Tetrakis(3,5-xylidinium) dihydrogen cyclohexaphosphate dihydrate

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In the title compound, $4C_8H_{12}N^+ \cdot H_2P_6O_{18}{}^{4-} \cdot 2H_2O$, the complete cyclohexaphosphate anion is generated by inversion symmetry. Crystal cohesion and stability are supported by electrostatic interactions which, together with N—H···O and O—H···O hydrogen bonds, build up a three-dimensional network.

Related literature

For related structures, see: Khederi *et al.* (2001); Rayes *et al.* (2004); Amri *et al.* (2008); Janiak *et al.* (2000). For a discussion on hydrogen bonding, see: Brown (1976). For tetrahedral distortions, see: Baur (1974). For the preparation of cyclohexaphosphoric acid, see: Schülke & Kayser (1985).
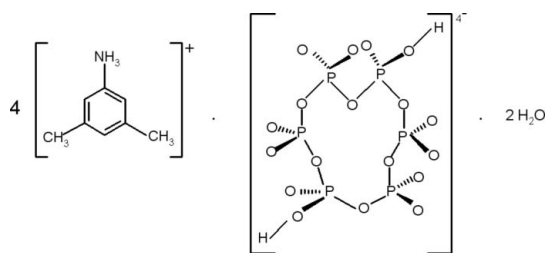
Experimental

Crystal data

$4C_8H_{12}N^+ \cdot H_2P_6O_{18}{}^{4-} \cdot 2H_2O$
$M_r = 1000.61$
Monoclinic, $P2_1/c$
$a = 17.254$ (3) Å
$b = 11.763$ (5) Å
$c = 11.556$ (2) Å
$\beta = 106.41$ (3)°

$V = 2249.9$ (11) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.32$ mm^{-1}
$T = 293$ K
$0.35 \times 0.20 \times 0.01$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
10097 measured reflections
9844 independent reflections

5567 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
2 standard reflections every 120 min
  intensity decay: 11%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.141$
$S = 1.02$
9844 reflections
295 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O6^i	0.82	1.71	2.421 (2)	144
O1W—H2W···O9	0.85 (1)	2.01 (1)	2.831 (2)	164 (2)
O1W—H1W···O5^{ii}	0.85 (1)	2.00 (1)	2.829 (2)	165 (2)
N1—H1A···O9^{iii}	0.89	2.03	2.910 (2)	170
N1—H1B···O1W	0.89	1.89	2.769 (2)	169
N1—H1C···O3	0.89	1.93	2.738 (2)	151
N2—H2A···O3^i	0.89	1.94	2.801 (2)	161
N2—H2B···O2^{iv}	0.89	1.97	2.768 (2)	148
N2—H2C···O5	0.89	1.83	2.719 (2)	175

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (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: HB5267).

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supporting information

Tetrakis(3,5-xylidinium) dihydrogen cyclohexaphosphate dihydrate

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S1. Comment

Following investigations of Schülke and Kayser (Schulke, *et al.*, 1985)on the condensation-cyclization on LiH_2PO_4 into $Li_6P_6O_{18}$, the crystal chemistry of cyclohexaphosphates developed rapidly. In the present investigation we report synthesis and crystal structure of a first cyclohexaphosphate acid, $[3,5-(CH_3)_2C_6H_3NH_3]_4H_2P_6O_{18}.2H_2O$, (I).

The title compound, is built up from $H_2P_6O_{18}^{4-}$ anion, four organic 3,5-xylidiniuium cations and two water molecules (Fig. 1). A half of the anion, two organic cations and a water molecule constitute the asymmetric unit of (I).

The atomic arrangement is a typical organization in layers as shows the figure 2. These corrugated layers are constituted of anions and water molecules that develop in the same way to plans (b,c) in $x = 0$. Charge compensation of these layers is achieved by the incorporation of the protonated 3,5-xylidinium cation in the interlayer spaces establishing H-bonds *via* their NH_3 groups with $H_2P_6O_{18}$ rings and water molecules. Inside such a structure, the phosphoric ring has an -1 internal symmetry. It develops around the inversion centers (0,0,0) and (0,1/2,1/2), so it is built up by only three independent tetrahedra. Among the P—O distances in PO_4 tetrahedra, we can distinguish three different types. The longest ones correspond to the bridging oxygen atom, the intermediate one, corresponds to the P—OH bonding and the shortest, correspond to the external oxygen atoms. The calculated average values of the distortion indices (Baur, 1974) corresponding to the different angles and distances in the PO_4 tetrahedra [DI (OPO) = 0.040; DI (PO) = 0.037; and DI (OO) = 0.016], show a pronounced distortion of the PO distances and OPO angles if compared to OO distances. So, the phosphate group can be considered as a rigid regular arrangement of oxygen atoms, with the phosphorus atom displaced from the gravity centre. It is worth noting that the strong H-bond between phosphoric rings (Table 1)(dO···O = 2.421 (2) Å < 2.73 Å) is never observed in cyclohexaphosphates.

With regards to the organic cation arrangement, these groups are in opposition, by creating thus a local invesion center. Interatomic bond lengths and angles of these groups spread within the respective ranges of 1.371 (3)–1.466 (2) Å and 118.2 (2)–122.1 (2)°. These values are similar to those obtained with the same isomers [Khederi, *et al.*, 2001, Rayes, *et al.*, 2004, Amri, *et al.*, 2008] The aromatic ring of the protonated used amine display an almost coplanar configuration with mean plane deviation of 0.000085 Å and 0.000245 Å. The interplanar distance between the aryl rings of the organic cations is in the vicinity of 4.00 Å, which is significantly longer than 3.80 Å for the $π$-$π$ interaction (Janiak, 2000). The cohesion forces in this compound are assured by electrostatic interactions, van der Waals contacts and hydrogen bonds (O—H···O, N—H···O).

S2. Experimental

The title compound, $[3,5-(CH_3)_2C_6H_3NH_3]_4H_2P_6O_{18}.2H_2O$ was synthesized by reaction of the cyclohexaphosphoric acid on 3,5-xylidine in an aqueous solution. The used acid was produced from a $Li_6P_6O_{18}$ (Schulke *et al.*, 1985) solution by cation exchange on resins (Amberlite IR 120). The obtained $H_6P_6O_{18}$ was added until the a pH between 1 and 2 in the final solution resulted. The same method of preparation was used for the synthesis of $[3,5-(CH_3)_2C_6H_3NH_3]_6P_6O_{18}.6H_2O$, but in

a less acidic medium (Khederi, *et al.*, 2001). Then this solution was slowly evaporated at room temperature for several days until the formation of transparent prisms of (I) were obtained.
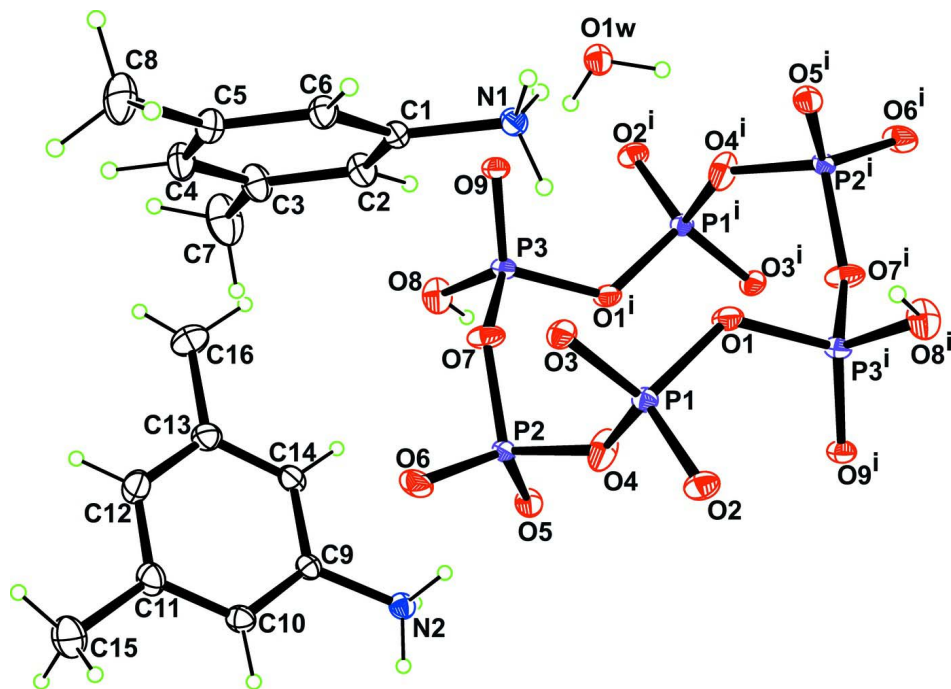

Figure 1

A view of (I) with displacement ellipsoids drawn at the 30% probability level. Symmetry code: (i) - *x*, - *y*, - *z*.
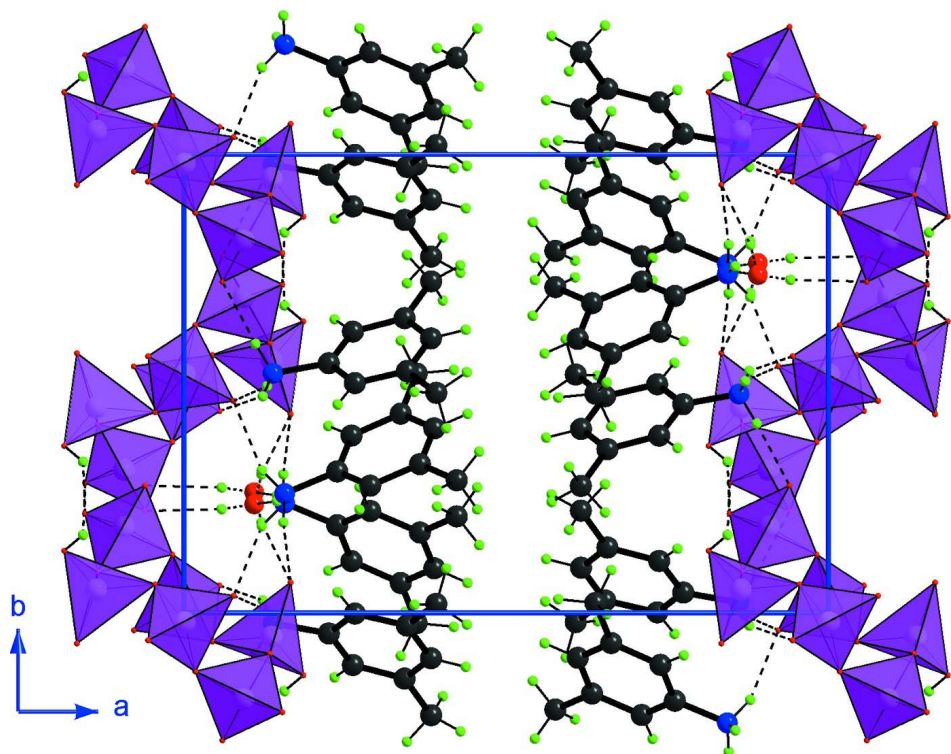
Figure 2

Projection of (I) along the *c* axis.

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Crystal data

$4C_8H_{12}N^+\cdot H_2P_6O_{18}^{4-}\cdot 2H_2O$	$F(000) = 1048$
$M_r = 1000.61$	$D_x = 1.477$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
$a = 17.254$ (3) Å	$\theta = 6.3$–10.1°
$b = 11.763$ (5) Å	$\mu = 0.32$ mm^{-1}
$c = 11.556$ (2) Å	$T = 293$ K
$\beta = 106.41$ (3)°	Prism, colourless
$V = 2249.9$ (11) Å^3	$0.35 \times 0.20 \times 0.01$ mm
$Z = 2$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.039$
Radiation source: Enraf Nonius FR590	$\theta_{max} = 35.0°$, $\theta_{min} = 3.0°$
Graphite monochromator	$h = 0\rightarrow 27$
non–profiled ω scans	$k = -18\rightarrow 0$
10097 measured reflections	$l = -18\rightarrow 17$
9844 independent reflections	2 standard reflections every 120 min
5567 reflections with $I > 2\sigma(I)$	intensity decay: 11%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.141$	$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.0078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	
9844 reflections	$(\Delta/\sigma)_{max} = 0.001$
295 parameters	$\Delta\rho_{max} = 0.50$ e Å^{-3}
3 restraints	$\Delta\rho_{min} = -0.51$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	

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	$U_{iso}*/U_{eq}$
P1	0.00136 (3)	0.52037 (4)	0.73695 (4)	0.02425 (10)

P2	0.09276 (3)	0.68827 (4)	0.63729 (4)	0.02327 (10)
P3	0.13602 (3)	0.55149 (4)	0.45087 (4)	0.02452 (10)
O1	−0.04452 (9)	0.43770 (12)	0.62693 (12)	0.0358 (3)
O2	−0.05542 (9)	0.55967 (13)	0.80125 (13)	0.0383 (3)
O3	0.07729 (8)	0.46237 (12)	0.80005 (12)	0.0331 (3)
O4	0.01792 (9)	0.62742 (15)	0.66154 (17)	0.0516 (5)
O5	0.06005 (9)	0.77774 (12)	0.54796 (13)	0.0355 (3)
O6	0.15290 (10)	0.71634 (14)	0.75342 (13)	0.0463 (4)
O7	0.13336 (10)	0.58897 (13)	0.58091 (12)	0.0411 (4)
O8	0.18549 (10)	0.63572 (15)	0.40517 (15)	0.0470 (4)
H8	0.1563	0.6703	0.3476	0.071*
O9	0.16580 (8)	0.43352 (11)	0.46188 (12)	0.0299 (3)
O1W	0.10923 (9)	0.23683 (13)	0.55367 (13)	0.0350 (3)
H2W	0.1207 (13)	0.3026 (12)	0.534 (2)	0.049 (8)*
H1W	0.0587 (6)	0.226 (2)	0.535 (2)	0.051 (8)*
N1	0.15651 (10)	0.26040 (14)	0.80220 (14)	0.0286 (3)
H1A	0.1545	0.1974	0.8438	0.043*
H1B	0.1444	0.2439	0.7240	0.043*
H1C	0.1211	0.3107	0.8146	0.043*
N2	0.13845 (10)	0.97750 (14)	0.54343 (15)	0.0330 (3)
H2A	0.1291	1.0056	0.4692	0.050*
H2B	0.1225	1.0275	0.5899	0.050*
H2C	0.1111	0.9129	0.5408	0.050*
C1	0.23806 (11)	0.30866 (16)	0.84216 (16)	0.0277 (3)
C2	0.26029 (13)	0.38692 (18)	0.76896 (19)	0.0374 (5)
H2	0.2242	0.4077	0.6958	0.045*
C3	0.33672 (15)	0.4345 (2)	0.8052 (2)	0.0442 (5)
C4	0.38869 (14)	0.4024 (2)	0.9153 (2)	0.0448 (5)
H4	0.4401	0.4342	0.9397	0.054*
C5	0.36648 (13)	0.3244 (2)	0.9902 (2)	0.0389 (5)
C6	0.28937 (12)	0.27755 (18)	0.95214 (18)	0.0338 (4)
H6	0.2726	0.2255	1.0007	0.041*
C7	0.3613 (2)	0.5219 (3)	0.7259 (3)	0.0738 (10)
H7A	0.3367	0.5936	0.7336	0.111*
H7B	0.3438	0.4974	0.6434	0.111*
H7C	0.4190	0.5301	0.7505	0.111*
C8	0.42344 (16)	0.2895 (3)	1.1093 (2)	0.0611 (8)
H8A	0.4552	0.2259	1.0973	0.092*
H8B	0.3930	0.2683	1.1638	0.092*
H8C	0.4584	0.3519	1.1428	0.092*
C9	0.22502 (12)	0.95537 (16)	0.59349 (17)	0.0301 (4)
C10	0.26548 (14)	0.99879 (18)	0.70482 (18)	0.0376 (5)
H10	0.2387	1.0454	0.7460	0.045*
C11	0.34615 (15)	0.9726 (2)	0.7550 (2)	0.0457 (5)
C12	0.38358 (15)	0.9013 (2)	0.6916 (2)	0.0508 (6)
H12	0.4376	0.8823	0.7253	0.061*
C13	0.34264 (15)	0.8571 (2)	0.5790 (2)	0.0454 (5)
C14	0.26241 (14)	0.88654 (19)	0.5298 (2)	0.0391 (5)

H14	0.2340	0.8598	0.4540	0.047*
C15	0.3918 (2)	1.0199 (3)	0.8766 (3)	0.0760 (10)
H15A	0.4073	1.0970	0.8674	0.114*
H15B	0.4392	0.9749	0.9101	0.114*
H15C	0.3579	1.0179	0.9296	0.114*
C16	0.3838 (2)	0.7769 (3)	0.5129 (3)	0.0766 (10)
H16A	0.3464	0.7193	0.4731	0.115*
H16B	0.4293	0.7419	0.5695	0.115*
H16C	0.4021	0.8186	0.4542	0.115*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0263 (2)	0.0223 (2)	0.0234 (2)	0.00168 (17)	0.00594 (16)	0.00207 (16)
P2	0.0260 (2)	0.01997 (19)	0.02237 (19)	−0.00016 (16)	0.00439 (16)	−0.00186 (15)
P3	0.0315 (2)	0.0211 (2)	0.01944 (19)	0.00324 (17)	0.00459 (17)	−0.00015 (15)
O1	0.0347 (7)	0.0350 (7)	0.0313 (7)	0.0121 (6)	−0.0010 (6)	−0.0107 (6)
O2	0.0436 (8)	0.0418 (8)	0.0324 (7)	0.0089 (7)	0.0154 (6)	−0.0037 (6)
O3	0.0305 (7)	0.0324 (7)	0.0315 (7)	0.0082 (6)	0.0007 (5)	0.0033 (5)
O4	0.0324 (8)	0.0516 (10)	0.0708 (11)	0.0029 (7)	0.0147 (8)	0.0358 (9)
O5	0.0356 (7)	0.0252 (6)	0.0419 (8)	0.0001 (6)	0.0045 (6)	0.0094 (6)
O6	0.0525 (10)	0.0407 (9)	0.0335 (8)	0.0069 (7)	−0.0078 (7)	−0.0156 (6)
O7	0.0591 (10)	0.0392 (8)	0.0218 (6)	0.0212 (7)	0.0061 (6)	−0.0037 (6)
O8	0.0470 (9)	0.0448 (9)	0.0454 (9)	−0.0054 (8)	0.0066 (7)	0.0210 (7)
O9	0.0348 (7)	0.0230 (6)	0.0306 (6)	0.0049 (5)	0.0073 (5)	−0.0017 (5)
O1W	0.0338 (8)	0.0331 (8)	0.0359 (7)	−0.0025 (6)	0.0064 (6)	0.0061 (6)
N1	0.0291 (8)	0.0261 (7)	0.0294 (7)	−0.0014 (6)	0.0061 (6)	−0.0001 (6)
N2	0.0352 (9)	0.0279 (8)	0.0350 (8)	−0.0015 (7)	0.0085 (7)	0.0026 (7)
C1	0.0264 (8)	0.0258 (8)	0.0294 (8)	−0.0014 (7)	0.0055 (7)	−0.0015 (7)
C2	0.0392 (11)	0.0373 (11)	0.0323 (10)	−0.0051 (9)	0.0044 (8)	0.0045 (8)
C3	0.0442 (12)	0.0455 (13)	0.0428 (12)	−0.0135 (10)	0.0122 (10)	0.0040 (10)
C4	0.0312 (11)	0.0496 (13)	0.0508 (13)	−0.0097 (10)	0.0067 (10)	−0.0007 (11)
C5	0.0313 (10)	0.0422 (12)	0.0380 (11)	0.0008 (9)	0.0015 (8)	−0.0005 (9)
C6	0.0322 (10)	0.0350 (10)	0.0324 (9)	−0.0008 (8)	0.0059 (8)	0.0036 (8)
C7	0.071 (2)	0.080 (2)	0.0672 (19)	−0.0334 (17)	0.0143 (16)	0.0215 (16)
C8	0.0433 (14)	0.078 (2)	0.0486 (14)	−0.0039 (13)	−0.0086 (11)	0.0127 (13)
C9	0.0334 (9)	0.0254 (8)	0.0316 (9)	−0.0034 (7)	0.0091 (7)	0.0019 (7)
C10	0.0438 (12)	0.0372 (11)	0.0306 (9)	0.0016 (9)	0.0087 (9)	−0.0024 (8)
C11	0.0446 (12)	0.0513 (14)	0.0341 (11)	−0.0006 (11)	−0.0003 (9)	−0.0010 (10)
C12	0.0362 (12)	0.0537 (15)	0.0579 (15)	0.0039 (11)	0.0057 (11)	0.0023 (12)
C13	0.0438 (12)	0.0412 (12)	0.0558 (14)	−0.0026 (10)	0.0217 (11)	−0.0076 (11)
C14	0.0423 (12)	0.0380 (11)	0.0377 (10)	−0.0082 (9)	0.0124 (9)	−0.0095 (9)
C15	0.070 (2)	0.090 (2)	0.0484 (15)	0.0066 (18)	−0.0148 (14)	−0.0129 (16)
C16	0.0589 (18)	0.082 (2)	0.098 (3)	0.0113 (17)	0.0369 (18)	−0.028 (2)

Geometric parameters (Å, º)

P1—O2	1.4619 (15)	C3—C7	1.515 (3)
P1—O3	1.4744 (14)	C4—C5	1.388 (3)
P1—O4	1.6024 (16)	C4—H4	0.9300
P1—O1	1.6187 (15)	C5—C6	1.392 (3)
P2—O5	1.4706 (15)	C5—C8	1.505 (3)
P2—O6	1.4832 (15)	C6—H6	0.9300
P2—O4	1.5692 (16)	C7—H7A	0.9600
P2—O7	1.5930 (15)	C7—H7B	0.9600
P3—O9	1.4728 (15)	C7—H7C	0.9600
P3—O8	1.4980 (16)	C8—H8A	0.9600
P3—O7	1.5790 (14)	C8—H8B	0.9600
P3—O1[i]	1.5859 (15)	C8—H8C	0.9600
O1—P3[i]	1.5859 (15)	C9—C14	1.371 (3)
O8—H8	0.8200	C9—C10	1.377 (3)
O1W—H2W	0.847 (9)	C10—C11	1.383 (3)
O1W—H1W	0.846 (9)	C10—H10	0.9300
N1—C1	1.466 (2)	C11—C12	1.387 (4)
N1—H1A	0.8900	C11—C15	1.510 (3)
N1—H1B	0.8900	C12—C13	1.393 (3)
N1—H1C	0.8900	C12—H12	0.9300
N2—C9	1.465 (3)	C13—C14	1.384 (3)
N2—H2A	0.8900	C13—C16	1.512 (4)
N2—H2B	0.8900	C14—H14	0.9300
N2—H2C	0.8900	C15—H15A	0.9600
C1—C2	1.376 (3)	C15—H15B	0.9600
C1—C6	1.377 (3)	C15—H15C	0.9600
C2—C3	1.384 (3)	C16—H16A	0.9600
C2—H2	0.9300	C16—H16B	0.9600
C3—C4	1.386 (3)	C16—H16C	0.9600
O2—P1—O3	121.63 (9)	C4—C5—C8	121.8 (2)
O2—P1—O4	106.02 (10)	C6—C5—C8	120.0 (2)
O3—P1—O4	111.25 (9)	C1—C6—C5	119.6 (2)
O2—P1—O1	109.87 (9)	C1—C6—H6	120.2
O3—P1—O1	106.24 (8)	C5—C6—H6	120.2
O4—P1—O1	99.66 (10)	C3—C7—H7A	109.5
O5—P2—O6	120.50 (10)	C3—C7—H7B	109.5
O5—P2—O4	106.25 (9)	H7A—C7—H7B	109.5
O6—P2—O4	109.92 (11)	C3—C7—H7C	109.5
O5—P2—O7	111.32 (9)	H7A—C7—H7C	109.5
O6—P2—O7	104.90 (9)	H7B—C7—H7C	109.5
O4—P2—O7	102.57 (10)	C5—C8—H8A	109.5
O9—P3—O8	115.72 (10)	C5—C8—H8B	109.5
O9—P3—O7	106.50 (8)	H8A—C8—H8B	109.5
O8—P3—O7	108.87 (10)	C5—C8—H8C	109.5
O9—P3—O1[i]	113.06 (8)	H8A—C8—H8C	109.5

O8—P3—O1ⁱ	108.80 (9)	H8B—C8—H8C	109.5
O7—P3—O1ⁱ	103.02 (9)	C14—C9—C10	121.9 (2)
P3ⁱ—O1—P1	125.79 (9)	C14—C9—N2	118.34 (18)
P2—O4—P1	137.54 (11)	C10—C9—N2	119.63 (18)
P3—O7—P2	136.74 (10)	C9—C10—C11	119.6 (2)
P3—O8—H8	109.5	C9—C10—H10	120.2
H2W—O1W—H1W	111.4 (19)	C11—C10—H10	120.2
C1—N1—H1A	109.5	C10—C11—C12	118.5 (2)
C1—N1—H1B	109.5	C10—C11—C15	120.4 (2)
H1A—N1—H1B	109.5	C12—C11—C15	121.1 (2)
C1—N1—H1C	109.5	C11—C12—C13	122.0 (2)
H1A—N1—H1C	109.5	C11—C12—H12	119.0
H1B—N1—H1C	109.5	C13—C12—H12	119.0
C9—N2—H2A	109.5	C14—C13—C12	118.4 (2)
C9—N2—H2B	109.5	C14—C13—C16	120.5 (2)
H2A—N2—H2B	109.5	C12—C13—C16	121.2 (2)
C9—N2—H2C	109.5	C9—C14—C13	119.6 (2)
H2A—N2—H2C	109.5	C9—C14—H14	120.2
H2B—N2—H2C	109.5	C13—C14—H14	120.2
C2—C1—C6	121.79 (18)	C11—C15—H15A	109.5
C2—C1—N1	118.35 (17)	C11—C15—H15B	109.5
C6—C1—N1	119.83 (17)	H15A—C15—H15B	109.5
C1—C2—C3	119.47 (19)	C11—C15—H15C	109.5
C1—C2—H2	120.3	H15A—C15—H15C	109.5
C3—C2—H2	120.3	H15B—C15—H15C	109.5
C2—C3—C4	118.8 (2)	C13—C16—H16A	109.5
C2—C3—C7	119.8 (2)	C13—C16—H16B	109.5
C4—C3—C7	121.4 (2)	H16A—C16—H16B	109.5
C3—C4—C5	122.1 (2)	C13—C16—H16C	109.5
C3—C4—H4	118.9	H16A—C16—H16C	109.5
C5—C4—H4	118.9	H16B—C16—H16C	109.5
C4—C5—C6	118.2 (2)		

Symmetry code: (i) −x, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O6ⁱⁱ	0.82	1.71	2.421 (2)	144
O1W—H2W···O9	0.85 (1)	2.01 (1)	2.831 (2)	164 (2)
O1W—H1W···O5ⁱ	0.85 (1)	2.00 (1)	2.829 (2)	165 (2)
N1—H1A···O9ⁱⁱⁱ	0.89	2.03	2.910 (2)	170
N1—H1B···O1W	0.89	1.89	2.769 (2)	169
N1—H1C···O3	0.89	1.93	2.738 (2)	151
N2—H2A···O3ⁱⁱ	0.89	1.94	2.801 (2)	161
N2—H2B···O2ⁱᵛ	0.89	1.97	2.768 (2)	148
N2—H2C···O5	0.89	1.83	2.719 (2)	175

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