2\text{O}_6)_6$ , 110  
 Mimetite,  $\text{Pb}_{10}\text{Cl}_2(\text{AsO}_4)_6$ , 66, 69  
 $\text{MoC}$ , 17  
 $\text{Mo}_2\text{C}$ , 25  
 Molybdenum,  $\text{Mo}$ , 10  
 Molybdenum Trioxide,  $\text{MoO}_3$ , 38  
 $\gamma$ -Monoacetylmethyl-l-Rhamnoside,  
 $\text{C}_9\text{H}_{16}\text{O}_6$ , 142  
 n-Monoamyl Ammonium Chloride,  
 $n\text{-C}_5\text{H}_{11}\text{NH}_2\text{Cl}$ , 133  
 Monomeric Butadiene Sulfone,  
 $\text{C}_4\text{H}_6\text{SO}_2$ , 159  
 Monomeric Dimethyl Butadiene Sulfone,  
 $\text{C}_6\text{H}_{10}\text{SO}_2$ , 160  
 Monomeric Isoprene Sulfone,  
 $\text{C}_6\text{H}_8\text{SO}_2$ , 159  
 Monomethyl Ammonium Cupric Chloride,  
 $(\text{NH}_2\text{CH}_3)_2\text{CuCl}_4$ , 133  
 Montmorillonite,  $\text{H}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ , 110  
 Mosandrite, 128  
 Mottramite, 74  
 Muscovite,  $\text{KAl}_2(\text{AlSi}_3)\text{O}_{10}(\text{OH})_2$ , 110  
  
 $\text{NaBi}$ , 17  
 $\text{NaIn}$ , 18  
 $\text{NaPb}_3$ , 39  
 $\text{Na}_{31}\text{Pb}_8$ , 40  
 $\text{NaTl}$ , 18  
 Na-W Bronze (blue),  $\text{Na}_2(\text{WO}_3)_5$ , 55  
 Na-W Bronze (cubic). See *Sodium Tung-*  
*state*  
 Nacrite,  $\text{Al}_2\text{Si}_2\text{O}_6(\text{OH})_4$ , 110  
 Naphthalene,  $\text{C}_{10}\text{H}_8$ , 150  
 Naphthazarin, 151  
 1, 2 Naphthoquinone,  $\text{C}_{10}\text{H}_6\text{O}_2$ , 151  
 1, 4 Naphthoquinone,  $\text{C}_{10}\text{H}_6\text{O}_2$ , 151  
 Narsarsukite,  $(\text{Si}_4\text{O}_{11})(\text{Ti}, \text{FeF})\text{Na}_2$ , 110  
 Natrolite,  $\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10} \cdot 2\text{H}_2\text{O}$ , 110  
 $\text{NdPMo}_{12}\text{O}_{40} \cdot 30\text{H}_2\text{O}$ , 86  
 Neodymium,  $\text{Nd}$ , 10  
 Neodymium Boride,  $\text{NdB}_6$ , 40  
 Neodymium Carbide,  $\text{NdC}_2$ , 25  
 Nephelite,  $\text{NaAlSi}_3\text{O}_8$ , 110  
 $\text{Ni}_2\text{SiMo}_{12}\text{O}_{40} \cdot 31\text{H}_2\text{O}$ , 86  
 Niccolite. See *Nickel Arsenide*  
 Nickel,  $\alpha$ -Ni, 10  
 Nickel,  $\beta$ -Ni, 10  
 Nickel Aluminate,  $\text{NiAl}_2\text{O}_4$ , 66  
 Nickel Arsenide,  $\text{NiAs}$ , 18  
 Nickel Bromide,  $\text{NiBr}_2$ , 25  
 Nickel Bromide Hexammoniate,  
 $\text{NiBr}_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Chloride Hexammoniate,  
 $\text{NiCl}_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Chromite,  $\text{NiCr}_2\text{O}_4$ , 66  
 Nickel Fluoborate Hexammoniate,  
 $\text{Ni}(\text{BF}_4)_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Fluophosphate Hexammoniate,  
 $\text{Ni}(\text{PF}_6)_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Fluosulfate Hexammoniate,  
 $\text{Ni}(\text{SO}_3\text{F})_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Hydroxide,  $\text{Ni}(\text{OH})_2$ , 25  
 Nickel Iodide,  $\text{NiI}_2$ , 25  
 Nickel Iodide Hexamethylamine,  
 $\text{NiI}_2 \cdot 6(\text{NH}_2\text{CH}_3)$ , 85  
 Nickel Iodide Hexammoniate,  
 $\text{NiI}_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Oxide,  $\text{NiO}$ , 18  
 Nickel Perchlorate Hexammoniate,  
 $\text{Ni}(\text{ClO}_4)_2 \cdot 6\text{NH}_3$ , 85  
 Nickel Sulfate Hexahydrate,  
 $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , 86  
 Nickel Sulfide,  $\text{NiS}$ , 18  
 Nickel Titanate,  $\text{NiTiO}_3$ , 55  
 Niobium. See *Columbium*  
 Nitrogen,  $\alpha$ -N<sub>2</sub>, 10  
 Nitrogen,  $\beta$ -N<sub>2</sub>, 10  
 Nitrogen Dioxide,  $\text{NO}_2$ , 25

- 4 Nitro-2-Methylaminotoluene, (red form) 147  
 4 Nitro-2-Methylaminotoluene, (yellow form), 147  
 p-Nitrostilbene,  
 $C_6H_5CH=CHC_6H_4(NO_2)$ , 149  
 Nitrous Oxide,  $N_2O$ , 25  
 Nordenskiöldite,  $CaSn(BO_3)_2$ , 54  
 Northupite,  $Na_2Mg(CO_3)_2 \cdot NaCl$ , 100  
 Noselite,  $Na_3Al_3Si_6O_{24} \cdot SO_4$ , 110
- Octadecyl Ammonium Chloride,  
 $C_{18}H_{37}NH_3Cl$ , 133  
 Osmium, Os, 10  
 Osmium Disulfide,  $OsS_2$ , 25  
 Oxalic Acid Dihydrate,  
 $(COOH)_2 \cdot 2H_2O$ , 138  
 Oxy-Apatite,  $Ca_{10}O(PO_4)_6$ , 69  
 Oxygen,  $O_2$ , 10
- Palladium, Pd, 10  
 Palladium Trifluoride,  $PdF_3$ , 39  
 Palladous Fluoride,  $PdF_2$ , 26  
 $\alpha$ -Palmitic Acid,  $CH_3(CH_2)_{14}COOH$ , 138  
 Parisite,  $CaCO_3 \cdot 2RfCO_3$ , 54  
 Partschinite, 128  
 Pectolite,  $NaHCa_2(SiO_3)_3$ , 110  
 Pentaerythritol Tetrabromide,  
 $C(CH_2Br)_4$ , 136  
 Pentaerythritol Tetrachloride,  
 $C(CH_2Cl)_4$ , 136  
 Pentaerythritol Tetraiodide,  
 $C(CH_2I)_4$ , 136  
 Pepsin, 163  
 d-Phenyl Alanine,  
 $C_6H_5CH_2 \cdot CH(NH_2)COOH$ , 146  
 Phenylaminoacetic Acid,  
 $C_6H_5CHNH_2COOH$ , 146  
 Phosphine,  $PH_3$ , 39  
 Phosphomolybdic Acid,  
 $H_3PMo_{12}O_{40} \cdot 30H_2O$ , 86  
 Phosphorus Triiodide,  $PI_3$ , 39  
 Phosphotungstic Acid Pentahydrate,  
 $H_3PW_{12}O_{40} \cdot 5H_2O$ , 83  
 Platinum, Pt, 11  
 Platinum Arsenide,  $PtAs_2$ , 26  
 Platinum Sulfide,  $PtS$ , 18  
 Pollucite,  
 $(CsAl_3H_4)Si_2O_6$ , 110  
 Polybasite,  $(Ag,Cu)_2Sb_2S_4$ , 40  
 Eu-Polyethylene Oxide,  
 $(CH_2CH_2O)_x$ , 159  
 $\beta$ -Polyoxymethylene, 159  
 Potassium Acid Tartrate,  
 $KHC_4H_4O_6$ , 131  
 Potassium Barium Cobalto-Hexanitrite,  
 $K_2BaCo(NO_2)_6$ , 78  
 Potassium Barium Nickel Hexanitrite,  
 $K_2BaNi(NO_2)_6$ , 78  
 Potassium Bromselenite,  $K_2SeBr_6$ , 78  
 Potassium Calcium Cobalto-Hexanitrite,  
 $K_2CaCo(NO_2)_6$ , 78  
 Potassium Calcium Nickel Hexanitrite,  
 $K_2CaNi(NO_2)_6$ , 78  
 Potassium Chloroplatinate,  $K_2PtCl_6$ , 78  
 Potassium Chlorostannate,  $K_2SnCl_6$ , 79  
 Potassium Chlorotellurite,  $K_2TeCl_6$ , 79  
 Potassium Chromate,  $K_2CrO_4$ , 65  
 Potassium Chromcyanide,  $K_3Cr(CN)_6$ , 78  
 Potassium Cobaltinitrite,  $K_3Co(NO_2)_6$ , 78  
 Potassium Columbate,  $KCbo_3$ , 54  
 Potassium Cupric Chloride Dihydrate,  
 $K_2CuCl_4 \cdot 2H_2O$ , 82  
 Potassium Cyanide,  $KCN$ , 17  
 Potassium Dithionate,  $K_2S_2O_6$ , 55  
 Potassium Ferricyanide,  $K_3Fe(CN)_6$ , 78  
 Potassium Ferrite,  $K_2Fe_2O_4$ , 41  
 Potassium Fluoborate,  $KBF_4$ , 65  
 Potassium Fluophosphate,  $KPF_6$ , 78  
 Potassium Hydrosulfide,  $KHS$  (low), 17  
 Potassium Hydrosulfide,  $KHS$  (high), 17  
 Potassium Iridium Cyanide,  $K_3Ir(CN)_6$ , 78  
 Potassium Iridium Nitrite,  $K_3Ir(NO_2)_6$ , 78  
 Potassium Lead Chloride Hydrate,  
 $3(KPbCl_4) \cdot H_2O$ , 82  
 Potassium Manganicyanide,  
 $K_3Mn(CN)_6$ , 78  
 Potassium Nickel Hexanitrite,  
 $K_4Ni(NO_2)_6$ , 78  
 Potassium Nitrate,  $KNO_3$ , 54  
 Potassium Osmiamate,  $KOsNO_3$ , 65  
 Potassium Osmyl Chloride,  
 $K_2OsO_2Cl_4$ , 78  
 Potassium Oxide,  $K_2O$ , 25  
 Potassium Pentachloronitrosimate,  
 $K_2OsNCl_6$ , 78  
 Potassium Perchlorate,  $KClO_4$  (low), 65  
 Potassium Permanganate,  $KMnO_4$ , 65  
 Potassium Pyrosulfite,  $K_2S_2O_5$ , 100  
 Potassium Rhodium Nitrite,  
 $K_3Rh(NO_2)_6$ , 78  
 Potassium Selenide,  $K_2Se$ , 25  
 Potassium Silver Cyanide,  $KAg(CN)_2$ , 41  
 Potassium Strontium Cobalto-Hexanitrite,  
 $K_2SrCo(NO_2)_6$ , 79  
 Potassium Strontium Nickel Hexanitrite,  
 $K_2SrNi(NO_2)_6$ , 79  
 Potassium Sulfide,  $K_2S$ , 25  
 Potassium Tantalate,  $KTaO_3$ , 55  
 Potassium Telluride,  $K_2Te$ , 25  
 Potassium Thiocyanate,  $KCNS$ , 38  
 Potassium Trithionate,  $K_2S_3O_6$ , 100  
 PrMg<sub>3</sub>, 39  
 Praseodymium, Pr, 11  
 Praseodymium Boride,  $PrB_6$ , 40  
 Praseodymium Carbide,  $PrC_2$ , 26  
 Pregnandiol, 162  
 Prehnite,  $Ca_2(SiO_3)_3(AlOH)AlO_2H$ , 110  
 Pseudoboleite, 100  
 d-Pseudococaine-l-Ephedrine-d-Tartrate  
 Monohydrate,  $C_{31}H_{42}O_{11}N_2 \cdot H_2O$ , 161  
 d-Pseudococaine-l-Methyl Ephedrine-d-  
 Tartrate Dihydrate,  
 $C_{32}H_{44}O_{11}N_2 \cdot 2H_2O$ , 161  
 d-Pseudoephedrine Hydrobromide,  
 $C_{10}H_{15}ON \cdot HBr$ , 160  
 Pseudoephedrine Hydrobromide (racemic)  
 $C_{10}H_{15}ON \cdot HBr$ , 161  
 d-Pseudoephedrine Hydrochloride,  
 $C_{10}H_{15}ON \cdot HCl$ , 161  
 Pseudoephedrine Hydrochloride (racemic),  
 $C_{10}H_{15}ON \cdot HCl$ , 161

- d-Pseudoephedrine Hydroiodide,  
 $C_{10}H_{15}ON, HI$ , 161  
 Pseudoephedrine Hydroiodide (racemic),  
 $C_{10}H_{15}ON, HI$ , 161  
 Psittacinite, 74  
 PtSn, 18  
 Pyramidon, 160  
 Pyrite. See *Iron Sulfide*  
 Pyroaurite,  $Fe(OH)_3 \cdot 3Mg(OH)_2 \cdot 3H_2O$ ,  
 100  
 Pyrochlore,  
 $(Na, Ca)_2(Cb, Ti)_2(O, F)_7$ , 100  
 Pyromorphite,  $Pb_{10}Cl_2(PO_4)_6$ , 66, 69  
 Pyrophyllite,  $Al_2Si_4O_{10}(OH)_2$ , 111  
 Pyrosmalite,  
 $Si_3O_7(Mn, Fe)3(Mn, Fe)(OH, Cl)_2$ , 111  
 Pyrrhite, 100  
  
 $\alpha$ -Quartz. See *Silicon Dioxide*  
 Quaterphenyl,  $C_6H_5(C_6H_4)_2C_6H_5$ , 152  
 Quebrachitol. See *Methyl-L-Inositol*  
 Quercitol,  $C_6H_7(OH)_5$ , 139  
 Quinhydrone,  $C_6H_4O_2 \cdot C_6H_4(OH)_2$ , 148  
 p-Quinone,  $C_6H_4O_2$ , 146  
  
 RbC<sub>8</sub> (brown), 40  
 RbC<sub>16</sub> (black), 40  
 Rb<sub>2</sub>(CrF<sub>5</sub>·H<sub>2</sub>O), 82  
 Resorcinol, m-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub>, 146  
 l-Rhamnose Monohydrate,  $C_6H_{12}O_5 \cdot H_2O$ ,  
 140  
 Rhenium, Re, 11  
 Rhenium Trioxide, ReO<sub>3</sub>, 39  
 Rhodium,  $\alpha$ -Rh, 11  
 Rhodium,  $\beta$ -Rh, 11  
 Rhodium Trifluoride, RhF<sub>3</sub>, 39  
 Rinkite,  $(SiO_4)_2[(Ti, Ce)F]Ca_2Na$ , 111  
 Rochelle Salt,  
 $NaOOC(CHOH)_2COOK \cdot 4H_2O$ , 131  
 Romeite,  $(Ca, Na, Mn)_2Sb_2(O, OH, F)_7$ , 100  
 Rubidium Chloroplatinate, Rb<sub>2</sub>PtCl<sub>6</sub>, 79  
 Rubidium Chloroplumbate, Rb<sub>2</sub>PbCl<sub>6</sub>, 79  
 Rubidium Chloroselenite, Rb<sub>2</sub>SeCl<sub>6</sub>, 79  
 Rubidium Chlorostannate, Rb<sub>2</sub>SnCl<sub>6</sub>, 79  
 Rubidium Chlorotellurite, Rb<sub>2</sub>TeCl<sub>6</sub>, 79  
 Rubidium Chlorotitanate, Rb<sub>2</sub>TiCl<sub>6</sub>, 79  
 Rubidium Chlorozirconate, Rb<sub>2</sub>ZrCl<sub>6</sub>, 79  
 Rubidium Cobaltinitrite, Rb<sub>3</sub>Co(NO<sub>2</sub>)<sub>6</sub>, 79  
 Rubidium Cyanide, RbCN, 18  
 Rubidium Dithionate, Rb<sub>2</sub>S<sub>2</sub>O<sub>6</sub>, 55  
 Rubidium Ferricyanide, Rb<sub>3</sub>Fe(CN)<sub>6</sub>, 79  
 Rubidium Hydrosulfide, RbHS (low), 18  
 Rubidium Hydrosulfide, RbHS (high), 18  
 Rubidium Iridium Nitrite, Rb<sub>3</sub>Ir(NO<sub>2</sub>)<sub>6</sub>, 79  
 Rubidium Nitrate, RbNO<sub>3</sub>, 55  
 Rubidium Osmiamate, RbOsNO<sub>3</sub>, 66  
 Rubidium Perrhenate, RbReO<sub>4</sub>, 66  
 Rubidium Rhodium Nitrite,  
 $Rb_3Rh(NO_2)_6$ , 79  
 Rubidium Trinitride, RbN<sub>3</sub>, 39  
 Ruthenium Disulfide, RuS<sub>2</sub>, 26  
  
 $Sa.PMO_{12}O_{40} \cdot 30H_2O$ , 86  
 Samarium Carbide, SaC<sub>2</sub>, 26  
 Sanidine, KAlSi<sub>3</sub>O<sub>8</sub>, 111, 128  
 Sb<sub>2</sub>Tl<sub>7</sub>, 40  
  
 SbZn, 18  
 Scandium Borate, ScBO<sub>3</sub>, 55  
 Scapolite, 111  
 Schizolite, 128  
 Schlippe's Salt, Na<sub>3</sub>SbS<sub>4</sub>·9H<sub>2</sub>O, 86  
 Schneebergite,  $(Ca, Na, Fe)_2Sb_2O_6(OH)$ , 100  
 Scolecite,  $CaAl_2Si_3O_{10} \cdot 3H_2O$ , 111  
 Selenium,  $\alpha$ -Se, 11  
 Selenium,  $\beta$ -Se, 11  
 Siderite. See *Ferrous Carbonate*  
 Silicon Carbide, SiC (II), 18  
 Silicon Dioxide, SiO<sub>2</sub>, 26  
 Silicon Tetrafluoride, SiF<sub>4</sub>, 39  
 Silicon Tetraiodide, SiI<sub>4</sub>, 39  
 Sillimanite, Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub>, 111  
 Silver, Ag, 9  
 Silver Antimony Sulfide, AgSbS<sub>2</sub>, 40  
 Silver Chlorite, AgClO<sub>2</sub>, 40  
 Silver Cyanide, AgCN, 16  
 Silver Fluorate, AgFO<sub>3</sub>, 54  
 Silver Iodide, AgI, 16  
 Silver Iodide, AgI (low), 16  
 Silver Iodide, AgI (high), 16  
 Silver Mercuric Iodide,  $\alpha$ -Ag<sub>2</sub>HgI<sub>4</sub>, 64  
 Silver Mercuric Iodide,  $\beta$ -Ag<sub>2</sub>HgI<sub>4</sub>, 64  
 Silver Nitrate Diammoniate,  
 $AgNO_3 \cdot 2NH_3$ , 82  
 Silver Nitrate Urea,  
 $AgNO_3 \cdot CO(NH_2)_2$ , 131  
 Silver Periodate, AgIO<sub>4</sub>, 64  
 Silver Perrhenate, AgReO<sub>4</sub>, 64  
 Silver Selenate, Ag<sub>2</sub>SeO<sub>4</sub>, 64  
 Silver Sulfate, Ag<sub>2</sub>SO<sub>4</sub>, 64  
 Silver Sulfate Tetrammoniate,  
 $Ag_2SO_4 \cdot 4NH_3$ , 83  
 Silver Sulfide, Ag<sub>2</sub>S, 24  
 Silver Telluride, Ag<sub>2</sub>Te, 24  
 Skolopsite, 125  
 Sodalite, Na<sub>4</sub>Al<sub>3</sub>Si<sub>3</sub>O<sub>12</sub>Cl, 111  
 Sodium Bicarbonate, NaHCO<sub>3</sub>, 55  
 Sodium Bromide Dihydrate,  
 $NaBr \cdot 2H_2O$ , 82  
 Sodium Carbonate Monohydrate,  
 $Na_2CO_3 \cdot H_2O$ , 82  
 Sodium Columbate, NaCbO<sub>3</sub>, 55  
 Sodium Cyanate, NaCNO, 38  
 Sodium Cyanide, NaCN, 17  
 Sodium Ferrite, Na<sub>2</sub>Fe<sub>2</sub>O<sub>4</sub>, 41  
 Sodium Fluophosphate,  
 $2Na_3PO_4 \cdot NaF \cdot 19H_2O$ , 86  
 Sodium Hydrosulfide, NaHS (low), 17  
 Sodium Hydrosulfide, NaHS (high), 18  
 Sodium Iodide Dihydrate, NaI·2H<sub>2</sub>O, 82  
 Sodium Nitrate, NaNO<sub>3</sub>, 55  
 Sodium Nitrite, NaNO<sub>2</sub>, 41  
 Sodium Selenide, Na<sub>2</sub>Se, 25  
 Sodium Sulfate, Na<sub>2</sub>SO<sub>4</sub>, 66  
 Sodium Sulfide, Na<sub>2</sub>S, 25  
 Sodium Sulfite, Na<sub>2</sub>SO<sub>3</sub>, 55  
 Sodium Tantalate, NaTaO<sub>3</sub>, 55  
 Sodium Telluride, Na<sub>2</sub>Te, 25  
 Sodium Trinitride, NaN<sub>3</sub>, 38  
 Sodium Tungstate, NaWO<sub>3</sub>, 55  
 Sperrylite. See *Platinum Arsenide*  
 Spessartite, 128  
 Spinel. See *Magnesium Aluminate*

- Spodumene,  $\text{LiAl}(\text{SiO}_3)_2$ , 111  
 SrPb<sub>3</sub>, 39  
 SrTl, 18  
 Stannite,  $\text{Cu}_2\text{FeSnS}_4$ , 65  
 Stannous Oxide,  $\text{SnO}$ , 18  
 $\alpha$ -Stearic Acid,  $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ , 138  
 $\beta$ -Stearic Acid,  $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ , 139  
 Stephanite,  $\text{Ag}_8\text{SbS}_4$ , 64  
 Stibiconite,  $\text{Sb}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , 82  
 Stibnite. See *Antimony Trisulfide*  
 Stilbene,  $\text{C}_6\text{H}_6\text{CH}=\text{CHC}_6\text{H}_5$ , 148  
 Stilbene+2 mol. 1, 3, 5 Trinitrobenzene,  
 $\text{C}_6\text{H}_5\text{CH}=\text{CHC}_6\text{H}_5 \cdot 2[\text{C}_6\text{H}_3(\text{NO}_2)_3]$ ,  
 149  
 Strontium Aluminate,  $\text{Sr}_3[\text{Al}(\text{OH})_6]_2$ , 79  
 Strontium Boride,  $\text{SrB}_6$ , 40  
 Strontium Bromide Hexahydrate,  
 $\text{SrBr}_2 \cdot 6\text{H}_2\text{O}$ , 86  
 Strontium Carbide,  $\text{SrC}_2$ , 26  
 Strontium Chloride Hexahydrate,  
 $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ , 86  
 Strontium Fluoride,  $\text{SrF}_2$ , 26  
 Strontium Hafnate,  $\text{SrHfO}_3$ , 56  
 Strontium Imide,  $\text{SrNH}$ , 18  
 Strontium Iodide Hexahydrate,  
 $\text{SrI}_2 \cdot 6\text{H}_2\text{O}$ , 86  
 Strontium Nickel Nitrite,  $\text{Sr}_2\text{Ni}(\text{NO}_2)_6$ , 79  
 Strontium Nitrate,  $\text{Sr}(\text{NO}_3)_2$ , 56  
 Strontium Oxide,  $\text{SrO}$ , 18  
 Strontium Peroxide Octahydrate,  
 $\text{SrO}_2 \cdot 8\text{H}_2\text{O}$ , 86  
 Strontium Zirconate,  $\text{SrZrO}_3$ , 56  
 Styphnic Acid,  $\text{C}_6\text{H}(\text{OH})_2(\text{NO}_2)_3$ , 147  
 $\alpha$ -Succinic Acid,  $\text{COOH}(\text{CH}_2)_2\text{COOH}$ , 138  
 $\beta$ -Succinic Acid,  $\text{COOH}(\text{CH}_2)_2\text{COOH}$ , 138  
 Sulfur, S, 11  
 Sulphohalite,  $2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$ , 100  
 Sulvanite. See *Copper Vanadium Sulfide*  
 Swedenborgite,  $\text{NaSbO}_3 \cdot 4\text{BeO}$ , 55  
 Synchisite,  $\text{CaCO}_3 \cdot \text{RFeCO}_3$ , 54  
  
 Ta<sub>2</sub>C, 26  
 Talc,  $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ , 111  
 Tantalum, Ta, 11  
 Tantalum Carbide, TaC, 18  
 Telluric Acid,  $\text{Te}(\text{OH})_6$ , 40  
 Telluric Acid,  $\text{Te}(\text{OH})_6$  (second form), 40  
 Tenorite. See *Cupric Oxide*  
 Terphenyl. See *p-Diphenylbenzene*  
 1, 3, 4, 5 Tetraacetyl- $\beta$ -d-Fructopyranose,  
 141  
 Tetradymite,  $\text{Bi}_2\text{Te}_2\text{S}$ , 100  
 Tetramethyl Ammonium Fluosilicate,  
 $[\text{N}(\text{CH}_3)_4]_2\text{SiF}_6$ , 133  
 1, 3, 4, 5 Tetramethyl- $\beta$ -d-Fructopyranose,  
 141  
 2, 3, 5, 6 Tetramethyl- $\gamma$ -d-Mannonolac-  
 tone, 142  
 Tetramminopalladous Chloride Monohy-  
 drate,  $\text{Pd}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O}$ , 83  
 Tetramminoplatinous Chloride Monohy-  
 drate,  $\text{Pt}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O}$ , 83  
 1, 2, 4, 6 Tetranitrobenzene,  
 $\text{C}_6\text{H}_2(\text{NO}_2)_4$ , 147  
 Tetraphosphonitrile,  $(\text{PNCl}_2)_4$ , 100  
 Thallium, Tl, 11  
 Thallium Arsenic Sulfide,  $\text{TlAsS}_3$ , 41  
 Thallium Chloride,  $\text{TlCl}$ , 18  
 Thallium Cyanide,  $\text{TlCN}$ , 18  
 Thallous Chloroplatinate,  $\text{Tl}_2\text{PtCl}_6$ , 80  
 Thallous Chlorostannate,  $\text{Tl}_2\text{SnCl}_6$ , 80  
 Thallous Chlorotellurite,  $\text{Tl}_2\text{TeCl}_6$ , 80  
 Thallous Cobaltinitrite,  $\text{Tl}_3\text{Co}(\text{NO}_2)_6$ , 79  
 Thallous Fluosilicate,  $\text{Tl}_2\text{SiF}_6$ , 80  
 Thallous Iridium Nitrite,  $\text{Tl}_3\text{Ir}(\text{NO}_2)_6$ , 80  
 Thallous Osmiamate,  $\text{TlOsNO}_3$ , 66  
 Thallous Perrhenate,  $\text{TlReO}_4$ , 66  
 Thallous Rhodium Nitrite,  $\text{Tl}_3\text{Rh}(\text{NO}_2)_6$ ,  
 80  
 Thallous Thiocyanate,  $\text{TlCNS}$ , 39  
 Thaumassite,  
 $\text{CaCO}_3 \cdot \text{CaSO}_4 \cdot \text{CaSiO}_3 \cdot 15\text{H}_2\text{O}$ , 111  
 Thiophene,  $\text{C}_4\text{H}_4\text{S}$ , 159  
 Thiourea,  $\text{CS}(\text{NH}_2)_2$ , 136  
 Thomsonite,  
 $\text{NaCa}_2\text{Al}_6\text{Si}_5\text{O}_{20} \cdot 6\text{H}_2\text{O}$ , 111  
 Thorium Boride,  $\text{ThB}_6$ , 40  
 TiAl<sub>3</sub>, 39  
 Tilasite,  $\text{CaMg}(\text{OH})\text{AsO}_4$ , 64  
 Tin, Sn, 11  
 Tin Arsenide,  $\text{SnAs}$ , 18  
 Titanium Carbide, TiC, 18  
 Titanium Tetrabromide,  $\text{TiBr}_4$ , 39  
 Titanium Tetraiodide,  $\text{TiI}_4$ , 39  
 $\text{Ti}_2(\text{VF}_6 \cdot \text{H}_2\text{O})$ , 82  
 Tolane,  $\text{C}_6\text{H}_5\text{C} \equiv \text{CC}_6\text{H}_5$ , 149  
 o-Tolidine,  $(\text{C}_6\text{H}_3\text{CH}_2\text{NH}_2)_2$ , 150  
 Tricalcium Phosphate Hydrate,  
 $\text{Ca}_3(\text{H}_2\text{O})_2(\text{PO}_4)_6$ , 69  
 Trihydroxyoestrin, 162  
 2, 3, 4 Trimethyl- $\delta$ -l-Arabanolactone, 141  
 2, 3, 5 Trimethyl- $\gamma$ -l-Rhamnonolactone,  
 142  
 2, 3, 4 Trimethyl- $\alpha$ -d-Xylopyranose, 141  
 2, 4, 6 Trinitroaniline,  
 $\text{C}_6\text{H}_2(\text{NH}_2)(\text{NO}_2)_3$ , 147  
 2, 4, 6 Trinitrobenzene,  
 $\text{C}_6\text{H}_3\text{Br}(\text{NO}_2)_3$ , 147  
 2, 4, 6 Trinitrochlorobenzene,  
 $\text{C}_6\text{H}_2\text{Cl}(\text{NO}_2)_3$ , 147  
 2, 4, 6 Trinitrodiphenylamine,  
 $\text{C}_6\text{H}_5(\text{NH})\text{C}_6\text{H}_2(\text{NO}_2)_3$ , 148  
 2, 4, 6 Trinitroiodobenzene,  
 $\text{C}_6\text{H}_2\text{I}(\text{NO}_2)_3$ , 147  
 2, 4, 6 Trinitrophenylglucitol,  
 $\text{C}_6(\text{OH})_3(\text{NO}_2)_3$ , 148  
 2, 4, 6 Trinitrotoluene,  
 $\text{C}_6\text{H}_2\text{CH}_3(\text{NO}_2)_3$ , 147  
 Triphenylbenzene,  $\text{C}_6\text{H}_3(\text{C}_6\text{H}_5)_3$ , 152  
 Triphenyl Bismuthine Dichloride, 131  
 Triphosphonitrile,  $(\text{PNCl}_2)_3$ , 100  
 Triphylite,  $\text{Li}(\text{Fe}, \text{Mn})\text{PO}_4$ , 65  
 Troilite. See *Ferrous Sulfide*  
 Tungsten, W, 11  
 Tungsten, W (second form), 11  
 Tungsten Carbide, WC, 18  
 Tungsten Oxide,  $\text{W}_4\text{O}_{11}$  (tetragonal), 40  
 Tungstic Trioxide,  $\text{WO}_3$ , 39  
 Tychite,  $2\text{MgCO}_3 \cdot 2\text{Na}_2\text{CO}_3 \cdot \text{Na}_2\text{SO}_4$ , 100  
 Tysonite,  $(\text{Ce}, \text{La}, \dots)\text{F}_3$ , 38

- Ultramarines, 112  
 Uranium, U, 11  
 Urea,  $\text{CO}(\text{NH}_2)_2$ , 136  
  
 Vanadinite,  $\text{Pb}_{10}\text{Cl}_2(\text{VO}_4)_6$ , 66, 69  
 Vanadium Oxide, VO, 18  
 Veramon, 160  
 Veronal. See *5, 5 Diethyl Barbituric Acid*  
 Vesuvianite, 112  
 Vitamin B<sub>1</sub> Hydrochloride,  
 $\text{C}_{12}\text{H}_{18}\text{O}_2\text{N}_4\text{S}_2\text{HCl}$ , 163  
 Voltaites, 100  
  
 Wavellite,  $4(\text{PO}_4)\text{Al}\cdot 2\text{Al}(\text{OH})_3\cdot 9\text{H}_2\text{O}$ , 86  
 Wöhlerite,  $(\text{SiO}_4)_2(\text{ZrF,CbO})\text{Ca}_2\text{Na}$ , 112  
 Wolfsbergite. See *Copper Antimony Sulfide*  
 Wollastonite,  $\text{CaSiO}_3$ , 112  
  
 Xenon, X, 11  
 l-Xylose,  $\text{C}_5\text{H}_{10}\text{O}_5$ , 140  
  
 Ytterbium Boride,  $\text{YtB}_6$ , 40  
 Yttrium, Y, 11  
 Yttrium Borate,  $\text{YBO}_3$ , 56  
 Yttrium Boride,  $\text{YB}_6$ , 40  
 Yttrium Carbide,  $\text{YC}_2$ , 26  
 Yttrium Vanadate,  $\text{YVO}_4$ , 66  
  
 Zinc, Zn, 11  
 Zinc Aluminate,  $\text{ZnAl}_2\text{O}_4$ , 66  
  
 Zinc Ammonium Sulfate Hexahydrate,  
 $\text{ZnSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , 86  
 Zinc Bromide Hexammoniate,  
 $\text{ZnBr}_2\cdot 6\text{NH}_3$ , 86  
 Zinc Carbonate,  $\text{ZnCO}_3$ , 56  
 Zinc Chloride (basic),  
 $\text{ZnCl}_2\cdot 4\text{Zn}(\text{OH})_2$ , 100  
 Zinc Chlorite Dihydrate,  
 $\text{Zn}(\text{ClO}_2)_2\cdot 2\text{H}_2\text{O}$ , 83  
 Zinc Chromite,  $\text{ZnCr}_2\text{O}_4$ , 66  
 Zinc Ferrite,  $\text{ZnFe}_2\text{O}_4$ , 66  
 Zinc Hydroxide,  $\text{Zn}(\text{OH})_2$ , 26  
 Zinc Iodide Hexammoniate,  
 $\text{ZnI}_2\cdot 6\text{NH}_3$ , 86  
 Zinc Oxide,  $\text{ZnO}$ , 18  
 Zinc Perchlorate Tetrammoniate,  
 $\text{Zn}(\text{ClO}_4)_2\cdot 4\text{NH}_3$ , 83  
 Zinc Phosphide,  $\text{Zn}_3\text{P}_2$ , 36  
 Zinc Stannate,  $\text{Zn}_2\text{SnO}_4$ , 67  
 Zinc Sulfate,  $\text{ZnSO}_4$ , 67  
 Zinc Thiochromite,  $\text{ZnCr}_2\text{S}_4$ , 66  
 Zinc Titanate,  $\text{Zn}_2\text{TiO}_4$ , 67  
 Zirconium, Zr, 11  
 Zirconium Carbide,  $\text{ZrC}$ , 18  
 Zirconium Tetrafluoride,  $\text{ZrF}_4$ , 39  
 $\text{Zn}_3\text{As}_2$ , 36  
 Zoisite,  $(\text{SiO}_4)_3\cdot \text{Al}_2\text{Ca}_2(\text{AlOH})$ , 112  
 ZrW<sub>2</sub>, 26  
 Zunyite,  $\text{Al}_{18}\text{Si}_6\text{O}_{20}(\text{OH},\text{F})_{18}\text{Cl}$ , 112



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