Discussion. Freudenbergite is a mineral first described by Frenzel (1961). McKie (1963) determined the cell dimensions ($a = 12\cdot305$, $b = 3\cdot822$, $c = 6\cdot500$ Å and $\beta = 107\cdot30°$). Wadsley (1964) commented on the close similarity of the powder pattern of freudenbergite to that of the synthetic sodium titanium dioxide 'bronze' $Na_{0\cdot2}TiO_2$. McKie & Long (1970) determined the chemical composition of freudenbergite by EPMA and calculated the density to be $3\cdot95$ 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 $Na_{2\cdot00}Fe_{2\cdot00}Ti_{6\cdot00}O_{16}$, 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 t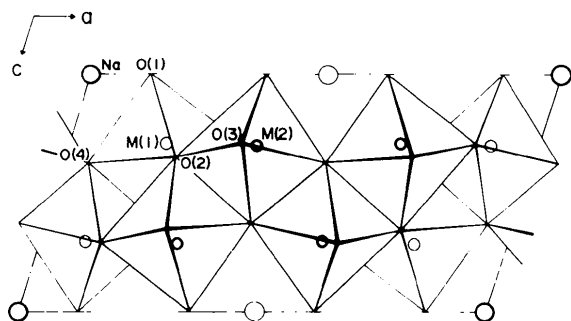he c direction by the octahedra sharing corners, thus forming a three-dimensional framework. The $M(1)$–O distances range from $1\cdot81$ to $2\cdot26$ Å (mean $1\cdot99$ Å), while the $M(2)$–O distances are in the range $1\cdot90$ to $2\cdot05$ Å (mean $1\cdot97$ Å). The Na site is almost fully occupied by Na$^+$ ions, with an environment of eight O atoms at the corners of a highly distorted cube. Four Na–O(1) bonds are $2\cdot46$ Å and the remaining four Na–O(4) bonds are $2\cdot97$ Å, so that there is a pronounced tendency towards a square-planar coordination. Atomic parameters and inter-atomic distances, as well as cell dimensions, are close to those of the sodium titanium bronze, $Na_{0\cdot2}TiO_2$, given by Wadsley (1964).

We are grateful to Professor Y. Iitaka for his permission to use an automated diffractometer in his laboratory at the University of Tokyo. Computations were carried out on HITAC 8700 and M-180 computers at the Computer Centre of Tokyo Institute of Technology.

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

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Abstract. $Na_{0\cdot5}K_{3\cdot5}[Te_2O_6(OH)_4].6H_2O$, orthorhombic, *Immm*, $a = 13\cdot023$ (6), $b = 17\cdot605$ (6), $c = 6\cdot876$ (5) Å, $Z = 4$, $D_m = 2\cdot84$, $D_c = 2\cdot85$ g cm^{-3}, μ(Mo $K\alpha$) = $48\cdot0$ 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 $K_4[Te_2O_6(OH)_4].7\cdot3H_2O$ (Lindqvist & Lundgren, 1966). The identity with Zikmund & Syneček's preparation was confirmed by means of a powder diagram. The density was measured pycnomet-

rically. A single crystal with dimensions $0.11 \times 0.13 \times 0.30$ mm was used for collecting all diffraction data. The intensities were estimated visually from equi-inclination Weissenberg films of the $hk0$–$hk7$ layers. The space group was determined on the basis of systematic absences $(h + k + l = 2n + 1)$ and by the morphological study of single crystals (Zikmund, 1961). Corrections were made for the Lp factor and for absorption. The Patterson synthesis yielded the position of the Te atom, and the K and O atoms were located by a Fourier synthesis. Further electron density maps also identified the Na atom. The presence of Na was not expected in the material and therefore a determination was also carried out by atomic absorption. The formula unit deduced was that previously reported by Lindqvist (1972). The unexpected presence of Na atoms in the structure of sodium potassium tellurate had already been found and discussed by Lindqvist (1969). The parameters were refined by a full-matrix least-squares method. The quantity minimized was $\Sigma w(F_o - |F_c|)^2$ with the Cruickshank weight $w = 1/(a + F_o + cF_o^2)$; $a = 10$ and $c = 0.002$. Values of $R = \Sigma|F_o - |F_c||/\Sigma F_o = 0.085$ and $R_w = (\Sigma w|F_o - |F_c||^2/\Sigma wF_o^2)^{1/2} = 0.111$ were obtained for 639 independent non-zero reflections.* The scattering factors for Te, K^+, Na^+ and O were those of Cromer & Waber (1965). The final positional parameters are given in Table 1. The following programs were used: refinement of cell parameters from powder diffractometer data, *MPEX* (Weiss, 1974); intensity data reduction, *KOREKCE* (Podlahová, Kratochvíl & Loub, 1975); Patterson and Fourier summations, *TLS* (Sklenář, 1974); refinements, *ORFLS* (Busing, Martin & Levy, 1962); interatomic distances and angles,

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32994 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

ORFFE (Busing, Martin & Levy, 1964). All computations were carried out on IBM 370/135 and Hewlett-Packard 9830A computers.

Discussion. The structure contains dimeric anions, $[Te_2O_6(OH)_4]^{4-}$, consisting of two octahedra sharing an edge, and K^+ and Na^+ cations and water molecules which are held together by electrostatic interactions and hydrogen bonds (Fig. 1). The distances and angles within the $[Te_2O_6(OH)_4]^{4-}$ anion are listed in Table 2. Terminal Te—O bonds are shorter than Te—O bridge bonds, owing to the partly multiplied bond character of terminal bonds. A comparison of the Te—O bond distances in $K_4[Te_2O_6(OH)_4].7.3H_2O$ (Lindqvist & Lundgren, 1966), $Na_2K_4[Te_2O_8(OH)_2].14H_2O$ (Lindqvist, 1969), and $Na_{0.5}K_{3.5}[Te_2O_6(OH)_4].6H_2O$ shows that there are no significant differences except a small expansion of all bonds in the $[Te_2O_8(OH)_2]^{6-}$ anion, following from the higher charge of this ion. A final difference electron density map did not enable the location of the positions of the H atoms. The H atoms

Table 2. *Interatomic distances (Å) and angles (°) within the $[Te_2O_6(OH)_4]^{4-}$ anion*

The notation is in accordance with Fig. 1.

Te^i–Te^{ii}	3.115 (2)	O(1)–Te–O(5)	89.3 (3)
Te–O(1)	1.98 (2)	O(3)–Te–O(5)	90.8 (3)
Te–O(2)	1.97 (2)	O(2)–Te–O(5)	89.1 (3)
Te–O(3)	1.83 (2)	O(4)–Te–O(5)	90.5 (3)
Te–O(4)	1.87 (2)	O(1)–Te–O(3)	91.5 (9)
Te–O(5) ⎫		O(1)–Te–O(2)	75.9 (9)
Te–O(5^i) ⎭	1.87 (1)	O(3)–Te–O(4)	103.2 (9)
O(1)–O(2)	2.43 (4)	O(4)–Te–O(2)	89.3 (9)
O(1)–O(3)	2.73 (3)	O(5)–Te–O(5^i)	177.9 (6)
O(1)–O(5)	2.70 (2)	O(4)–Te–O(1)	165.3 (9)
O(2)–O(4)	2.70 (3)	O(3)–Te–O(2)	167.4 (9)
O(2)–O(5)	2.69 (2)		
O(3)–O(4)	2.90 (3)		
O(3)–O(5)	2.63 (2)		
O(4)–O(5)	2.66 (2)		

Table 3. *Distances (Å) indicating possible hydrogen bonds*

O(4)–O(6)	2.76 (2)
O(4)–O(7)	2.68 (3)
O(5)–O(3^i)	2.79 (2)
O(3^i)–O(7)	2.91 (3)

Table 4. *K—O and Na—O distances (Å)*

		Distance to next-nearest O atom
K(1)–O	(4×) 2.80 (1), (4×) 2.90 (2)	3.48 (1)
K(2)–O	(4×) 2.81 (1), (4×) 3.05 (2)	3.48 (1)
K(3)–O	(4×) 2.73 (2), (2×) 2.85 (3)	3.90 (2)
K(4)–O	(8×) 3.00 (2)	4.32 (2)
Na–O	(4×) 2.43 (2), (2×) 2.29 (2)	3.36 (3)

Table 1. *Fractional coordinates*

The e.s.d.'s are given in parentheses.

	x	y	z
Te	0.1196 (1)	0.1987 (1)	0
K(1)	0	0.2978 (7)	$\frac{1}{2}$
K(2)	0	0.0982 (6)	$\frac{1}{2}$
K(3)	0.3075 (9)	0	$\frac{1}{2}$
K(4)	$\frac{1}{2}$	0	0
Na	0	0	0
O(1)	0	0.2678 (17)	0
O(2)	0	0.1298 (17)	0
O(3)	0.2090 (19)	0.2788 (12)	0
O(4)	0.2065 (15)	0.1141 (11)	0
O(5)	0.1170 (8)	0.1983 (6)	0.2719 (17)
O(6)	0.1365 (17)	0	0.2408 (33)
O(7)	0.3699 (15)	0.1060 (9)	0.2363 (29)
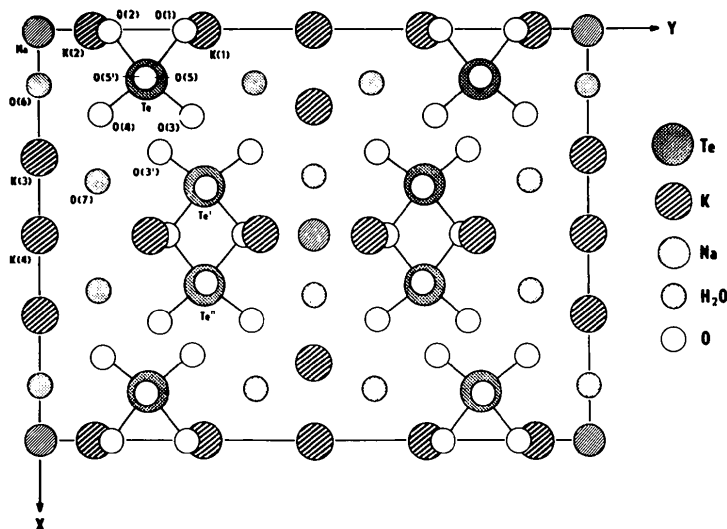
Fig. 1. The structure of $Na_{0.5}K_{3.5}[Te_2O_6(OH)_4].6H_2O$ projected on to the xy plane.

are presumably situated so that they form hydrogen bonds connecting O atoms outside the tellurate anions. All O—O distances indicating possible hydrogen bonds are given in Table 3. The K^+ ions [K(1), K(2) and K(4)] have eight O neighbours and K(3) has six. The Na^+ ion shows a very regular octahedral environment. Table 4 lists K—O and Na—O distances.

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