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Structure of a Potassium Diphosphate Tellurate Hydrate, $K_3HP_2O_7.Te(OH)_6.H_2O$

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Abstract. $M_r = 539·9$, triclinic, $P\bar{1}$, $a = 15·98$ (8), $b = 7·226$ (5), $c = 6·253$ (5) Å, $\alpha = 109·49$ (6), $\beta = 84·34$ (7), $\gamma = 101·83$ (7)°, $V = 665·9$ Å^3, $Z = 2$, $D_x = 2·692$ Mg m^{-3}, λ(Ag $K\alpha$) = 0·5608 Å, $R = 0·036$ for 2629 independent reflexions. This compound provides the first evidence for the existence of diphosphate–tellurates.

Introduction. Phosphate–tellurates are addition compounds between telluric acid and various kinds of phosphates, whether condensed or not. Up to now, numerous monophosphate–tellurates and trimetaphosphate–tellurates have been described and one example of a tetrametaphosphate–tellurate is known (Averbuch-Pouchot, 1980, 1981; Averbuch-Pouchot & Durif, 1981; Boudjada, Averbuch-Pouchot & Durif, 1981; Durif & Averbuch-Pouchot, 1981*a,b;* Durif, Averbuch-Pouchot & Guitel, 1982).

In the course of a systematic investigation of these compounds the present work deals with the chemical preparation and the crystal structure of $K_3HP_2O_7.Te(OH)_6.H_2O$, the first example of a diphosphate–tellurate.

Experimental. Large triclinic prisms of $K_3HP_2O_7.Te(OH)_6.H_2O$ are easily obtained by evaporating, at room temperature, an aqueous solution of normal potassium diphosphate and telluric acid with a 1/1 ratio. From single-crystal X-ray techniques, the symmetry was proved to be triclinic with the unit-cell dimensions reported in the *Abstract*.

The crystal used was an irregular prismatic fragment, approximately 0·19 × 0·24 × 0·24 mm, Philips PW 1100 automatic four-circle diffractometer, Ag $K\alpha$ radiation monochromatized by a graphite plate, 2816 independent reflexions collected between 3 and 28° (θ), each reflexion measured with an ω scan in a domain of 1·50° at a speed of 0·03° s^{-1}, background measured for 10 s at each extremity of this range, two standard intensity and orientation reflexions (060 and $0\bar{6}0$) measured every 2 h without any significant variation.

The crystal structure was solved by interpretation of a three-dimensional Patterson map, followed by successive Fourier syntheses. After refinement cycles the R value was 0·044 for the complete set of 2816 reflexions. After elimination of 187 reflexions with $F_o < 2\sigma(F), |F_o - F_c| > 10$ in a scale 0–150, the final R value is 0·036 for the remaining 2629 reflexions; $F(000) = 520$; unit weights; no absorption correction; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); the Enraf–Nonius (1979) *SDP* programs used.

Discussion. Table 1 reports the final atomic coordinates.*

As in all the phosphate–tellurates previously described, the coexistence of $Te(OH)_6$ and phosphate groups (here P_2O_7) as independent units in the atomic arrangement is observed.

$Te(OH)_6$ *groups.* Two independent $Te(OH)_6$ groups exist in this structure; both are centrosymmetric and

Table 1. *Atomic coordinates and* B_{eq} *values for* $K_3HP_2O_7.Te(OH)_6.H_2O$

Standard deviations are given in parentheses. $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
Te(1)	0	$\frac{1}{2}$	$\frac{1}{2}$	0·76 (1)
Te(2)	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	0·79 (1)
K(1)	0·58827 (10)	0·1845 (2)	0·8186 (2)	2·43 (2)
K(2)	0·93837 (9)	0·8386 (2)	0·1833 (3)	2·34 (2)
K(3)	0·26224 (7)	0·4958 (2)	0·6347 (2)	1·63 (2)
P(1)	0·18418 (8)	1·0001 (2)	0·1536 (2)	0·95 (2)
P(2)	0·32059 (8)	0·0334 (2)	0·8024 (2)	1·02 (2)
O(1)	0·3995 (2)	0·4566 (6)	0·3362 (7)	1·80 (6)
O(2)	0·5047 (3)	0·7826 (5)	0·5800 (7)	1·60 (6)
O(3)	0·4285 (2)	0·5096 (6)	0·7667 (7)	1·51 (6)
O(4)	0·0922 (3)	0·5450 (7)	0·6940 (8)	2·53 (7)
O(5)	0·0736 (3)	0·4082 (7)	0·2440 (8)	2·60 (8)
O(6)	0·0321 (3)	0·7681 (7)	0·4904 (8)	2·56 (8)
O(*E*11)	0·1885 (3)	0·9039 (6)	0·3359 (7)	1·56 (6)
O(*E*12)	0·1078 (3)	0·9016 (6)	0·0094 (7)	1·67 (6)
O(*E*13)	0·1943 (3)	0·2231 (5)	0·2510 (7)	1·69 (6)
O(*L*)	0·2692 (2)	0·9478 (6)	−0·0057 (7)	1·67 (6)
O(*E*21)	0·3489 (2)	0·8571 (6)	0·6278 (7)	1·54 (6)
O(*E*22)	0·2552 (3)	0·1150 (6)	0·7055 (7)	1·62 (6)
O(*E*23)	0·3914 (3)	0·1988 (6)	−0·0827 (7)	1·83 (6)
O(*W*)	0·2568 (3)	0·5499 (6)	0·0872 (8)	2·29 (8)

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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 38154 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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located around the $(0,0,\frac{1}{2})$ and $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ inversion centers. For the first, Te(1) $(OH)_6$, the average Te—O value is 1·910 (4) Å, while O—Te—O angles range from 89·3 (2) to 90·8 (2)°. For the second, Te(2) $(OH)_6$, slightly more distorted, these values are respectively 1·914 (3) Å, 87·3 to 88·9°. Table 2 reports detailed numerical values for these two octahedral groups.

The P_2O_7 *group.* The main geometrical features of this group are reported in Table 3. The average P—O distances are, respectively, 1·542 (3) and 1·540 (3) Å in the P(1)O_4 and P(2)O_4 tetrahedra. The bonding distances P(1)—O(*L*) and P(2)—O(*L*) are, respectively, 1·635 (3) and 1·609 (3) Å with a P(1)—O(*L*)—P(2) angle of 132·6 (2)°.

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *in the two independent* Te$(OH)_6$ *groups*

Te(1)$(OH)_6$ group			
2 × Te(1)—O(4)	1·908 (3)	2 × Te(1)—O(6)	1·919 (4)
2 × Te(1)—O(5)	1·904 (4)		
O(4)—Te—O(4)′	180 (4)	O(4)—Te(1)—O(5)	89·3 (2)
O(5)—Te—O(5)′	180 (4)	O(4)—Te(1)—O(6)	90·5 (2)
O(6)—Te—O(6)′	180 (2)	O(5)—Te(1)—O(6)	90·8 (2)
O(4)—O(4)′	3·815 (7)	O(5)—O(5)′	3·809 (7)
O(4)—O(5)	2·678 (5)	O(5)—O(6)	2·722 (5)
O(4)—O(5)′	2·713 (5)	O(5)—O(6)′	2·686 (5)
O(4)—O(6)	2·717 (5)	O(6)—O(6)′	3·838 (7)
O(4)—O(6)′	2·694 (5)		
Te(2)$(OH)_6$ group			
2 × Te(2)—O(1)	1·906 (3)	2 × Te(2)—O(3)	1·916 (3)
2 × Te(2)—O(2)	1·921 (3)		
O(1)—Te(2)—O(1)′	180 (3)	O(1)—Te(2)—O(2)	88·9 (1)
O(2)—Te(2)—O(2)′	180 (2)	O(1)—Te(2)—O(3)	88·4 (1)
O(3)—Te(3)—O(3)′	180 (3)	O(2)—Te(2)—O(3)	87·3 (1)
O(1)—O(1)′	3·812 (6)	O(2)—O(2)′	3·842 (6)
O(1)—O(2)	2·681 (4)	O(2)—O(3)	2·648 (4)
O(1)—O(2)′	2·731 (4)	O(2)—O(3)′	2·777 (4)
O(1)—O(3)	2·664 (5)	O(3)—O(3)′	3·833 (6)
O(1)—O(3)′	2·741 (4)		

Table 3. *Main interatomic distances* (Å) *and bond angles* (°) *in the* P_2O_7 *groups*

P(1)O_4 tetrahedron				
P(1)	O(*E*11)	O(*E*12)	O(*E*13)	O(*L*)
O(*E*11)	1·534 (3)	112·1 (2)	112·1 (2)	102·0 (2)
O(*E*12)	2·514 (4)	1·497 (3)	114·3 (2)	107·6 (2)
O(*E*13)	2·516 (4)	2·518 (4)	1·501 (3)	107·9 (2)
O(*L*)	2·464 (4)	2·528 (4)	2·537 (4)	1·635 (3)
P(2)O_4 tetrahedron				
P(2)	O(*E*21)	O(*E*22)	O(*E*23)	O(*L*)
O(*E*21)	1·494 (3)	111·4 (2)	115·3 (2)	105·0 (2)
O(*E*22)	2·520 (4)	1·556 (3)	110·9 (2)	106·3 (2)
O(*E*23)	2·531 (4)	2·520 (4)	1·503 (3)	107·2 (2)
O(*L*)	2·463 (4)	2·533 (4)	2·506 (4)	1·609 (3)
P(1)—P(2)	2·971 (1)	P(1)—O(*L*)—P(2)	132·6 (2)	

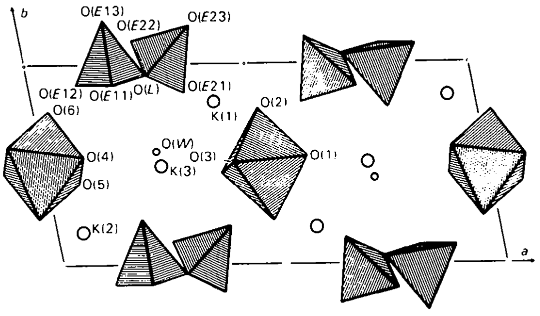

Fig. 1. Projection of the atomic arrangement of $K_3HP_2O_7.Te(OH)_6.H_2O$ along the **c** direction.

Table 4. K—O *distances* (Å) *in the three independent* KO_n *polyhedra*

K(1)—O(1)	3·022 (4)	K(2)—O(4)	3·050 (4)
K(1)—O(2)	2·890 (3)	K(2)—O(5)	2·671 (4)
K(1)—O(2)	3·122 (4)	K(2)—O(6)	2·779 (4)
K(1)—O(3)	2·822 (3)	K(2)—O(6)	2·870 (4)
K(1)—O(*L*)	3·104 (3)	K(2)—O(*E*12)	2·823 (3)
K(1)—O(*E*21)	2·811 (3)	K(2)—O(*E*12)	2·789 (3)
K(1)—O(*E*23)	3·162 (4)	K(2)—O(*E*22)	3·165 (3)
K(1)—O(*W*)	2·783 (4)		
K(3)—O(1)	2·733 (3)	K(3)—O(*L*)	3·276 (3)
K(3)—O(3)	2·832 (3)	K(3)—O(*E*21)	2·705 (3)
K(3)—O(4)	2·786 (3)	K(3)—O(*E*22)	2·909 (3)
K(3)—O(*E*13)	2·702 (3)	K(3)—O(*W*)	2·719 (4)

As shown in Fig. 1, this structure can be described as approximately (210) planes containing both P_2O_7 and Te$(OH)_6$ groups, alternating with planes containing water molecules and K atoms.

K—O distances in the KO_n polyhedra are reported in Table 4. The location of the H atoms has not been possible in this structure.

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