indicated. The hexafluoroarsenate ions occupy the corners of the cell and centers of the *ab* faces. The S_2N^+ cations occupy the remaining four faces of the cell.

The S_2N^+ cation is linear and has six weak F—S contacts (see Table 2), at approximately 90% of the sum of the van der Waals radii. The S—N bond distance in the S_2N^+ cation of $S_2N^+.AsF_6^-$ [1·480(3) Å] is longer than or equal to the corresponding bond distances in $S_2N^+.SbCl_6^-$ [1·463(4) Å] (Faggiani, Gillespie, Lock & Tyrer, 1978) or $S_2N^+.AlCl_4^-$ [1·464(5), 1·472(5) Å] (Thewalt, Berhalter & Müller, 1982).

The strengths of the cation–anion contacts generally increase with the Lewis basicity of the anions ($AsF_6^- < SbCl_6^- < AlCl_4^-$), and therefore it was anticipated that the S—N distance in the AsF_6^- salt might be shorter than the others. On the other hand, the averages of the symmetric and asymmetric S—N stretching frequencies in the AsF_6^- and $SbCl_6^-$ salts are almost the same, 1095 and 1093 cm^{-1}, respectively (Banister, Hey, MacLean & Passmore, 1982). Consistently, stretching frequencies and force contacts for the AsF_6^-, SbF_6^-, SO_3F^- and NO_3^- salts of the related NO_2^+ are also very similar (Qureshi, Carter & Aubke, 1971). On this basis, the S—N distances in the $SbCl_6^-$ and AsF_6^- salts would be expected to be about the same. A correction for thermal effects using the 'riding model' (Busing & Levy, 1963) gives S—N distances of 1·490 [SbF_6^-], 1·510 [AsF_6^-], 1·494 and 1·517 Å for the two S—N distances in the $AlCl_4^-$ salt (Thewalt, Berhalter & Müller, 1982). No noticeable trend is observable in these bond distances and they are not significantly different from one another.

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Crystal Structure of a New Adduct Between Telluric Acid and Alkali *cyclo*-Triphosphates: $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$

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Abstract. $M_r = 889·74$, monoclinic, $C2/c$, $a = 18·42\,(1)$, $b = 10·644\,(5)$, $c = 12·348\,(8)$ Å, $\beta = 119·76\,(5)$°, $V = 2102\,(2)$ Å^3, $Z = 4$, $D_x = 2·811$ Mg m^{-3}, $\lambda(\text{Ag }K\bar{\alpha}) = 0·5608$ Å, $\mu = 1·38$ mm^{-1}, $F(000) = 1720$, $T = 298$ K, $R = 0·017$ for 2553 independent reflexions. The crystal structure is built up of planes ($z \simeq 0·0$ and 0·5) of a hexagonal network of P_3O_9 groups centred by $Te(OH)_6$ groups, alternating with planes ($z \simeq 0·25$ and 0·75) of the associated cations.

Introduction. Addition compounds between telluric acid and alkali *cyclo*-triphosphates have been widely investigated. Structural investigations have been reported for $Te(OH)_6.2Na_3P_3O_9.6H_2O$ (Boudjada, Averbuch-Pouchot & Durif, 1981*a*; Boudjada, Lambert-Andron & Boucherle, 1985; Boudjada, 1981), $Te(OH)_6.K_3P_3O_9.2H_2O$ (Boudjada, 1981; Boudjada, Averbuch-Pouchot & Durif, 1981*b*), $Te(OH)_6.Rb_3P_3O_9.H_2O$ (Boudjada & Durif, 1982), and $Te(OH)_6.2(NH_4)_3P_3O_9$ (Boudjada, Boudjada & Guitel, 1983). Up to now, only one adduct between a mixed-alkali *cyclo*-triphosphate and telluric acid has been reported: $Te(OH)_6.Cs_2NaP_3O_9$ (Boudjada, 1985). As in all the adducts between telluric acid and various kinds of inorganic phosphates, condensed or not, one observes in their atomic frameworks the coexistence of independent $Te(OH)_6$ and phosphoric anionic groups. In the present work we

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Table 1. *Final atomic coordinates for* $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$ *and* B_{eq} *for non-*H *atoms,* B_{iso} *for* H *atoms*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \mathbf{a}_i.\mathbf{a}_j\beta_{ij}.$$

	x	*y*	*z*	B_{eq}/B_{iso}(Å²)
Te	¼	¼	0	1·07 (1)
K(1)	½	0·14423 (8)	¼	1·75 (2)
K(2)	0·19707 (3)	0·05296 (6)	0·24996 (5)	1·77 (2)
P(1)	0·33257 (3)	0·33279 (6)	0·47715 (5)	0·99 (1)
P(2)	0·48865 (3)	0·32332 (6)	0·47711 (5)	0·99 (1)
P(3)	0·09408 (3)	0·40606 (6)	0·52284 (5)	0·99 (1)
Na(1)	0·09947 (6)	0·3495 (1)	0·24998 (9)	1·62 (3)
Na(2)	½	0·5510 (2)	¼	1·61 (3)
O(1)	0·33408 (10)	0·3676 (2)	0·1046 (2)	1·90 (4)
O(2)	0·20158 (10)	0·2649 (2)	0·1048 (1)	1·91 (4)
O(3)	0·31902 (11)	0·1174 (2)	0·1049 (2)	1·93 (4)
O(*L*12)	0·39884 (9)	0·3845 (2)	0·4375 (1)	1·17 (3)
O(*E*11)	0·25159 (10)	0·3925 (2)	0·3908 (2)	1·45 (3)
O(*E*12)	0·13088 (10)	0·1578 (2)	0·3858 (1)	1·47 (3)
O(*L*13)	0·32711 (9)	0·1871 (2)	0·4374 (1)	1·17 (3)
O(*E*21)	0·04350 (10)	0·1678 (2)	0·1142 (1)	1·46 (3)
O(*E*22)	0·51584 (9)	0·3718 (2)	0·3906 (1)	1·45 (3)
O(*L*23)	0·46155 (9)	0·1783 (2)	0·4374 (1)	1·17 (3)
O(*E*31)	0·12650 (10)	0·4856 (2)	0·1090 (1)	1·45 (3)
O(*E*32)	0·04861 (10)	0·4242 (2)	0·3860 (1)	1·45 (3)
H(1)	0·370 (2)	0·364 (4)	0·093 (3)	2·7 (8)
H(2)	0·178 (2)	0·323 (4)	0·095 (3)	3·0 (9)
H(3)	0·293 (2)	0·067 (4)	0·087 (3)	2·9 (9)

describe the second example of an adduct between a mixed-alkali *cyclo*-triphosphate and telluric acid: $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$.

Experimental. When a concentrated aqueous solution of $Na_3P_3O_9$, $K_3P_3O_9$ and $Te(OH)_6$ (2/1/3) is slowly evaporated at room temperature the first crystallization occurring after some days gives large hexagonal prisms of $Te(OH)_6.2Na_3P_3O_9.6H_2O$. At the end of its crystallization this phase is removed and the remaining aqueous solution again kept at room temperature for some days. A second crystallization of slightly distorted octahedra of $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$ then occurs.

Crystal size: almost regular octahedron (edge = 0·16 mm); Enraf–Nonius CAD-4 diffractometer; graphite monochromator; θ range 3–35°; ω scan; scan width 1·20°; scan speed 0·01–0·04° s⁻¹; two standard reflexions (444 and $\bar{4}\bar{4}\bar{4}$), no significant variation; unit-cell refinement with 18 reflexions ($10 < \theta < 11°$); 8816 reflexions collected (hkl, $\bar{h}kl$); $h_{\max} = 32$, $k_{\max} = 20$, $l_{\max} = 19$; systematic absences: hkl ($h + k = 2n$), $h0l$ ($h = 2n$, $l = 2n$); Lorentz–polarization correction, no absorption correction. Structure solved by classical methods: three-dimensional Patterson function and successive Fourier syntheses; H atoms from difference Fourier map; final refinements with 2553 independent reflexions ($F > 4\sigma_F$); full-matrix refinements on F with anisotropic thermal parameters for non-H atoms; unit weights; max. $\Delta/\sigma = 0{\cdot}36$ [x of H(2)]; max. $\Delta\rho = 0{\cdot}36$ e Å^{-3}; final $R = 0{\cdot}017$, $wR = 0{\cdot}021$, $S = 1{\cdot}036$. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* used for all calculations. Computer: PDP 11-70.

Table 1 reports the final atomic coordinates with B_{eq} and B_{iso} for the H atoms and Table 2 the main interatomic distances and bond angles of this atomic arrangement.*

Discussion. As in the other adducts between telluric acid and inorganic phosphates, condensed or not, one observes the coexistence of two distinct types of anions in the atomic arrangement: phosphoric anions (here

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *in the atomic arrangement of* $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$

P(1)O_4 tetrahedron

P(1)	O(*L*12)	O(*L*13)	O(*E*11)	O(*E*12)
O(*L*12)	1·620 (2)	2·482 (2)	2·479 (2)	2·539 (2)
O(*L*13)	100·23 (9)	1·614 (2)	2·500 (2)	2·532 (2)
O(*E*11)	106·1 (1)	107·8 (1)	1·478 (2)	2·579 (2)
O(*E*12)	109·8 (1)	109·6 (1)	121·2 (1)	1·481 (2)

P(2)O_4 tetrahedron

P(2)	O(*L*12)	O(*L*23)	O(*E*21)	O(*E*22)
O(*L*12)	1·612 (2)	2·481 (2)	2·529 (2)	2·498 (2)
O(*L*23)	100·23 (9)	1·621 (2)	2·541 (2)	2·480 (2)
O(*E*21)	109·6 (1)	109·8 (1)	1·482 (2)	2·581 (2)
O(*E*22)	107·7 (1)	106·1 (1)	121·3 (1)	1·480 (2)

P(3)O_4 tetrahedron

P(3)	O(*L*13)	O(*L*23)	O(*E*31)	O(*E*32)
O(*L*13)	1·620 (2)	2·479 (2)	2·479 (2)	2·538 (2)
O(*L*23)	100·16 (9)	1·612 (2)	2·499 (2)	2·528 (2)
O(*E*31)	106·2 (1)	107·8 (1)	1·479 (2)	2·577 (2)
O(*E*32)	109·9 (1)	109·6 (1)	121·2 (1)	1·480 (2)

P(1)–P(2)	2·8771 (8)	P(2)–P(3)	2·8782 (9)
P(1)–P(3)	2·8790 (9)		
P(1)–O(*L*12)–P(2)	125·8 (1)	P(2)–P(1)–P(3)	60·00 (2)
P(1)–O(*L*13)–P(3)	125·8 (1)	P(1)–P(2)–P(3)	60·03 (2)
P(2)–O(*L*23)–P(3)	125·8 (1)	P(1)–P(3)–P(2)	59·96 (2)

TeO_6 octahedron

Te–O(1)	1·910 (2) (×2)	Te–O(3)	1·911 (2) (×2)
Te–O(2)	1·909 (2 (×2)		

KO_8 polyhedra

K(1)–O(3)	2·911 (2) (×2)	K(2)–O(1)	2·910 (2)
K(1)–O(*E*22)	2·909 (2) (×2)	K(2)–O(2)	2·907 (2)
K(1)–O(*L*23)	2·759 (2) (×2)	K(2)–O(*L*12)	2·763 (2)
K(1)–O(*E*32)	2·759 (2) (×2)	K(2)–O(*E*11)	2·910 (2)
		K(2)–O(*E*12)	2·753 (2)
		K(2)–O(*L*13)	2·762 (2)
		K(2)–O(*E*21)	2·757 (2)
		K(2)–O(*E*31)	2·914 (2)

NaO_6 polyhedra

Na(1)–O(*E*11)	2·499 (2)	Na(2)–O(*E*12)	2·430 (2) (×2)
Na(1)–O(*E*12)	2·519 (2)	Na(2)–O(*E*21)	2·517 (2) (×2)
Na(1)–O(*E*21)	2·429 (2)	Na(2)–O(*E*22)	2·497 (2) (×2)
Na(1)–O(*E*31)	2·497 (2)		
Na(1)–O(*E*32)	2·427 (2)		
Na(1)–O(*E*32)	2·519 (2)		

	O–H (Å)	H···O (Å)	O–H···O (°)	O–O (Å)
O(1)–H(1)···O(*E*22)	0·74 (4)	2·02 (4)	164 (4)	2·736 (3)
O(2)–H(2)···O(*E*31)	0·73 (4)	2·02 (4)	167 (4)	2·740 (3)
O(3)–H(3)···O(*E*11)	0·68 (4)	2·10 (4)	156 (4)	2·737 (3)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43976 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

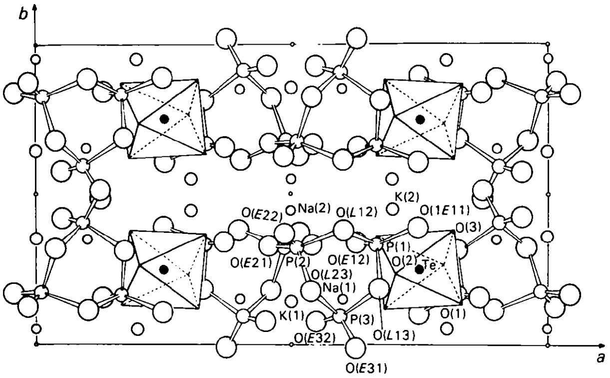

Fig. 1. Projection along the *c* axis of the atomic arrangement of $Te(OH)_6.Na_3P_3O_9.K_3P_3O_9$.

P_3O_9) and $Te(OH)_6$ groups, linked only by hydrogen bonds.

Fig. 1 is a projection of the atomic arrangement on the *ab* plane. H atoms of the $Te(OH)_6$ groups are not represented.

The P_3O_9 *ring anion*

The P_3O_9 ring anions have no internal symmetry. The three P atoms and the bonding O atom (O*L*) are almost coplanar in a plane perpendicular to the *c* axis. These ring planes are distributed in $z \simeq 0{\cdot}0$ and $0{\cdot}5$. All geometrical features of the P_3O_9 group are reported in Table 2 and do not depart significantly from previous observations of other *cyclo*-triphosphates.

The $Te(OH)_6$ *octahedron*

The $Te(OH)_6$ group is centrosymmetric and located around the ($\frac{1}{4}\frac{1}{4}0$, $\frac{1}{4}\frac{3}{4}0$; $\frac{1}{4}\frac{1}{4}\frac{1}{2}$, $\frac{1}{4}\frac{3}{4}\frac{1}{2}$) inversion centres. This group is quite regular, Te—O distances ranging from 1·909 to 1·911 Å.

Associated cation polyhedra

Within a range of 3 Å, the two K atoms are coordinated to eight O atoms. The $K(1)O_8$ polyhedron has a binary symmetry. The two Na atoms have an octahedral oxygen coordination; one of the NaO_6 polyhedra [$Na(2)O_6$] has twofold symmetry.

Geometrical details for the $Te(OH)_6$ group and the associated cation polyhedra are given in Table 2.

The general arrangement of this atomic framework can be simply described by considering the respective locations of $Te(OH)_6$ groups and P_3O_9 rings in planes $z \simeq 0{\cdot}0$ and $0{\cdot}5$. In such planes P_3O_9 anions form an almost regular hexagonal network, each $(P_3O_9)_6$ hexagon being centred by a $Te(OH)_6$ group. The six O atoms of the central $Te(OH)_6$ group are connected by hydrogen bonds to external O atoms of the six different P_3O_9 rings building the surrounding hexagon. Numerical details of the hydrogen-bond scheme are reported in Table 2.

Associated cations are all situated in the intermediate planes ($z \simeq 0{\cdot}25$ and $0{\cdot}75$).

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Structural Phase Transitions in Chevrel Phases Containing Divalent Metal Cations. I. Structure Refinement of Rhombohedral MMo_6S_8 (M = Eu, Sr, Ba) at Room Temperature

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Abstract. Single-crystal X-ray diffraction data on MMo_6S_8 (M = Eu, Sr, Ba) at $T = 294$ K and ambient pressure are reported: $M_r = 984{\cdot}1$, $919{\cdot}8$, $969{\cdot}5$, space group $R\bar{3}$, Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å), $a_{\rm rh} =$ 6·5535 (2), 6·5702 (3), 6·6507 (3) Å, $\alpha = 88{\cdot}918$ (3), 89·003 (5), 88·584 (6)°, $V = 281{\cdot}32$ (2), 283·50 (3), 293·90 (4) Å^3, $Z = 1$, $D_x = 5{\cdot}809$, 5·387, 5·477 Mg m^{-3}, $\mu = 13{\cdot}21$, 12·18, 10·68 mm^{-1}, $F(000)$

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