

1.479 (2) and 1.465 (1), [O(2)—B(2)] = 1.439 (3) and 1.436 (3), for Cr—I and Cr—Cl, respectively. The shortest separation between the oxygen atoms occurs within the BO_4 group {[O(2)—O(2)] = 2.393 (2) Å for Cr—I and 2.388 (2) Å for Cr—Cl}. As expected from the topology of the structure and the bonding, the metal ions vibrate mainly along the fourfold axes [$\langle u^2 \rangle_{\parallel} = 0.0109$ (3), $\langle u^2 \rangle_{\perp} = 0.0040$ (3) Å²], i.e. in directions approximately perpendicular to the metal–oxygen bonds, and towards the nearest halogen neighbours (Fig. 1). These are the directions of greatest atomic shift during the structural phase transitions in boracites which lead from cubic to lower symmetry.

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Structure of a New Adduct Between Telluric Acid and a Condensed Phosphate: $\text{K}_4\text{P}_4\text{O}_{12}\cdot\text{Te}(\text{OH})_6\cdot 2\text{H}_2\text{O}$

BY M. T. AVERBUCH-POUCHOT AND A. DURIF

Laboratoire de Cristallographie, Centre National de la Recherche Scientifique, Laboratoire Associé à l'USTMG, 166X, 38042 Grenoble CEDEX, France

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Abstract. Tetrapotassium *cyclo*-tetraphosphate–tellurium hexahydroxide–water (1/1/2), $M_r = 737.96$, monoclinic, $C2/c$, $a = 9.731$ (5), $b = 11.43$ (1), $c = 17.16$ (1) Å, $\beta = 99.45$ (5)°, $V = 1883$ (2) Å³, $Z = 4$, D_m not measured, $D_x = 2.598$ Mg m^{−3}, $\lambda(\text{Ag K}\alpha) = 0.5608$ Å, $\mu = 1.51$ mm^{−1}, $F(000) = 1432$, $T = 293$ K, final R value 0.024 for 2353 independent reflexions. Planes of $\text{Te}(\text{OH})_6$ groups at $z = 0.0$ and 0.5 alternate with planes of P_4O_{12} ring anions at $z = 0.25$ and 0.75 . The set of hydrogen bonds, which involves H_2O molecules as well as P_4O_{12} and $\text{Te}(\text{OH})_6$ groups, spreads in a one-dimensional way along the b direction. The P_4O_{12} rings have twofold symmetry.

Introduction. Telluric acid has the property to form adducts with many kinds of inorganic phosphates, condensed or not. Up to now only one example of an adduct between $\text{Te}(\text{OH})_6$ and an alkali *cyclo*-tetraphosphate has been reported: $2\text{Te}(\text{OH})_6\cdot(\text{NH}_4)_4\text{P}_4$

$\text{O}_{12}\cdot 2\text{H}_2\text{O}$ (Durif, Averbuch-Pouchot & Guitel, 1982). In the present work we describe the second example of an adduct between telluric acid and an alkali *cyclo*-tetraphosphate: $\text{K}_4\text{P}_4\text{O}_{12}\cdot\text{Te}(\text{OH})_6\cdot 2\text{H}_2\text{O}$.

Experimental. $\text{Te}(\text{OH})_6\cdot\text{K}_4\text{P}_4\text{O}_{12}\cdot 2\text{H}_2\text{O}$ is readily prepared by slow evaporation at room temperature of a solution of potassium tetrametaphosphate and telluric acid with an approximate molar ratio 1/1. Large monoclinic prisms up to 5 mm in length are obtained after some days of evaporation.

Crystal size: $0.32 \times 0.24 \times 0.24$ mm; Philips PW 1100 diffractometer; graphite monochromator; systematic absences: hkl ($h + k = 2n$), $h0l$ ($h = 2n$, $l = 2n$); 16 reflexions ($11 < \theta < 14^\circ$) for refining the unit cell; ω scan; scan speed: $0.02^\circ \text{ s}^{-1}$; scan width: 1.20° ; total background measuring time: 20 s; intensity and orientation reflexions: 800 and $\bar{8}00$; θ range: $3\text{--}27^\circ$; 3423 reflexions measured ($\pm h, k, l$); $h_{\text{max}} = 15$,

Table 1. Final atomic coordinates for K₄P₄O₁₂·Te(OH)₆·2H₂O Table 2. Main interatomic distances (Å) and bond angles (°) in K₄P₄O₁₂·Te(OH)₆·2H₂O

B_{eq} and σ for non-H atoms, B_{iso} for H atoms.
 $B_{eq} = \frac{1}{3} \sum_i \sum_j \mathbf{a}_i \cdot \mathbf{a}_j \beta_{ij}$

	x	y	z	$B_{eq}/B_{iso}(\text{\AA}^2)$
Te	0	0	0	0.912 (3)
P(1)	-0.01124 (8)	0.35775 (7)	0.13535 (5)	0.92 (1)
P(2)	0.20354 (8)	0.43673 (7)	0.26261 (5)	0.94 (1)
K(1)	$\frac{1}{2}$	0.4216 (1)	$\frac{1}{2}$	1.97 (2)
K(2)	0.32913 (8)	0.12355 (7)	0.43095 (5)	1.59 (1)
K(3)	$\frac{1}{2}$	0.2087 (1)	$\frac{1}{2}$	2.59 (2)
O(1)	0.5959 (3)	0.3939 (2)	0.5760 (2)	1.73 (4)
O(2)	0.3704 (3)	0.3793 (2)	0.4605 (2)	1.49 (4)
O(3)	0.6202 (3)	0.4588 (2)	0.4261 (2)	1.81 (4)
O(L1)	0.6217 (2)	0.1583 (2)	0.7042 (1)	1.22 (4)
O(L2)	0.3979 (2)	0.0473 (2)	0.6726 (1)	1.10 (3)
O(E11)	0.5320 (3)	0.0923 (2)	0.5642 (1)	1.60 (4)
O(E12)	0.4169 (2)	0.2578 (2)	0.6299 (2)	1.46 (4)
O(E21)	0.3356 (2)	0.3810 (2)	0.2997 (2)	1.52 (4)
O(E22)	0.2929 (3)	0.0520 (2)	0.2769 (1)	1.50 (4)
O(W)	0.6289 (3)	0.2058 (3)	0.4250 (2)	2.63 (6)
H(1)	0.522 (7)	0.331 (6)	0.601 (4)	6 (2)
H(2)	0.348 (5)	0.380 (4)	0.410 (3)	1.8 (9)
H(3)	0.651 (5)	0.400 (4)	0.438 (3)	1.8 (9)
H(W1)	0.731 (6)	0.171 (6)	0.433 (4)	4 (1)
H(W2)	0.618 (6)	0.195 (6)	0.473 (4)	5 (1)

$k_{max} = 18$, $l_{max} = 25$; Lorentz and polarization correction; no absorption correction; conventional methods for structure determination: Patterson function and successive Fourier syntheses; H atoms located from difference-Fourier synthesis; anisotropic full-matrix least-squares refinements on F for non-H atoms, isotropic for H atoms; unitary weighting scheme; total number of unique reflexions: 3297; final refinements with a set of 2353 reflexions corresponding to the rejection of 922 reflexions ($F_o < 3\sigma$) and 22 very strong ones badly measured; extinction not taken into account; final R value: 0.024 ($wR = 0.031$); R factor for the 3297 unique reflexions: 0.034; scattering factors for neutral atoms, f' and f'' from *International Tables for X-ray Crystallography* (1974). *SDP* (Enraf-Nonius, 1977) used for all calculations. Computer: PDP11-70; $S = 1.389$; max. $\Delta\rho = 0.8 \text{ e \AA}^{-3}$; max. $\Delta/\sigma = 0.48$ (scale factor).*

Discussion. Table 1 reports the final atomic coordinates. Fig. 1 is a projection of the atomic arrangement along the b axis. The distribution of the anionic groups: Te(OH)₆ and P₄O₁₂ is very regular; the former are all located in planes $z = 0$ and 0.5 , the latter in planes $z = 0.25$ and 0.75 .

The cyclic P₄O₁₂ anions are located around the

P(1)O₄ tetrahedron

P(1)	O(L1)	O(L2)	O(E11)	O(E12)
O(L1)	1.613 (2)	2.502 (3)	2.533 (3)	2.462 (3)
O(L2)	102.3 (1)	1.598 (2)	2.496 (3)	2.531 (3)
O(E11)	110.4 (1)	108.8 (1)	1.470 (2)	2.553 (3)
O(E12)	104.9 (1)	110.0 (1)	119.1 (1)	1.491 (2)

P(2)O₄ tetrahedron

P(2)	O(L1)	O(L2)	O(E21)	O(E22)
O(L1)	1.599 (2)	2.499 (3)	2.470 (3)	2.547 (3)
O(L2)	102.2 (1)	1.614 (2)	2.532 (3)	2.477 (3)
O(E21)	106.6 (1)	109.7 (1)	1.481 (2)	2.564 (3)
O(E22)	111.3 (1)	106.1 (1)	119.7 (1)	1.485 (2)

P(1)—P(2)	2.908 (1)	P(1)—O(L1)—P(2)	129.7 (2)
P(2)—P(1)	2.909 (1)	P(1)—O(L2)—P(2)	129.8 (2)

P(2)—P(1)—P(2)	84.58 (3)
P(1)—P(2)—P(1)	84.37 (3)

Te(OH)₆ group

2 × Te—O(1)	1.908 (2)
2 × Te—O(2)	1.916 (2)
2 × Te—O(3)	1.920 (2)

O(1)—O(1)	3.816 (4)	O(1)—Te—O(1)	180
O(1)—O(2)	2.709 (3)	O(1)—Te—O(2)	90.2 (1)
O(1)—O(3)	2.700 (3)	O(1)—Te—O(3)	89.8 (1)
O(1)—O(3)	2.689 (3)	O(1)—Te—O(3)	89.3 (1)
O(1)—O(3)	2.724 (3)	O(1)—Te—O(3)	90.7 (1)
O(2)—O(2)	3.833 (4)	O(2)—Te—O(2)	180
O(2)—O(3)	2.749 (3)	O(2)—Te—O(3)	91.5 (1)
O(2)—O(3)	2.676 (3)	O(2)—Te—O(3)	88.5 (1)
O(3)—O(3)	3.839 (4)	O(3)—Te—O(3)	180

	O—H	H...O	∠O—H...O	∠Te—O—H
Te—O(1)—H(1)...O(E12)	1.15 (6)	1.47 (6)	175 (5)	113 (3)
Te—O(2)—H(2)...O(E21)	0.85 (4)	1.88 (4)	169 (4)	113 (3)
Te—O(3)—H(3)...O(W)	0.75 (4)	2.23 (4)	147 (4)	108 (3)

KO_n polyhedra

2 × K(1)—O(1)	3.288 (3)	K(2)—O(2)	2.982 (2)
2 × K(1)—O(3)	3.349 (3)	K(2)—O(2)	2.903 (2)
2 × K(1)—O(L1)	3.373 (2)	K(2)—O(3)	2.762 (2)
2 × K(1)—O(E12)	2.803 (2)	K(2)—O(E11)	2.788 (2)
2 × K(1)—O(E21)	2.971 (2)	K(2)—O(E11)	2.808 (2)
2 × K(1)—O(E22)	2.828 (2)	K(2)—O(E22)	2.734 (2)
		K(2)—O(W)	3.083 (3)
2 × K(3)—O(L2)	3.298 (2)		
2 × K(3)—O(E21)	2.759 (2)		
2 × K(3)—O(E22)	2.791 (2)		
2 × K(3)—O(W)	3.060 (3)		

Hydrogen-bond scheme of water molecule

	O(W)—H	H...O	∠O(W)—H...O
O(W)—H(W1)...O(1)	1.06 (6)	1.87 (6)	2.913 (4)
O(W)—H(W2)...O(E11)	0.86 (6)	2.23 (6)	3.003 (4)
			151 (5)
H(W1)—O(W)—H(W2)			95 (5)

twofold axis. Such a local symmetry for a P₄O₁₂ ring has, up to now, never been observed. Main interatomic distances and bond angles in this ring are reported in Table 2. Located on inversion centres, the Te(OH)₆ groups are almost undistorted octahedra with 1.908 < Te—O < 1.920 Å and 88.5 < O—Te—O < 91.5°. The

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43872 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

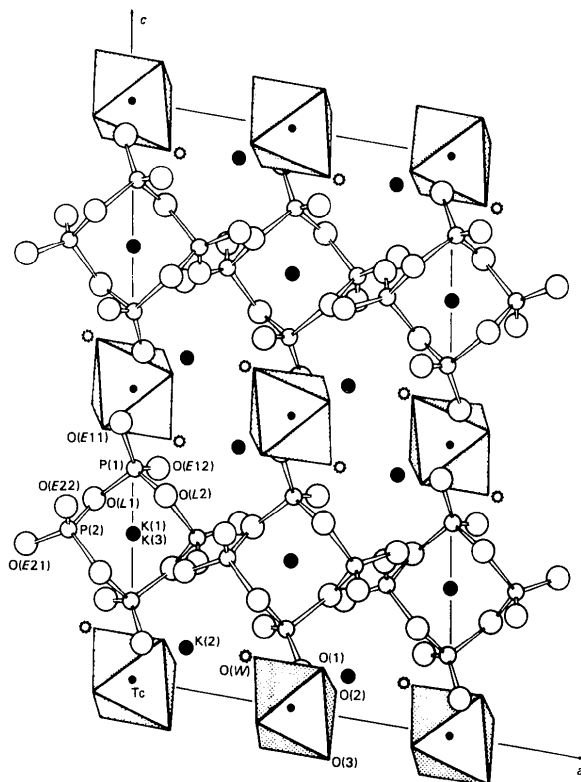


Fig. 1. Projection along the *b* axis of $K_4P_4O_{12}.Te(OH)_6.2H_2O$

geometrical details concerning this group are reported in Table 2.

On the twofold axes, the K(1) and K(2) atoms alternate with the P_4O_{12} groups located almost half way

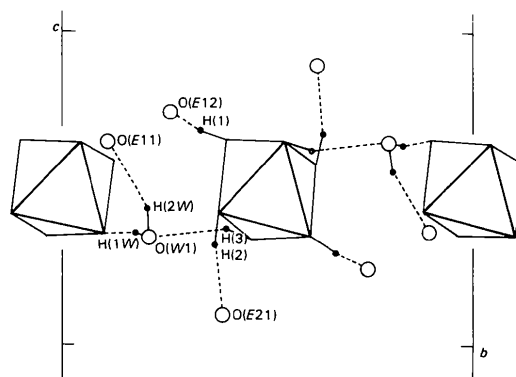


Fig. 2. Hydrogen-bond scheme in projection along the *a* axis. O atoms not involved in the hydrogen bonds are omitted.

between K(1) and K(3). In Fig. 1 these two kinds of K atom are superimposed in projection.

Atoms K(2), in general position, and water molecules are situated in planes $z \sim \pm 0.08$. K—O distances are reported in Table 2.

The hydrogen-bond scheme is depicted in Fig. 2 and in Table 2.

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Synthèse et Structure du Borouranate de Calcium: $CaB_2U_2O_{10}$

PAR MADELEINE GASPERIN

Laboratoire de Minéralogie–Cristallographie associé au CNRS, Université P. et M. Curie, 4 place Jussieu, 75252 Paris CEDEX 05, France

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Abstract. $M_r = 697.78$, monoclinic, $C2$, $a = 16.512(3)$, $b = 8.169(2)$, $c = 6.582(1)$ Å, $\beta = 96.97(3)^\circ$, $V = 881(1)$ Å³, $Z = 4$, $D_x = 5.26$ Mg m^{−3}, $\lambda(Mo K\alpha) = 0.7107$ Å, $\mu = 35.4$ mm^{−1}, $F(000) = 1176$, $T = 290$ K; 3516 contributing reflexions, $R = 0.028$, $wR = 0.033$. Three independent U^{6+} with different surroundings: U(1) has four oxygen atoms closer ($U-O = 1.85-2.05$ Å) than two others ($U-O = 2.30$ Å); U(2) has a normal uranyl bond ($U-O$

$= 1.81$ Å) perpendicular to four other neighbours ($U-O = 2.20-2.23$ Å); U(3) forms a uranyl bond ($U-O = 1.80$ and 1.84 Å) perpendicular to five other atoms ($U-O = 2.22-2.41$ Å). B atoms are in BO_3 triangles ($B-O = 1.34-1.41$ Å) and seven Ca—O distances vary between 2.31 and 2.61 Å.

Introduction. Les composés que l'uranium est susceptible de former avec les borates présentent un intérêt