

Bis(homopiperazine-1,4-dium) cyclo-tetraphosphate–telluric acid (1/2)

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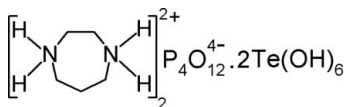
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.033; wR factor = 0.084; data-to-parameter ratio = 28.5.

The title compound, $2\text{C}_5\text{H}_{14}\text{N}_2^{2+} \cdot \text{P}_4\text{O}_{12}^{4-} \cdot 2\text{Te}(\text{OH})_6$, involves doubly protonated homopiperazinium cations, cyclotetraphosphate anions and telluric acid molecules. The framework possesses very large channels wherein the organic cations reside. A network of $\text{O}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds consolidates the crystal packing.

Related literature

For the properties of materials containing telluric acid, see: Chabchoub *et al.* (2006); Khemakhem, (1999). For related structures containing phosphate rings and telluric acid, see: Averbuch-Pouchot & Durif (1987*a,b*); Durif *et al.* (1982). For hydrogen bonding, see: Blessing (1986); Brown (1976). For deviations in four-membered phosphate rings having the same $\bar{1}$ internal symmetry, see: Durif (1995); A similar conformation for the same organic molecule was observed in $(\text{C}_5\text{H}_{14}\text{N}_2)(\text{H}_2\text{AsO}_4)_2$, see: Wilkinson & Harrison (2006). For the synthesis, see: Ondik (1964).



Experimental

Crystal data

$\text{C}_5\text{H}_{14}\text{N}_2^{2+} \cdot 0.5\text{P}_4\text{O}_{12}^{4-} \cdot \text{Te}(\text{OH})_6$
 $M_r = 489.77$
 Monoclinic, $C2/c$
 $a = 20.826$ (3) Å
 $b = 8.3600$ (13) Å
 $c = 17.030$ (8) Å
 $\beta = 101.65$ (3)°
 $V = 2903.9$ (14) Å³
 $Z = 8$
 Ag $K\alpha$ radiation
 $\lambda = 0.56083$ Å
 $\mu = 1.24$ mm⁻¹
 $T = 293$ K
 $0.2 \times 0.18 \times 0.16$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 8219 measured reflections
 6346 independent reflections
 4847 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 2 standard reflections every 120 min
 intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.084$
 $S = 1.01$
 6346 reflections
 223 parameters
 12 restraints
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.99$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1} \cdots \text{O12}^i$	0.86 (2)	1.85 (2)	2.686 (3)	165 (2)
$\text{N1}-\text{H1B} \cdots \text{O4}^{ii}$	0.90	2.15	2.898 (3)	140
$\text{N1}-\text{H1B} \cdots \text{O9}^{iii}$	0.90	2.42	3.002 (3)	123
$\text{N1}-\text{H1A} \cdots \text{O2}^{iv}$	0.90	1.89	2.763 (3)	162
$\text{O2}-\text{H2} \cdots \text{O9}^v$	0.86 (2)	1.83 (2)	2.688 (3)	172 (3)
$\text{N2}-\text{H2A} \cdots \text{O12}$	0.90	1.89	2.769 (3)	167
$\text{N2}-\text{H2B} \cdots \text{O11}^{vi}$	0.90	1.85	2.739 (3)	172
$\text{O3}-\text{H3} \cdots \text{O8}^v$	0.84 (3)	2.05 (3)	2.881 (3)	170 (4)
$\text{O4}-\text{H4} \cdots \text{O1}^{vii}$	0.85 (3)	1.85 (3)	2.695 (3)	175 (3)
$\text{O5}-\text{H5} \cdots \text{O8}^{viii}$	0.87 (3)	1.85 (3)	2.719 (3)	172 (3)
$\text{O6}-\text{H6} \cdots \text{O9}^{ix}$	0.83 (3)	1.90 (2)	2.722 (3)	171 (3)
$\text{C2}-\text{H2C} \cdots \text{O1}^x$	0.97	2.60	3.162 (3)	117
$\text{C4}-\text{H4B} \cdots \text{O5}^i$	0.97	2.43	3.256 (4)	142
$\text{C5}-\text{H5B} \cdots \text{O11}^{xi}$	0.97	2.31	3.265 (4)	168

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x, y, z + 1$; (v) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (vi) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (vii) $-x, y, -z + \frac{1}{2}$; (viii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (ix) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (x) $x, -y, z + \frac{1}{2}$; (xi) $x, -y + 1, z + \frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2159).

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supplementary materials

Acta Cryst. (2010). E66, o2712 [doi:10.1107/S1600536810038225]

Bis(homopiperazine-1,4-dium) cyclotetraphosphate-telluric acid (1/2)

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Comment

Materials containing telluric acid exhibit many interesting physical properties such as ferroelectricity and protonic conduction (Chabchoub *et al.*, 2006; Khemakhem, 1999). On the chemical plane, telluric acid has the property to form adduct compounds with many kinds of phosphates and was extensively investigated during the past thirty years. With cyclotriphosphate, several adduct compounds of telluric acid have been identified whereas with cyclotetraphosphate only three inorganic telluric acid-adduct compounds are reported: $(\text{NH}_4)_4\text{P}_4\text{O}_{12} \cdot \text{Te}(\text{OH})_6 \cdot 2\text{H}_2\text{O}$ (Durif *et al.*, 1982), $\text{K}_4\text{P}_4\text{O}_{12} \cdot \text{Te}(\text{OH})_6 \cdot 2\text{H}_2\text{O}$ and $\text{Rb}_4\text{P}_4\text{O}_{12} \cdot \text{Te}(\text{OH})_6 \cdot 2\text{H}_2\text{O}$ (Averbuch-Pouchot & Durif, 1987a b). In the present work, we report the first telluric acid-adduct compound of an organic cyclotetraphosphate $(\text{C}_5\text{H}_{14}\text{N}_2)_2\text{P}_4\text{O}_{12} \cdot 2\text{Te}(\text{OH})_6$ (I). The configuration of different components of (I) is shown in Figure 1. In the crystal structure of this compound, $\text{P}_4\text{O}_{12}^{4-}$ and $\text{Te}(\text{OH})_6$ form an anionic three-dimensional framework. The negative charge of this is compensated by the diprotonated amine $(\text{C}_5\text{H}_{14}\text{N}_2)^{2+}$. Figure 2 shows the total atomic arrangement projected along the [1 0 1] direction. This projection shows that the inorganic entities form very large channels wherein the organic cations are located. The telluric acid molecules $\text{Te}(\text{OH})_6$ are linked pair-wise by two symmetric strong $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds, with an $\text{O} \cdots \text{O}$ distance of 2.695 (3) Å, into dimers where the $\text{Te} \cdots \text{Te}$ distance is 5.102 (5) Å. The tellurium atom is surrounded by six OH groups in a rather regular octahedral arrangement as indicated by the Te-O distances ranging from 1.905 (2) to 1.916 (2) Å, the *cis* and *trans* O-Te-O angles which are found in the 87.4 (1)-92.94 (9) ° and 175.03 (9)-178.8 (1) ° ranges, respectively, and Te—O—H angles ranging from 107 (3) to 116 (3) °. Each $\text{Te}(\text{OH})_6$ dimer is linked to six P_4O_{12} rings and two homopiperazinium $(\text{C}_5\text{H}_{14}\text{N}_2)^{2+}$ through a three-dimensional hydrogen bonding network $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ (Fig. 3). The P_4O_{12} ring anions are centrosymmetric and are built by only two crystallographically independent PO_4 tetrahedra with P-O distances ranging from 1.477 (2) to 1.605 (2) Å and O-P-O angles ranging from 103.9 (1) to 120.2 (2) ° with a mean value (109.22°) very close to the ideal value (109.28°). In spite of these large ranges in P-O distances and O-P-O angles, which can be explained by different environments of oxygen atoms, the PO_4 tetrahedron is described by regular oxygen atom arrangement with the phosphorus atom shifted by 0.122 and 0.149 Å from the centre of gravity. The cyclic anion is distorted as evidenced by P—P—P angles (81.60 (1) and 98.40 (1) °) which show a pronounced deviation from the ideal value (90°). Such deviation is commonly observed in tetramembered phosphoric rings having the same -1 internal symmetry (Durif, 1995). Only one homopiperazinium cation $(\text{C}_5\text{H}_{14}\text{N}_2)^{2+}$ exists in the asymmetric unit and adopts a chair conformation as evidenced by the mean deviation (± 0.0251 Å) from the least square plane defined by the four constituent atoms N1, N2, C1 and C3 and the remaining atoms C2, C4 and C5 displaced from the plane by 0.7495 (3), 0.6175 (3) and -0.2661 (3) Å, respectively. A similar conformation for the same organic molecule was observed in $(\text{C}_5\text{H}_{14}\text{N}_2)(\text{H}_2\text{AsO}_4)_2$ (Wilkinson & Harrison, 2006) with important difference that the "seat" chair was defined by one N atom and three C atoms rather than two N atoms and two C atoms as found here. The organic and inorganic components exert between them different interactions (electrostatic, hydrogen bonds and Van der Waals) to form a stable three-dimensional network. The examination of the hydrogen-bond scheme shows the existence of four strong hydrogen bonds with distances $\text{O} \cdots \text{O}$ ranging from 2.686 (3) to 2.722 (3) Å. The other bonds are weaker, with $\text{O}(\text{N},\text{C}) \cdots \text{O}$ distances falling from 2.739 (3) to 3.265 (4) Å (Blessing, 1986; Brown, 1976).

Experimental

Crystals of the title compound were prepared by adding ethanolic solution (5 ml) of homopiperazine (10 mmol) dropwise to an aqueous solution of cyclotetraphosphoric acid (5 mmol, 20 ml). The obtained solution was added to an aqueous solution of telluric acid (10 mmol, 15 ml). Good quality of colourless prisms were obtained after a slow evaporation during few days at ambient temperature. The cyclotetraphosphoric acid $H_4P_4O_{12}$, was produced from $Na_4P_4O_{12} \cdot 4H_2O$, prepared according to the Ondik process (Ondik, 1964), through an ion-exchange resin in H-state (Amberlite IR 120).

Refinement

All H atoms of the organic molecule were positioned geometrically and treated as riding on their parent atoms, [N–H = 0.89, C–H = 0.96 Å (CH₃) with $U_{iso}(H) = 1.5U_{eq}$ and C–H = 0.96 Å (Ar–H), with $U_{iso}(H) = 1.5U_{eq}$]. The H-atoms of telluric acid, located in difference Fourier maps, were refined with a distance restraint of O–H = 0.86 (2) Å; their temperature factors were refined.

Figures

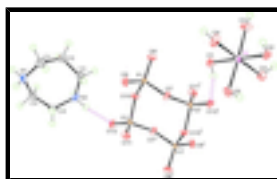


Fig. 1. An *ORTEP* view of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. [Symmetry codes: (i) $1/2 - x, 1/2 - y, 1 - z$].

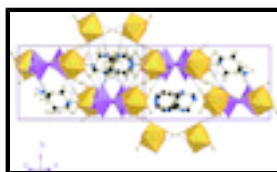


Fig. 2. Projection of (I) along [1 0 1] direction. Some hydrogen atoms are omitted for clarity.

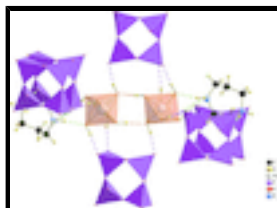


Fig. 3. A perspective view of the atomic arrangement of (I).

Bis(piperazine-1,4-dium) cyclotetraphosphate–telluric acid (1/2)

Crystal data

$C_5H_{14}N_2^{2+} \cdot 0.5P_4O_{12}^{4-} \cdot Te(OH)_6$

$M_r = 489.77$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 20.826 (3) \text{ \AA}$

$b = 8.3600 (13) \text{ \AA}$

$F(000) = 1936$

$D_x = 2.241 \text{ Mg m}^{-3}$

Ag $K\alpha$ radiation, $\lambda = 0.56083 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 9.0\text{--}10.9^\circ$

$\mu = 1.24 \text{ mm}^{-1}$

$c = 17.030 (8) \text{ \AA}$
 $\beta = 101.65 (3)^\circ$
 $V = 2903.9 (14) \text{ \AA}^3$
 $Z = 8$

$T = 293 \text{ K}$
 Prism, colourless
 $0.2 \times 0.18 \times 0.16 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 Radiation source: Enraf Nonius FR590 graphite
 Non-profiled ω scans
 8219 measured reflections
 6346 independent reflections
 4847 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.1^\circ$
 $h = -33 \rightarrow 33$
 $k = 0 \rightarrow 13$
 $l = -5 \rightarrow 27$
 2 standard reflections every 120 min
 intensity decay: 5%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.084$
 $S = 1.01$
 6346 reflections
 223 parameters
 12 restraints

Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.99 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.19 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C5	0.17222 (12)	0.3358 (3)	0.92873 (16)	0.0227 (5)
H5A	0.1782	0.2247	0.9450	0.027*

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H5B	0.1909	0.4014	0.9747	0.027*
C4	0.20920 (13)	0.3672 (3)	0.86247 (18)	0.0253 (5)
H4A	0.1880	0.4538	0.8291	0.030*
H4B	0.2535	0.4010	0.8860	0.030*
H3	-0.0202 (13)	0.058 (5)	0.080 (2)	0.060 (14)*
H4	-0.0415 (18)	0.298 (4)	0.1851 (19)	0.039 (11)*
H1	0.1244 (7)	0.217 (4)	0.2504 (13)	0.027 (9)*
H6	0.0994 (18)	0.505 (3)	0.1086 (14)	0.053 (13)*
H2	-0.0171 (9)	0.333 (4)	0.0087 (11)	0.024 (8)*
H5	0.1230 (13)	0.094 (3)	0.068 (2)	0.047 (12)*
Te1	0.053963 (7)	0.257312 (16)	0.128185 (8)	0.01399 (4)
P1	0.35495 (3)	0.28147 (6)	0.49522 (3)	0.01199 (9)
P2	0.26836 (3)	0.30733 (7)	0.61119 (3)	0.01214 (9)
O10	0.32281 (8)	0.22730 (19)	0.56907 (10)	0.0161 (3)
O7	0.29316 (8)	0.3056 (2)	0.42301 (10)	0.0167 (3)
O9	0.39402 (8)	0.1419 (2)	0.47840 (11)	0.0199 (3)
O1	0.08267 (8)	0.2069 (2)	0.23924 (11)	0.0200 (3)
O11	0.25530 (8)	0.4740 (2)	0.58395 (11)	0.0197 (3)
N2	0.21224 (11)	0.2239 (2)	0.81152 (15)	0.0223 (4)
H2A	0.2422	0.2419	0.7812	0.027*
H2B	0.2264	0.1404	0.8438	0.027*
O4	-0.02504 (9)	0.3469 (2)	0.15028 (12)	0.0239 (4)
O6	0.09666 (11)	0.4585 (2)	0.15090 (12)	0.0273 (4)
O8	0.38702 (8)	0.4392 (2)	0.51103 (11)	0.0197 (3)
O12	0.28855 (8)	0.2710 (2)	0.69789 (10)	0.0217 (3)
O2	0.02429 (9)	0.3119 (3)	0.01751 (11)	0.0229 (4)
O5	0.13208 (8)	0.1739 (3)	0.10185 (12)	0.0254 (4)
C3	0.14949 (14)	0.1782 (3)	0.75778 (16)	0.0268 (5)
H3A	0.1564	0.0813	0.7292	0.032*
H3B	0.1368	0.2621	0.7183	0.032*
N1	0.10076 (10)	0.3697 (3)	0.90552 (13)	0.0211 (4)
H1A	0.0817	0.3340	0.9451	0.025*
H1B	0.0955	0.4765	0.9031	0.025*
C2	0.09384 (13)	0.1504 (3)	0.80154 (17)	0.0245 (5)
H2C	0.0595	0.0911	0.7665	0.029*
H2D	0.1099	0.0841	0.8481	0.029*
O3	0.01479 (9)	0.0496 (2)	0.11451 (13)	0.0253 (4)
C1	0.06439 (13)	0.3009 (4)	0.82876 (18)	0.0289 (5)
H1C	0.0619	0.3812	0.7871	0.035*
H1D	0.0199	0.2775	0.8344	0.035*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.0248 (11)	0.0218 (10)	0.0203 (11)	0.0009 (9)	0.0014 (9)	-0.0054 (9)
C4	0.0217 (10)	0.0176 (10)	0.0379 (15)	-0.0035 (8)	0.0093 (10)	-0.0031 (10)
Te1	0.01393 (6)	0.01490 (6)	0.01358 (6)	0.00039 (5)	0.00382 (4)	0.00058 (5)
P1	0.0102 (2)	0.0138 (2)	0.0119 (2)	-0.00096 (16)	0.00219 (17)	-0.00043 (17)

P2	0.0121 (2)	0.0149 (2)	0.0091 (2)	-0.00041 (17)	0.00137 (17)	-0.00098 (18)
O10	0.0179 (7)	0.0176 (7)	0.0150 (7)	0.0030 (5)	0.0081 (6)	0.0033 (5)
O7	0.0144 (6)	0.0184 (7)	0.0153 (7)	-0.0029 (5)	-0.0016 (5)	0.0042 (6)
O9	0.0178 (7)	0.0238 (8)	0.0187 (8)	0.0042 (6)	0.0051 (6)	-0.0050 (6)
O1	0.0178 (7)	0.0266 (8)	0.0151 (7)	0.0017 (6)	0.0023 (6)	0.0054 (6)
O11	0.0218 (8)	0.0140 (7)	0.0224 (8)	0.0010 (6)	0.0023 (7)	-0.0022 (6)
N2	0.0252 (9)	0.0183 (8)	0.0275 (11)	0.0030 (7)	0.0152 (9)	0.0046 (7)
O4	0.0236 (8)	0.0291 (9)	0.0213 (8)	0.0106 (7)	0.0101 (7)	0.0080 (7)
O6	0.0435 (11)	0.0192 (8)	0.0191 (9)	-0.0103 (8)	0.0060 (8)	-0.0019 (7)
O8	0.0191 (7)	0.0189 (7)	0.0198 (8)	-0.0068 (6)	0.0010 (6)	-0.0013 (6)
O12	0.0173 (7)	0.0394 (10)	0.0084 (6)	-0.0014 (7)	0.0021 (6)	0.0014 (6)
O2	0.0170 (7)	0.0376 (10)	0.0142 (7)	0.0013 (7)	0.0034 (6)	0.0046 (7)
O5	0.0146 (7)	0.0335 (10)	0.0282 (10)	0.0016 (7)	0.0045 (7)	-0.0091 (8)
C3	0.0350 (13)	0.0285 (12)	0.0182 (11)	0.0024 (10)	0.0083 (10)	-0.0008 (10)
N1	0.0248 (9)	0.0209 (9)	0.0197 (10)	0.0009 (7)	0.0094 (8)	-0.0039 (7)
C2	0.0254 (11)	0.0217 (11)	0.0261 (12)	-0.0026 (9)	0.0042 (10)	-0.0058 (9)
O3	0.0218 (8)	0.0196 (8)	0.0326 (11)	-0.0040 (6)	0.0009 (8)	0.0022 (7)
C1	0.0222 (11)	0.0325 (13)	0.0306 (14)	0.0044 (10)	0.0021 (10)	-0.0068 (11)

Geometric parameters (Å, °)

C5—N1	1.488 (3)	O7—P2 ⁱ	1.6033 (17)
C5—C4	1.512 (4)	O1—H1	0.855 (13)
C5—H5A	0.9700	N2—C3	1.486 (4)
C5—H5B	0.9700	N2—H2A	0.9000
C4—N2	1.488 (3)	N2—H2B	0.9000
C4—H4A	0.9700	O4—H4	0.846 (18)
C4—H4B	0.9700	O6—H6	0.830 (17)
Te1—O5	1.9050 (18)	O2—H2	0.862 (16)
Te1—O6	1.9050 (18)	O5—H5	0.874 (17)
Te1—O1	1.9119 (19)	C3—C2	1.517 (4)
Te1—O3	1.9124 (18)	C3—H3A	0.9700
Te1—O4	1.9130 (18)	C3—H3B	0.9700
Te1—O2	1.916 (2)	N1—C1	1.488 (4)
P1—O8	1.4784 (17)	N1—H1A	0.9000
P1—O9	1.4831 (17)	N1—H1B	0.9000
P1—O7	1.6028 (18)	C2—C1	1.513 (4)
P1—O10	1.6046 (18)	C2—H2C	0.9700
P2—O11	1.4766 (18)	C2—H2D	0.9700
P2—O12	1.4825 (19)	O3—H3	0.840 (19)
P2—O7 ⁱ	1.6033 (17)	C1—H1C	0.9700
P2—O10	1.6052 (17)	C1—H1D	0.9700
N1—C5—C4	113.7 (2)	P1—O10—P2	132.59 (11)
N1—C5—H5A	108.8	P1—O7—P2 ⁱ	131.58 (11)
C4—C5—H5A	108.8	Te1—O1—H1	107.4 (15)
N1—C5—H5B	108.8	C3—N2—C4	115.5 (2)
C4—C5—H5B	108.8	C3—N2—H2A	108.4
H5A—C5—H5B	107.7	C4—N2—H2A	108.4

supplementary materials

N2—C4—C5	112.5 (2)	C3—N2—H2B	108.4
N2—C4—H4A	109.1	C4—N2—H2B	108.4
C5—C4—H4A	109.1	H2A—N2—H2B	107.5
N2—C4—H4B	109.1	Te1—O4—H4	116 (3)
C5—C4—H4B	109.1	Te1—O6—H6	110.3 (17)
H4A—C4—H4B	107.8	Te1—O2—H2	109.4 (13)
O5—Te1—O6	89.15 (9)	Te1—O5—H5	110.6 (16)
O5—Te1—O1	92.34 (9)	N2—C3—C2	113.6 (2)
O6—Te1—O1	87.36 (8)	N2—C3—H3A	108.8
O5—Te1—O3	90.15 (9)	C2—C3—H3A	108.8
O6—Te1—O3	175.03 (9)	N2—C3—H3B	108.8
O1—Te1—O3	87.75 (9)	C2—C3—H3B	108.8
O5—Te1—O4	177.38 (9)	H3A—C3—H3B	107.7
O6—Te1—O4	89.95 (9)	C1—N1—C5	117.7 (2)
O1—Te1—O4	90.08 (8)	C1—N1—H1A	107.9
O3—Te1—O4	90.96 (8)	C5—N1—H1A	107.9
O5—Te1—O2	88.67 (9)	C1—N1—H1B	107.9
O6—Te1—O2	91.96 (9)	C5—N1—H1B	107.9
O1—Te1—O2	178.78 (8)	H1A—N1—H1B	107.2
O3—Te1—O2	92.94 (9)	C1—C2—C3	114.8 (2)
O4—Te1—O2	88.91 (8)	C1—C2—H2C	108.6
O8—P1—O9	119.45 (10)	C3—C2—H2C	108.6
O8—P1—O7	106.91 (10)	C1—C2—H2D	108.6
O9—P1—O7	109.72 (10)	C3—C2—H2D	108.6
O8—P1—O10	110.71 (10)	H2C—C2—H2D	107.5
O9—P1—O10	105.15 (10)	Te1—O3—H3	107 (3)
O7—P1—O10	103.86 (9)	N1—C1—C2	115.1 (2)
O11—P2—O12	120.22 (11)	N1—C1—H1C	108.5
O11—P2—O7 ⁱ	111.00 (9)	C2—C1—H1C	108.5
O12—P2—O7 ⁱ	106.65 (10)	N1—C1—H1D	108.5
O11—P2—O10	110.78 (10)	C2—C1—H1D	108.5
O12—P2—O10	106.01 (10)	H1C—C1—H1D	107.5
O7 ⁱ —P2—O10	100.20 (9)		

Symmetry codes: (i) $-x+1/2, -y+1/2, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O12 ⁱ	0.86 (2)	1.85 (2)	2.686 (3)	165 (2)
N1—H1B \cdots O4 ⁱⁱ	0.90	2.15	2.898 (3)	140
N1—H1B \cdots O9 ⁱⁱⁱ	0.90	2.42	3.002 (3)	123
N1—H1A \cdots O2 ^{iv}	0.90	1.89	2.763 (3)	162
O2—H2 \cdots O9 ^v	0.86 (2)	1.83 (2)	2.688 (3)	172 (3)
N2—H2A \cdots O12	0.90	1.89	2.769 (3)	167
N2—H2B \cdots O11 ^{vi}	0.90	1.85	2.739 (3)	172
O3—H3 \cdots O8 ^v	0.84 (3)	2.05 (3)	2.881 (3)	170 (4)
O4—H4 \cdots O1 ^{vii}	0.85 (3)	1.85 (3)	2.695 (3)	175 (3)

O5—H5...O8 ^{viii}	0.87 (3)	1.85 (3)	2.719 (3)	172 (3)
O6—H6...O9 ^{ix}	0.83 (3)	1.90 (2)	2.722 (3)	171 (3)
C2—H2C...O1 ^x	0.97	2.60	3.162 (3)	117
C4—H4B...O5 ⁱ	0.97	2.43	3.256 (4)	142
C5—H5B...O11 ^{xi}	0.97	2.31	3.265 (4)	168

Symmetry codes: (i) $-x+1/2, -y+1/2, -z+1$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1/2, y+1/2, -z+3/2$; (iv) $x, y, z+1$; (v) $x-1/2, -y+1/2, z-1/2$; (vi) $-x+1/2, y-1/2, -z+3/2$; (vii) $-x, y, -z+1/2$; (viii) $-x+1/2, y-1/2, -z+1/2$; (ix) $-x+1/2, y+1/2, -z+1/2$; (x) $x, -y, z+1/2$; (xi) $x, -y+1, z+1/2$.

Fig. 1

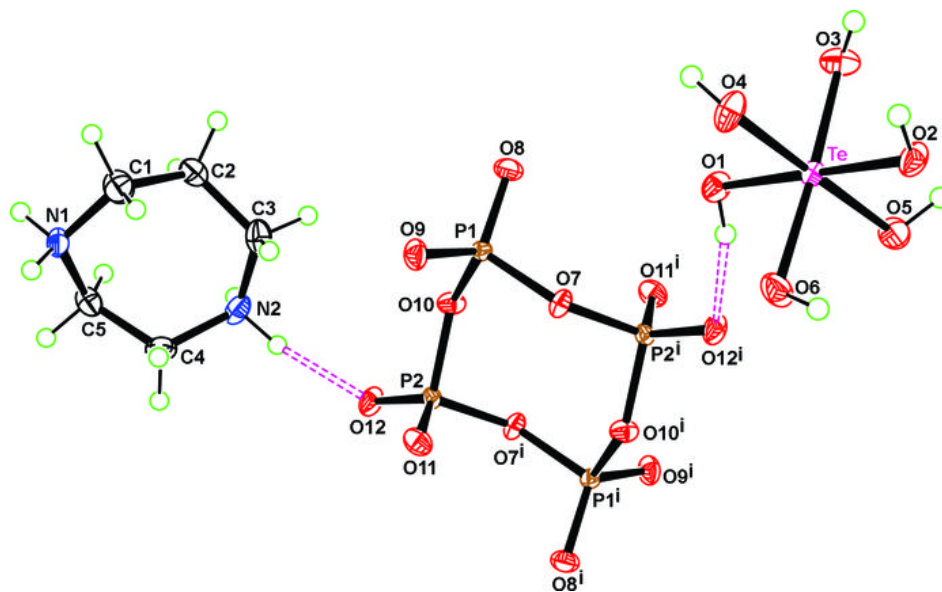


Fig. 2

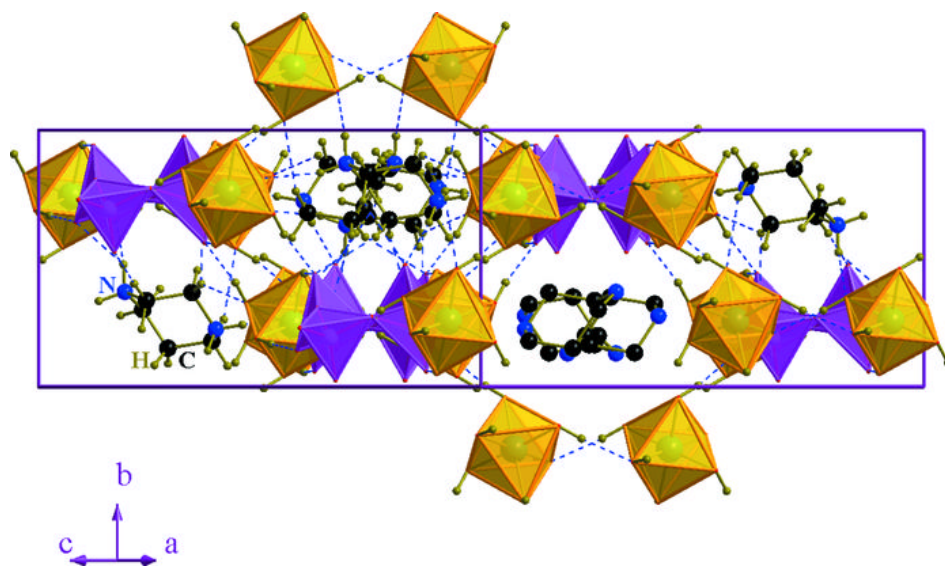


Fig. 3

