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Unit cell and space group of $NiK_2(SO_4)_2.6H_2O$.

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No X-ray data have been reported for $NiK_2(SO_4)_2.6H_2O$, one of the Tutton salts. Optical studies of Tutton and of Murmann and Rotter (Mellor, 1936) show the blue green prismatic crystals to be monoclinic. Data obtained in this laboratory confirm the symmetry and revise previously reported axial ratios.

The d values and relative intensities given in Table 1 were obtained from X-ray powder photographs using cobalt radiation. Lattice constants were determined by superposition of single crystal reflections of quartz on zero level Weissenberg photographs about the a and b axes. The monoclinic angle was taken from a zero level precession photograph. These data show

$$a = 6\cdot130 \pm 0\cdot002,\ b = 12\cdot185 \pm 0\cdot004,\ c = 8\cdot991 \pm 0\cdot002\ \text{Å},\ \beta = 104°\ 59' \pm 3'.$$

The axial ratios found here are 0·5031:1:0·7379 as compared to 0·5020:1:0·7379 by Tutton and 0·4965:1:0·7374 by Murmann and Rotter (Mellor, 1936). For $Z = 2$ the calculated density is 2·237 g.cm.$^{-3}$ which agrees with the observed density 2·232 g.cm.$^{-3}$ obtained by flotation methods. The single crystal photographs indicate the following conditions for non-extinction: $h0l$ present only for $l = 2n$ and $0k0$ present only for $k = 2n$. The space group therefore is $P2_1/c$–C_{2h}^5.

Reference

MELLOR, J. W. (1936). *A Comprehensive Treatise on Inorganic and Theoretical Chemistry*, Vol. XV, p. 469. London: Longmans, Green, and Co.

Table 1. *Powder diffraction data for* $NiK_2(SO_4)_2.6H_2O$

d_o	I/I_0	hkl
6·08	20	020
5·90	5	100
5·31	20	110
5·11	10	$11\bar{1}$
4·97	5	021
4·35	20	002
4·24	15	120
4·13	75	111 $12\bar{1}$
4·03	75	$10\bar{2}$
3·82	5	$11\bar{2}$
3·67	100	031
3·54	15	121 022
3·35	10	130
3·29	20	$13\bar{1}$
3·13	15	102
3·04	30	040 112
2·962	55	$21\bar{1}$ 200
2·870	5	041
2·800	20	$20\bar{2}$
2·776	15	122
2·727	15	$22\bar{1}$
2·672	5	$14\bar{1}$
2·628	5	$12\bar{3}$
2·546	5	$22\bar{2}$ 211
2·485	10	042 132
2·433	5	$23\bar{1}$ $14\bar{2}$
2·386	5	221 230
2·364	50	$13\bar{3}$
2·249	5	150
2·231	5	$15\bar{1}$ $10\bar{4}$
2·182	20	142
2·158	5	212 $24\bar{1}$
2·121	10	240
2·100	5	$14\bar{3}$ 043 $12\bar{4}$
2·090	5	$15\bar{2}$
2·063	10	222 $24\bar{2}$
2·039	15	024
2·000	5	$30\bar{2}$
1·974	10	$31\bar{2}$ 300
1·917	10	160
1·881	5	250
1·866	5	$15\bar{3}$ 114
1·847	5	$31\bar{3}$
1·825	5	$33\bar{1}$ 213
1·813	10	$16\bar{2}$
1·799	5	$14\bar{4}$ $33\bar{2}$

Additional lines not listed.

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The beryllides of Ti, V, Cr, Zr, Nb, Mo, Hf, and Ta.*

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(*Received* 1 *April* 1960)

A thorough X-ray diffraction study has been made of the beryllides of Ti, V, Cr, Zr, Nb, Mo, Hf, and Ta. Table 1 shows the complete list of intermetallic compounds that are stable or metastable at room temperatures.

Cell dimensions heretofore not reported are given in

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* This work was performed under the auspices of the U.S. Atomic Energy Commission.

Table 1. *Structure types and stoichiometry of some transition element beryllides*

Type	V	Cr	Mo	Nb	Ta	Ti	Hf	Zr
U_3Si_2	—	—	—	Nb_3Be_2(8)	Ta_3Be_2(*)	—	—	—
AlB_2	—	—	—	—	—	—	$HfBe_2$(*)	$ZrBe_2$(3)
$MgCu_2$	—	—	—	$NbBe_2$(7)	$TaBe_2$(*)	$TiBe_2$(1. 2)	—	—
$MgZn_2$	VBe_2(1)	$CrBe_2$(1, 12, 13)	$MoBe_2$(1, 10)	—	—	—	—	—
$NbBe_3$	—	—	—	$NbBe_3$(7)	$TaBe_3$(*)	$TiBe_3$(*)	—	—
$CaZn_5$	—	—	—	—	—	—	$HfBe_5$(*)	$ZrBe_5$(4)
Nb_2Be_{17}	—	—	—	Nb_2Be_{17}(9)	Ta_2Be_{17}(*)	α Ti_2Be_{17}(*)	α-$HfBe_{17}$(*)	Zr_2Be_{17}(4)
Th_2Ni_{17}	—	—	—	—	—	β-Ti_2Be_{17}(*)	β-Hf_2Be_{17}(*)	—
$ThMn_{12}$	VBe_{12}(6, 14)	$CrBe_{12}$(6, 14)	$MoBe_{12}$(1, 10, 11)	$NbBe_{12}$(6)	$TaBe_{12}$(1, 14)	$TiBe_{12}$(*)	—	—
$NaZn_{13}$	—	—	—	—	—	—	$HfBe_{13}$(*)	$ZrBe_{13}$(5)

1. Misch (1936).
2. Ehrlich (1949).
3. Nielsen & Baenziger (1954).
4. Zalkin, Bedford & Sands (1959).
5. Baenziger & Rundle (1949).
6. Kripyakevich & Gladyschevskii (1955).
7. Sands, Zalkin & Krikorian (1959).
8. Zalkin, Sands & Krikorian (1960).
9. Zalkin, Sands & Krikorian (1959).
10. Gordon, McGurty, Klein & Koshuba (1951).
11. Rauechle & Batchelder (1955).
12. Zaharova & Dalnov (1938).
13. Edwards & Johnstone (1956).
14. Batchelder & Raeuchle (1957).

* See Table 2.

Table 2. Rhombohedral types are reported with the dimensions of their triply primitive hexagonal cells.

Table 2. *Cell dimensions of some transition element beryllides*

Compound	*a*	*c*	Lattice type
$HfBe_2$	3·787 Å	3·159 Å	hexagonal
$TaBe_2$	6·51		cubic
Ta_3Be_2	6·50	3·32	tetragonal
$TiBe_3$	4·49	21·32	rhombohedral
$TaBe_3$	4·53	20·95	rhombohedral
$HfBe_5$	4·519	3·465	hexagonal
Ta_2Be_{17}	7·388	10·74	rhombohedral
α-Ti_2Be_{17}	7·392	10·79	rhombohedral
α-Hf_2Be_{17}	7·494	10·93	rhombohedral
β-Ti_2Be_{17}	7·36	7·30	hexagonal
β-Hf_2Be_{17}	7·44	7·38	hexagonal
$TiBe_{12}$	7·35	4·19	tetragonal
$HfBe_{13}$	10·00		cubic

The cell lengths in Table 2 were taken from powder patterns photographed with chromium or copper *Kα* X-rays. The methods of preparation of the materials are similar to those previously reported by Zalkin, Bedford & Sands (1959); Sands, Zalkin & Krikorian (1959); and Zalkin, Sands & Krikorian (1959).

Both the Ti and the Hf systems exhibit two different structures for the M_2Be_{17} composition. The α form is isomorphous with the Nb_2Be_{17} type (Zalkin, Sands & Krikorian, 1959). The β form is isomorphous with the Th_2Ni_{17} type (Florio, Baenziger & Rundle, 1956), and is a new type for beryllides.

Table 3 shows the observed and calculated powder pattern of β-Hf_2Be_{17}. Only the geometrical part of the structure factor of the Hf atom positions was used for the calculated intensities. Powder patterns of β-Ti_2Be_{17} are very similar in appearance to those of β-Hf_2Be_{17}. Further proof of structure was found in the good agreement of the formular volumes of the α and β forms to each other; for the α- and β-Hf_2Be_{17} the formular volumes are 177·2 and 176·9 Å³, respectively; for the α- and β-Ti_2Be_{17} they are 170·3 and 171·2 Å³, respectively.

At the present time we do not know which of the two forms of M_2Be_{17} is the room-temperature-stable form.

Table 3. *Observed and calculated powder patterns for* β-Hf_2Be_{17}

hkl	I_c†	I_o*	*d*
001	0	< 0·2	
100	1·5	0·5	6·45 Å
101	9	5	4·82
110	24	12	3·72
002	8	3	3·68
111	0	< 1	
200	1·5	1	3·18
102	3		
201	9	10	2·95
112	48	31	2·61
003	0	< 3	
210	3	3	2·43
202	3	< 3	
211	18	17	2·31
103	9	9	2·29
300	24	29	2·15
301	0	< 3	
113	0	< 3	
212	6	5	2·03
203	9	16	1·95
220	24	72	1·86
302	48		
004	8	12	1·84

* Cu *Kα* X-rays, $\lambda = 1{\cdot}5418$ Å; corrected for Lorenz and polarization effects; no absorption correction.

† Includes only the geometrical factor of the heavy-metal atom position.

Powder patterns of pure α, pure β, and α, β mixtures were obtained in both the Ti and Hf systems. The β patterns seemed to be associated with the Be-rich side and the α patterns with the Be-poor side.

The disordered hexagonal $TiBe_{12}$ reported by Raeuchle & Rundle (1952) was not observed in the powder patterns.

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Crystalline hydrous tin (II) oxide.

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Hydrous tin (II) oxide (tin (II) hydroxide) is normally prepared by the addition of a solution of an alkali or alkali carbonate to the solution of a tin (II) salt under a non-oxidising atmosphere (Ditte, 1882; Bury & Partington, 1922; Britton, 1925; Weiser & Milligan, 1932). The product is a powder with crystallites, which are not large enough to be seen under a microscope.

It was found, however, that a crystalline material with an X-ray diffraction powder pattern identical with that of hydrous tin (II) oxide can be prepared by the hydrolysis of a basic tin (II) nitrate (Donaldson, 1959). The basic nitrate is hydrolysed by storage under oxygen-free distilled water until yellow nuggets of hydrous tin (II) oxide are deposited. The preparation presumably depends upon the fact that the rate of hydrolysis of basic tin (II) nitrate is slow enough to permit orderly crystal formation. The crystalline product contains some tin (IV) material but is sufficiently pure for study by X-ray diffraction.

In a recent publication, Weber (1959) deals with crystalline tin (II) materials containing silica but which do not have the same X-ray diffraction powder data as hydrous tin (II) oxide. These materials cannot, therefore, be crystalline tin (II) hydroxide as suggested by the title of the publication.

From X-ray diffraction single-crystal rotation, oscillation and Weissenberg photographs with Cu $K\alpha$ radiation it was found that the crystals of hydrous tin (II) oxide were triclinic with unit-cell dimensions:

$$a=11{\cdot}5,\ b=6{\cdot}03,\ c=19{\cdot}8\ \text{Å};$$
$$\alpha=99^\circ,\ \beta=60^\circ\ 30',\ \gamma=88^\circ\ 30';$$
$$U=1175\ \text{Å}^3.$$

The reciprocal cell dimensions are:

$$a^*=0{\cdot}1005,\ b^*=0{\cdot}1686,\ c^*=0{\cdot}0590\ \text{Å}^{-1};$$
$$\alpha^*=79^\circ\ 30',\ \beta^*=120^\circ,\ \gamma^*=96^\circ.$$

Most of the lines in the powder pattern of hydrous tin (II) oxide were found to be due to more than one reflection and thus indexing was impracticable.

Chemical analyses of hydrous tin (II) oxide have given three formulae; $3\,SnO.2\,H_2O$ (Bury & Partington, 1922), $4\,SnO.2\,H_2O$ (Weiser & Milligan, 1932) and more recently $5\,SnO.2\,H_2O$ (Donaldson, 1959). Unit-cell contents based on each of these formulae, together with the observed density of $4{\cdot}71 \pm 0{\cdot}06$ g.cm.$^{-3}$ (by displacement of various liquids) and the cell volume are as follows:

	Cell contents		
Composition	Tin	Oxygen	Hydrogen
$3\,SnO.2\,H_2O$	22·5	37·5	30·0
$4\,SnO.2\,H_2O$	23·2	34·8	23·2
$5\,SnO.2\,H_2O$	23·5	32·9	18·8

Of these the nearest to a possible cell is $5\,SnO.2\,H_2O$ (a cell content of $Sn_{24}O_{14}(OH)_{20}$ is possible from lattice considerations). The composition $Sn_{24}O_{14}(OH)_{20}$, which can also be written $24\,SnO.10\,H_2O$, could not easily be distinguished from $5\,SnO.2\,H_2O$ by chemical analysis. The density calculated for the idealised formula is 4·82 g.cm.$^{-3}$.

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