SHORT STRUCTURAL PAPERS

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Freudenbergite

By T. Ishiguro, K. Tanaka, F. Marumo, M. G. M. U. Ismail, S. Hirano and S. Sōmiya

Research Laboratory of Engineering Materials, Tokyo Institute of Technology, O-okayama, Meguro-ku, Tokyo 152, *Japan*

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Abstract. $Na_2Fe_2Ti_6O_{16}$, monoclinic, $C2/m$, a = 12·267 (6), b = 3·823 (5), c = 6·483 (3) Å, β = 107·16 (5)°, Z = 4, D_x = 3·97 g cm^{-3}. The crystals were prepared by heating a mixture of Fe and TiO_2 in 10 M NaOH solution, sealed in a gold tube, at 650 °C and 1000 atm for 21 days. Black platy crystals were obtained. The substance is isostructural with $Na_{0.2}TiO_2$, with mean M(1)—O, M(2)—O and Na—O distances of 1·993, 1·973 and 2·715 Å respectively.

Introduction. The systematic absences observed on Weissenberg and precession photographs were hkl for $h + k$ odd, indicating the possible space groups Cm, $C2$ and $C2/m$. $C2/m$ gave a satisfactory result for the structure refinement. A platy crystal with dimensions about 0·09 × 0·16 × 0·04 mm was used for intensity collection. Intensities were measured on a Philips automated four-circle diffractometer, with Mo $K\alpha$ radiation monochromated with graphite, up to 2θ = 100° by the ω–2θ scan technique. The scan speed was 4° min^{-1} in ω and scanning was repeated twice when the total counts were less than 1000. The scan width was determined according to $(0·8 + 0·3\tan\theta)°$. Intensities were corrected for Lorentz–polarization and absorption effects (μ = 75·73 cm^{-1}). 960 independent reflection data, whose $|F|$'s were larger than $3\sigma(|F|)$, were obtained and used for the structure determination.

Table 1. *Final positional parameters of freudenbergite*

The y value is zero for all atoms.

		Population	x	z
M(1)	Ti	0·73 (2)	0·29759 (8)	0·7106 (2)
	Fe	0·27 (2)		
M(2)	Ti	0·77 (2)	0·39774 (8)	0·3023 (2)
	Fe	0·23 (2)		
Na		1·00 (4)	0	0
O(1)		1	0·3725 (4)	0·9958 (8)
O(2)		1	0·2366 (4)	0·3465 (8)
O(3)		1	0·1349 (4)	0·7094 (8)
O(4)		1	0·4412 (4)	0·6333 (8)

Table 2. *Interatomic distances* (Å)

Symmetry code

None	x,	y,	z	(ii)	x,	y,	$z - 1$
(i)	$\frac{1}{2} - x$,	$\frac{1}{2} + y$,	$1 - z$	(iii)	$1 - x$,	y,	$1 - z$

M(1)—O(1)	1·807 (5)	M(2)—O(4^{iii})	1·896 (5)
M(1)—O(3)	1·993 (5)	M(2)—O(4)	2·053 (5)
M(1)—O(2)	2·257 (5)	M(2)—O(2)	2·078 (5)
M(1)—O(4)	1·967 (5)	M(2)—O(3^{i})	1·947 (1) × 2
M(1)—O(2^{i})	1·966 (1) × 2	O(1^{ii})—O(4^{iii})	2·790 (6)
O(1)—O(3)	2·962 (6)	O(4^{iii})—O(4)	2·553 (8)
O(3)—O(2)	2·967 (8)	O(4)—O(2)	2·651 (6)
O(2)—O(4)	2·651 (6)	O(2)—O(1^{ii})	3·187 (8)
O(4)—O(1)	2·717 (8)	O(1^{ii})—O(3^{i})	2·720 (5) × 2
O(1)—O(2^{i})	2·935 (5) × 2	O(4^{iii})—O(3^{i})	2·974 (5) × 2
O(3)—O(2^{i})	2·568 (5) × 2	O(4)—O(3^{i})	2·869 (5) × 2
O(2)—O(2^{i})	2·707 (5) × 2	O(2)—O(3^{i})	2·568 (5) × 2
O(4)—O(2^{i})	2·930 (5) × 2	Na—O(1^{i})	2·463 (3) × 4
M(2)—O(1^{ii})	1·918 (5)	Na—O(4^{i})	2·967 (4) × 4

The positions of all atoms were obtained from the Patterson maps. The structure was refined with the full-matrix least-squares program *LINUS* (Coppens & Hamilton, 1970) and anisotropic temperature factors. The site populations of the Na^+, Fe^{3+} and Ti^{4+} ions were also varied by constraining the net charge of the crystal to be neutral and by assuming that no vacancy exists at either of the two crystallographically independent octahedral sites which are occupied by Fe^{3+} and Ti^{4+} ions in disorder. The final conventional R value became 0·053 after correction for the isotropic-extinction effect. The atomic scattering and dispersion-correction factors for Na^+, Fe^{3+} and Ti^{4+} were taken from *International Tables for X-ray Crystallography* (1974); for O^{2-}, values given by Tokonami (1965) were used. Final positional parameters are given in Table 1.* Interatomic distances are given in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33007 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Freudenbergite is a mineral first described by Frenzel (1961). McKie (1963) determined the cell dimensions (a = 12·305, b = 3·822, c = 6·500 Å and β = 107·30°). Wadsley (1964) commented on the close similarity of the powder pattern of freudenbergite to that of the synthetic sodium titanium dioxide 'bronze' $Na_{0.2}TiO_2$. McKie & Long (1970) determined the chemical composition of freudenbergite by EPMA and calculated the density to be 3·95 g cm^{-3}.

The structure viewed along the b axis is shown in Fig. 1. A population analysis of the metal sites indicated random distribution of the Ti^{4+} and Fe^{3+} ions over the two octahedral sites, M(1) and M(2), yielding the chemical formula $Na_{2.00}Fe_{2.00}Ti_{6.00}O_{16}$, which is exactly the same as that given by McKie & Long (1970). The (Ti,Fe)O_6 octahedra share edges to form double sheets parallel to (001). The sheets are further connected in the **c** direction by the octahedra sharing corners, thus forming a three-dimensional framework. The M(1)—O distances range from 1·81 to 2·26 Å (mean 1·99 Å), while the M(2)—O distances are in the range 1·90 to 2·05 Å (mean 1·97 Å). The Na site is almost fully occupied by Na^+ ions, with an environment of eight O atoms at the corners of a highly distorted cube. Four Na—O(1) bonds are 2·46 Å and the remaining four Na—O(4) bonds are 2·97 Å, so that there is a pronounced tendency towards a square-planar coordination. Atomic parameters and interatomic distances, as well as cell dimensions, are close to those of the sodium titanium bronze, $Na_{0.2}TiO_2$, given by Wadsley (1964).

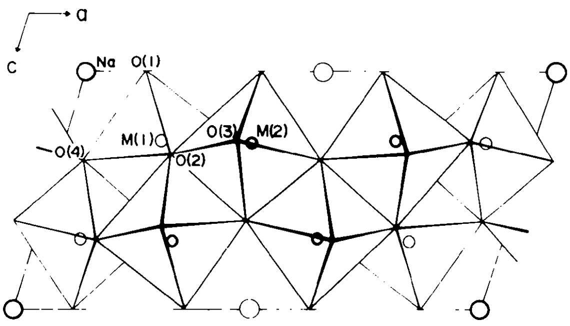

Fig. 1. The crystal structure of freudenbergite viewed along **b**, showing the linkage of the (Ti,Fe)O_6 octahedra.

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Sodium Potassium Ditellurate(VI) Hexahydrate

By Bohumil Kratochvíl, Jana Podlahová and Lubor Jenšovský

Department of Inorganic Chemistry, Charles University, Albertov 2030, 128 40 *Praha* 2, *Czechoslovakia*

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Abstract. $Na_{0.5}K_{3.5}[Te_2O_6(OH)_4].6H_2O$, orthorhombic, *Immm*, a = 13·023 (6), b = 17·605 (6), c = 6·876 (5) Å, Z = 4, D_m = 2·84, D_c = 2·85 g cm^{-3}, μ(Mo $K\alpha$) = 48·0 cm^{-1}. The basic structural motif consists of pairs of tellurate octahedra with a common edge.

Introduction. The structure of $K_2TeO_3(OH)_2.xH_2O$ was studied by Zikmund & Syneček (1967). A structure redetermination was carried out because the published results were only approximate. The crystals were prepared by gradual crystallization from a solution containing $Te(OH)_6$, KOH and KCl or KBr after removing $K_4[Te_2O_6(OH)_4].7{\cdot}3H_2O$ (Lindqvist & Lundgren, 1966). The identity with Zikmund & Syneček's preparation was confirmed by means of a powder diagram. The density was measured pycnomet-