

Table 2. Bond lengths (Å), inter-bond angles (°) and probable hydrogen bonds (Å)

| | | | |
|--------------------------|----------|-------------------|----------|
| I(1)—C(1) | 2.10 (2) | C(8)—C(9) | 1.53 (2) |
| N(1)—C(2) | 1.45 (2) | C(8)—C(10) | 1.49 (2) |
| N(1)—C(7) | 1.36 (2) | C(8)—C(11) | 1.59 (2) |
| N(2)—C(7) | 1.33 (2) | C(9)—C(13) | 1.53 (2) |
| N(2)—C(8) | 1.47 (2) | C(10)—C(12) | 1.54 (2) |
| N(3)—C(7) | 1.32 (2) | C(11)—C(14) | 1.55 (2) |
| C(1)—C(2) | 1.38 (2) | C(12)—C(16) | 1.49 (2) |
| C(1)—C(6) | 1.39 (2) | C(12)—C(17) | 1.53 (2) |
| C(2)—C(3) | 1.38 (2) | C(13)—C(15) | 1.52 (3) |
| C(3)—C(4) | 1.39 (2) | C(13)—C(17) | 1.52 (3) |
| C(4)—C(5) | 1.32 (2) | C(14)—C(15) | 1.53 (2) |
| C(5)—C(6) | 1.40 (3) | C(14)—C(16) | 1.53 (2) |
| C(2)—N(1)—C(7) | 122 (1) | C(9)—C(8)—C(10) | 112 (1) |
| C(7)—N(2)—C(8) | 128 (1) | C(9)—C(8)—C(11) | 107 (1) |
| I(1)—C(1)—C(2) | 121 (1) | C(10)—C(8)—C(11) | 109 (1) |
| I(1)—C(1)—C(6) | 120 (1) | C(8)—C(9)—C(13) | 109 (1) |
| C(2)—C(1)—C(6) | 118 (1) | C(8)—C(10)—C(12) | 109 (1) |
| N(1)—C(2)—C(1) | 120 (1) | C(8)—C(11)—C(14) | 108 (1) |
| N(1)—C(2)—C(3) | 119 (1) | C(10)—C(12)—C(16) | 112 (2) |
| C(1)—C(2)—C(3) | 120 (1) | C(10)—C(12)—C(17) | 108 (1) |
| C(2)—C(3)—C(4) | 121 (2) | C(16)—C(12)—C(17) | 111 (1) |
| C(3)—C(4)—C(5) | 120 (2) | C(9)—C(13)—C(15) | 110 (2) |
| C(4)—C(5)—C(6) | 121 (2) | C(9)—C(13)—C(17) | 109 (1) |
| C(1)—C(6)—C(5) | 120 (2) | C(15)—C(13)—C(17) | 110 (2) |
| N(1)—C(7)—N(2) | 115 (2) | C(11)—C(14)—C(15) | 108 (1) |
| N(1)—C(7)—N(3) | 120 (2) | C(11)—C(14)—C(16) | 111 (1) |
| N(2)—C(7)—N(3) | 125 (2) | C(15)—C(14)—C(16) | 111 (2) |
| N(2)—C(8)—C(9) | 111 (1) | C(13)—C(15)—C(14) | 108 (1) |
| N(2)—C(8)—C(10) | 114 (1) | C(12)—C(16)—C(14) | 108 (1) |
| N(2)—C(8)—C(11) | 103 (1) | C(12)—C(17)—C(13) | 109 (2) |
| Cl(1)⋯N(1 ⁱ) | 3.13 (2) | Cl(1)⋯N(3) | 3.12 (2) |
| Cl(1)⋯N(2 ⁱ) | 3.20 (2) | | |

Symmetry code: (i) $x, y, 1+z$.

lished observation) has shown that (i) the N₃C group and attached C(phenyl) and C(adamantyl) atoms are nearly coplanar; (ii) the phenyl ring is at nearly 90° to the N₃C plane and (iii) the phenyl group is *syn*, and the adamantyl group is *anti*, to the terminal N atom.

A molecular geometry similar to that of the free base has been reported in the crystal structure of *N,N'*-bis(2-methylphenyl)guanidine (DTG) free base (Brown & Gash, 1984). The dihedral angle between

the N₃C and the phenyl groups was found to be 106°. The two phenyl rings of this compound were found to lie *syn* and *anti*, respectively, to the unsubstituted nitrogen.

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Crystal Chemistry of *cyclo*-Hexaphosphates. VII. Structure of Tris(ethylene-diammonium) *cyclo*-Hexaphosphate Tellurate Dihydrate

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Abstract. (C₂H₁₀N₂)₃[P₆O₁₈][Te(OH)₆]₂·2H₂O, *M*_r = 1151.458, triclinic, *PI*, *a* = 10.945 (3), *b* = 11.252 (3), *c* = 8.042 (7) Å, α = 90.90 (5), β = 92.97 (5), γ =

116.82 (5)°, *V* = 881.8 Å³, *Z* = 1, *D*_x = 2.168 Mg m⁻³, λ (Ag *K*α) = 0.5608 Å, μ = 1.090 mm⁻¹, *F*(000) = 574, room temperature, final *R* = 0.022 for

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Table 1. Final atomic coordinates and B_{eq} for non-H atoms with *e.s.d.*'s in parentheses
$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

| | x | y | z | $B_{\text{eq}}(\text{\AA}^2)$ |
|--------|-------------|---------------|-------------|-------------------------------|
| Te(1) | 0 | 0 | 0 | 1.378 (3) |
| Te(3) | 0 | $\frac{1}{2}$ | 0 | 1.230 (3) |
| P(1) | 0.77337 (5) | 0.76346 (5) | 0.50832 (7) | 1.365 (8) |
| P(2) | 0.31382 (4) | 0.52399 (5) | 0.51025 (7) | 1.255 (8) |
| P(3) | 0.54099 (5) | 0.77259 (5) | 0.66419 (7) | 1.435 (8) |
| O(1) | 0.9873 (2) | 0.0551 (2) | 0.7806 (2) | 2.13 (3) |
| O(2) | 0.1899 (2) | 0.0407 (2) | 0.9807 (2) | 2.61 (4) |
| O(3) | 0.9485 (2) | 0.8202 (2) | 0.9207 (2) | 2.43 (3) |
| O(4) | 0.9475 (2) | 0.5446 (2) | 0.7892 (2) | 1.87 (3) |
| O(5) | 0.8180 (2) | 0.4419 (2) | 0.0693 (2) | 2.56 (4) |
| O(6) | 0.9428 (2) | 0.3221 (2) | 0.9062 (2) | 2.46 (3) |
| O(L13) | 0.6921 (1) | 0.7828 (1) | 0.6582 (2) | 1.57 (2) |
| O(L12) | 0.6802 (2) | 0.6117 (1) | 0.4425 (2) | 1.80 (3) |
| O(E11) | 0.0940 (1) | 0.2219 (2) | 0.4156 (2) | 1.93 (3) |
| O(E21) | 0.2323 (2) | 0.1552 (2) | 0.6339 (2) | 2.26 (3) |
| O(L23) | 0.5457 (2) | 0.3822 (2) | 0.3855 (3) | 2.34 (3) |
| O(E12) | 0.8023 (1) | 0.4686 (2) | 0.4103 (2) | 1.90 (3) |
| O(E22) | 0.6761 (2) | 0.4566 (2) | 0.6717 (2) | 2.49 (3) |
| O(E13) | 0.4752 (2) | 0.2071 (2) | 0.1564 (2) | 2.93 (4) |
| O(E23) | 0.5160 (2) | 0.8565 (2) | 0.5419 (3) | 2.71 (3) |
| O(W) | 0.3657 (3) | 0.8675 (3) | 0.0654 (4) | 4.67 (7) |
| N(1) | 0.5445 (2) | 0.6598 (2) | 0.1221 (3) | 2.54 (4) |
| N(2) | 0.9103 (2) | 0.2975 (2) | 0.5504 (3) | 2.02 (3) |
| N(3) | 0.3614 (2) | 0.9779 (2) | 0.7231 (3) | 2.45 (4) |
| C(1) | 0.4327 (3) | 0.4465 (3) | 0.9622 (3) | 2.49 (5) |
| C(2) | 0.7916 (2) | 0.1644 (2) | 0.5108 (3) | 2.01 (4) |
| C(3) | 0.2374 (2) | 0.8532 (2) | 0.6767 (3) | 2.07 (4) |

5222 reflections. The $\text{Te}(\text{OH})_6$ groups located on inversion centres at 0,0,0 and $0, \frac{1}{2}, 0$ form chains along the *b* axis. The centrosymmetrical P_6O_{18} ring anion develops around the inversion centre at $\frac{1}{2}, \frac{3}{2}, \frac{1}{2}$. Two kinds of ethylenediammonium cations co-exist in the arrangement, one with $\bar{1}$ internal symmetry, the other with no internal symmetry. The hydrogen-bond scheme is described.

Introduction. A great number of adducts of telluric acid with various inorganic phosphates, condensed or not, have been well characterized. A review of this domain of crystal chemistry has been given recently by Boudjada (1985). Some adducts of telluric acid with organic compounds have also been reported by Tranqui, Vicat & Durif (1984), Averbuch-Pouchot (1988) and Loub, Haase & Merghehn (1979). The title compound is the first example of a phosphate tellurate with an organic cation.

Experimental. The title compound is easily obtained by slow evaporation at room temperature of an aqueous solution of telluric acid and ethylenediammonium *cyclo*-hexaphosphate in a stoichiometric ratio. Crystals appear as large triclinic prisms.

Crystal size: $0.60 \times 0.55 \times 0.40$ mm. Density not measured. Nonius CAD-4 diffractometer, graphite monochromator. 24 reflections ($10.5 < \theta < 16.5^\circ$) for refining unit-cell dimensions. ω scan; scan width 1.20° , scan speed variable between 0.02 and $0.06^\circ \text{ s}^{-1}$, total background measuring time between

30 and 10 s. 5718 reflections collected ($3 < \theta < 27.5^\circ$), $\pm h, \pm k, l$; $h_{\text{max}} = 19$, $k_{\text{max}} = 20$, $l_{\text{max}} = 12$. $2\theta_{\text{max}} = 55^\circ$. Two orientation (470 and 470) and two intensity (520 and $\bar{5}20$) control reflections. The decrease of intensity (3%) observed during the 10 days of the data collection has been corrected. 5376 unique reflections kept after averaging Friedel pairs ($R_{\text{int}} = 0.019$).

Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods (*MULTAN77*; Main, Lessinger, Woolfson, Germain & Declercq, 1977). H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinements (on *F*), isotropic for H atoms. Unit weights. Final refinement cycles with 5222 reflections ($I > 4\sigma_I$). Final $R = 0.022$ ($wR = 0.026$), $S = 0.816$, max. $\Delta/\sigma = 0.06$. Max. peak height in the final difference Fourier synthesis 1.135 e \AA^{-3} . No extinction correction. Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). Enraf-Nonius (1979) *SDP* used for all calculations. Computer used: MicroVAX II.

Discussion. Table 1 reports the final atomic coordinates.* A projection of the atomic arrangement along the *c* axis is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, bond angles and lengths involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53104 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

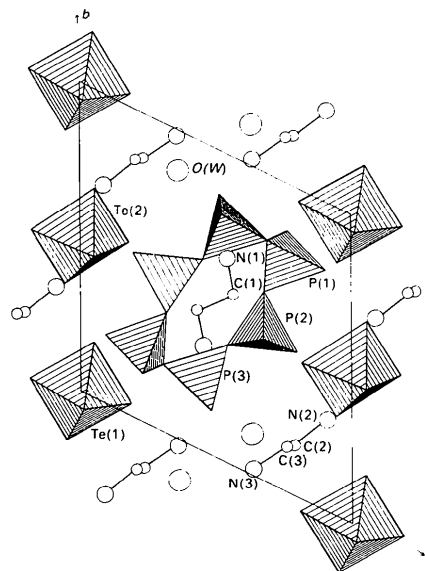


Fig. 1. Projection along the *c* axis of the atomic arrangement of $2\text{Te}(\text{OH})_6 \cdot (\text{C}_2\text{N}_2\text{H}_{10})_3\text{P}_6\text{O}_{18} \cdot 2\text{H}_2\text{O}$. The hydrogen atoms have been omitted for clarity.

Table 2. Main interatomic distances (Å) and bond angles (°) in the atomic arrangement of $2Te(OH)_6 \cdot (C_2N_2H_{10})_3P_6O_{18} \cdot 2H_2O$ with e.s.d.'s in parentheses

| | | | | |
|-------------------------------------|-------------|------------------|-----------------|-----------|
| P(1)O4 tetrahedron | | | | |
| P(1) | O(L13) | O(L12) | O(E11) | O(E21) |
| O(L13) | 1.603 (2) | 2.525 (2) | 2.468 (2) | 2.534 (2) |
| O(L12) | 103.78 (8) | 1.606 (1) | 2.526 (2) | 2.453 (2) |
| O(E11) | 106.17 (9) | 109.68 (10) | 1.482 (2) | 2.594 (3) |
| O(E21) | 109.87 (11) | 104.65 (9) | 121.38 (9) | 1.492 (2) |
| P(2)O4 tetrahedron | | | | |
| P(2) | O(L12) | O(L23) | O(E12) | O(E22) |
| O(L12) | 1.608 (2) | 2.347 (2) | 2.533 (3) | 2.546 (3) |
| O(L23) | 94.08 (9) | 1.599 (2) | 2.519 (2) | 2.561 (2) |
| O(E12) | 109.79 (9) | 109.44 (10) | 1.486 (2) | 2.547 (2) |
| O(E22) | 110.73 (11) | 112.23 (9) | 117.99 (12) | 1.485 (2) |
| P(3)O4 tetrahedron | | | | |
| P(3) | O(L13) | O(L23) | O(E13) | O(E23) |
| O(L13) | 1.609 (2) | 2.423 (2) | 2.461 (3) | 2.561 (3) |
| O(L23) | 98.26 (9) | 1.595 (2) | 2.498 (2) | 2.543 (3) |
| O(E13) | 105.10 (10) | 108.17 (10) | 1.489 (2) | 2.550 (3) |
| O(E23) | 112.37 (10) | 111.95 (10) | 118.84 (13) | 1.472 (2) |
| P(1)—P(2) | 2.9290 (7) | | | |
| P(1)—P(3) | 2.9350 (8) | P(2)—P(3) | 2.9725 (6) | |
| P(2)—P(1)—P(3) | 101.09 (2) | P(1)—O(L13)—P(3) | 132.07 (10) | |
| P(1)—P(2)—P(3) | 139.52 (3) | P(1)—O(L12)—P(2) | 131.35 (11) | |
| P(1)—P(3)—P(2) | 99.53 (2) | P(2)—O(L23)—P(3) | 137.16 (14) | |
| Te(OH)₆ octahedra | | | | |
| Te(1)—O(1) | 1.898 (2) | (× 2) | Te(2)—O(4) | 1.909 (2) |
| Te(1)—O(2) | 1.932 (2) | (× 2) | Te(2)—O(5) | 1.912 (2) |
| Te(1)—O(3) | 1.926 (2) | (× 2) | Te(2)—O(6) | 1.934 (2) |
| Te(1)—O(1)—H(1) | 111 (3) | | Te(2)—O(4)—H(4) | 107 (2) |
| Te(1)—O(2)—H(2) | 117 (3) | | Te(2)—O(5)—H(5) | 103 (3) |
| Te(1)—O(3)—H(3) | 104 (2) | | Te(2)—O(6)—H(6) | 107 (2) |
| Ethylenediammonium groups | | | | |
| N(1)—C(1) | 1.488 (4) | N(1)—C(1)—C(1) | 109.7 (2) | |
| C(1)—C(1) | 1.507 (3) | | | |
| N(2)—C(2) | 1.489 (2) | N(2)—C(2)—C(3) | 108.5 (2) | |
| C(2)—C(3) | 1.516 (3) | N(2)—C(3)—N(3) | 111.1 (2) | |
| C(3)—N(3) | 1.471 (3) | | | |
| Hydrogen bonds | | | | |
| O(N)—H...O | O(N)—H | H...O | O(N)—O | O(N)...O |
| O(1)—H(1)...O(E21) | 0.84 (4) | 1.96 (4) | 2.738 (2) | 155 (4) |
| O(2)—H(2)...O(E13) | 0.74 (3) | 2.41 (3) | 3.072 (2) | 149 (4) |
| O(3)—H(3)...O(E11) | 0.90 (3) | 1.84 (4) | 2.718 (2) | 164 (4) |
| O(4)—H(4)...O(E22) | 0.78 (3) | 2.06 (3) | 2.778 (2) | 155 (3) |
| O(5)—H(5)...O(E12) | 0.79 (4) | 2.01 (4) | 2.777 (2) | 163 (4) |
| O(6)—H(6)...O(3) | 0.80 (4) | 1.96 (4) | 2.745 (3) | 167 (4) |
| O(W)—H(1W)...O(E13) | 0.99 (5) | 1.96 (5) | 2.918 (4) | 161 (3) |
| O(W)—H(2W)...O(5) | 0.86 (3) | 2.48 (4) | 3.267 (3) | 152 (4) |
| O(W)—H(2W)...O(6) | 0.86 (3) | 2.39 (4) | 3.098 (3) | 140 (4) |
| N(1)—H(1N1)...O(E13) | 0.82 (4) | 1.96 (3) | 2.766 (3) | 165 (4) |
| N(1)—H(2N1)...O(E21) | 0.87 (3) | 2.14 (3) | 2.989 (2) | 166 (4) |
| N(1)—H(3N1)...O(E22) | 0.89 (3) | 1.93 (3) | 2.816 (3) | 174 (3) |
| N(2)—H(1N2)...O(6) | 0.92 (4) | 1.97 (4) | 2.858 (3) | 164 (3) |
| N(2)—H(2N2)...O(E12) | 0.94 (4) | 1.98 (4) | 2.891 (3) | 162 (2) |
| N(2)—H(3N2)...O(E11) | 0.96 (4) | 1.91 (4) | 2.772 (3) | 148 (3) |
| N(3)—H(1N3)...O(E23) | 0.89 (4) | 2.23 (4) | 2.825 (3) | 148 (3) |
| N(3)—H(2N3)...O(W) | 0.96 (4) | 2.15 (4) | 3.045 (4) | 156 (3) |
| N(3)—H(2N3)...O(E21) | 0.84 (4) | 2.21 (4) | 2.995 (4) | 156 (3) |

The $Te(OH)_6$ groups. The two independent $Te(OH)_6$ groups are located around inversion centres at 0,0,0 and $0, \frac{1}{2}, 0$ separated by a distance of 5.626 Å. As usual they appear as almost regular octahedra. Their internal geometry, Te—O distances and O—Te—O angles are reported in Table 2. The $Te(OH)_6$ groups are interconnected by a hydrogen bond [O(6)—H(6)...O(3)] to form a chain spreading along the *b* axis.

The P_6O_{18} anion. The centrosymmetric phosphoric ring anion is located around the inversion centre at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. As it has been frequently observed for P_6O_{18} ring anions with $\bar{1}$ internal symmetry the P—P—P angles vary in a wide range, e.g. from 99.53 (2) to 139.52 (3)°. Main interatomic distances and bond angles in this ring are reported in Table 2. The P—P distances [2.9290 (7)–2.9725 (6) Å] as well as the P—O—P angles [131.35 (11)–137.16 (14)°] are in accordance with values normally observed in condensed phosphates.

The ethylenediammonium groups. Two kinds of ethylenediammonium groups co-exist in this atomic arrangement. The first one built up by C(1) and N(1) atoms is centrosymmetrical and located around the inversion centre at $\frac{1}{2}, \frac{1}{2}, 0$. The second one built up by C(2), C(3), N(2) and N(3) has no internal symmetry. In spite of this difference, the main geometrical features of these two groups, reported in Table 2, do not differ significantly.

The hydrogen-bond scheme. The main characteristics of the three-dimensional network of hydrogen-bonds reported in Table 2 show no special features for the N—H...O or O(W)—H...O bonds, but it must be noted that the $Te(OH)_6$ groups spreading along the *b* axis are interconnected by the O(6)—H(6)...O(3) bond.

The drawing was prepared using the STRUPLO program (Fischer, 1985).

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