

THE AMERICAN MINERALOGIST, VOL. 55, MARCH-APRIL, 1970

CELL DIMENSIONS OF THE HYDRATED PHOSPHATE, KINGITE

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ABSTRACT

Cell dimensions of kingite, $\text{Al}_3[(\text{OH})_3/(\text{PO}_4)_2] \cdot 9\text{H}_2\text{O}$, have been determined using electron and X-ray powder diffraction data: $a=9.15 \pm 0.01 \text{ \AA}$, $b=10.00 \pm 0.01 \text{ \AA}$, $c=7.24 \pm 0.02 \text{ \AA}$, $\alpha=98.6 \pm 0.1^\circ$, $\beta=93.6 \pm 0.1^\circ$, $\gamma=93.2 \pm 0.1^\circ$, $V=652 \pm 2 \text{ \AA}^3$, $Z=2$, $\rho_{\text{calc.}}=2.465$, $\rho_{\text{meas.}}=2.2-2.3$.

Kingite, $\text{Al}_3[(\text{OH})_3/(\text{PO}_4)_2] \cdot 9\text{H}_2\text{O}$, is a hydrated aluminum phosphate mineral first described and named by Norrish *et al.* (1957) who studied a specimen from the Fairview phosphate workings, Robertstown, South Australia.

Physical, chemical, differential thermal and X-ray diffraction data were recorded by Norrish *et al.* but its powder diffraction pattern could not be indexed by them for the low symmetry of kingite.

Fine dispersions of kingite type specimen have now been examined by electron microscopy and single-crystal electron diffraction patterns recorded for the preferred orientation taken up by the crystals on the stage of the microscope. Individual crystals are either platelets or irregular thick fragments and are about $0.5 \mu\text{m}$ to $1.0 \mu\text{m}$ in size. These crystals are, however, unstable to longer exposure under the strong beam due to the heating in the electron beam; this is consistent with the dehydration data of Norrish *et al.*

The obtained electron diffraction patterns show an oblique array suggesting monoclinic or triclinic symmetry. The kingite was heavily shadowed by aluminum to give reference diffraction rings for calibration purposes.

This led to the following trial values for certain unit cell parameters, viz., $c=7.14$, $b=9.78 \text{ \AA}$, $\alpha=98^\circ$. X-ray powder diffraction data were obtained using a Philips wide-angle diffractometer, and one of the three longest reflections was assumed to be $d(100)=9.08 \text{ \AA}$ for the trial base. (The other two reflections, 9.8 \AA and 7.2 \AA were assumed to be (010) and (001), respectively.)

These trial parameters were then used as input data for two computer programs which generate all possible spacings within the monoclinic and triclinic systems. Observed and calculated spacings were compared and indices assigned where there was a reasonable fit. These indexed reflections were subsequently used in a least squares programs for refinement of lattice constants, and the whole trial and error process repeated, in

TABLE. X-RAY POWDER DATA FOR KINGITE

Calculated		Measured ^a		Calculated		Measured ^a	
<i>hkl</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>hkl</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)
010	9.866	9	9.8	122	2.529	3	2.529
100	9.112	100	9.1	32 $\bar{1}$	2.480	7	2.476
001	7.140	6	7.2	0 $\bar{4}$ 1	2.450	5	2.446
$\bar{1}$ 10	6.926	24	6.85			1	2.404
0 $\bar{1}$ 1	6.259	14	6.27	11 $\bar{3}$	2.353	1	2.365
101	5.433	28	5.43			5	2.336
11 $\bar{1}$	5.211	52	5.28	231	2.315	7	2.300
1 $\bar{1}$ 1	5.109	24	5.10			2	2.292
		4	4.96			2	2.274
$\bar{1}$ 11	4.845	5	4.86			3	2.269
1 $\bar{2}$ 0	4.577	18	4.61			1	2.255
200	4.556	5	4.55			3	2.248
0 $\bar{2}$ 1	4.386	8	4.36	3 $\bar{3}$ 1	2.232	7	2.233
12 $\bar{1}$	3.930	15	3.93			4	2.216
		1	3.75			4	2.208
$\bar{1}$ 21	3.620	6	3.60			3	2.196
220	3.463	65	3.48			1	2.189
121	3.417	80	3.45			1	2.182
11 $\bar{2}$	3.350	27	3.38	331	2.163	2	2.159
		2	3.29			2	2.148
1 $\bar{1}$ 2	3.244	2	3.24			3	2.127
221	3.183	39	3.17	322	2.116	14	2.112
$\bar{1}$ 30	3.161	15	3.14			3	2.054
1 $\bar{1}$ 2	3.107	30	3.108			1	2.041
221	3.053	14	3.066	421	2.035	6	2.026
131	3.031	10	3.018			2	2.005
13 $\bar{1}$	2.972	24	2.975	332	1.988	8	1.990
112	2.944	4	2.940	050	1.973	13	1.974
122	2.941	2	2.893	15 $\bar{1}$	1.918	3	1.922
310	2.850	9	2.846	233	1.912	1	1.914
		6	2.837	042	1.898	5	1.900
311	2.757	7	2.759	233	1.882	1	1.882
3 $\bar{1}$ 1	2.710	3	2.712	412	1.875	1	1.875
		2	2.667	340	1.856	3	1.858
131	2.637	12	2.640	051	1.831	13	1.835
		9	2.617	510	1.814	1	1.814
230	2.587	5	2.590	1 $\bar{1}$ 4	1.806	3	1.805
		8	2.568	25 $\bar{1}$	1.787	2	1.788
222	2.554	4	2.551			4	1.722

^a Data measured by Norrish *et al.* Co-K α radiation, $\lambda = 1.7889$ Å.

both crystal systems. The calculation assuming monoclinic symmetry did not converge. The cell parameters obtained from the final least squares adjustment, assuming triclinic symmetry, are as follows:

$a = 9.15 \pm 0.01 \text{ \AA}$, $b = 10.00 \pm 0.01 \text{ \AA}$, $c = 7.24 \pm 0.02 \text{ \AA}$, $\alpha = 98.6 \pm 0.1^\circ$, $\beta = 93.6 \pm 0.1^\circ$, $\gamma = 93.2 \pm 0.1^\circ$, $V = 652 \pm 2 \text{ \AA}^3$, $Z = 2$ (using above described chemical formula), $\rho_{\text{calc}} = 2.465$, $\rho_{\text{meas}} = 2.2\text{--}2.3$. (by Norrish *et al.*)

Meta-kingite, which is obtained by dehydrating kingite at 160°C and gives the same X-ray pattern as that reported by Norrish *et al.*, gives insufficient diffraction spots in the electron diffraction pattern to allow a similar calculation of unit-cell parameters.

The author would like to acknowledge Drs. K. Norrish and E. W. Radoslovich, Soils Division (Adelaide), C.S.I.R.O., for the sample, X-ray data and facilities. The C.D.C. 3600 computer at the Computing Centre of C.S.I.R.O. and HITAC 5020E computer at the Computing Centre of University of Tokyo were used.

REFERENCE

- NORRISH, K., L. E. R. ROGERS AND R. E. SHAPTER (1957) Kingite, a new hydrated aluminum phosphate mineral from Robertstown, South Australia. *Mineral. Mag.* **31**, 351-357.

THE AMERICAN MINERALOGIST, VOL. 55, MARCH-APRIL, 1970

THE SYNTHESIS OF CHLORITOID AT LOW PRESSURES

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ABSTRACT

At oxygen fugacities along the Ni-NiO buffer, triclinic chloritoid has been synthesized from oxide mixes at water pressures between 8.7 kbar and 1 kbar and at temperatures between 650°C and 525°C respectively. The data indicates that the 7 \AA chlorite assemblage, previously considered to be the low pressure metastable equivalent of chloritoid, is unstable with respect to chloritoid.

The mineral chloritoid, $\text{FeAl}_2\text{SiO}_5(\text{OH})_2$, has been the subject of several experimental studies in recent years. (Halferdahl, 1961, Ganguly and Newton, 1968). Although it has been described from various apparently low-pressure geological environments, including contact metamorphic aureoles, chloritoid has never been successfully synthesized below pressures of the order of 10 kbar. The lowest pressure of synthesis reported is at 9.2 kbar and 650°C by Halferdahl (1961).

In previous low pressure studies on chloritoid by Halferdahl and on iron cordierite (composition: chloritoid + SiO_2 , Fig. 1) by Schreyer: