Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetrakis(triethanolammonium) cyclotetraphosphate

El Hassane Soumhi,[a]* Ismael Saadoune[a] and Ahmed Driss[b]

[a]Equipe de Chimie des Matériaux et de l'Environnement, FSTG-Marrakech, Université Cadi Ayyad, Boulevard Abdelkrim Khattabi, BP 549, Marrakech, Morocco, and [b]Laboratoire de Matériaux et Cristallochimie, Faculté des Sciences de Tunis, Université de Tunis El Manar, 2092 El Manar II, Tunis, Tunisia
Correspondence e-mail: eh_soumhi@yahoo.fr

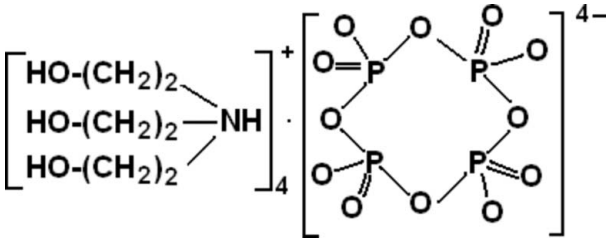
Received 18 April 2007; accepted 27 April 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(P–O) = 0.002 Å; disorder in main residue; R factor = 0.046; wR factor = 0.146; data-to-parameter ratio = 14.3.

In the title compound, $4C_6H_{16}NO_3^+P_4O_{12}^{4-}$, the asymmetric unit consists of two organic cations and two linked PO_4 tetrahedra. These tetrahedra are also linked with symmetry-equivalents to form centrosymmetric P_4O_{12} rings. The organic cations are linked to the cyclotetraphosphate rings by O—H···O═P hydrogen bonds to form a three-dimensional network.

Related literature

For related structures, see: Nahouane *et al.* (2005) and Soumhi *et al.* (1998, 2005, 2006).



Experimental

Crystal data

$4C_6H_{16}NO_3^+\cdot O_{12}P_4^{4-}$
M_r = 916.68
Monoclinic, $P2_1/n$
a = 9.945 (2) Å
b = 14.591 (3) Å
c = 14.428 (3) Å
β = 101.12 (2)°
V = 2054.3 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
5112 measured reflections
4483 independent reflections
3545 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections
frequency: 60 min
intensity decay: 1.2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.146
S = 1.05
4483 reflections
314 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O21	0.82	1.90	2.718 (3)	172
O2—H2···O11^{i}	0.82	1.98	2.771 (3)	164
O3—H3···O22^{ii}	0.82	1.85	2.658 (3)	170
O4—H4···O12^{iii}	0.82	1.85	2.669 (3)	177
O5—H5···O21	0.82	1.85	2.659 (3)	170
O6—H6···O22	0.82	1.91	2.722 (3)	174
N1—H7···O2	0.91	2.26	2.743 (3)	113
N2—H8···O4	0.91	2.27	2.703 (3)	109

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y, -z+1$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS for Windows* (Dowty, 1995); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2299).

References

Dowty, E. (1995). *ATOMS for Windows*. Version 3.2. Shape Software, Kingsport, Tennessee, USA.
Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.
Macíček, J. & Yordanov, A. (1992). *J. Appl. Cryst.* **25**, 73–80.
Nahouane, R., Soumhi, E. H., Saadoune, I. & Driss, A. (2005). *Acta Cryst.* E**61**, o2850–o2852.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Soumhi, E. H., Saadoune, I. & Driss, A. (2006). *Acta Cryst.* E**62**, o212–o214.
Soumhi, E. H., Saadoune, I., Driss, A. & Jouini, T. (1998). *Eur. J. Solid State Inorg. Chem.* **35**, 629–637.
Soumhi, E. H., Saadoune, I., Nahouane, R. & Driss, A. (2005). *Acta Cryst.* E**61**, o2847–o2849.

 doi:10.1107/S1600536807021083

supplementary materials

Acta Cryst. (2007). E**63**, o2827 [**doi:10.1107/S1600536807021083**]

Tetrakis(triethanolammonium) cyclotetraphosphate

E. H. Soumhi, I. Saadoune and A. Driss

Comment

The present description of the stucture of $2(C_6H_{16}NO_3)^{+}0.5(P_4O_{12})^{4-}$ is part of an investigation of materials resulting from interaction between cyclotetraphosphoric acid and organic molecules such as amines, amino-acids and aminoalcohols, with the aim to select a new protonic conductors which are of interest as solid electrolytes.

As shown in figure 2, the crystal structure of bis(triethanolammonium) hemi(cyclotetraphosphate) could be described as a succession of organic and inorganic sheets parallel to the (101) plane. These layers are inteconnected by O2—H2..O11 H-bonds to form a three-dimensional network.

The P_4O_{12} rings lie about inversion centers at (0 1/2 0) and (1/2 0 1/2). The cyclotetraphosphate anion is, as usual, made up of four PO_4 tetrahedra sharing corners. It presents a strong distortion as shown by the P···P···P angles within the rings with values of 82.80° and 97.20°. Inside each PO_4 tetrahedron, two types of P—O distances are observed. The longest ones correspond to the bridging oxygen atoms OL and the shortest ones correpond to those for the terminal P—O. P—O distances vary from 1.475 (2) Å to 1.606 (2) Å, while the O—P—O angle values vary from 99.77 (9)° to 120.6 (2)°. Such values agree with those previously reported for other studied cyclotetraphosphates (Nahouane *et al.*, 2005; Soumhi, 2006). In addition the asymmetric unit contains two independent organic cations. C—C, C—N, C—O distances and C—C—C, C—C—N, and O—C—C angles are in good agreement with those observed in similar condensed phosphate salts of organic cations (Soumhi, 1998; 2005).

The structure contains six O—H···O H-bonds involving the six hydroxyl groups of the organic cations and external oxygen atoms of P_4O_{12} rings. The O···O distances range from 2.658 (3) to 2.771 (3) Å, Table 1. Thus, this study confirms that the network stability as with most organic cation phosphates is due essentially to the H-bonding system.

Experimental

The title compound was prepared by neutralization of $H_4P_4O_{12}$ with triethanolamine in a 1:4 molar ratio. The $H_4P_4O_{12}$ solution was prepared using an aqueous solution of $Na_4P_4O_{12}.4H_2O$ and an ion-exchange resin (Amberlite IR-120). Colourless single crystals appeared after evaporation of the solution at room temperature for a few days.

Refinement

The C1, C3 and C5 atoms of one organic cation and the C7, C9 and C11 atoms of other are equally disordered over two positions a and b. They were refined with fixed occupancy factors. All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.97 Å, $U_{iso} = 1.2U_{eq}$ (C) for CH_2 atoms, d(O—H)0.82 Å, $U_{iso} = 1.2U_{eq}$ (O) for OH groups and 0.91 Å, $U_{iso} = 1.2U_{eq}$ (N) for the NH groups.

Figures

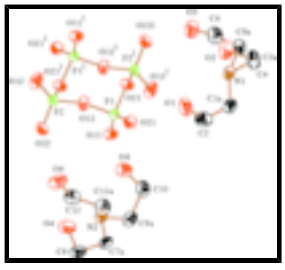


Fig. 1. The asymmetric unit of (I) with 50% probability ellipsoids. H atoms are omitted. Only one disorder component of the cations is shown with the atom label ending in a.

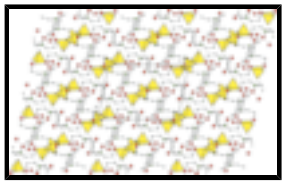


Fig. 2. Projection along the *b* axis of the $2(C_6H_{16}NO_3)^{+}0.5(P_4O_{12})^{4-}$ structure. By order of decreasing size, the circles represent O, N and C atoms. H atoms are omitted. Only one component of the disordered cations is shown.

Tetrakis(triethanolammonium) cyclotetraphosphate

Crystal data

$4C_6H_{16}NO_3^{+}\cdot O_{12}P_4^{4-}$	F_{000} = 976
M_r = 916.68	D_x = 1.482 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 9.945 (2) Å	θ = 14–16º
b = 14.591 (3) Å	μ = 0.27 mm^{-1}
c = 14.428 (3) Å	T = 298 (2) K
β = 101.12 (2)º	Plate, colourless
V = 2054.3 (7) Å^3	0.30 × 0.20 × 0.10 mm
Z = 2	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.021
Radiation source: fine-focus sealed tube	θ_{max} = 27.0º
Monochromator: graphite	θ_{min} = 2.0º
T = 298(2) K	h = 0→12
ω/2θ scans	k = −1→18
Absorption correction: none	l = −18→18
5112 measured reflections	3 standard reflections
4483 independent reflections	every 60 min
3545 reflections with $I > 2\sigma(I)$	intensity decay: 1.2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.146$

$S = 1.05$

4483 reflections

314 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0821P)^2 + 1.2338P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.33$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0050 (13)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}	Occ. (<1)
P1	0.46835 (6)	0.13183 (4)	0.50552 (4)	0.02529 (17)	
P2	0.61592 (6)	0.02263 (4)	0.38538 (4)	0.03044 (18)	
O11	0.33994 (17)	0.12216 (13)	0.43540 (12)	0.0377 (4)	
O21	0.5100 (2)	0.22260 (11)	0.54932 (12)	0.0392 (4)	
O12	0.76309 (19)	−0.00145 (15)	0.40132 (16)	0.0516 (5)	
O22	0.5464 (2)	0.05786 (13)	0.29148 (12)	0.0474 (5)	
OL12	0.59842 (17)	0.09636 (12)	0.46476 (12)	0.0354 (4)	
OL21	0.47489 (17)	0.06256 (11)	0.59256 (11)	0.0322 (4)	
O1	0.4594 (2)	0.2708 (2)	0.72131 (15)	0.0675 (7)	
H1	0.4698	0.2521	0.6696	0.081*	
O2	0.6311 (2)	0.26453 (16)	0.96937 (17)	0.0553 (6)	
H2	0.6877	0.3049	0.9672	0.066*	
O3	0.4951 (2)	0.03254 (17)	0.87152 (16)	0.0569 (6)	
H3	0.4891	0.0000	0.8246	0.068*	
O4	0.5562 (2)	0.46793 (18)	0.20999 (17)	0.0599 (7)	
H4	0.6097	0.4782	0.1745	0.072*	

O5	0.5151 (2)	0.37927 (14)	0.45598 (15)	0.0496 (5)	
H5	0.5199	0.3285	0.4808	0.060*	
O6	0.4491 (2)	0.22622 (15)	0.23101 (19)	0.0604 (6)	
H6	0.4726	0.1744	0.2494	0.072*	
N1	0.3754 (2)	0.20659 (15)	0.88240 (15)	0.0367 (5)	
H7	0.4591	0.1971	0.8673	0.044*	
N2	0.3440 (2)	0.39342 (14)	0.27570 (15)	0.0323 (4)	
H8	0.4322	0.3732	0.2858	0.039*	
C1A	0.3012 (6)	0.2844 (5)	0.8161 (5)	0.0528 (15)	0.50
C3A	0.3969 (6)	0.2338 (5)	0.9816 (4)	0.0474 (14)	0.50
C5A	0.2908 (6)	0.1181 (5)	0.8594 (5)	0.0546 (15)	0.50
C1B	0.2742 (6)	0.2149 (6)	0.7979 (4)	0.0590 (18)	0.50
H9	0.1908	0.2394	0.8138	0.071*	
H10	0.2534	0.1544	0.7711	0.071*	
C3B	0.3979 (5)	0.2948 (4)	0.9414 (4)	0.0430 (12)	0.50
H11	0.3932	0.3464	0.8986	0.052*	
H12	0.3229	0.3011	0.9751	0.052*	
C5B	0.3521 (6)	0.1277 (4)	0.9459 (4)	0.0435 (12)	0.50
H13	0.4180	0.1314	1.0049	0.052*	
H14	0.2610	0.1325	0.9603	0.052*	
C2	0.3187 (3)	0.2775 (3)	0.7222 (2)	0.0626 (9)	
H15	0.2805	0.3311	0.6869	0.075*	
H16	0.2713	0.2237	0.6928	0.075*	
C4	0.5219 (3)	0.3005 (2)	1.0075 (2)	0.0508 (7)	
H17	0.4973	0.3609	0.9815	0.061*	
H18	0.5487	0.3060	1.0755	0.061*	
C6	0.3667 (3)	0.0367 (2)	0.8992 (2)	0.0538 (7)	
H19	0.3801	0.0386	0.9676	0.065*	
H20	0.3141	−0.0178	0.8776	0.065*	
C7A	0.3159 (6)	0.4427 (5)	0.1794 (4)	0.0519 (15)	0.50
C9A	0.3333 (6)	0.4627 (4)	0.3538 (4)	0.0477 (13)	0.50
C11A	0.2560 (6)	0.3128 (4)	0.2764 (4)	0.0482 (14)	0.50
C7B	0.3360 (5)	0.4895 (3)	0.2464 (4)	0.0374 (11)	0.50
H21	0.3649	0.5283	0.3013	0.045*	
H22	0.2418	0.5048	0.2190	0.045*	
C9B	0.2908 (5)	0.3752 (4)	0.3648 (4)	0.0413 (12)	0.50
H23	0.1996	0.4011	0.3585	0.050*	
H24	0.2836	0.3096	0.3732	0.050*	
C11B	0.2728 (5)	0.3292 (4)	0.1986 (4)	0.0405 (11)	0.50
H25	0.1745	0.3372	0.1915	0.049*	
H26	0.2981	0.3466	0.1394	0.049*	
C8	0.4285 (3)	0.5083 (2)	0.1729 (2)	0.0461 (7)	
H27	0.4249	0.5251	0.1075	0.055*	
H28	0.4171	0.5635	0.2080	0.055*	
C10	0.3815 (3)	0.4153 (2)	0.4507 (2)	0.0498 (7)	
H29	0.3818	0.4592	0.5012	0.060*	
H30	0.3187	0.3662	0.4583	0.060*	
C12	0.3050 (3)	0.2332 (2)	0.2160 (2)	0.0483 (7)	
H31	0.2673	0.1755	0.2325	0.058*	

H32	0.2696	0.2445	0.1495	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0268 (3)	0.0232 (3)	0.0260 (3)	0.0011 (2)	0.0053 (2)	−0.0010 (2)
P2	0.0333 (3)	0.0292 (3)	0.0334 (3)	0.0001 (2)	0.0176 (2)	0.0008 (2)
O11	0.0302 (9)	0.0444 (10)	0.0355 (9)	0.0044 (7)	−0.0010 (7)	0.0000 (7)
O21	0.0549 (11)	0.0243 (8)	0.0385 (9)	−0.0021 (8)	0.0091 (8)	−0.0042 (7)
O12	0.0367 (10)	0.0536 (12)	0.0730 (14)	0.0034 (9)	0.0316 (10)	0.0003 (10)
O22	0.0770 (14)	0.0370 (10)	0.0321 (9)	0.0083 (9)	0.0204 (9)	0.0054 (8)
OL12	0.0283 (8)	0.0402 (9)	0.0399 (9)	−0.0046 (7)	0.0124 (7)	−0.0109 (7)
OL21	0.0327 (8)	0.0327 (9)	0.0305 (8)	−0.0057 (7)	0.0043 (6)	0.0047 (7)
O1	0.0585 (14)	0.103 (2)	0.0415 (12)	0.0086 (13)	0.0101 (10)	−0.0176 (12)
O2	0.0385 (10)	0.0581 (13)	0.0727 (14)	−0.0143 (9)	0.0188 (10)	−0.0170 (11)
O3	0.0519 (12)	0.0640 (15)	0.0547 (13)	−0.0008 (10)	0.0100 (10)	−0.0193 (11)
O4	0.0311 (10)	0.0838 (17)	0.0691 (14)	0.0108 (10)	0.0202 (9)	0.0380 (13)
O5	0.0515 (12)	0.0408 (11)	0.0568 (12)	−0.0044 (9)	0.0108 (9)	0.0113 (9)
O6	0.0525 (13)	0.0374 (11)	0.0906 (17)	0.0029 (9)	0.0120 (12)	0.0165 (11)
N1	0.0257 (10)	0.0425 (12)	0.0422 (11)	−0.0007 (8)	0.0071 (8)	−0.0071 (9)
N2	0.0237 (9)	0.0330 (10)	0.0415 (11)	0.0001 (8)	0.0091 (8)	0.0043 (9)
C1A	0.040 (3)	0.066 (4)	0.052 (3)	0.017 (3)	0.008 (3)	−0.001 (3)
C3A	0.041 (3)	0.068 (4)	0.035 (3)	−0.009 (3)	0.012 (2)	−0.015 (3)
C5A	0.037 (3)	0.061 (4)	0.066 (4)	−0.015 (3)	0.014 (3)	−0.012 (3)
C1B	0.036 (3)	0.072 (5)	0.058 (4)	−0.011 (3)	−0.019 (3)	0.011 (3)
C3B	0.035 (3)	0.041 (3)	0.055 (3)	0.004 (2)	0.014 (2)	−0.010 (3)
C5B	0.037 (3)	0.052 (3)	0.045 (3)	0.001 (2)	0.015 (2)	0.001 (2)
C2	0.0521 (18)	0.078 (2)	0.0516 (17)	0.0115 (17)	−0.0051 (14)	0.0115 (17)
C4	0.0520 (17)	0.0578 (18)	0.0457 (15)	−0.0121 (14)	0.0174 (13)	−0.0161 (14)
C6	0.0604 (19)	0.0489 (17)	0.0554 (18)	−0.0114 (14)	0.0190 (15)	−0.0072 (14)
C7A	0.036 (3)	0.063 (4)	0.054 (3)	0.002 (3)	0.002 (2)	0.029 (3)
C9A	0.045 (3)	0.040 (3)	0.060 (3)	0.004 (2)	0.015 (3)	−0.002 (3)
C11A	0.045 (3)	0.041 (3)	0.062 (4)	−0.018 (2)	0.018 (3)	−0.004 (3)
C7B	0.030 (2)	0.030 (2)	0.054 (3)	0.0073 (19)	0.013 (2)	0.004 (2)
C9B	0.036 (3)	0.046 (3)	0.047 (3)	0.001 (2)	0.022 (2)	0.004 (2)
C11B	0.036 (3)	0.043 (3)	0.038 (3)	0.000 (2)	−0.004 (2)	0.001 (2)
C8	0.0347 (13)	0.0455 (15)	0.0596 (18)	0.0043 (12)	0.0130 (12)	0.0209 (13)
C10	0.0582 (17)	0.0468 (16)	0.0479 (16)	−0.0050 (13)	0.0192 (13)	−0.0085 (13)
C12	0.0502 (16)	0.0406 (15)	0.0510 (16)	−0.0083 (12)	0.0017 (13)	0.0015 (12)

Geometric parameters (Å, °)

P1—O11	1.475 (2)	C3A—C4	1.566 (6)
P1—O21	1.491 (2)	C5A—C6	1.464 (8)
P1—OL21	1.604 (2)	C1B—C2	1.552 (7)
P1—OL12	1.606 (2)	C1B—H9	0.9700
P2—O12	1.479 (2)	C1B—H10	0.9700
P2—O22	1.489 (2)	C3B—C4	1.408 (6)

P2—OL21^{i}	1.604 (2)	C3B—H11	0.9700
P2—OL12	1.605 (2)	C3B—H12	0.9700
OL21—P2^{i}	1.6040 (17)	C5B—C6	1.509 (6)
O1—C2	1.405 (4)	C5B—H13	0.9700
O1—H1	0.8200	C5B—H14	0.9700
O2—C4	1.410 (3)	C2—H15	0.9700
O2—H2	0.8200	C2—H16	0.9700
O3—C6	1.411 (4)	C4—H17	0.9700
O3—H3	0.8200	C4—H18	0.9700
O4—C8	1.408 (3)	C6—H19	0.9700
O4—H4	0.8200	C6—H20	0.9700
O5—C10	1.417 (4)	C7A—C8	1.490 (7)
O5—H5	0.8200	C9A—C10	1.551 (7)
O6—C12	1.412 (4)	C11A—C12	1.584 (7)
O6—H6	0.8200	C7B—C8	1.556 (6)
N1—C1B	1.429 (5)	C7B—H21	0.9700
N1—C3A	1.461 (5)	C7B—H22	0.9700
N1—C5B	1.516 (6)	C9B—C10	1.504 (6)
N1—C3B	1.535 (6)	C9B—H23	0.9700
N1—C5A	1.542 (6)	C9B—H24	0.9700
N1—C1A	1.574 (7)	C11B—C12	1.447 (6)
N1—H7	0.9100	C11B—H25	0.9700
N2—C7B	1.463 (5)	C11B—H26	0.9700
N2—C11A	1.467 (6)	C8—H27	0.9700
N2—C9B	1.505 (5)	C8—H28	0.9700
N2—C11B	1.521 (5)	C10—H29	0.9700
N2—C9A	1.533 (6)	C10—H30	0.9700
N2—C7A	1.541 (6)	C12—H31	0.9700
N2—H8	0.9100	C12—H32	0.9700
C1A—C2	1.401 (7)		
O11—P1—O21	120.6 (2)	C1A—C2—H16	109.9
O11—P1—OL21	112.1 (1)	O1—C2—H16	109.9
O21—P1—OL21	105.2 (1)	C1B—C2—H16	69.6
O11—P1—OL12	111.7 (1)	H15—C2—H16	108.3
O21—P1—OL12	105.3 (1)	C3B—C4—O2	110.7 (3)
OL21—P1—OL12	99.77 (9)	O2—C4—C3A	108.1 (3)
O12—P2—O22	119.7 (2)	C3B—C4—H17	70.7
O12—P2—OL21^{i}	111.3 (2)	O2—C4—H17	110.1
O22—P2—OL21^{i}	105.5 (2)	C3A—C4—H17	110.1
O12—P2—OL12	106.8 (2)	C3B—C4—H18	136.4
O22—P2—OL12	108.8 (1)	O2—C4—H18	110.1
OL21^{i}—P2—OL12	103.60 (9)	C3A—C4—H18	110.1
P2—OL12—P1	133.1 (2)	H17—C4—H18	108.4
P1—OL21—P2^{i}	134.8 (2)	O3—C6—C5A	110.5 (3)
C2—O1—H1	109.5	O3—C6—C5B	109.5 (3)
C4—O2—H2	109.5	O3—C6—H19	109.6
C6—O3—H3	109.5	C5A—C6—H19	109.6

C8—O4—H4	109.5	C5B—C6—H19	61.7
C10—O5—H5	109.5	O3—C6—H20	109.6
C12—O6—H6	109.5	C5A—C6—H20	109.6
C1B—N1—C5B	114.6 (4)	C5B—C6—H20	140.7
C1B—N1—C3B	113.9 (4)	H19—C6—H20	108.1
C5B—N1—C3B	108.9 (3)	C8—C7A—N2	110.4 (4)
C3A—N1—C5A	114.1 (4)	N2—C9A—C10	108.4 (4)
C3A—N1—C1A	111.3 (4)	N2—C11A—C12	109.7 (4)
C5A—N1—C1A	107.5 (4)	N2—C7B—C8	111.0 (3)
C1B—N1—H7	109.6	N2—C7B—H21	109.4
C3A—N1—H7	107.9	C8—C7B—H21	109.4
C5B—N1—H7	106.4	N2—C7B—H22	109.4
C3B—N1—H7	102.6	C8—C7B—H22	109.4
C5A—N1—H7	107.9	H21—C7B—H22	108.0
C1A—N1—H7	107.9	C10—C9B—N2	112.4 (4)
C7B—N2—C9B	114.0 (3)	C10—C9B—H23	109.1
C7B—N2—C11B	112.9 (3)	N2—C9B—H23	109.1
C9B—N2—C11B	109.0 (3)	C10—C9B—H24	109.1
C11A—N2—C9A	113.4 (4)	N2—C9B—H24	109.1
C11A—N2—C7A	112.0 (4)	H23—C9B—H24	107.8
C9A—N2—C7A	109.2 (4)	C12—C11B—N2	114.4 (3)
C7B—N2—H8	110.7	C12—C11B—H25	108.7
C11A—N2—H8	107.3	N2—C11B—H25	108.7
C9B—N2—H8	107.0	C12—C11B—H26	108.7
C11B—N2—H8	102.5	N2—C11B—H26	108.7
C9A—N2—H8	107.3	H25—C11B—H26	107.6
C7A—N2—H8	107.3	O4—C8—C7A	110.0 (3)
C2—C1A—N1	113.9 (4)	O4—C8—C7B	106.0 (3)
N1—C3A—C4	111.2 (4)	O4—C8—H27	109.7
C6—C5A—N1	112.1 (4)	C7A—C8—H27	109.7
N1—C1B—C2	113.6 (4)	C7B—C8—H27	142.4
N1—C1B—H9	108.8	O4—C8—H28	109.7
C2—C1B—H9	108.8	C7A—C8—H28	109.7
N1—C1B—H10	108.8	C7B—C8—H28	69.3
C2—C1B—H10	108.8	H27—C8—H28	108.2
H9—C1B—H10	107.7	O5—C10—C9B	107.9 (3)
C4—C3B—N1	116.1 (4)	O5—C10—C9A	109.7 (3)
C4—C3B—H11	108.3	O5—C10—H29	109.7
N1—C3B—H11	108.3	C9B—C10—H29	142.2
C4—C3B—H12	108.3	C9A—C10—H29	109.7
N1—C3B—H12	108.3	O5—C10—H30	109.7
H11—C3B—H12	107.4	C9B—C10—H30	60.6
C6—C5B—N1	111.0 (4)	C9A—C10—H30	109.7
C6—C5B—H13	109.4	H29—C10—H30	108.2
N1—C5B—H13	109.4	O6—C12—C11B	106.3 (3)
C6—C5B—H14	109.4	O6—C12—C11A	112.3 (3)
N1—C5B—H14	109.4	O6—C12—H31	109.1
H13—C5B—H14	108.0	C11B—C12—H31	143.1
C1A—C2—O1	108.9 (3)	C11A—C12—H31	109.1

O1—C2—C1B	112.7 (3)	O6—C12—H32	109.1
C1A—C2—H15	109.9	C11A—C12—H32	109.1
O1—C2—H15	109.9	H31—C12—H32	107.9
C1B—C2—H15	135.1		
O12—P2—OL12—P1	−161.95 (16)	N1—C5A—C6—O3	−53.1 (5)
O22—P2—OL12—P1	67.58 (19)	N1—C5A—C6—C5B	46.0 (4)
OL21^{i}—P2—OL12—P1	−44.37 (18)	N1—C5B—C6—O3	54.5 (5)
O11—P1—OL12—P2	−26.5 (2)	N1—C5B—C6—C5A	−46.5 (4)
O21—P1—OL12—P2	−159.04 (16)	C7B—N2—C7A—C8	54.3 (4)
OL21—P1—OL12—P2	92.13 (17)	C11A—N2—C7A—C8	−165.6 (4)
O11—P1—OL21—P2^{i}	9.38 (19)	C9B—N2—C7A—C8	120.7 (6)
O21—P1—OL21—P2^{i}	142.11 (16)	C11B—N2—C7A—C8	−145.3 (5)
OL12—P1—OL21—P2^{i}	−108.96 (16)	C9A—N2—C7A—C8	67.9 (5)
C1B—N1—C1A—C2	−58.4 (6)	C7B—N2—C9A—C10	−161.5 (5)
C3A—N1—C1A—C2	159.3 (5)	C11A—N2—C9A—C10	62.0 (5)
C5B—N1—C1A—C2	−119.3 (6)	C9B—N2—C9A—C10	42.4 (4)
C3B—N1—C1A—C2	139.7 (6)	C11B—N2—C9A—C10	105.8 (7)
C5A—N1—C1A—C2	−75.0 (6)	C7A—N2—C9A—C10	−172.2 (4)
C1B—N1—C3A—C4	−116.6 (6)	C7B—N2—C11A—C12	118.9 (5)
C5B—N1—C3A—C4	140.9 (5)	C9B—N2—C11A—C12	−145.4 (5)
C3B—N1—C3A—C4	−48.9 (4)	C11B—N2—C11A—C12	46.3 (4)
C5A—N1—C3A—C4	159.9 (4)	C9A—N2—C11A—C12	−163.0 (4)
C1A—N1—C3A—C4	−78.2 (5)	C7A—N2—C11A—C12	72.8 (5)
C1B—N1—C5A—C6	154.0 (6)	C11A—N2—C7B—C8	−120.4 (5)
C3A—N1—C5A—C6	−70.3 (5)	C9B—N2—C7B—C8	163.7 (3)
C5B—N1—C5A—C6	−47.0 (4)	C11B—N2—C7B—C8	−71.2 (4)
C3B—N1—C5A—C6	−105.8 (7)	C9A—N2—C7B—C8	142.9 (5)
C1A—N1—C5A—C6	165.8 (4)	C7A—N2—C7B—C8	−51.4 (4)
C3A—N1—C1B—C2	111.4 (7)	C7B—N2—C9B—C10	−69.2 (5)
C5B—N1—C1B—C2	−164.4 (5)	C11A—N2—C9B—C10	154.7 (5)
C3B—N1—C1B—C2	69.3 (6)	C11B—N2—C9B—C10	163.7 (4)
C5A—N1—C1B—C2	−146.9 (7)	C9A—N2—C9B—C10	−45.6 (4)
C1A—N1—C1B—C2	50.2 (5)	C7A—N2—C9B—C10	−115.2 (6)
C1B—N1—C3B—C4	−160.8 (5)	C7B—N2—C11B—C12	166.3 (4)
C3A—N1—C3B—C4	60.4 (5)	C11A—N2—C11B—C12	−54.9 (5)
C5B—N1—C3B—C4	70.0 (5)	C9B—N2—C11B—C12	−66.0 (5)
C5A—N1—C3B—C4	113.7 (7)	C9A—N2—C11B—C12	−115.3 (6)
C1A—N1—C3B—C4	−147.8 (5)	C7A—N2—C11B—C12	151.2 (5)
C1B—N1—C5B—C6	66.2 (6)	N2—C7A—C8—O4	42.5 (6)
C3A—N1—C5B—C6	−158.2 (5)	N2—C7A—C8—C7B	−50.5 (4)
C3B—N1—C5B—C6	−165.0 (4)	N2—C7B—C8—O4	−47.6 (5)
C5A—N1—C5B—C6	44.8 (4)	N2—C7B—C8—C7A	54.8 (4)
C1A—N1—C5B—C6	105.6 (6)	N2—C9B—C10—O5	−56.4 (4)
N1—C1A—C2—O1	−52.6 (6)	N2—C9B—C10—C9A	45.2 (4)
N1—C1A—C2—C1B	50.7 (5)	N2—C9A—C10—O5	55.3 (4)
N1—C1B—C2—C1A	−58.3 (5)	N2—C9A—C10—C9B	−42.7 (4)
N1—C1B—C2—O1	35.1 (7)	N2—C11B—C12—O6	−55.2 (5)
N1—C3B—C4—O2	40.0 (6)	N2—C11B—C12—C11A	50.4 (4)

N1—C3B—C4—C3A	−54.3 (5)	N2—C11A—C12—O6	40.9 (5)
N1—C3A—C4—C3B	55.2 (5)	N2—C11A—C12—C11B	−50.5 (4)
N1—C3A—C4—O2	−45.8 (5)		

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O21	0.82	1.90	2.718 (3)	172
O2—H2···O11^{ii}	0.82	1.98	2.771 (3)	164
O3—H3···O22^{i}	0.82	1.85	2.658 (3)	170
O4—H4···O12^{iii}	0.82	1.85	2.669 (3)	177
O5—H5···O21	0.82	1.85	2.659 (3)	170
O6—H6···O22	0.82	1.91	2.722 (3)	174
N1—H7···O2	0.91	2.26	2.743 (3)	113
N2—H8···O4	0.91	2.27	2.703 (3)	109

Symmetry codes: (ii) *x*+1/2, −*y*+1/2, *z*+1/2; (i) −*x*+1, −*y*, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1

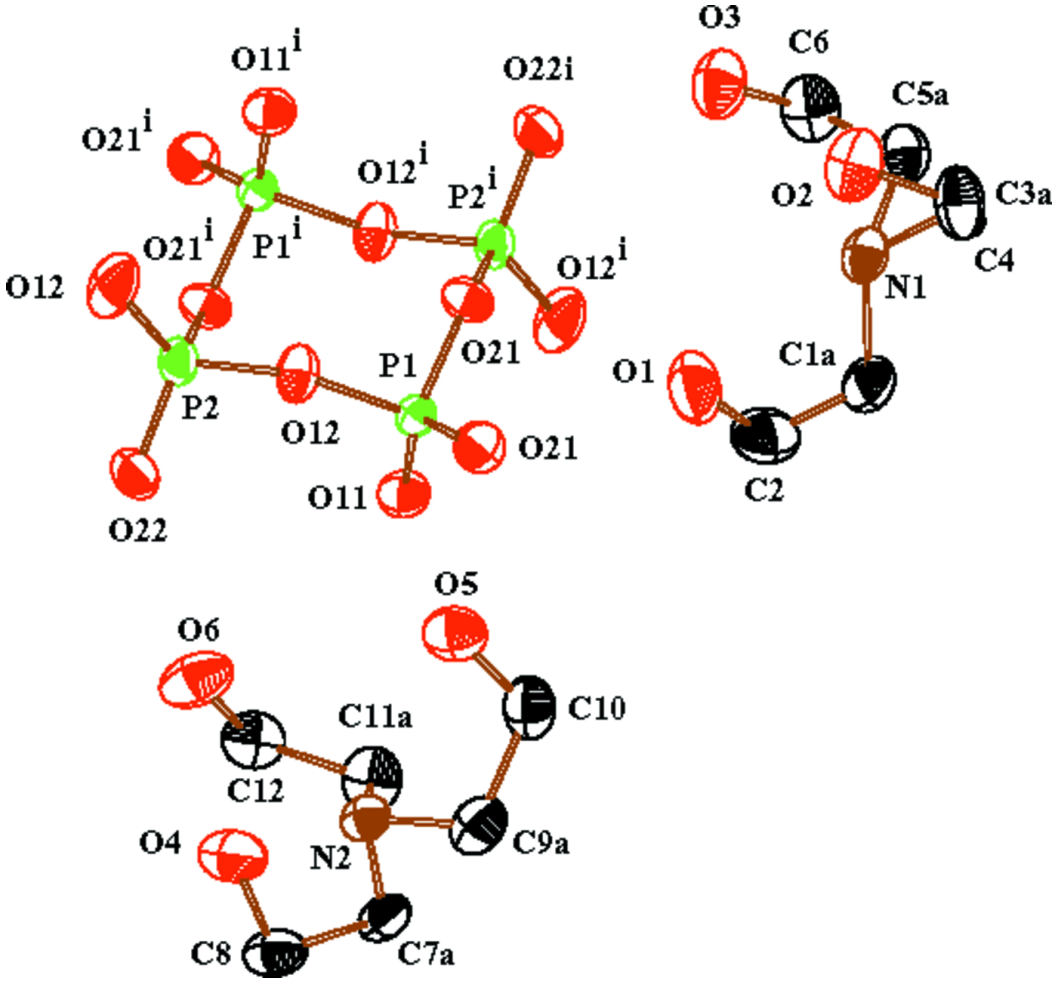

O11i
O21i
O22i
O12i
P2i
O21i
P1i
O12
O12i
P1
O21
P2
O12
O21
O11
O22
O3
C6
C5a
O2
C3a
C4
N1
C1a
O1
C2
O5
O6
C10
C11a
C12
O4
N2
C9a
C8
C7a

Fig. 2