

square-planar geometry, with no deviation greater than  $2.1^\circ$  from ideal angles. The Au—Cl bond lengths vary from 2.264 (8) to 2.296 (8) Å, with an average of 2.277 Å [ $\sigma(\text{mean}) = 0.002$  Å] over 16 bonds; *cf.* 2.271, 2.277 (5) Å in  $\text{TiAuCl}_4$  (Jones *et al.*, 1987), and 2.260 (6)–2.288 (5), ave. 2.277 Å in  $\text{NaAuCl}_4 \cdot 2\text{H}_2\text{O}$  (Bonamico *et al.*, 1966).

The crystal packing is shown in Fig. 1. The Na ions are each coordinated by seven chlorines, with  $\text{Na} \cdots \text{Cl}$  2.797 (15)–3.188 (16) Å. All Cl atoms are involved in these  $\text{Na} \cdots \text{Cl}$  contacts. There are no other  $\text{Na} \cdots \text{Cl}$  contacts under 4 Å. In  $\text{NaAuCl}_4 \cdot 2\text{H}_2\text{O}$  (Bonamico *et al.*, 1966) the Na ions are also seven-coordinate, with three Cl and four O ligands.

The shortest  $\text{Au} \cdots \text{Cl}$  non-bonded contact is 3.280 Å between Cl(43) and Au(1) at  $0.5-x$ ,  $-0.5+y$ ,  $1.5-z$ .

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## Structure of a New Adduct Between Telluric Acid and a Condensed Phosphate: $\text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$

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**Abstract.**  $M_r = 883.28$ , monoclinic,  $P2_1/c$ ,  $a = 7.279$  (2),  $b = 13.984$  (8),  $c = 17.071$  (4) Å,  $\beta = 90.42$  (2) $^\circ$ ,  $V = 1738$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_m$  not measured,  $D_x = 3.376$  Mg m<sup>-3</sup>,  $\lambda(\text{Ag } K\alpha) = 0.5608$  Å,  $\mu = 4.33$  mm<sup>-1</sup>,  $F(000) = 1592$ ,  $T = 293$  K, final  $R$  value 0.024 for 4512 independent observed reflexions. Planes of  $\text{Te}(\text{OH})_6$  groups at  $z = 0$  and  $0.5$  alternate with planes of  $\text{P}_3\text{O}_9$  ring anions at  $z = 0.25$  and  $0.75$ . The H atoms were located by difference-Fourier synthesis and their positions have been refined. A complete scheme of the hydrogen bonds is given.

**Introduction.** A few adducts of telluric acid with *cyclo*-triphosphates of monovalent cations have already been investigated:  $\text{Te}(\text{OH})_6 \cdot 2\text{Na}_3\text{P}_3\text{O}_9 \cdot 6\text{H}_2\text{O}$  (Boudjada, Averbuch-Pouchot & Durif, 1981a),  $\text{Te}(\text{OH})_6 \cdot \text{K}_3\text{P}_3\text{O}_9 \cdot 2\text{H}_2\text{O}$  (Boudjada, Averbuch-Pouchot & Durif, 1981b),  $\text{Te}(\text{OH})_6 \cdot \text{Rb}_3\text{P}_3\text{O}_9 \cdot \text{H}_2\text{O}$  (Boudjada & Durif, 1982) and  $\text{Te}(\text{OH})_6 \cdot 2(\text{NH}_4)_3\text{P}_3\text{O}_9$  (Boudjada, Boudjada & Guitel, 1983).

Up to now, all attempts to prepare a similar caesium adduct have been unsuccessful because of the difficulty in preparing large amounts of caesium *cyclo*-triphosphate. This starting material is normally prepared by Boullé's (1941) metathesis reaction according to the

following scheme:  $3\text{CsCl} + \text{Ag}_3\text{P}_3\text{O}_9 \rightarrow \text{Cs}_3\text{P}_3\text{O}_9 + 3\text{AgCl}$ .

In fact it is very difficult to prepare sodium-free  $\text{Ag}_3\text{P}_3\text{O}_9$  and usually the reaction leads to the formation of a very stable compound of the  $\text{Na}_3\text{P}_3\text{O}_9$ – $\text{Cs}_3\text{P}_3\text{O}_9$ – $\text{H}_2\text{O}$  system:  $\text{CsNa}_2\text{P}_3\text{O}_9 \cdot 2\text{H}_2\text{O}$  (Boudjada, 1985) with a very poor yield of  $\text{Cs}_3\text{P}_3\text{O}_9$ . The use of such a sodium-contaminated starting material invariably leads to the adduct  $\text{Cs}_2\text{NaP}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6$  (Boudjada, 1985). The pure caesium adduct  $\text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$ , whose structure is described in the present paper, has been prepared as follows involving the preparation of an almost sodium-free  $\text{Ag}_3\text{P}_3\text{O}_9$ .  $\text{Ag}_3\text{P}_3\text{O}_9$  is normally prepared by adding a silver nitrate solution to a solution of  $\text{Na}_3\text{P}_3\text{O}_9$ :  $3\text{AgNO}_3 + \text{Na}_3\text{P}_3\text{O}_9 \rightarrow \text{Ag}_3\text{P}_3\text{O}_9 + 3\text{NaNO}_3$ .

The precipitated compound was well crystallized and sparingly water soluble. The solubility of the crystals seems proportional to their sodium content, so several washings with water eliminated the greatest part of the sodium-rich crystals. The salt so obtained is pure enough for preparing the desired caesium adduct according to the stoichiometric reaction in aqueous solution:  $\text{Cs}_3\text{P}_3\text{O}_9 + \text{Te}(\text{OH})_6 + \text{H}_2\text{O} \rightarrow \text{Cs}_3\text{P}_3\text{O}_9 \cdot \text{Te}(\text{OH})_6 \cdot \text{H}_2\text{O}$ .

Table 1. Atomic coordinates for Cs<sub>3</sub>P<sub>3</sub>O<sub>9</sub>Te(OH)<sub>6</sub>H<sub>2</sub>O

Starred atoms were refined isotropically. E.s.d.'s are in parentheses.

$$B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}/B_{iso}(\text{\AA}^2)$
Cs(1)	0.58237 (4)	0.28393 (2)	0.23924 (2)	2.206 (4)
Cs(2)	0.24451 (4)	0.07367 (2)	0.93093 (2)	2.739 (5)
Cs(3)	0.26971 (4)	0.41182 (2)	0.04547 (2)	2.509 (5)
Te	0.22268 (4)	0.74742 (2)	0.00313 (1)	1.374 (3)
P(1)	0.7101 (2)	0.55387 (8)	0.34332 (6)	1.56 (2)
P(2)	-0.0194 (2)	0.85108 (8)	0.75326 (6)	1.67 (2)
P(3)	0.6852 (2)	0.55245 (8)	0.17527 (6)	1.75 (2)
O(1)	0.0095 (4)	0.2405 (3)	0.4452 (2)	2.28 (6)
O(2)	0.8649 (5)	0.1384 (2)	0.0457 (2)	2.23 (5)
O(3)	0.4495 (4)	0.7509 (2)	0.9471 (2)	2.34 (6)
O(4)	0.3078 (5)	0.6336 (2)	0.0529 (2)	2.51 (6)
O(5)	0.3308 (4)	0.8227 (2)	0.0847 (2)	2.14 (5)
O(6)	0.1139 (5)	0.8271 (2)	0.4215 (2)	2.35 (6)
O(L12)	0.1835 (4)	0.3502 (2)	0.6830 (2)	1.75 (5)
O(E11)	0.4369 (5)	0.0810 (3)	0.1024 (2)	2.83 (6)
O(E12)	0.8416 (5)	0.0205 (3)	0.8677 (2)	3.11 (7)
O(L23)	0.0999 (4)	0.0717 (2)	0.3086 (2)	1.92 (5)
O(E21)	0.8517 (5)	0.1085 (3)	0.2106 (2)	2.55 (6)
O(E22)	-0.0118 (5)	0.7443 (2)	0.2164 (2)	2.52 (6)
O(L13)	0.3817 (5)	0.0234 (3)	0.2383 (2)	2.64 (6)
O(E31)	0.3336 (5)	0.9676 (3)	0.3753 (2)	2.66 (6)
O(E32)	0.5958 (5)	0.8578 (3)	0.6502 (2)	2.97 (6)
O(W)	0.2224 (6)	0.4083 (3)	0.2348 (3)	3.91 (9)
H(1W)	0.143 (9)	0.130 (5)	0.728 (4)	2 (1)*
H(2W)	0.813 (9)	0.964 (5)	0.252 (4)	2 (2)*
H(1)	1.00 (1)	0.266 (7)	0.903 (5)	5 (2)*
H(2)	0.13 (1)	0.857 (5)	0.902 (4)	3 (2)*
H(3)	0.533 (9)	0.202 (5)	0.066 (4)	2 (1)*
H(4)	0.62 (1)	0.351 (5)	0.920 (4)	2 (2)*
H(5)	0.28 (1)	0.855 (5)	0.091 (4)	2 (2)*
H(6)	0.85 (1)	0.370 (8)	0.086 (6)	7 (3)*

**Experimental.** Crystal size: 0.27 × 0.24 × 0.27 mm; Nonius CAD-4 diffractometer; graphite monochromator; systematic absences:  $h0l$  ( $l = 2n$ ),  $0k0$  ( $k = 2n$ ); 16 reflexions ( $10.40 < \theta < 11.30^\circ$ ) for refining the unit cell;  $\omega$  scan; scan speed: 0.02–0.04° s<sup>-1</sup>; scan width: 1.20°; total background measuring time: 15–30 s; two orientation ( $491$  and  $49\bar{1}$ ) and two intensity ( $49\bar{1}$  and  $491$ ) reference reflexions, no significant variation. 7815 reflexions measured ( $3 < \theta < 25^\circ$ ),  $\pm h, k, l$ ,  $h_{\max} = 9$ ,  $k_{\max} = 24$ ,  $l_{\max} = 30$ ; 7011 independent reflexions,  $R_{\text{int}} = 0.02$  on  $F$ . Lorentz and polarization corrections; no absorption correction.

Crystal structure solved by direct methods with *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977); H atoms located by difference-Fourier map; anisotropic full-matrix least-squares refinement (on  $F$ ) for non-H atoms, isotropic for H atoms. Unit weights. Final refinement with 4512 reflexions ( $I > 9\sigma$ ). Final  $R = 0.024$  ( $wR = 0.027$ ). Extinction coefficient refined,  $g = 4.18 \times 10^{-7}$  (Stout & Jensen, 1968).  $S = 2.108$ . Max.  $\Delta/\sigma = 0.01$  (atomic coordinates and  $B_{\text{iso}}$  of some H atoms). Max. peak height in final difference-Fourier synthesis:  $1.034 \text{ e \AA}^{-3}$ . The final  $R$  value = 0.043 for the complete set of 7011 independent reflexions. Scattering factors for neutral atoms and  $f'$ ,  $f''$  from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius (1977) *SDP* employed for all calculations. Computer used: MICROVAXII.

Table 2. Main interatomic distances (Å) and bond angles (°) in Cs<sub>3</sub>P<sub>3</sub>O<sub>9</sub>Te(OH)<sub>6</sub>H<sub>2</sub>O

E.s.d.'s are in parentheses.

P(1)O <sub>4</sub> tetrahedron						
P(1)	O(L12)	O(E11)	O(E12)	O(L13)		
O(L12)	1.614 (3)	2.502 (5)	2.539 (5)	2.465 (5)		
O(E11)	108.3 (2)	1.470 (4)	2.529 (5)	2.491 (5)		
O(E12)	110.7 (2)	118.6 (2)	1.471 (4)	2.500 (5)		
O(L13)	100.2 (2)	108.5 (2)	109.0 (2)	1.599 (4)		
P(2)O <sub>4</sub> tetrahedron						
P(2)	O(L12)	O(L23)	O(E21)	O(E22)		
O(L12)	1.621 (3)	2.484 (4)	2.530 (4)	2.508 (4)		
O(L23)	100.2 (2)	1.617 (3)	2.506 (4)	2.532 (5)		
O(E21)	109.5 (2)	108.7 (2)	1.458 (4)	2.548 (5)		
O(E22)	108.1 (2)	109.8 (2)	119.4 (2)	1.476 (4)		
P(3)O <sub>4</sub> tetrahedron						
P(3)	O(L23)	O(L13)	O(E31)	O(E32)		
O(L23)	1.609 (3)	2.478 (5)	2.506 (5)	2.520 (5)		
O(L13)	100.7 (2)	1.609 (3)	2.492 (5)	2.531 (5)		
O(E31)	108.7 (2)	107.8 (2)	1.473 (4)	2.533 (5)		
O(E32)	109.5 (2)	110.2 (2)	118.5 (2)	1.475 (4)		
P(1)–O(L13)–P(3)	127.2 (2)	P(1)–P(2)–P(3)	60.58 (4)			
P(1)–O(L12)–P(2)	122.6 (2)	P(2)–P(3)–P(1)	59.34 (4)			
P(2)–O(L23)–P(3)	124.8 (2)	P(3)–P(1)–P(2)	60.08 (2)			
P(1)–P(2)	2.837 (2)					
P(2)–P(3)	2.859 (2)					
P(3)–P(1)	2.873 (2)					
TeO <sub>6</sub> octahedron						
Te	O(1)	O(2)	O(3)	O(4)	O(5)	O(6)
O(1)	1.914 (3)	2.636 (5)	3.828 (4)	2.751 (5)	2.775 (5)	2.627 (5)
O(2)	87.2 (1)	1.909 (3)	2.767 (5)	3.813 (4)	2.689 (4)	2.702 (5)
O(3)	177.1 (1)	92.7 (1)	1.915 (3)	2.653 (5)	2.702 (5)	2.707 (5)
O(4)	92.2 (2)	179.3 (1)	88.0 (2)	1.905 (6)	2.704 (4)	2.697 (5)
O(5)	93.0 (1)	89.5 (1)	89.9 (1)	90.3 (1)	1.910 (3)	3.817 (4)
O(6)	86.9 (1)	90.2 (1)	90.2 (1)	90.1 (1)	179.6 (1)	1.907 (3)
Te–O(1)–H(1)	112 (9)			Te–O(4)–H(4)	106 (6)	
Te–O(2)–H(2)	110 (5)			Te–O(5)–H(5)	109 (7)	
Te–O(3)–H(3)	110 (6)			Te–O(6)–H(6)	116 (9)	

Cs<sub>3</sub>O<sub>9</sub> polyhedra

Cs(1)–O(3)	3.224 (3)	Cs(2)–O(1)	3.122 (3)	Cs(3)–O(1)	3.317 (3)
Cs(1)–O(5)	3.115 (3)	Cs(2)–O(2)	3.097 (3)	Cs(3)–O(3)	3.061 (3)
Cs(1)–O(E21)	3.180 (4)	Cs(2)–O(5)	3.426 (3)	Cs(3)–O(4)	3.117 (3)
Cs(1)–O(E22)	3.258 (4)	Cs(2)–O(E11)	3.237 (3)	Cs(3)–O(6)	3.089 (3)
Cs(1)–O(L13)	3.380 (4)	Cs(2)–O(E11)	3.225 (4)	Cs(3)–O(E31)	3.274 (4)
Cs(1)–O(E31)	3.288 (4)	Cs(2)–O(E12)	3.205 (4)	Cs(3)–O(E31)	3.394 (3)
Cs(1)–O(E32)	3.035 (4)	Cs(2)–O(W)	3.360 (5)	Cs(3)–O(W)	3.254 (5)
Cs(1)–O(W)	3.145 (4)				

## Hydrogen-bond scheme

O–H...O	O–H	H...O	O...O	O–H...O
O(1)–H(1)...O(E22)	0.72 (9)	2.05 (9)	2.759 (4)	166 (10)
O(2)–H(2)...O(E21)	0.89 (7)	1.99 (7)	2.849 (5)	161 (7)
O(3)–H(3)...O(E11)	0.71 (7)	1.93 (7)	2.634 (5)	169 (8)
O(4)–H(4)...O(E32)	0.76 (7)	1.94 (7)	2.668 (5)	161 (8)
O(5)–H(5)...O(E12)	0.58 (7)	2.09 (7)	2.656 (5)	168 (8)
O(6)–H(6)...O(E31)	0.68 (11)	2.01 (10)	2.657 (5)	161 (11)
O(W)–H(1W)...O(E22)	0.80 (7)	2.21 (7)	2.885 (6)	142 (6)
O(W)–H(2W)...O(E21)	0.85 (7)	2.16 (7)	3.001 (6)	170 (7)

H(1W)–O(W)–H(2W) 116 (7)

**Discussion.** Table 1 reports the final atomic coordinates and Table 2 the main interatomic distances and bond angles.\* Fig. 1 is a projection of the atomic

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44878 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

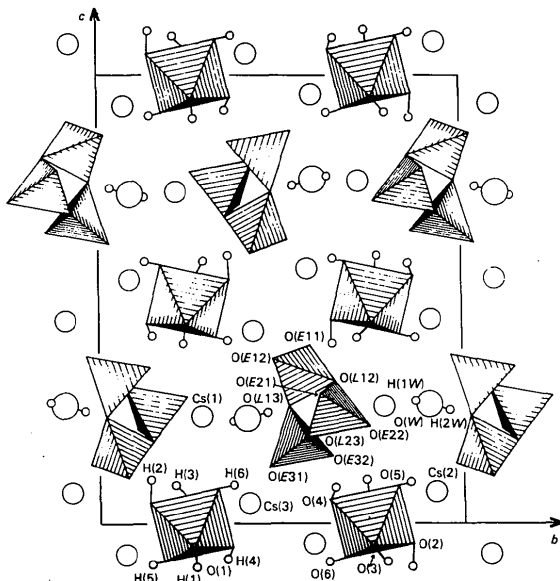


Fig. 1. Projection of the structure along the *a* axis.

arrangement along the *a* axis. The distribution of anionic groups is very regular; Te(OH)<sub>6</sub> groups are located in planes *z* = 0 and 0.5, and P<sub>3</sub>O<sub>9</sub> anions in planes *z* = 0.25 and 0.75. The cohesions between these planes are assumed by the hydrogen bonds and the CsO<sub>*n*</sub> coordination polyhedra, in a three-dimensional way. Each Te(OH)<sub>6</sub> group is linked to six different P<sub>3</sub>O<sub>9</sub> anions. The H atoms of the water molecules connect two neighbouring phosphoric anions. The interatomic distances and bond angles found for the P<sub>3</sub>O<sub>9</sub> groups (Table 2) are those generally encountered in inorganic

cyclo-tri-phosphates. P—O mean distances are 1.538, 1.543 and 1.541 Å in P(1)O<sub>4</sub>, P(2)O<sub>4</sub> and P(3)O<sub>4</sub> tetrahedra respectively, the general mean being 1.543 Å for these compounds.

The TeO<sub>6</sub> group forms a very regular octahedron with 1.905 < Te—O < 1.915 Å and 86.9 < O—Te—O < 93.0°. One can remark, considering the atomic coordinates, that the Te atom is practically a non-crystallographic inversion centre for the TeO<sub>6</sub> octahedron.

The Cs—O distances in the different caesium coordination polyhedra are given in Table 2. Within a range of 3.5 Å Cs(1) has eight neighbours, Cs(2) and Cs(3) only seven.

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## Structure of Potassium Magnesium Hydrogenbiscarbonate Tetrahydrate\*

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**Abstract.** KMgH(CO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, *M<sub>r</sub>* = 256.5, triclinic, *P* $\bar{1}$ , *Z* = 1,  $\lambda(\text{Mo } K\alpha)$  = 0.71069 Å,  $\mu$  = 0.725 mm<sup>-1</sup>, *F*(000) = 132: (1) *T* = 143 K, *a* = 6.697 (1), *b* = 7.267 (1), *c* = 5.336 (1) Å,  $\alpha$  = 107.04 (2),  $\beta$  = 107.83 (2),  $\gamma$  = 59.88 (2)°, *V* = 210.6 Å<sup>3</sup>, *D<sub>x</sub>* =

2.02 Mg m<sup>-3</sup>, *R* = 0.0230, 2616 unique reflections; (2) *T* = 295 K, *a* = 6.7125 (7), *b* = 7.3439 (9), *c* = 5.3677 (8) Å,  $\alpha$  = 107.267 (8),  $\beta$  = 107.965 (7),  $\gamma$  = 59.546 (8)°, *V* = 213.77 Å<sup>3</sup>, *D<sub>x</sub>* = 1.99 Mg m<sup>-3</sup>, *R* = 0.0331, 1215 unique reflections. Two carbonate groups are linked by a hydrogen bond to form a slightly twisted anion, H(CO<sub>3</sub>)<sub>2</sub><sup>3-</sup>, with an O...O distance of 2.447 (1) Å at 143 K and 2.461 (3) Å at 295 K; the distance between the planes of the carbonate groups is 0.512 (1) Å at 143 K and 0.503 (2) Å at 295 K.

\* Hydrogen Bond Studies. 154. Part 153: Olovsson & Jaskólski (1986).

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