

Discussion. The K_2PtCl_6 -type structure (Fig. 1) was confirmed with the final structural parameters given in Table 1; a positional disorder of the NH_4^+ ion should be assumed. For the first time a high-quality atomic distance Se—Cl for the hexacoordinated complex ion is available (interatomic distances are in Table 2). A subsequent correction of the positional parameter of the Cl atom was applied because of the shortening of the Se—Cl bond due to thermal motion (Schomaker & Trueblood, 1968) on the assumption that the rigid $SeCl_6^{2-}$ ion is librating about the central Se atom. The new corrected atomic distance Se—Cl is now 2.411 (1) Å. The correctness of the rigid-body model can be checked by calculating a generalized R index for the agreement of observed and calculated U_{ij} [program *XANADU* by Roberts & Sheldrick (1975): $R_G = 0.026$]. The angular displacement of the libration is 3.44° off the center position.

Although the rigid-body model fits rather well, the broad T_{1u} deformation vibration band of the $SeCl_6^{2-}$ ion (Stufkens, 1970) indicates slightly distorted octahedra averaged by the X-ray diffraction experiments. As already pointed out for the TeX_6^{2-} salts (Abriel, 1986), the high-symmetry crystal field yielding at least a center of symmetry for the Se position stabilizes this dynamic structure.

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Hexasodium Hexatungstotellurate(VI) 22-Hydrate

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Abstract. $Na_6[TeW_6O_{24}] \cdot 22H_2O$, $M_r = 2149.0$, triclinic, space group $P\bar{1}$, $a = 10.269$ (3), $b = 10.575$ (2), $c = 11.104$ (5) Å, $\alpha = 90.95$ (3), $\beta = 115.13$ (4), $\gamma = 104.96$ (2)°, $U = 1044$ (2) Å³, $Z = 1$, $D_x = 3.42$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 17.7$ mm⁻¹, $F(000) = 974$, $T = 298$ K, $R_1 = 0.0638$ ($wR = 0.0698$) for 3774 reflections with $I > 3\sigma(I)$. The lattice contains sodium ions and a centrosymmetric Anderson type II anion $TeW_6O_{24}^{6-}$ surrounded by 22 water molecules. The hexatungstotellurate(VI) anion has approximate D_3 symmetry and is isostructural with the known molybdenum analogue. The central tellurium atom is in a

slightly flattened octahedral environment with an average Te—O distance of 1.924 (11) Å.

Introduction. Heteropolynuclear oxometallates of Mo and W have been known in solution for some time and several crystal structures have been determined (Evans, 1971; Nomiya & Miwa, 1984). With recent advances in the field of multinuclear magnetic resonance spectroscopy and the general accessibility of nearly all NMR-active nuclides to observation by the technique, suitable reference compounds are not always readily available. Although the problem generally does not arise for spin- $\frac{1}{2}$ nuclides, difficulties may arise when the nuclide chosen for study possesses a spin greater than $\frac{1}{2}$.

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The spin-lattice relaxation times of the latter quadrupolar nuclides are usually dominated by and are severely broadened by quadrupole relaxation when the nucleus in question occurs in a noncubic environment. The heteropolynuclear oxometallates, in which the incorporated heteroatoms occupy octahedral or tetrahedral sites, therefore offer some promise for providing suitable reference compounds in which the quadrupolar NMR-active nuclide (the heteroatom) is in an environment of sufficiently high symmetry to provide an NMR line width suitable for reference purposes.

Experimental. The title compound was prepared using a modified version of the method of Roy & Mishra (1978). Approximately spherical crystals sealed in 0.2–0.3 mm Lindemann capillaries with mother liquor present. Precession photographs to check crystal quality. Further work on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Cell constants by least-squares refinement of the diffracting positions of 25 well centred reflections ($4.8 < \theta < 15.6^\circ$). Intensity data collected using ω – 2θ scans over ω scan ranges ($0.85 + 0.35 \tan\theta$)°. Scan rates conditional on information collected in prescans were selected to give an $I/\sigma(I)$ ratio of 25 within a max. scan time of 75 s. Three standard reflections monitored every 12000 s indicated that the crystal had moved after 3912 reflections. Crystal recentered and data collection then proceeded smoothly with only statistical fluctuations in the intensities of the standards. Backgrounds by extending scan by 25% on either side of peak were measured for half the time taken to collect the peak. 5144 reflections in the quadrants $h, \pm k, \pm l$ with $2\theta \leq 55^\circ$ collected followed by the recollection of 120 reflections before the shift of the crystal. [At a later stage in the refinement a section of 174 reflections ($7, \bar{1}, \bar{1}4$ to $7, \bar{1}0, 9$) were considered suspect owing to crystal movement and were rejected]. Lorentz, polarization and spherical absorption corrections ($\mu = 17.7 \text{ mm}^{-1}$, $\mu R = 2.7$, transmissions: 3.5–5.7%) applied to all data.

Structure solution: Patterson function for Te and W; other atoms using least squares, Fourier and ΔF Fourier maps. Te and W refined anisotropically. Least squares minimizing $\sum w |F_o| - |F_c|^2$ converged (max. $\Delta/\sigma = 0.002$) to final agreement indices $R_1 = 0.0638$ ($wR = 0.0698$) for 3774 reflections with $I > 3\sigma(I)$. Weights: $w = [\sigma^2(F) + 0.01125F^2]^{-1}$. Most significant features in a final difference Fourier synthesis are some diffraction ripples ($ca 5 e \text{ \AA}^{-3}$) close to W atoms (somewhat large owing to problems associated with the absorption correction). Programs: Enraf–Nonius *SDP* (Frenz, 1981) and *SHELX* (Sheldrick, 1976) on PDP 11/23 and Gould 9705 computers. Scattering factors for neutral atoms were from *International Tables for X-ray Crystallography* (1974).

Table 1. Final atomic positional ($\times 10^4$; $\times 10^5$ for W) and thermal parameters ($\times 10^3$) for $\text{Na}_6[\text{TeW}_6\text{O}_{24}] \cdot 22\text{H}_2\text{O}$

	x	y	z	U or U_{eq}^\dagger
W(1)	15458 (6)	29573 (5)	17698 (5)	11.6 (2)
W(2)	–4787 (6)	5841 (5)	26785 (5)	11.3 (2)
W(3)	–20159 (6)	–23663 (5)	9117 (5)	11.5 (2)
Te	0 (0)	0 (0)	0 (0)	7.7 (3)
Na(1)	3920 (8)	–1597 (7)	4330 (7)	29.1 (14)
Na(2)	5614 (7)	2426 (6)	8442 (7)	25.9 (13)
Na(3)	7997 (8)	4112 (7)	4505 (7)	32 (2)
O(1)	1234 (10)	755 (9)	1848 (9)	12 (2)
O(2)	97 (10)	1811 (9)	–322 (10)	13 (2)
O(3)	–1652 (10)	–244 (9)	444 (10)	13 (2)
O(11)	2904 (11)	2836 (10)	1016 (10)	18 (2)
O(12)	–255 (11)	2237 (10)	1996 (10)	19 (2)
O(13)	2886 (14)	3338 (12)	3443 (13)	27 (2)
O(14)	1192 (14)	4450 (12)	1287 (12)	28 (2)
O(21)	–2191 (14)	517 (13)	2733 (13)	32 (3)
O(22)	925 (13)	1095 (12)	4288 (13)	26 (2)
O(31)	–3691 (15)	–2355 (13)	959 (14)	35 (3)
O(32)	–547 (12)	–1242 (11)	2588 (11)	20 (2)
O(33)	–1642 (14)	–3816 (12)	1442 (13)	29 (3)
O(w1)	–2257 (14)	2732 (12)	–2135 (12)	27 (2)
O(w2)	5556 (16)	188 (14)	8818 (15)	40 (3)
O(w3)	–203 (14)	4747 (12)	3502 (12)	28 (2)
O(w4)	3994 (15)	6248 (13)	4767 (14)	32 (3)
O(w5)	6103 (15)	–1276 (13)	3783 (14)	32 (3)
O(w6)	4078 (14)	465 (13)	3517 (13)	31 (3)
O(w7)	2602 (17)	–3408 (15)	–564 (15)	44 (3)
O(w8)	4814 (19)	5664 (17)	2470 (18)	50 (4)
O(w9)	1762 (15)	–1958 (13)	4817 (13)	33 (3)
O(w10)	2150 (20)	3681 (18)	5799 (19)	55 (4)
O(w11)	3908 (20)	–2955 (17)	7637 (18)	51 (4)

† For W and Te atoms $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$.

Discussion. The final atomic positional and thermal parameters are given in Table 1, selected bond lengths and bond angles in Table 2.* The unit cell contains the Anderson type II polyanion $\text{TeW}_6\text{O}_{24}^{6-}$ (Fig. 1), six Na^+ ions and 22 water molecules. The centrosymmetric anion is isostructural with the hexamolybdotellurate(VI) anion which has been determined as a complex with telluric acid (Evans, 1974) and related anions including $\text{K}_2[\text{IMo}_6\text{O}_{24}] \cdot 5\text{H}_2\text{O}$ (Kondo, Kobayashi & Sasaki, 1980) and $\text{Na}_2\text{K}_6[\text{MnW}_6\text{O}_{24}] \cdot 12\text{H}_2\text{O}$ (Sergienko, Molchanov & Porai-Koshits, 1979). Each anion thus consists of a slightly flattened central octahedron surrounded by an almost hexagonal planar array of distorted MoO_6 or WO_6 octahedra with approximate overall symmetry $D_{3d}(\bar{3}m)$. The distortion of the MoO_6 or WO_6 octahedra is due to an outwards expansion of the Mo(W) atoms thus reducing $M \cdots M$ repulsions (Evans, 1974).

The average Te–O distance of 1.924 (11) Å in the present anion is comparable to that observed in $(\text{NH}_4)_6[\text{TeMo}_6\text{O}_{24}] \cdot \text{Te}(\text{OH})_6 \cdot 7\text{H}_2\text{O}$ [1.93 (1) Å in the anion, 1.91 (1) Å in the telluric acid; Evans, 1974], in

* Lists of structure factor amplitudes, anisotropic thermal parameters and Fig. 2 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42936 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

telluric acid itself [1.909 (1) Å; Lindqvist & Lehmann, 1973] and in $\text{Te}(\text{OTeF}_5)_6$ [1.903 (3) Å (two forms); Lentz, Pritzkow & Seppelt, 1978]. W—O bond lengths are conditional on the coordination numbers of the O atoms and average values for the three types of bond [of lengths 1.74, 1.93, and 2.28 (1) Å] are normal. The O—Te—O_{cis} angles are 85.0–95.0 (4)°.

The Na^+ ions are surrounded by approximate octahedra of water molecules or terminal oxygen atom O(31) of the $\text{TeW}_6\text{O}_{24}^{6-}$ anion with *cis* and *trans* angles of 77.3–103.3 (5) and 165.2–178.5 (5)°, respectively. [The bond-valence sums for each Na^+ are reasonably close to the expected value of 1.0 (Table 2).] All the oxygen atoms of the anion, with the exception of O(31) (interacting with the Na^+ ion), are involved in possible hydrogen-bonding interactions with the water molecules with $\text{O}_{\text{anion}} \cdots \text{O}(\text{wn})$ distances as short as 2.82 (2) Å [O(1)···O(w6)] (Table 2). A view of the crystal packing is given in Fig. 2 (deposited).

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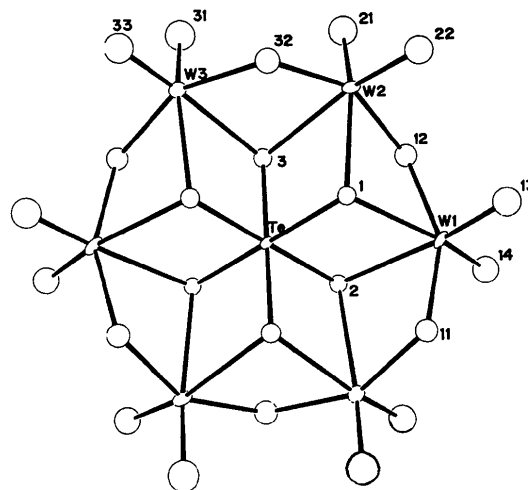


Fig. 1. ORTEP view (Johnson, 1965) of the centrosymmetric $\text{TeW}_6\text{O}_{24}^{6-}$ anion showing the atomic numbering scheme used.

Table 2. Selected bond and contact distances (Å), bond angles (°), and hydrogen-bond distances (Å)

Values in square brackets are bond valences calculated using the expression $S = S_o (R/R_o)^{-N}$ with $S_o = 0.166$, $R_o = 2.449$ and $N = 5.6$ (Brown & Shannon, 1973). Primed atoms are related to corresponding unprimed atoms by the inversion operation.

Bond and contact distances			
W(1)···W(2)	3.2879 (7)	W(2)···W(3)	3.2710 (7)
W(1)···Te	3.2781 (5)	W(2)···Te	3.2873 (5)
W(1)—O(1)	2.275 (9)	W(2)—O(1)	2.280 (9)
W(1)—O(2)	2.262 (10)	W(2)—O(3)	2.285 (10)
W(1)—O(11)	1.935 (10)	W(2)—O(12)	1.915 (10)
W(1)—O(12)	1.933 (10)	W(2)—O(21)	1.768 (13)
W(1)—O(13)	1.740 (13)	W(2)—O(22)	1.715 (13)
W(1)—O(14)	1.752 (12)	W(2)—O(32)	1.914 (11)
Te—O(1)	1.915 (9)	Te—O(2)	1.939 (9)
Na(1)···O(w1)	2.368 [2.004]	Na(2)···O(w1)	2.474 [2.242]
Na(1)···O(w4)	2.351 [2.087]	Na(2)···O(w2)	2.401 [1.565]
Na(1)···O(w5)	2.506 [1.459]	Na(2)···O(31)	2.322 [1.855]
Na(1)···O(w6)	2.367 [2.009]	Na(2)···O(w5)	2.405 [1.837]
Na(1)···O(w6')	2.416 [1.791]	Na(2)···O(w7)	2.293 [2.400]
Na(1)···O(w9)	2.437 [1.706]	Na(2)···O(w8)	2.333 [2.173]
	1.1056		1.2072
		E.s.d.'s 0.014–0.020 Å	
Bond angles			
O(1)—Te—O(2)	85.0 (4)	O(2)—Te—O(3)	94.5 (4)
O(1)—Te—O(3)	85.2 (4)	O(1)—Te—O(2')	95.0 (4)
O(1)—W(1)—O(2)	70.1 (3)	O(1)—W(2)—O(3)	69.3 (3)
O(1)—W(1)—O(11)	84.7 (4)	O(1)—W(2)—O(12)	72.7 (4)
O(1)—W(1)—O(12)	72.5 (4)	O(1)—W(2)—O(21)	159.8 (5)
O(1)—W(1)—O(13)	91.2 (5)	O(1)—W(2)—O(22)	91.4 (5)
O(1)—W(1)—O(14)	161.3 (5)	O(1)—W(2)—O(32)	83.2 (4)
O(2)—W(1)—O(11)	73.6 (4)	O(3)—W(2)—O(12)	82.3 (4)
O(2)—W(1)—O(12)	83.0 (4)	O(3)—W(2)—O(21)	93.0 (5)
O(2)—W(1)—O(13)	159.3 (5)	O(3)—W(2)—O(22)	159.5 (5)
O(2)—W(1)—O(14)	94.1 (5)	O(3)—W(2)—O(32)	74.0 (4)
O(11)—W(1)—O(12)	151.8 (4)	O(12)—W(2)—O(21)	95.9 (5)
O(11)—W(1)—O(13)	96.6 (5)	O(12)—W(2)—O(22)	99.1 (5)
O(11)—W(1)—O(14)	100.7 (5)	O(12)—W(2)—O(32)	150.9 (5)
O(12)—W(1)—O(13)	100.1 (5)	O(21)—W(2)—O(22)	107.1 (6)
O(12)—W(1)—O(14)	96.4 (5)	O(21)—W(2)—O(32)	101.9 (5)
O(13)—W(1)—O(14)	105.8 (6)	O(22)—W(2)—O(32)	97.4 (5)
		O(1)—Te—O(3')	94.8 (4)
		O(2)—Te—O(3')	85.5 (4)
		O(2)—W(3)—O(3)	70.1 (3)
		O(2)—W(3)—O(11)	73.4 (4)
		O(2)—W(3)—O(31)	158.2 (5)
		O(2)—W(3)—O(32)	83.8 (5)
		O(2)—W(3)—O(33)	93.2 (5)
		O(3)—W(3)—O(11)	84.2 (4)
		O(3)—W(3)—O(31)	90.6 (5)
		O(3)—W(3)—O(32)	73.6 (4)
		O(3)—W(3)—O(33)	160.5 (5)
		O(11)—W(3)—O(31)	95.3 (5)
		O(11)—W(3)—O(32)	152.5 (5)
		O(11)—W(3)—O(33)	100.9 (5)
		O(31)—W(3)—O(32)	100.8 (6)
		O(31)—W(3)—O(33)	107.5 (6)
		O(32)—W(3)—O(33)	95.4 (5)
Hydrogen-bond distances (<3 Å)			
O(1)···O(w6)	2.818	O(14)···O(w1)	2.883
O(2)···O(w1)	2.827	O(21)···O(w5)	2.849
O(3)···O(w2)	2.828	O(21)···O(w9)	2.901
O(11)···O(w11)	2.931	O(22)···O(w5)	2.842
O(12)···O(w7)	2.867	O(22)···O(w10)	2.856
O(13)···O(w4)	2.872	O(31)···Na(2)	2.322]
		O(32)···O(w9)	2.885
		O(33)···O(w3)	2.851
		O(33)···O(w7)	2.852
		O(w2)···O(w6)	2.871
		O(w2)···O(w11)	2.827
		O(w2)···O(w10)	2.765

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Acta Cryst. (1986). C42, 1118–1120

Hydrated Aluminophosphate (AlPO₄·1.5H₂O) with PO₄, AlO₄ and AlO₄(H₂O)₂ Groups and Encapsulated Water

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Abstract. Aluminium phosphate hydrate, AlPO₄·1.5H₂O, $M_r = 148.98$, orthorhombic, *Pbca*, $a = 19.3525$ (13), $b = 9.7272$ (7), $c = 9.7621$ (8) Å, $V = 1837.7$ (2) Å³, $Z = 16$, $D_x = 2.15$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 68.2$ cm⁻¹, $F(000) = 1200$, $T \sim 295$ K, $R = 0.033$ for 1530 diffractions. A 4-connected framework contains PO₄ tetrahedra interposed between AlO₄ tetrahedra and AlO₄(H₂O)₂ octahedra at the nodes of cross-linked alternate 6³ and 4.8² nets. A two-dimensional channel system, limited by 8-rings, lies between adjacent 6³ nets. One H₂O of each octahedron lies in a 6-ring, and the other forms a continuous chain with a third H₂O which is held in place only by hydrogen bonds.

Introduction. Variscite, metavariscite, and new hydrates of AlPO₄ (including H_1 – H_4) were synthesized by d'Yvoire (1961). We have determined the crystal structure of a hydrated aluminophosphate, AlPO₄·1.5H₂O, whose new type of framework topology is described by Pluth & Smith (1985) and which resembles the species H_3 of d'Yvoire.

Experimental. Crystals of AlPO₄·1.5H₂O were isolated from a synthetic mixture exhibiting X-ray powder diffraction lines similar to those of the H_3 reported by d'Yvoire. Square prismatic needle elongated on **b** (0.04 × 0.04 × 0.22 mm) after Weissenberg photographic examination for space group and preliminary cell-parameter determination coated with oil to control humidity, mounted on automated Picker–Krisel 4-circle

diffractometer with **b** offset 7° from ϕ axis. Refinement using 20 diffractions ($41 < 2\theta < 77^\circ$), each the average of automatic centering of the 8 possible settings, gave cell parameters. 19 551 measured intensities averaged to produce 1530 merged intensities ($R_{\text{int}} = 0.027$), all with $I > 0$, max. $\sin \theta/\lambda = 0.58$ Å⁻¹; data collection range $h \pm 22$, $k \pm 11$, $l \pm 11$; max. intensity variation of 3 standard reflections 1.7%. Absorption correction using analytical method gave transmission factors 0.62–0.78. Initial phase determination from *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) followed by Fourier refinement. Positions for four H atoms attached to O(9) and O(10) from difference Fourier synthesis, but least-squares refinement unsatisfactory. In final model, isotropic H atoms constrained at 0.85 Å from neighboring O atom. In contrast to these two water molecules, which are bonded to Al(2), the last oxygen O(11), is not within bonding distance of any Al. It is presumed to be part of a water molecule, held only by hydrogen bonds, whose H atoms are too disordered to be located from the Fourier synthesis. Final least-squares refinement* minimized all F 's with σ_F computed from σ , the square root of [total counts + (2% of total counts)²], $w = \sigma_F^{-2}$, $R = 0.033$, $wR = 0.039$, $S = 4.8$;

* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42992 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.