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# Bis(4-chlorobenzylammonium) tetrakis(2,6-diethylanilinium) cyclohexaphosphate tetrahydrate

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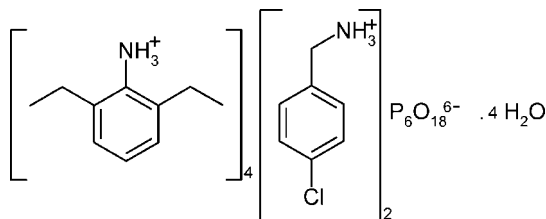
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.053;  $wR$  factor = 0.144; data-to-parameter ratio = 20.3.

In the crystal of the title hydrated molecular salt,  $2\text{C}_7\text{H}_9\text{ClN}^+ \cdot 4\text{C}_{10}\text{H}_{16}\text{N}^+ \cdot \text{P}_6\text{O}_{18}^{6-} \cdot 4\text{H}_2\text{O}$ , the packing consists of a three-dimensional  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{N}-\text{H} \cdots \text{O}$  hydrogen-bonded network resulting from the association of anionic layers built up from centrosymmetric cyclohexaphosphate ions and water molecules and the two types of organic cations.

## Related literature

For related structures, see: Amri *et al.* (2007, 2008); Marouani & Rzaigui (2002). For background, see: Kresge *et al.* (1992); Katsoulis (1998).



## Experimental

## Crystal data

 $2\text{C}_7\text{H}_9\text{ClN}^+ \cdot 4\text{C}_{10}\text{H}_{16}\text{N}^+ \cdot \text{P}_6\text{O}_{18}^{6-} \cdot 4\text{H}_2\text{O}$  $M_r = 1432.04$ Monoclinic,  $C2/c$  $a = 31.437$  (2) Å $b = 14.178$  (2) Å $c = 16.034$  (2) Å $\beta = 99.60$  (2)° $V = 7046.5$  (14) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.30$  mm<sup>-1</sup> $T = 293$  K

0.20 × 0.18 × 0.16 mm

## Data collection

Enraf–Nonius TurboCAD-4 diffractometer  
Absorption correction: none  
11637 measured reflections  
8452 independent reflections

5321 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$   
2 standard reflections  
frequency: 120 min  
intensity decay: 5%

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$   
 $wR(F^2) = 0.144$   
 $S = 1.02$   
8452 reflections

417 parameters  
H-atom parameters not refined  
 $\Delta\rho_{\text{max}} = 0.57$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.44$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1A} \cdots \text{O9}^{\text{i}}$	0.89	1.80	2.685 (3)	171
$\text{N1}-\text{H1B} \cdots \text{O2}$	0.89	2.03	2.758 (3)	138
$\text{N1}-\text{H1C} \cdots \text{O4}$	0.89	2.13	2.935 (3)	151
$\text{N2}-\text{H2A} \cdots \text{O4}^{\text{ii}}$	0.89	1.94	2.827 (3)	172
$\text{N2}-\text{H2B} \cdots \text{O1}$	0.89	2.05	2.887 (3)	156
$\text{N2}-\text{H2C} \cdots \text{O7}$	0.89	1.94	2.809 (3)	166
$\text{N3}-\text{H3A} \cdots \text{O5}^{\text{iii}}$	0.89	1.89	2.762 (3)	165
$\text{N3}-\text{H3B} \cdots \text{O9}$	0.89	1.93	2.799 (3)	164
$\text{N3}-\text{H3C} \cdots \text{O10}$	0.89	1.99	2.800 (5)	151
$\text{O10}-\text{H1} \cdots \text{O11}$	0.85	2.22	2.923 (6)	140
$\text{O10}-\text{H2} \cdots \text{O11}^{\text{iv}}$	0.86	2.25	2.831 (5)	125
$\text{O11}-\text{H6} \cdots \text{O2}^{\text{v}}$	0.85	2.23	2.740 (4)	118
$\text{O11}-\text{H7} \cdots \text{O5}^{\text{iii}}$	0.85	2.17	2.781 (4)	129

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

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: *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: HB2918).

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**supplementary materials**

*Acta Cryst.* (2009). E65, o654 [ doi:10.1107/S1600536809006655 ]

## Bis(4-chlorobenzylammonium) tetrakis(2,6-diethylanilinium) cyclohexaphosphate tetrahydrate

O. Amri, S. Abid and M. Rzaigui

### Comment

The synthesis and characterization of organic–inorganic solid state hybrid materials has attracted great attention due to their structural diversity (Kresge *et al.*, 1992) and widely promising potential areas in chemistry, biology and material science (Katsoulis, 1998). As part of our studies in this area, we report here synthesis and crystal structure of the the title compound, (I), (Fig. 1).

The centrosymmetric cyclohexaphosphate anion and the two water molecules are linked together by O—H···O hydrogen bonds to form inorganic layers parallel to the (b,c) plane. On both sides of each inorganic layer, are grafted the organic cations compensating their negatives charges (Fig. 2). Inside this arrangement, the geometry of the phosphoric rings is comparable to those found in [2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>3</sub>]<sub>4</sub>[2,6-(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>3</sub>]<sub>2</sub>P<sub>6</sub>O<sub>18</sub>·4H<sub>2</sub>O (Amri *et al.* 2007) and (1,6NH<sub>3</sub>C<sub>6</sub>H<sub>12</sub>NH<sub>3</sub>)(C<sub>6</sub>H<sub>5</sub>NH<sub>3</sub>)<sub>4</sub>P<sub>6</sub>O<sub>18</sub>·6H<sub>2</sub>O (Marouani *et al.* 2002). In the title compound, the phosphoric rings have the same internal symmetry and thus built up by only three independent PO<sub>4</sub>tetrahedra P1O<sub>4</sub>, P2O<sub>4</sub> and P3O<sub>4</sub>. In the PO<sub>4</sub> tetrahedra, the P—O distances range in [1.470 (3) - 1.610 (2) Å] and the O—P—O bond angles in [99.4 (2) - 121.2 (2)°]. It is the same for the P—P distances ranging from 2.901 (1) and 2.937 (1) Å which are comparable to values generally measured. For the organic cations, the main features measured are similar to distances and angles usually reported for such molecules (Amri *et al.* 2007, Amri *et al.* 2008). The phenyl rings of these groups are almost planar, with mean deviations of ± 0.004 and ± 0.011 Å for 2,6- diethylphenylammonium and ± 0.003 Å for 4-chlorobenzylanilinium.

### Experimental

Cyclohexaphosphoric acid (4.8 mmol) was slowly added to an ethanolic solution of 2,6-diethylaniline (3.16 ml; 19.2 mmol) and 4-chlorobenzylaniline (1.16 ml, 9.6 mmol) in a molar ratio of 2:1 respectively. The obtained solution was slowly evaporated at room temperature. After some days, colourless prisms of (I) were formed. The cyclohexaphosphoric acid is freshly prepared by passing a solution of Li<sub>6</sub>P<sub>6</sub>O<sub>18</sub>(3 g, 4.8 mmol) through an ion exchange resin in its H-state (Amberlite IR 120).

### Refinement

The water H atoms were located in a difference map and freely refined. The other H atoms were positioned geometrically (N—H = 0.89, C—H = 0.93–0.96 Å) and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ .

## Figures

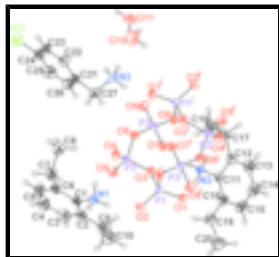


Fig. 1. View of (I) with displacement ellipsoids for the non-H atoms drawn at the 30% probability level (symmetry code: (i)  $1/2 - x, 1/2 - y, 1 - z$ ).

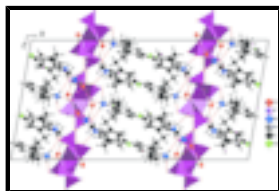
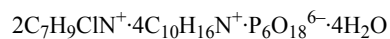


Fig. 2. Projection of (I) along  $b$  axis.

## Bis(4-chlorobenzylammonium) tetrakis(2,6-diethylanilinium) cyclohexaphosphate tetrahydrate

### Crystal data



$M_r = 1432.04$

Monoclinic,  $C2/c$

Hall symbol:  $-C 2yc$

$a = 31.437 (2) \text{ \AA}$

$b = 14.178 (2) \text{ \AA}$

$c = 16.034 (2) \text{ \AA}$

$\beta = 99.60 (2)^\circ$

$V = 7046.5 (14) \text{ \AA}^3$

$Z = 4$

$F_{000} = 3024$

$D_x = 1.350 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 9\text{--}11^\circ$

$\mu = 0.30 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, colourless

$0.20 \times 0.18 \times 0.16 \text{ mm}$

### Data collection

Enraf–Nonius TurboCAD-4 diffractometer

Monochromator: graphite

non-profiled  $\omega$  scans

Absorption correction: none

11637 measured reflections

8452 independent reflections

5321 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\text{max}} = 28.0^\circ$

$\theta_{\text{min}} = 2.1^\circ$

$h = -41 \rightarrow 41$

$k = 0 \rightarrow 18$

$l = -5 \rightarrow 21$

2 standard reflections

every 120 min

intensity decay: 5%

### Refinement

Refinement on  $F^2$

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.053$$

$$wR(F^2) = 0.144$$

$$S = 1.02$$

8452 reflections

417 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters not refined

$$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 2.2936P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

### 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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.01382 (3)	0.69163 (8)	0.12825 (8)	0.0880 (3)
P1	0.24807 (2)	0.07584 (5)	0.44736 (4)	0.03164 (16)
P2	0.23420 (2)	0.23566 (5)	0.33302 (4)	0.03092 (15)
P3	0.293786 (19)	0.38486 (4)	0.40635 (4)	0.03042 (15)
O1	0.28646 (5)	0.04571 (12)	0.50699 (11)	0.0400 (4)
O2	0.22756 (7)	0.01330 (14)	0.37887 (13)	0.0541 (5)
O3	0.26078 (5)	0.17399 (13)	0.40739 (12)	0.0414 (4)
O4	0.18744 (5)	0.22936 (14)	0.33497 (12)	0.0428 (4)
O5	0.24898 (7)	0.21336 (15)	0.25302 (12)	0.0524 (5)
O6	0.24927 (6)	0.33887 (13)	0.36243 (14)	0.0485 (5)
O7	0.33084 (5)	0.32302 (13)	0.39994 (12)	0.0423 (4)
O8	0.28898 (6)	0.38724 (15)	0.50341 (11)	0.0462 (5)
O9	0.29240 (6)	0.48264 (13)	0.37325 (13)	0.0454 (5)
O10	0.19365 (13)	0.6949 (3)	0.4542 (2)	0.1059 (10)
H1	0.2147	0.7334	0.4620	0.21 (4)*
H2	0.1988	0.6545	0.4948	0.15 (3)*
O11	0.25125 (15)	0.8271 (2)	0.38834 (19)	0.1238 (15)
H6	0.2411	0.8642	0.3478	0.37 (7)*
H7	0.2659	0.7851	0.3679	0.58 (12)*
N1	0.16392 (7)	0.05296 (16)	0.24302 (14)	0.0422 (5)
H1A	0.1792	0.0362	0.2034	0.063*
H1B	0.1734	0.0214	0.2905	0.063*

## supplementary materials

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H1C	0.1669	0.1146	0.2527	0.063*
N2	0.35074 (6)	0.19299 (15)	0.53288 (14)	0.0363 (5)
H2A	0.3395	0.2228	0.5729	0.054*
H2B	0.3377	0.1376	0.5221	0.054*
H2C	0.3470	0.2278	0.4860	0.054*
N3	0.21272 (7)	0.57295 (18)	0.32809 (16)	0.0500 (6)
H3A	0.2205	0.6199	0.2970	0.075*
H3B	0.2360	0.5407	0.3512	0.075*
H3C	0.1998	0.5965	0.3688	0.075*
C1	0.11824 (8)	0.0310 (2)	0.21416 (17)	0.0415 (6)
C2	0.10027 (9)	-0.0469 (2)	0.24718 (19)	0.0507 (7)
C3	0.05691 (11)	-0.0647 (3)	0.2167 (2)	0.0688 (10)
H3	0.0436	-0.1163	0.2373	0.083*
C4	0.03350 (11)	-0.0076 (3)	0.1571 (3)	0.0799 (12)
H4	0.0045	-0.0206	0.1382	0.096*
C5	0.05230 (11)	0.0683 (3)	0.1251 (2)	0.0753 (11)
H5	0.0360	0.1059	0.0843	0.090*
C6	0.09543 (10)	0.0899 (2)	0.15295 (19)	0.0530 (8)
C7	0.11533 (14)	0.1759 (3)	0.1192 (3)	0.0777 (11)
H7A	0.1459	0.1645	0.1207	0.093*
H7B	0.1023	0.1853	0.0606	0.093*
C8	0.10995 (18)	0.2644 (3)	0.1678 (3)	0.1045 (16)
H8A	0.0799	0.2806	0.1609	0.157*
H8B	0.1256	0.3149	0.1470	0.157*
H8C	0.1209	0.2543	0.2267	0.157*
C9	0.12659 (12)	-0.1088 (3)	0.3131 (2)	0.0683 (10)
H9A	0.1434	-0.0686	0.3551	0.082*
H9B	0.1467	-0.1448	0.2860	0.082*
C10	0.10082 (16)	-0.1772 (3)	0.3583 (3)	0.0936 (13)
H10A	0.0811	-0.1425	0.3864	0.140*
H10B	0.1202	-0.2128	0.3991	0.140*
H10C	0.0849	-0.2194	0.3177	0.140*
C11	0.39714 (8)	0.17773 (19)	0.56208 (17)	0.0378 (6)
C12	0.42153 (9)	0.2542 (2)	0.59790 (19)	0.0459 (7)
C13	0.46474 (11)	0.2379 (3)	0.6290 (2)	0.0671 (10)
H13	0.4818	0.2869	0.6545	0.081*
C14	0.48285 (10)	0.1505 (3)	0.6226 (3)	0.0786 (12)
H14	0.5119	0.1412	0.6438	0.094*
C15	0.45849 (10)	0.0770 (3)	0.5855 (2)	0.0669 (10)
H15	0.4713	0.0186	0.5808	0.080*
C16	0.41455 (9)	0.0887 (2)	0.5545 (2)	0.0461 (7)
C17	0.40416 (10)	0.3535 (2)	0.6000 (2)	0.0547 (8)
H17A	0.3729	0.3512	0.5935	0.066*
H17B	0.4150	0.3817	0.6545	0.066*
C18	0.41691 (12)	0.4142 (2)	0.5311 (2)	0.0675 (10)
H18A	0.4478	0.4147	0.5360	0.101*
H18B	0.4067	0.4773	0.5364	0.101*
H18C	0.4044	0.3891	0.4769	0.101*
C19	0.38797 (10)	0.0071 (2)	0.5151 (3)	0.0635 (9)

H19A	0.3622	0.0033	0.5407	0.076*
H19B	0.3788	0.0210	0.4555	0.076*
C20	0.40905 (15)	-0.0872 (3)	0.5220 (3)	0.0992 (15)
H20A	0.4322	-0.0877	0.4897	0.149*
H20B	0.3882	-0.1346	0.5006	0.149*
H20C	0.4203	-0.1002	0.5803	0.149*
C21	0.14034 (9)	0.5570 (2)	0.23882 (18)	0.0453 (7)
C22	0.13925 (9)	0.6432 (2)	0.19784 (18)	0.0470 (7)
H22	0.1651	0.6734	0.1935	0.056*
C23	0.10094 (9)	0.6850 (2)	0.16347 (19)	0.0488 (7)
H23	0.1007	0.7427	0.1358	0.059*
C24	0.06256 (9)	0.6395 (2)	0.1708 (2)	0.0535 (8)
C25	0.06304 (11)	0.5540 (3)	0.2110 (2)	0.0649 (9)
H25	0.0372	0.5238	0.2154	0.078*
C26	0.10184 (11)	0.5128 (2)	0.2447 (2)	0.0579 (8)
H26	0.1021	0.4548	0.2718	0.070*
C27	0.18274 (10)	0.5098 (2)	0.2740 (2)	0.0574 (8)
H27A	0.1771	0.4550	0.3067	0.069*
H27B	0.1963	0.4881	0.2274	0.069*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0443 (4)	0.0973 (8)	0.1139 (9)	0.0033 (5)	-0.0115 (5)	-0.0025 (7)
P1	0.0321 (3)	0.0326 (3)	0.0294 (3)	-0.0001 (3)	0.0026 (3)	0.0001 (3)
P2	0.0332 (3)	0.0313 (3)	0.0282 (3)	-0.0024 (3)	0.0053 (2)	0.0001 (3)
P3	0.0303 (3)	0.0308 (3)	0.0303 (3)	-0.0005 (2)	0.0055 (2)	0.0023 (3)
O1	0.0369 (9)	0.0413 (10)	0.0400 (10)	0.0045 (8)	0.0013 (8)	0.0042 (8)
O2	0.0625 (13)	0.0426 (11)	0.0508 (12)	0.0012 (10)	-0.0090 (10)	-0.0127 (10)
O3	0.0355 (9)	0.0458 (10)	0.0419 (10)	-0.0050 (8)	0.0034 (8)	0.0130 (9)
O4	0.0337 (9)	0.0540 (11)	0.0390 (10)	-0.0053 (8)	0.0009 (8)	0.0041 (9)
O5	0.0746 (13)	0.0520 (12)	0.0342 (10)	0.0114 (10)	0.0196 (10)	-0.0005 (9)
O6	0.0363 (9)	0.0353 (10)	0.0686 (14)	0.0020 (8)	-0.0063 (9)	-0.0110 (10)
O7	0.0352 (9)	0.0416 (10)	0.0509 (11)	0.0047 (8)	0.0091 (8)	-0.0015 (9)
O8	0.0359 (9)	0.0754 (14)	0.0281 (9)	0.0093 (9)	0.0074 (8)	0.0112 (9)
O9	0.0457 (10)	0.0401 (10)	0.0529 (12)	0.0012 (8)	0.0149 (9)	0.0137 (9)
O10	0.110 (3)	0.124 (3)	0.077 (2)	0.029 (2)	-0.0021 (17)	-0.005 (2)
O11	0.245 (5)	0.0640 (17)	0.0534 (16)	0.041 (2)	-0.001 (2)	-0.0106 (15)
N1	0.0381 (11)	0.0459 (13)	0.0421 (13)	0.0004 (10)	0.0054 (10)	-0.0121 (11)
N2	0.0335 (10)	0.0355 (11)	0.0391 (12)	0.0028 (9)	0.0036 (9)	-0.0012 (10)
N3	0.0403 (12)	0.0528 (15)	0.0560 (15)	0.0045 (11)	0.0052 (11)	0.0182 (13)
C1	0.0349 (13)	0.0500 (16)	0.0398 (15)	0.0021 (12)	0.0070 (11)	-0.0160 (13)
C2	0.0473 (16)	0.0577 (18)	0.0482 (17)	-0.0016 (14)	0.0107 (14)	-0.0150 (15)
C3	0.0480 (18)	0.079 (2)	0.082 (3)	-0.0122 (18)	0.0180 (18)	-0.010 (2)
C4	0.0417 (18)	0.110 (3)	0.084 (3)	-0.006 (2)	-0.0015 (19)	-0.011 (3)
C5	0.0507 (19)	0.104 (3)	0.066 (2)	0.018 (2)	-0.0057 (17)	-0.006 (2)
C6	0.0488 (16)	0.064 (2)	0.0454 (17)	0.0071 (15)	0.0059 (14)	-0.0089 (16)
C7	0.088 (3)	0.077 (3)	0.063 (2)	0.001 (2)	-0.002 (2)	0.014 (2)

## supplementary materials

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C8	0.128 (4)	0.077 (3)	0.096 (3)	0.013 (3)	-0.019 (3)	0.003 (3)
C9	0.068 (2)	0.062 (2)	0.074 (2)	-0.0057 (18)	0.0119 (19)	0.0038 (19)
C10	0.121 (4)	0.079 (3)	0.084 (3)	-0.007 (3)	0.025 (3)	0.007 (2)
C11	0.0330 (12)	0.0441 (15)	0.0357 (14)	0.0007 (11)	0.0037 (11)	0.0040 (12)
C12	0.0408 (14)	0.0540 (18)	0.0434 (16)	-0.0078 (13)	0.0081 (12)	-0.0010 (14)
C13	0.0469 (18)	0.073 (2)	0.076 (2)	-0.0179 (17)	-0.0053 (16)	0.001 (2)
C14	0.0331 (16)	0.083 (3)	0.112 (3)	-0.0008 (17)	-0.0095 (18)	0.017 (2)
C15	0.0386 (16)	0.061 (2)	0.099 (3)	0.0131 (15)	0.0040 (17)	0.012 (2)
C16	0.0367 (13)	0.0446 (16)	0.0562 (18)	0.0054 (12)	0.0053 (13)	0.0064 (14)
C17	0.0558 (18)	0.0472 (17)	0.063 (2)	-0.0112 (14)	0.0162 (16)	-0.0176 (16)
C18	0.069 (2)	0.055 (2)	0.078 (2)	-0.0131 (17)	0.0102 (19)	-0.0027 (19)
C19	0.0516 (18)	0.0449 (18)	0.091 (3)	0.0065 (14)	0.0014 (18)	-0.0006 (18)
C20	0.096 (3)	0.062 (2)	0.134 (4)	0.014 (2)	0.000 (3)	-0.017 (3)
C21	0.0502 (16)	0.0464 (16)	0.0386 (15)	-0.0045 (13)	0.0054 (13)	0.0021 (13)
C22	0.0412 (14)	0.0513 (17)	0.0488 (17)	-0.0047 (13)	0.0086 (13)	0.0067 (14)
C23	0.0460 (15)	0.0502 (17)	0.0491 (17)	-0.0025 (13)	0.0048 (13)	0.0070 (14)
C24	0.0416 (15)	0.062 (2)	0.0537 (19)	-0.0031 (14)	-0.0010 (14)	-0.0070 (16)
C25	0.0508 (18)	0.068 (2)	0.075 (2)	-0.0224 (17)	0.0086 (17)	-0.0006 (19)
C26	0.064 (2)	0.0505 (18)	0.059 (2)	-0.0121 (16)	0.0089 (16)	0.0057 (16)
C27	0.0620 (19)	0.0477 (18)	0.061 (2)	0.0079 (15)	0.0047 (16)	0.0074 (16)

### *Geometric parameters (Å, °)*

O7—P3	1.4751 (18)	C1—C6	1.393 (4)
O6—P2	1.5854 (19)	C2—C3	1.392 (4)
O6—P3	1.5970 (19)	C2—C9	1.510 (5)
O1—P1	1.4724 (18)	C6—C5	1.389 (5)
O3—P2	1.5975 (19)	C6—C7	1.512 (5)
O3—P1	1.6101 (19)	C5—C4	1.368 (6)
O4—P2	1.4783 (18)	C5—H5	0.9300
O8—P3	1.5889 (18)	C3—C4	1.369 (5)
O8—P1 <sup>i</sup>	1.6003 (19)	C3—H3	0.9300
O9—P3	1.4825 (19)	C4—H4	0.9300
O5—P2	1.470 (2)	C9—C10	1.522 (5)
O2—P1	1.474 (2)	C9—H9A	0.9700
P1—O8 <sup>i</sup>	1.6003 (19)	C9—H9B	0.9700
N2—C11	1.472 (3)	C7—C8	1.501 (6)
N2—H2A	0.8900	C7—H7A	0.9700
N2—H2B	0.8900	C7—H7B	0.9700
N2—H2C	0.8900	C10—H10A	0.9600
C11—C16	1.389 (4)	C10—H10B	0.9600
C11—C12	1.395 (4)	C10—H10C	0.9600
C12—C13	1.386 (4)	C8—H8A	0.9600
C12—C17	1.512 (4)	C8—H8B	0.9600
C16—C15	1.398 (4)	C8—H8C	0.9600
C16—C19	1.503 (4)	C11—C24	1.735 (3)
C17—C18	1.506 (5)	N3—C27	1.473 (4)
C17—H17A	0.9700	N3—H3A	0.8900
C17—H17B	0.9700	N3—H3B	0.8900

C19—C20	1.489 (5)	N3—H3C	0.8900
C19—H19A	0.9700	C23—C22	1.372 (4)
C19—H19B	0.9700	C23—C24	1.390 (4)
C15—C14	1.369 (5)	C23—H23	0.9300
C15—H15	0.9300	C22—C21	1.385 (4)
C14—C13	1.374 (5)	C22—H22	0.9300
C14—H14	0.9300	C21—C26	1.380 (4)
C13—H13	0.9300	C21—C27	1.514 (4)
C18—H18A	0.9600	C24—C25	1.372 (5)
C18—H18B	0.9600	C25—C26	1.379 (5)
C18—H18C	0.9600	C25—H25	0.9300
C20—H20A	0.9600	C27—H27A	0.9700
C20—H20B	0.9600	C27—H27B	0.9700
C20—H20C	0.9600	C26—H26	0.9300
N1—C1	1.467 (3)	O11—H6	0.8600
N1—H1A	0.8900	O11—H7	0.8500
N1—H1B	0.8900	O10—H1	0.8500
N1—H1C	0.8900	O10—H2	0.8600
C1—C2	1.385 (4)		
P2—O6—P3	134.79 (12)	C2—C1—C6	123.5 (3)
P2—O3—P1	129.53 (11)	C2—C1—N1	119.3 (3)
P3—O8—P1 <sup>i</sup>	133.32 (12)	C6—C1—N1	117.2 (3)
O5—P2—O4	117.76 (12)	C1—C2—C3	116.7 (3)
O5—P2—O6	109.61 (12)	C1—C2—C9	121.3 (3)
O4—P2—O6	107.36 (11)	C3—C2—C9	122.0 (3)
O5—P2—O3	109.36 (11)	C5—C6—C1	117.0 (3)
O4—P2—O3	110.45 (10)	C5—C6—C7	120.2 (3)
O6—P2—O3	100.95 (10)	C1—C6—C7	122.7 (3)
O7—P3—O9	120.48 (11)	C4—C5—C6	120.8 (4)
O7—P3—O8	106.62 (11)	C4—C5—H5	119.6
O9—P3—O8	109.20 (12)	C6—C5—H5	119.6
O7—P3—O6	111.75 (11)	C4—C3—C2	121.2 (4)
O9—P3—O6	104.64 (11)	C4—C3—H3	119.4
O8—P3—O6	102.77 (11)	C2—C3—H3	119.4
O1—P1—O2	121.24 (11)	C5—C4—C3	120.7 (3)
O1—P1—O8 <sup>i</sup>	111.08 (10)	C5—C4—H4	119.6
O2—P1—O8 <sup>i</sup>	107.48 (12)	C3—C4—H4	119.6
O1—P1—O3	106.16 (10)	C2—C9—C10	115.5 (3)
O2—P1—O3	109.28 (11)	C2—C9—H9A	108.4
O8 <sup>i</sup> —P1—O3	99.39 (11)	C10—C9—H9A	108.4
C11—N2—H2A	109.5	C2—C9—H9B	108.4
C11—N2—H2B	109.5	C10—C9—H9B	108.4
H2A—N2—H2B	109.5	H9A—C9—H9B	107.5
C11—N2—H2C	109.5	C8—C7—C6	113.5 (4)
H2A—N2—H2C	109.5	C8—C7—H7A	108.9
H2B—N2—H2C	109.5	C6—C7—H7A	108.9
C16—C11—C12	123.0 (2)	C8—C7—H7B	108.9
C16—C11—N2	119.3 (2)	C6—C7—H7B	108.9

## supplementary materials

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C12—C11—N2	117.7 (2)	H7A—C7—H7B	107.7
C13—C12—C11	117.2 (3)	C9—C10—H10A	109.5
C13—C12—C17	118.9 (3)	C9—C10—H10B	109.5
C11—C12—C17	123.8 (3)	H10A—C10—H10B	109.5
C11—C16—C15	117.2 (3)	C9—C10—H10C	109.5
C11—C16—C19	122.4 (2)	H10A—C10—H10C	109.5
C15—C16—C19	120.3 (3)	H10B—C10—H10C	109.5
C18—C17—C12	112.0 (3)	C7—C8—H8A	109.5
C18—C17—H17A	109.2	C7—C8—H8B	109.5
C12—C17—H17A	109.2	H8A—C8—H8B	109.5
C18—C17—H17B	109.2	C7—C8—H8C	109.5
C12—C17—H17B	109.2	H8A—C8—H8C	109.5
H17A—C17—H17B	107.9	H8B—C8—H8C	109.5
C20—C19—C16	116.8 (3)	C27—N3—H3A	109.5
C20—C19—H19A	108.1	C27—N3—H3B	109.5
C16—C19—H19A	108.1	H3A—N3—H3B	109.5
C20—C19—H19B	108.1	C27—N3—H3C	109.5
C16—C19—H19B	108.1	H3A—N3—H3C	109.5
H19A—C19—H19B	107.3	H3B—N3—H3C	109.5
C14—C15—C16	120.8 (3)	C22—C23—C24	118.8 (3)
C14—C15—H15	119.6	C22—C23—H23	120.6
C16—C15—H15	119.6	C24—C23—H23	120.6
C15—C14—C13	120.7 (3)	C23—C22—C21	121.4 (3)
C15—C14—H14	119.7	C23—C22—H22	119.3
C13—C14—H14	119.7	C21—C22—H22	119.3
C14—C13—C12	121.1 (3)	C26—C21—C22	118.7 (3)
C14—C13—H13	119.4	C26—C21—C27	120.1 (3)
C12—C13—H13	119.4	C22—C21—C27	121.2 (3)
C17—C18—H18A	109.5	C25—C24—C23	120.5 (3)
C17—C18—H18B	109.5	C25—C24—C11	120.1 (3)
H18A—C18—H18B	109.5	C23—C24—C11	119.4 (3)
C17—C18—H18C	109.5	C24—C25—C26	119.9 (3)
H18A—C18—H18C	109.5	C24—C25—H25	120.1
H18B—C18—H18C	109.5	C26—C25—H25	120.1
C19—C20—H20A	109.5	N3—C27—C21	112.8 (2)
C19—C20—H20B	109.5	N3—C27—H27A	109.0
H20A—C20—H20B	109.5	C21—C27—H27A	109.0
C19—C20—H20C	109.5	N3—C27—H27B	109.0
H20A—C20—H20C	109.5	C21—C27—H27B	109.0
H20B—C20—H20C	109.5	H27A—C27—H27B	107.8
C1—N1—H1A	109.5	C25—C26—C21	120.6 (3)
C1—N1—H1B	109.5	C25—C26—H26	119.7
H1A—N1—H1B	109.5	C21—C26—H26	119.7
C1—N1—H1C	109.5	H6—O11—H7	107.00
H1A—N1—H1C	109.5	H1—O10—H2	106.00
H1B—N1—H1C	109.5		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...O9 <sup>ii</sup>	0.89	1.80	2.685 (3)	171
N1—H1B...O2	0.89	2.03	2.758 (3)	138
N1—H1C...O4	0.89	2.13	2.935 (3)	151
N2—H2A...O4 <sup>i</sup>	0.89	1.94	2.827 (3)	172
N2—H2B...O1	0.89	2.05	2.887 (3)	156
N2—H2C...O7	0.89	1.94	2.809 (3)	166
N3—H3A...O5 <sup>iii</sup>	0.89	1.89	2.762 (3)	165
N3—H3B...O9	0.89	1.93	2.799 (3)	164
N3—H3C...O10	0.89	1.99	2.800 (5)	151
O10—H1...O11	0.85	2.22	2.923 (6)	140
O10—H2...O11 <sup>iv</sup>	0.86	2.25	2.831 (5)	125
O11—H6...O2 <sup>v</sup>	0.85	2.23	2.740 (4)	118
O11—H7...O5 <sup>iii</sup>	0.85	2.17	2.781 (4)	129

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

Fig. 1

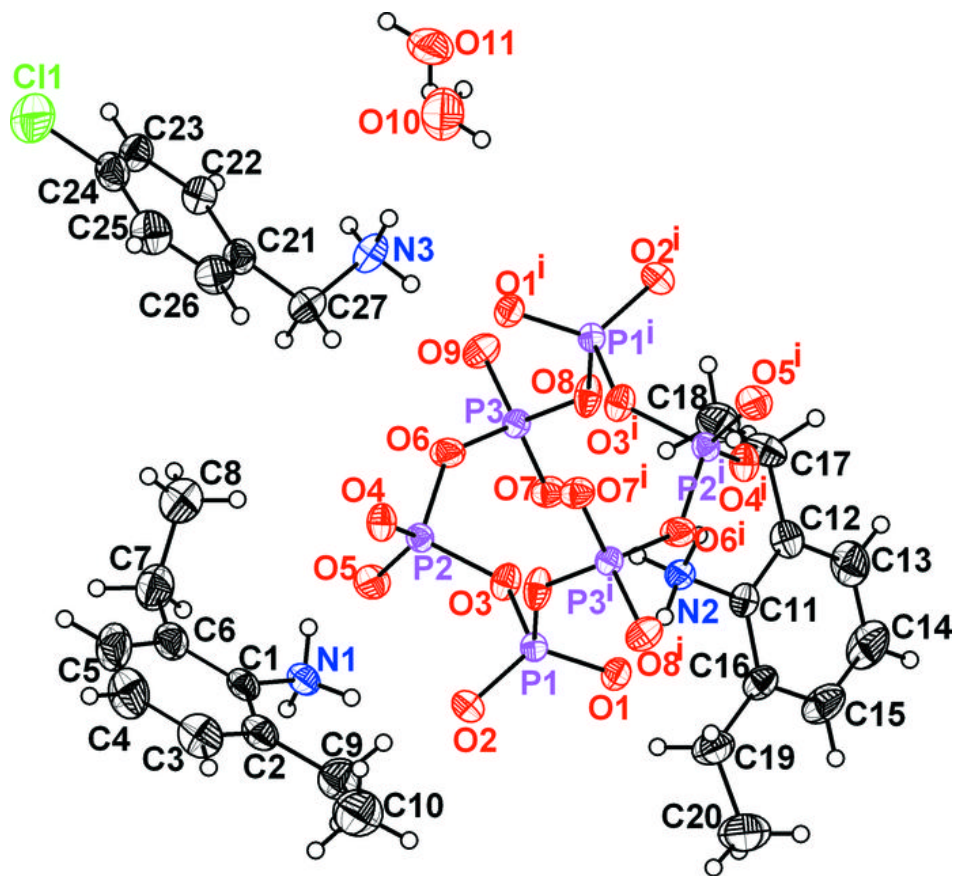


Fig. 2

