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4',4',6',6'-Tetrachloro-2-(6-methylpyridin-2-yl)-1*H*,2*H*-spiro[naphtho[1,2-*e*][1,3,2]oxazaphosphinine-3,2'-[1,3,5,2,4,6]triazatriphosphinine]

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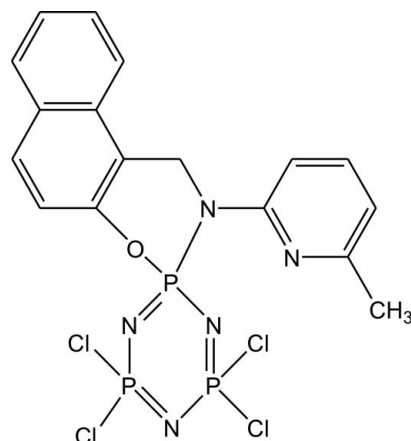
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.048; wR factor = 0.129; data-to-parameter ratio = 19.5.

The title compound, $\text{C}_{17}\text{H}_{14}\text{Cl}_4\text{N}_5\text{OP}_3$, is a spiro-phosphazene derivative with bulky naphthalene and pyridine rings. The phosphazene and the six-membered N/O rings are in flattened-boat and twisted-boat conformations, respectively. The naphthalene ring system and the pyridine ring are oriented at a dihedral angle of $18.06(8)^\circ$. In the crystal, weak π - π stacking between the pyridine rings and between the pyridine rings and the naphthalene ring system [centroid-centroid distances = $3.594(2)$ and $3.961(2)$ Å, respectively] occur. Weak $\text{C}-\text{H}\cdots\pi$ interactions are also observed. These interactions link the molecules into a three-dimensional supramolecular network.

Related literature

For products from the reaction of $\text{N}_3\text{P}_3\text{Cl}_6$ with bifunctional reagents, see: Bešli *et al.* (2006). For N/O donor type bifunctional reagents used for the reaction of hexachlorocyclo-triphosphazene giving *spiro* derivatives, see: Bešli *et al.* (2007); Sournies *et al.* (1997); Deutsch & Shaw (1990); Işıklan *et al.* (2010); İter *et al.* (2004, 2007); Tercan *et al.* (2004). For industrial applications of cyclophosphazenes, see: Omotowa *et al.* (2004); Barbera *et al.* (2005); Schneider *et al.* (1999); Ding *et al.* (2005); Allcock (2006); Xu *et al.* (2006); Allcock & Wood (2006); Li *et al.* (2004); Singh *et al.* (2006); Greish *et al.* (2005). For bond-length data, see: Allen *et al.* (1987). For the standard compound, $\text{N}_3\text{P}_3\text{Cl}_6$, see: Bullen (1971). For ring-puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{14}\text{Cl}_4\text{N}_5\text{OP}_3$
 $M_r = 539.04$

 Orthorhombic, *Pbca*
 $a = 21.7784(5)$ Å

 $b = 7.8573(3)$ Å

 $c = 25.1034(5)$ Å

 $V = 4295.7(2)$ Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.80$ mm⁻¹
 $T = 100$ K

 $0.38 \times 0.28 \times 0.08$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Absorption correction: multi-scan

 (*SADABS*; Bruker, 2005)

 $T_{\min} = 0.752$, $T_{\max} = 0.939$

22083 measured reflections

5297 independent reflections

 3446 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.070$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.129$
 $S = 1.01$

5297 reflections

272 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.73$ e Å⁻³
 $\Delta\rho_{\min} = -0.77$ e Å⁻³
Table 1

Selected bond lengths (Å).

P1—O1	1.580 (2)	P2—N1	1.571 (3)
P1—N1	1.596 (3)	P2—N2	1.580 (3)
P1—N3	1.598 (3)	P3—N2	1.581 (3)
P1—N4	1.665 (3)	P3—N3	1.571 (3)

Table 2

Hydrogen-bond geometry (Å, °).

 $Cg3$ is the centroid of the C8-benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C7-H7\cdots Cg3^i$	0.95	2.82	3.507 (3)	130

 Symmetry code: (i) $x, -y - \frac{1}{2}, z - \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5701).

References

- Allcock, H. R. (2006). *Curr. Opin. Solid State Mater. Sci.* **10**, 231–240.
- Allcock, H. R. & Wood, R. M. (2006). *J. Polym. Sci. Part B Polym. Phys.* **44**, 2358–2368.
- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Barbera, J., Bardaji, M., Jimenez, J., Laguna, A., Martinez, M. P., Oriol, L., Serrano, J. L. & Zaragoza, I. (2005). *J. Am. Chem. Soc.* **127**, 8994–9002.
- Beşli, S., Coles, S. J., Davies, D. B., Eaton, R. J., Kılıç, A. & Shaw, R. A. (2006). *Polyhedron*, **25**, 963–974.
- Beşli, S., Coles, S. J., Davies, D. B., Hursthouse, M. B., Kılıç, A. & Shaw, R. A. (2007). *Dalton Trans.* pp. 2792–2801.
- Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bullen, G. J. (1971). *J. Chem. Soc. A*, pp. 1450–1453.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Deutsch, W. F. & Shaw, R. A. (1990). *Phosphorus Sulfur Silicon*, **47**, 119–140.
- Ding, J., Liang, H., Shi, W. & Shen, X. (2005). *J. Appl. Polym. Sci.* **97**, 1776–1782.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Greish, Y. E., Bender, J. E., Lakshmi, S., Brown, P. W., Allcock, H. R. & Laurencin, C. T. (2005). *Biomaterials*, **26**, 1–9.
- İlter, E. E., Asmafiliz, N., Kılıç, Z., Işıklan, M., Hökelek, T., Çaylak, N. & Şahin, E. (2007). *Inorg. Chem.* **46**, 9931–9944.
- İlter, E. E., Çaylak, N., Işıklan, M., Asmafiliz, N., Kılıç, Z. & Hökelek, T. (2004). *J. Mol. Struct.* **697**, 119–129.
- Işıklan, M., Asmafiliz, N., Özalp, E. E., İlter, E. E., Kılıç, Z., Coşut, B., Yeşilot, S., Kılıç, A., Öztürk, A., Hökelek, T., Koç, L. Y., Açıık, L. & Akyüz, E. (2010). *Inorg. Chem.* **49**, 7057–7071.
- Li, Z., Huang, C., Hua, J., Qin, J., Yang, Z. & Ye, C. (2004). *Macromolecules*, **37**, 371–376.
- Omotowa, B. A., Phillips, B. S., Zabinski, J. S. & Shreeve, J. M. (2004). *Inorg. Chem.* **43**, 5466–5471.
- Schneider, R., Köllner, C., Weber, I. & Togni, A. (1999). *Chem. Commun.* **23**, 2415–2416.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Singh, A., Krogman, N. R., Sethurman, S., Nair, L. S., Sturgeon, J. L., Brown, P. W., Laurencin, C. T. & Allcock, H. R. (2006). *Biomacromolecules*, **7**, 914–918.
- Sournies, F., Zai, K., Vercruyse, K., Labarre, M. C. & Labarre, J. F. (1997). *J. Mol. Struct.* **412**, 19–26.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Tercan, B., Hökelek, T., Işıklan, M., İlter, E. E. & Kılıç, Z. (2004). *Acta Cryst.* **E60**, o971–o973.
- Xu, G.-X., Lu, Q., Yu, B. T. & Wen, L. (2006). *Solid State Ionics*, **177**, 305–309.

supplementary materials

Acta Cryst. (2013). E69, o861–o862 [doi:10.1107/S1600536813012348]

4',4',6',6'-Tetrachloro-2-(6-methylpyridin-2-yl)-1*H*,2*H*-spiro[naphtho[1,2-*e*][1,3,2]oxazaphosphinine-3,2'-[1,3,5,2,4,6]triazatriphosphinine]

Muhammet Işıklan, Ömer Sonkaya and Tuncer Hökelek

Comment

Cyclophosphazenes are known as inorganic heterocyclic rings containing an (N=PX₂)_n repeating unit. Hexachlorocyclo-triphosphazene, N₃P₃Cl₆, can also be considered as a trimer, which can be obtained by the reaction of phosphorus pentachloride and ammonium chloride. It has been observed that reactions of N₃P₃Cl₆ with bifunctional reagents can afford *spiro*-, *ansa*-, *bino*- and open chain (dangling) products (Bešli *et al.*, 2006). Reaction of hexachlorocyclo-triphosphazene with N/O donor type bifunctional reagents such as aminoalcohols (Bešli *et al.*, 2007; Sournies *et al.*, 1997; Deutsch & Shaw, 1990), aminophenols (Işıklan *et al.*, 2010; İter *et al.*, 2007) and aminonaphthols (İter *et al.*, 2004; Tercan *et al.*, 2004) predominantly gave *spiro* derivatives. Cyclophosphazenes have found industrial applications such as in the production of ionic liquids (Omotowa *et al.*, 2004), liquid crystals (Barbera *et al.*, 2005), dendrimers having chiral ligands for asymmetric catalysis (Schneider *et al.*, 1999), flame retardants (Ding *et al.*, 2005), advanced elastomers (Allcock, 2006), rechargeable lithium batteries and polymer electrolytes (Xu *et al.*, 2006; Allcock & Wood, 2006), non-linear optics (Li *et al.*, 2004), biomedical membranes (Singh *et al.*, 2006), and biomedical materials including synthetic bones (Greish *et al.*, 2005). The present study was undertaken to ascertain the crystal structure of the title compound.

In the title compound, (Fig. 1), the phosphazene ring (*A*) is in flattened-boat conformation [$\varphi = 31.7 (9)^\circ$ and $\theta = 101.8 (8)^\circ$] having total puckering amplitude Q_T of 0.137 (2) Å (Cremer & Pople, 1975). Atoms N1, N2 and N3 are displaced from the plane through the P atoms by 0.112 (3), -0.154 (3) and -0.029 (3) Å, respectively. As expected, the naphthalene and pyridine rings are planar, and they are oriented at a dihedral angle of 18.06 (8)°. Ring *B* (P1/O1/C9—C11/N4) is in twisted-boat conformation [$\varphi = 147.1 (7)^\circ$ and $\theta = 96.9 (8)^\circ$] having total puckering amplitude Q_T of 0.482 (7) Å.

In the phosphazene ring, the P—N bond lengths are in the range of 1.571 (3)–1.598 (3) Å [average value is 1.583 (3) Å] (Table 1), exhibiting a regular variation with distances from P1: P1—N1 \approx P1—N3 \succ P2—N2 \approx P3—N2 \succ P2—N1 \approx P3—N3, and showing double-bond character. However, the exocyclic P1—N4 bond [1.665 (3) Å] is at the lower limit of the single bond length. In the phosphazene compounds, the P—N and P=N bonds are generally in the ranges of 1.628–1.691 and 1.571–1.604 Å, respectively (Allen *et al.*, 1987). The shortening in the P1—N4 bond is probably due to electron transfer from N4 to the phosphazene ring.

In the phosphazene ring, the endocyclic N1—P1—N3 angle [117.31 (14)°] is decreased and the exocyclic O1—P1—N4 angle [102.37 (12)°] is increased, with electron donation and withdrawal by the substituents, relative to the 'standard compound' N₃P₃Cl₆ (Bullen, 1971). In the latter compound, the corresponding angles are 118.3, 118.5, 101.2 and 101.6 °, respectively.

The P1—N1—P1, P2—N2—P3 and P1—N3—P3 angles are 121.72 (17), 119.79 (17) and 120.78 (17) °, respectively; P2—N2—P3 and P1—N3—P3 are decreased, with electron donation and withdrawal by the N₃P₃ ring. They can be compared with the average value reported for N₃P₃Cl₆, viz. 121.4 (3)°.

In the crystal, molecules are stacked nearly parallel to (101) [Fig. 2]. Two weak C—H··· π interactions (Table 2) and two weak π — π contacts between the pyridine rings and between the pyridine and naphthalene rings, Cg5—Cg5ⁱ and Cg5—Cg3ⁱⁱ [symmetry codes: (i) - x, - y, 1 - z, (ii) - x, 1 - y, 1 - z, where Cg3 and Cg5 are the centroids of the rings (C1/C6—C10) and (N5/C12—C16), respectively] may stabilize the structure, with centroid-centroid distances of 3.594 (2) and 3.961 (2) Å, respectively.

Experimental

Hexachlorocyclotriphosphazene (1.00 g, 2.88 mmol) in dry THF (100 ml) was introduced into a 250 ml three-necked round-bottomed flask. One equivalent of 1-[(6-methylpyridin-2-ylamino)methyl]naphthalene-2-ol (0.76 g, 2.88 mmol) and triethylamine (5 ml, 36.00 mmol) were dissolved in dry THF (50 ml) and placed in an addition funnel. The 1-[(6-methylpyridin-2-ylamino)methyl]-naphthalene-2-ol and triethylamine solution was added dropwise to the hexachlorocyclotriphosphazene solution and allowed to react for 24 h. The progress of the reaction was monitored by TLC. The precipitated triethylamine hydrochloride was filtered off. The solvent was evaporated and the product was purified through a silica gel column with a mobile phase of CHCl₃. The oily product was recrystallized in n-hexane [m.p. 495.5 K, yield: 0.70 g, 45%].

Refinement

H atoms were positioned geometrically with C—H = 0.95, 0.99 and 0.98 Å for aromatic, methylene and methyl H atoms, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{C})$, where $k = 1.5$ for methyl H-atoms and $k = 1.2$ for all other H-atoms.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE* (Bruker, 2007); 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, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

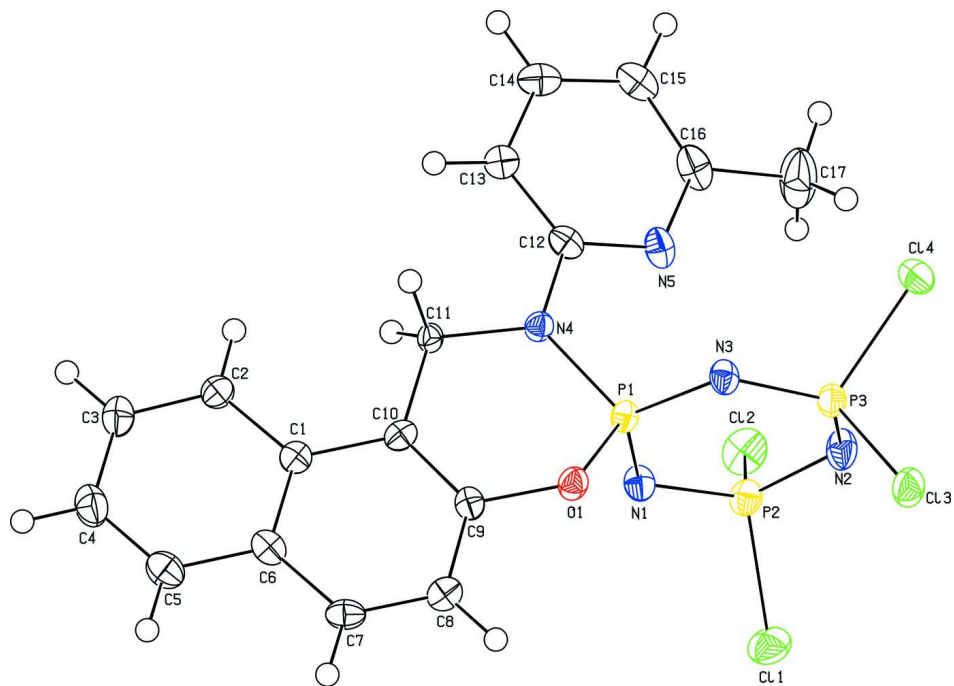
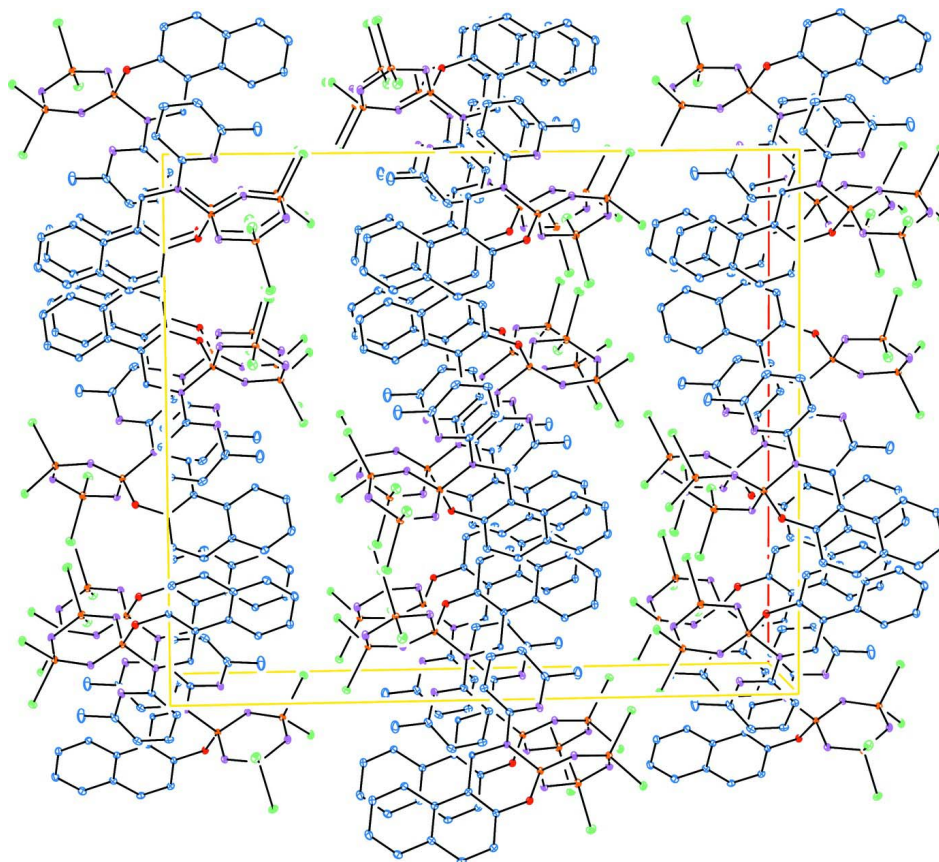


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A view along the *b* axis of the crystal packing of the title compound (*a* axis vertical; *c* axis horizontal). Hydrogen atoms have been omitted for clarity.

**4',4',6',6'-Tetrachloro-2-(6-methylpyridin-2-yl)-1*H*,2*H*-spiro[naphtho[1,2-*e*]
[1,3,2]oxazaphosphinine-3,2'-[1,3,5,2,4,6]triazatriphosphinine]**

Crystal data

$C_{17}H_{14}Cl_4N_5OP_3$

$M_r = 539.04$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 21.7784$ (5) Å

$b = 7.8573$ (3) Å

$c = 25.1034$ (5) Å

$V = 4295.7$ (2) Å³

$Z = 8$

$F(000) = 2176$

$D_x = 1.667$ Mg m⁻³

Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 2741 reflections

$\theta = 2.5$ – 26.6°

$\mu = 0.80$ mm⁻¹

$T = 100$ K

Plate, colorless

$0.38 \times 0.28 \times 0.08$ mm

Data collection

Bruker Kappa APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.752$, $T_{\max} = 0.939$

22083 measured reflections

5297 independent reflections

3446 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.070$
 $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -25 \rightarrow 29$

$k = -10 \rightarrow 6$
 $l = -31 \rightarrow 33$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.129$
 $S = 1.01$
 5297 reflections
 272 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 1.3984P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.73 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.77 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.23736 (4)	0.09286 (12)	0.33683 (4)	0.0310 (2)
Cl2	0.12400 (4)	-0.12370 (11)	0.36507 (4)	0.0315 (2)
Cl3	0.12654 (4)	0.54678 (12)	0.27056 (3)	0.0268 (2)
Cl4	0.00013 (4)	0.37802 (12)	0.28234 (3)	0.0277 (2)
P1	0.10765 (4)	0.37524 (10)	0.42559 (3)	0.01570 (18)
P2	0.14946 (4)	0.11972 (11)	0.35899 (3)	0.0203 (2)
P3	0.08161 (4)	0.38196 (11)	0.31822 (3)	0.01836 (19)
O1	0.15089 (10)	0.5220 (3)	0.44668 (8)	0.0188 (5)
N1	0.14830 (12)	0.2097 (3)	0.41488 (10)	0.0195 (6)
N2	0.11206 (13)	0.2007 (4)	0.31138 (11)	0.0243 (7)
N3	0.07526 (12)	0.4618 (3)	0.37531 (10)	0.0183 (6)
N4	0.06038 (12)	0.3449 (3)	0.47683 (10)	0.0161 (6)
N5	0.00488 (13)	0.1705 (3)	0.42067 (11)	0.0220 (6)
C1	0.15699 (15)	0.5167 (4)	0.59375 (12)	0.0170 (7)
C2	0.12413 (15)	0.4649 (4)	0.63968 (12)	0.0197 (7)
H2	0.0861	0.4068	0.6357	0.024*
C3	0.14637 (16)	0.4972 (4)	0.68968 (13)	0.0241 (8)
H3	0.1242	0.4585	0.7199	0.029*
C4	0.20163 (17)	0.5872 (5)	0.69686 (14)	0.0279 (8)
H4	0.2163	0.6104	0.7317	0.033*
C5	0.23409 (16)	0.6410 (4)	0.65326 (14)	0.0248 (8)
H5	0.2713	0.7021	0.6581	0.030*
C6	0.21268 (15)	0.6066 (4)	0.60098 (13)	0.0202 (7)

C7	0.24658 (15)	0.6603 (4)	0.55567 (13)	0.0213 (7)
H7	0.2839	0.7211	0.5606	0.026*
C8	0.22672 (15)	0.6265 (4)	0.50547 (13)	0.0203 (7)
H8	0.2499	0.6612	0.4753	0.024*
C9	0.17088 (14)	0.5387 (4)	0.49938 (12)	0.0167 (6)
C10	0.13573 (14)	0.4829 (4)	0.54063 (12)	0.0164 (6)
C11	0.07536 (14)	0.3917 (4)	0.53259 (11)	0.0147 (6)
H11A	0.0758	0.2865	0.5543	0.018*
H11B	0.0420	0.4651	0.5464	0.018*
C12	0.00836 (14)	0.2393 (4)	0.46936 (13)	0.0184 (7)
C13	-0.03482 (15)	0.2132 (4)	0.50891 (13)	0.0182 (7)
H13	-0.0292	0.2574	0.5438	0.022*
C14	-0.08672 (15)	0.1197 (4)	0.49558 (14)	0.0229 (7)
H14	-0.1179	0.1002	0.5213	0.027*
C15	-0.09263 (16)	0.0552 (4)	0.44471 (14)	0.0244 (8)
H15	-0.1287	-0.0045	0.4346	0.029*
C16	-0.04548 (17)	0.0783 (4)	0.40862 (14)	0.0263 (8)
C17	-0.0465 (2)	-0.0028 (6)	0.35445 (15)	0.0493 (12)
H17A	-0.0384	0.0838	0.3273	0.074*
H17B	-0.0869	-0.0539	0.3481	0.074*
H17C	-0.0149	-0.0913	0.3526	0.074*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0266 (5)	0.0330 (5)	0.0333 (5)	0.0062 (4)	0.0064 (4)	-0.0064 (4)
C12	0.0411 (6)	0.0177 (4)	0.0357 (5)	-0.0001 (4)	0.0043 (4)	-0.0053 (4)
C13	0.0293 (5)	0.0327 (5)	0.0184 (4)	-0.0093 (4)	0.0004 (3)	0.0037 (3)
C14	0.0211 (4)	0.0374 (5)	0.0245 (4)	-0.0040 (4)	-0.0045 (3)	0.0029 (4)
P1	0.0188 (4)	0.0146 (4)	0.0137 (4)	0.0009 (4)	0.0004 (3)	-0.0004 (3)
P2	0.0245 (5)	0.0173 (4)	0.0192 (4)	0.0029 (4)	0.0014 (3)	-0.0034 (3)
P3	0.0199 (4)	0.0200 (4)	0.0151 (4)	-0.0005 (4)	-0.0005 (3)	0.0000 (3)
O1	0.0211 (12)	0.0192 (12)	0.0161 (11)	-0.0042 (10)	0.0013 (9)	0.0005 (9)
N1	0.0233 (15)	0.0169 (14)	0.0182 (14)	0.0053 (12)	-0.0021 (11)	-0.0008 (11)
N2	0.0315 (17)	0.0241 (15)	0.0174 (14)	0.0053 (13)	-0.0010 (11)	-0.0054 (12)
N3	0.0222 (15)	0.0169 (14)	0.0159 (13)	0.0050 (12)	-0.0021 (11)	0.0026 (11)
N4	0.0170 (14)	0.0163 (14)	0.0152 (12)	-0.0027 (11)	0.0010 (10)	-0.0001 (10)
N5	0.0297 (16)	0.0186 (14)	0.0179 (13)	-0.0036 (13)	-0.0055 (11)	0.0005 (11)
C1	0.0191 (17)	0.0131 (16)	0.0189 (15)	0.0029 (13)	0.0002 (12)	0.0018 (12)
C2	0.0201 (17)	0.0192 (17)	0.0199 (16)	-0.0004 (14)	0.0011 (13)	0.0006 (13)
C3	0.030 (2)	0.0240 (18)	0.0185 (16)	0.0026 (15)	-0.0009 (13)	0.0006 (13)
C4	0.032 (2)	0.031 (2)	0.0212 (17)	0.0033 (16)	-0.0050 (14)	-0.0046 (14)
C5	0.0244 (18)	0.0215 (18)	0.0286 (18)	-0.0027 (16)	-0.0045 (14)	-0.0024 (14)
C6	0.0216 (17)	0.0159 (17)	0.0233 (16)	0.0003 (14)	-0.0043 (13)	0.0003 (13)
C7	0.0166 (17)	0.0177 (18)	0.0295 (18)	-0.0027 (13)	0.0016 (13)	-0.0011 (14)
C8	0.0221 (17)	0.0166 (16)	0.0223 (16)	-0.0005 (15)	0.0031 (13)	0.0002 (13)
C9	0.0198 (17)	0.0139 (16)	0.0164 (15)	0.0033 (13)	-0.0017 (12)	-0.0019 (12)
C10	0.0180 (16)	0.0122 (15)	0.0191 (15)	0.0029 (13)	0.0032 (12)	0.0007 (12)
C11	0.0161 (15)	0.0161 (15)	0.0118 (13)	0.0012 (13)	-0.0002 (11)	0.0025 (12)
C12	0.0187 (17)	0.0129 (16)	0.0235 (16)	0.0020 (14)	-0.0044 (13)	0.0027 (13)

C13	0.0205 (17)	0.0118 (15)	0.0225 (16)	0.0012 (14)	0.0006 (13)	0.0000 (13)
C14	0.0191 (17)	0.0146 (16)	0.0349 (19)	0.0023 (14)	0.0019 (14)	0.0056 (14)
C15	0.0239 (18)	0.0155 (16)	0.0337 (19)	-0.0032 (15)	-0.0093 (15)	0.0058 (14)
C16	0.035 (2)	0.0184 (18)	0.0251 (18)	-0.0069 (16)	-0.0101 (15)	0.0037 (14)
C17	0.069 (3)	0.054 (3)	0.025 (2)	-0.034 (2)	-0.003 (2)	-0.0021 (19)

Geometric parameters (Å, °)

C11—P2	2.0047 (12)	C6—C5	1.419 (4)
C12—P2	1.9972 (13)	C6—C7	1.420 (5)
C13—P3	2.0165 (12)	C7—C8	1.358 (4)
C14—P3	1.9902 (12)	C7—H7	0.9500
P1—O1	1.580 (2)	C8—H8	0.9500
P1—N1	1.596 (3)	C9—C8	1.407 (4)
P1—N3	1.598 (3)	C10—C1	1.436 (4)
P1—N4	1.665 (3)	C10—C9	1.360 (4)
P2—N1	1.571 (3)	C11—C10	1.511 (4)
P2—N2	1.580 (3)	C11—H11A	0.9900
P3—N2	1.581 (3)	C11—H11B	0.9900
P3—N3	1.571 (3)	C12—N5	1.339 (4)
O1—C9	1.399 (4)	C12—C13	1.383 (4)
N4—C11	1.483 (4)	C13—C14	1.389 (5)
N4—C12	1.416 (4)	C13—H13	0.9500
C1—C2	1.417 (4)	C14—C15	1.380 (5)
C1—C6	1.415 (4)	C14—H14	0.9500
C2—C3	1.369 (4)	C15—C16	1.381 (5)
C2—H2	0.9500	C15—H15	0.9500
C3—H3	0.9500	C16—N5	1.348 (4)
C4—C3	1.407 (5)	C16—C17	1.502 (5)
C4—H4	0.9500	C17—H17A	0.9800
C5—C4	1.370 (5)	C17—H17B	0.9800
C5—H5	0.9500	C17—H17C	0.9800
O1—P1—N1	108.71 (14)	C1—C6—C7	119.4 (3)
O1—P1—N3	102.53 (13)	C5—C6—C7	120.9 (3)
O1—P1—N4	102.37 (12)	C6—C7—H7	119.4
N1—P1—N3	117.31 (14)	C8—C7—C6	121.3 (3)
N1—P1—N4	110.87 (14)	C8—C7—H7	119.4
N3—P1—N4	113.47 (14)	C7—C8—C9	118.2 (3)
C12—P2—C11	100.69 (5)	C7—C8—H8	120.9
N1—P2—C11	108.08 (11)	C9—C8—H8	120.9
N1—P2—C12	110.98 (11)	O1—C9—C8	114.7 (3)
N1—P2—N2	119.09 (15)	C10—C9—O1	121.0 (3)
N2—P2—C11	108.95 (12)	C10—C9—C8	124.2 (3)
N2—P2—C12	107.47 (12)	C1—C10—C11	119.5 (3)
C14—P3—C13	100.03 (5)	C9—C10—C1	117.8 (3)
N2—P3—C13	108.10 (12)	C9—C10—C11	122.7 (3)
N2—P3—C14	108.11 (11)	N4—C11—C10	115.8 (2)
N3—P3—C13	109.12 (11)	N4—C11—H11A	108.3
N3—P3—C14	109.92 (11)	N4—C11—H11B	108.3

N3—P3—N2	119.75 (14)	C10—C11—H11A	108.3
C9—O1—P1	124.78 (19)	C10—C11—H11B	108.3
P2—N1—P1	121.72 (17)	H11A—C11—H11B	107.4
P2—N2—P3	119.79 (17)	N5—C12—N4	113.8 (3)
P3—N3—P1	120.78 (17)	N5—C12—C13	123.8 (3)
C11—N4—P1	123.9 (2)	C13—C12—N4	122.4 (3)
C12—N4—P1	118.4 (2)	C12—C13—C14	117.3 (3)
C12—N4—C11	116.5 (2)	C12—C13—H13	121.3
C12—N5—C16	117.9 (3)	C14—C13—H13	121.3
C6—C1—C2	118.2 (3)	C13—C14—H14	120.2
C6—C1—C10	119.2 (3)	C15—C14—C13	119.5 (3)
C2—C1—C10	122.6 (3)	C15—C14—H14	120.2
C1—C2—H2	119.5	C14—C15—C16	119.3 (3)
C3—C2—C1	120.9 (3)	C14—C15—H15	120.4
C3—C2—H2	119.5	C16—C15—H15	120.4
C2—C3—C4	120.9 (3)	N5—C16—C15	121.9 (3)
C2—C3—H3	119.6	N5—C16—C17	116.3 (3)
C4—C3—H3	119.6	C15—C16—C17	121.8 (3)
C3—C4—H4	120.2	C16—C17—H17A	109.5
C5—C4—C3	119.6 (3)	C16—C17—H17B	109.5
C5—C4—H4	120.2	C16—C17—H17C	109.5
C4—C5—C6	120.7 (3)	H17A—C17—H17B	109.5
C4—C5—H5	119.6	H17A—C17—H17C	109.5
C6—C5—H5	119.6	H17B—C17—H17C	109.5
C1—C6—C5	119.7 (3)		
N1—P1—O1—C9	-81.1 (3)	C10—C1—C2—C3	179.0 (3)
N3—P1—O1—C9	154.0 (2)	C2—C1—C6—C5	0.4 (5)
N4—P1—O1—C9	36.2 (3)	C2—C1—C6—C7	-179.9 (3)
O1—P1—N1—P2	-123.90 (19)	C10—C1—C6—C5	179.9 (3)
N3—P1—N1—P2	-8.3 (3)	C10—C1—C6—C7	-0.4 (5)
N4—P1—N1—P2	124.32 (19)	C1—C2—C3—C4	1.8 (5)
O1—P1—N3—P3	121.15 (19)	C5—C4—C3—C2	-0.9 (5)
N1—P1—N3—P3	2.2 (3)	C6—C5—C4—C3	-0.2 (5)
N4—P1—N3—P3	-129.23 (18)	C1—C6—C5—C4	0.5 (5)
O1—P1—N4—C11	-22.8 (3)	C7—C6—C5—C4	-179.2 (3)
O1—P1—N4—C12	170.1 (2)	C1—C6—C7—C8	-0.3 (5)
N1—P1—N4—C11	93.0 (3)	C5—C6—C7—C8	179.4 (3)
N1—P1—N4—C12	-74.1 (3)	C6—C7—C8—C9	1.0 (5)
N3—P1—N4—C11	-132.5 (2)	O1—C9—C8—C7	174.6 (3)
N3—P1—N4—C12	60.4 (3)	C10—C9—C8—C7	-1.0 (5)
C11—P2—N1—P1	128.10 (17)	C9—C10—C1—C2	179.9 (3)
C12—P2—N1—P1	-122.37 (17)	C9—C10—C1—C6	0.4 (4)
N2—P2—N1—P1	3.2 (3)	C11—C10—C1—C2	1.3 (5)
C11—P2—N2—P3	-116.35 (18)	C11—C10—C1—C6	-178.2 (3)
C12—P2—N2—P3	135.37 (17)	C1—C10—C9—O1	-175.1 (3)
N1—P2—N2—P3	8.2 (3)	C1—C10—C9—C8	0.3 (5)
C13—P3—N2—P2	111.53 (18)	C11—C10—C9—O1	3.5 (5)
C14—P3—N2—P2	-141.03 (17)	C11—C10—C9—C8	178.9 (3)

N3—P3—N2—P2	-14.2 (3)	N4—C11—C10—C1	-173.4 (3)
C13—P3—N3—P1	-116.34 (17)	N4—C11—C10—C9	8.1 (4)
C14—P3—N3—P1	134.90 (16)	N4—C12—N5—C16	-175.2 (3)
N2—P3—N3—P1	8.9 (3)	C13—C12—N5—C16	4.0 (5)
P1—O1—C9—C8	154.1 (2)	N4—C12—C13—C14	174.3 (3)
P1—O1—C9—C10	-30.1 (4)	N5—C12—C13—C14	-4.7 (5)
P1—N4—C11—C10	4.4 (4)	C12—C13—C14—C15	1.1 (5)
C12—N4—C11—C10	171.6 (3)	C13—C14—C15—C16	2.9 (5)
P1—N4—C12—N5	2.2 (4)	C14—C15—C16—N5	-3.8 (5)
P1—N4—C12—C13	-177.0 (2)	C14—C15—C16—C17	174.1 (4)
C11—N4—C12—N5	-165.9 (3)	C15—C16—N5—C12	0.4 (5)
C11—N4—C12—C13	15.0 (4)	C17—C16—N5—C12	-177.6 (3)
C6—C1—C2—C3	-1.5 (5)		

Hydrogen-bond geometry (Å, °)

Cg3 is the centroid of the C8-benzene ring.

<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>
C5—H5...Cg2 ⁱ	0.95	2.97	3.746 (4)	140
C7—H7...Cg3 ⁱ	0.95	2.82	3.507 (3)	130

Symmetry code: (i) *x*, *-y*-1/2, *z*-1/2.