

N,N'-Dibenzyl-*N''*-(2-chloro-2,2-difluoroacetyl)-*N,N'*-dimethylphosphoric triamide

Akbar Raissi Shabari,^{a*} Mehrdad Pourayoubi^b and Anahid Saneei^b

^aFaculty of Chemistry, Islamic Azad University, North Tehran Branch, Tehran, Iran, and ^bDepartment of Chemistry, Ferdowsi University of Mashhad, Mashhad 91779, Iran

Correspondence e-mail: a.raissi_shabari@yahoo.com

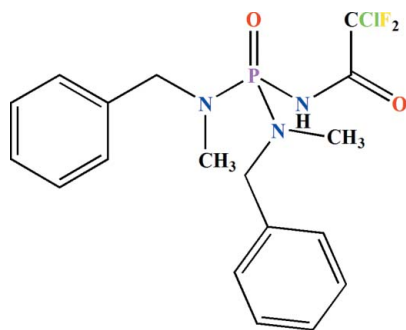
Received 2 February 2011; accepted 14 February 2011

Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.049; wR factor = 0.111; data-to-parameter ratio = 16.8.

In the title molecule, $\text{C}_{18}\text{H}_{21}\text{ClF}_2\text{N}_3\text{O}_2\text{P}$, the $\text{P}=\text{O}$ and $\text{N}-\text{H}$ groups are *syn* to each other. The P atom adopts a slightly distorted tetrahedral environment and the N atoms of the tertiary amine groups are bonded in an essentially planar geometry. In the crystal, pairs of intermolecular $\text{N}-\text{H}\cdots\text{O}(\text{P})$ hydrogen bonds form centrosymmetric dimers.

Related literature

For metal complexes with phosphoryl donor ligands, see: Gholivand *et al.* (2010). For a polyoxometalate-based inorganic-organic compound containing a phosphoryl ligand, see: Niu *et al.* (1996). For phosphoric triamide compounds having a $\text{C}(=\text{O})\text{NHP}(=\text{O})$ skeleton, see: Pourayoubi & Sabbaghi (2009) and references cited therein. For bond lengths in related structures, see: Sabbaghi *et al.* (2010). For hydrogen-bond motifs, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For the synthesis of the starting material, $\text{CClF}_2\text{C}(\text{O})\text{NHP}(\text{O})\text{Cl}_2$, see: Iriarte *et al.* (2008).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{21}\text{ClF}_2\text{N}_3\text{O}_2\text{P}$
 $M_r = 415.80$
 Triclinic, $P\bar{1}$
 $a = 10.3059$ (9) Å
 $b = 10.5030$ (9) Å
 $c = 10.9473$ (9) Å
 $\alpha = 71.743$ (2)°
 $\beta = 67.294$ (2)°

$\gamma = 63.265$ (2)°
 $V = 962.15$ (14) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.32$ mm⁻¹
 $T = 120$ K
 $0.28 \times 0.22 \times 0.15$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1998)
 $T_{\min} = 0.916$, $T_{\max} = 0.954$

9258 measured reflections
 4148 independent reflections
 3359 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.111$
 $S = 1.00$
 4148 reflections

247 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Selected bond angles (°).

O1—P1—N2	112.34 (9)	C1—N1—C2	114.36 (17)
O1—P1—N1	117.13 (9)	C1—N1—P1	126.21 (15)
N2—P1—N1	107.02 (9)	C2—N1—P1	119.42 (14)
O1—P1—N3	105.06 (9)	C9—N2—C10	115.45 (17)
N2—P1—N3	110.57 (9)	C9—N2—P1	121.56 (14)
N1—P1—N3	104.38 (9)	C10—N2—P1	122.42 (15)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N3}-\text{H3N}\cdots\text{O1}^i$	0.87	1.91	2.772 (3)	174

Symmetry code: (i) $-x + 1, -y + 1, -z$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1998); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Support of this investigation by Islamic Azad University-North Tehran Branch is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5205).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Bruker (1998). *SAINTE-Plus* and *SMART*, Bruker AXS Inc., Madison, Wisconsin, USA.
 Etter, M. C., MacDonal, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Gholivand, K., Mahzouni, H. R., Pourayoubi, M. & Amiri, S. (2010). *Inorg. Chim. Acta*, **363**, 2318–2324.

- Iriarte, A. G., Erben, M. F., Gholivand, K., Jios, J. L., Ulic, S. E. & Védova, C. O. D. (2008). *J. Mol. Struct.* **886**, 66–71.
- Niu, J.-Y., You, X.-Z., Duan, C.-Y., Fun, H.-K. & Zhou, Z.-Y. (1996). *Inorg. Chem.* **35**, 4211–4217.
- Pourayoubi, M. & Sabbaghi, F. (2009). *J. Chem. Crystallogr.* **39**, 874–880.
- Sabbaghi, F., Pourayoubi, M., Toghraee, M. & Divjakovic, V. (2010). *Acta Cryst.* **E66**, o344.
- Sheldrick, G. M. (1998). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2011). E67, o663-o664 [doi:10.1107/S1600536811005423]

N,N'-Dibenzyl-*N'*-(2-chloro-2,2-difluoroacetyl)-*N,N'*-dimethylphosphoric triamide

A. Raissi Shabari, M. Pourayoubi and A. Saneei

Comment

Phosphoryl donor ligands have been used to prepare coordination complexes (Gholivand *et al.*, 2010) and polyoxometalate-based inorganic-organic hybrid compounds (Niu *et al.*, 1996). The structure determination of title compound was performed as a part of a project on the synthesis of new potential phosphoric triamide ligands having a C(=O)NHP(=O) skeleton (Pourayoubi & Sabbaghi, 2009).

In the title compound, C₁₈H₂₁ClF₂N₃O₂P, the phosphoryl group and NH unit are *syn* to each other and the phosphorus atom has a slightly distorted tetrahedral configuration (Fig. 1). The P atom adopts a slightly distorted tetrahedral environment and the N atoms of the tertiary amine groups are bonded in an essentially planar geometry (see Table 1). The P=O bond length is comparable to those in similar compounds e.g. in P(O)[NHC(O)C₆H₄(4-NO₂)]₂ (Sabbaghi *et al.*, 2010). In the (CClF₂)C(O) unit, the O—C—C—F dihedral angles showing the orientation of fluorine atoms relative to carbonyl group are 17.7 (3) and 137.1 (2)° and the O—C—C—Cl dihedral angle is -101.9 (2)°.

The phosphoryl is a better H-acceptor relative to the carbonyl counterpart (Pourayoubi & Sabbaghi, 2009); so, the hydrogen atom of the C(=O)NHP(=O) group is involved in an intermolecular —P=O···H—N— hydrogen bond (see Table 2) to form a centrosymmetric dimeric aggregate [graph set: R₂²(8) (Etter *et al.*, 1990; Bernstein *et al.*, 1995)], Fig. 2.

Experimental

Synthesis of CClF₂C(O)NHP(O)Cl₂: CClF₂C(O)NHP(O)Cl₂ was prepared according to procedure reported by Iriarte *et al.* (2008) from a reaction between phosphorus pentachloride (16.91 mmol) and CClF₂C(O)NH₂ (16.91 mmol) in dry CCl₄ at 358 K (3 h) and then the treatment of formic acid (16.91 mmol) at ice bath temperature; then removing of solvent in vacuum to yield CClF₂C(O)NHP(O)Cl₂.

Synthesis of title compound: To a solution of CClF₂C(O)NHP(O)Cl₂ (1.04 mmol) in dry CHCl₃, a solution of *N*-methylbenzylamine (4.16 mmol) in dry CHCl₃ was added dropwise and stirred at 273 K. After 4 h, the solvent was removed at room temperature. The solid was washed with H₂O. The product was obtained after recrystallization from a methanol/*n*-heptane mixture (4:1) after a slow evaporation at room temperature. IR (KBr, cm⁻¹): 3066, 2886, 1729 (C=O), 1592, 1481, 1359, 1286, 1218, 1150, 1009, 965, 863, 809, 698. ¹⁹F NMR (470.59 MHz, DMSO-d₆, 300 K, CFCl₃): -63.69 p.p.m. (s). ³¹P{¹H} NMR (202.46 MHz, DMSO-d₆, 300 K, 85% H₃PO₄): 12.80 p.p.m. (s). ¹H NMR (500.13 MHz, DMSO-d₆, 300 K, TMS): 2.48 (s, 3H, CH₃), 2.50 (s, 3H, CH₃), 4.16 (m, 4H, CH₂), 7.25–7.38 (m, 10H, Ar-H), 10.60 p.p.m. (s, 1H, NH). ¹³C NMR (125.76 MHz, DMSO-d₆, 300 K, TMS): 33.16 (d, ²J(P,C) = 4.6 Hz, 2 C, CH₃), 51.78 (d, ²J(P,C) = 5.1 Hz, 2 C, CH₂), 118.08 (dt, 1C, CClF₂), 127.17 (s), 127.96 (s), 128.32 (s), 137.68 (d, ³J(P,C) = 3.9 Hz, 2 C, C_{ipso}), 159.81 p.p.m. (t, ²J(F,C) = 35.0 Hz, 1 C, C=O).

Refinement

H atoms were placed in calculated positions with C—H = 0.95 - 0.99 Å and N—H = 0.87 Å and were included in the refinement with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The $U_{\text{iso}}(\text{H})$ value of the H atom bonded to N3 was refined.

Figures

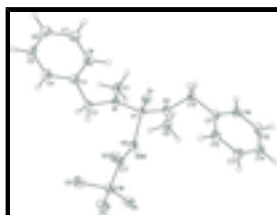


Fig. 1. The molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

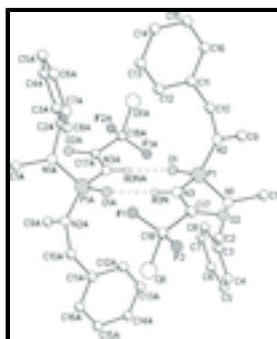


Fig. 2. A view of the H-bonded (dashed lines) centrosymmetric dimer (symmetry code: (A) 1-x, 1-y, -z).

N,N'-Dibenzyl-*N''*-(2-chloro-2,2-difluoroacetyl)-*N,N'*-dimethylphosphoric triamide

Crystal data

$\text{C}_{18}\text{H}_{21}\text{ClF}_2\text{N}_3\text{O}_2\text{P}$

$M_r = 415.80$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 10.3059$ (9) Å

$b = 10.5030$ (9) Å

$c = 10.9473$ (9) Å

$\alpha = 71.743$ (2)°

$\beta = 67.294$ (2)°

$\gamma = 63.265$ (2)°

$V = 962.15$ (14) Å³

$Z = 2$

$F(000) = 432$

$D_x = 1.435$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3946 reflections

$\theta = 2.3\text{--}29.1^\circ$

$\mu = 0.32$ mm⁻¹

$T = 120$ K

Prism, colourless

$0.28 \times 0.22 \times 0.15$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

4148 independent reflections

3359 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

φ and ω scans	$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.1^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1998)	$h = -13 \rightarrow 13$
$T_{\min} = 0.916$, $T_{\max} = 0.954$	$k = -13 \rightarrow 13$
9258 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: mixed
$wR(F^2) = 0.111$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2 + 1.6759P]$
4148 reflections	where $P = (F_o^2 + 2F_c^2)/3$
247 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.24133 (6)	0.55399 (6)	0.07097 (5)	0.01766 (13)
Cl1	0.40224 (7)	0.93801 (7)	-0.06620 (6)	0.03255 (16)
F1	0.45008 (15)	0.79512 (15)	0.15810 (14)	0.0304 (3)
F2	0.25207 (16)	0.99322 (15)	0.16926 (15)	0.0346 (3)
O1	0.37544 (16)	0.44767 (15)	-0.00684 (15)	0.0207 (3)
O2	0.09472 (17)	0.85802 (17)	0.16326 (17)	0.0290 (4)
N1	0.11179 (19)	0.66717 (19)	-0.00324 (17)	0.0194 (4)
N2	0.1560 (2)	0.47741 (19)	0.21311 (17)	0.0205 (4)
N3	0.3102 (2)	0.66075 (19)	0.09736 (18)	0.0197 (4)
H3N	0.4088	0.6297	0.0731	0.039 (8)*
C1	-0.0432 (3)	0.6755 (3)	0.0358 (2)	0.0300 (5)
H1A	-0.0678	0.6791	-0.0435	0.045*
H1B	-0.1129	0.7630	0.0751	0.045*
H1C	-0.0534	0.5902	0.1021	0.045*

supplementary materials

C2	0.1497 (3)	0.7723 (2)	-0.1223 (2)	0.0232 (4)
H2A	0.2436	0.7795	-0.1257	0.028*
H2B	0.0671	0.8685	-0.1125	0.028*
C3	0.1726 (2)	0.7347 (2)	-0.2540 (2)	0.0215 (4)
C4	0.1249 (2)	0.8454 (2)	-0.3561 (2)	0.0241 (5)
H4A	0.0693	0.9420	-0.3396	0.029*
C5	0.1583 (3)	0.8152 (3)	-0.4825 (2)	0.0281 (5)
H5A	0.1265	0.8912	-0.5521	0.034*
C6	0.2378 (3)	0.6744 (3)	-0.5067 (2)	0.0299 (5)
H6A	0.2612	0.6537	-0.5929	0.036*
C7	0.2835 (3)	0.5633 (3)	-0.4041 (2)	0.0293 (5)
H7A	0.3371	0.4666	-0.4204	0.035*
C8	0.2511 (3)	0.5929 (2)	-0.2784 (2)	0.0254 (5)
H8A	0.2825	0.5165	-0.2088	0.030*
C9	0.0398 (3)	0.5614 (2)	0.3182 (2)	0.0254 (5)
H9A	-0.0450	0.5282	0.3573	0.038*
H9B	0.0031	0.6641	0.2790	0.038*
H9C	0.0839	0.5478	0.3885	0.038*
C10	0.2044 (3)	0.3195 (2)	0.2526 (2)	0.0235 (4)
H10A	0.2779	0.2738	0.1736	0.028*
H10B	0.1151	0.2927	0.2808	0.028*
C11	0.2766 (2)	0.2604 (2)	0.3657 (2)	0.0217 (4)
C12	0.4040 (3)	0.2854 (2)	0.3528 (2)	0.0249 (5)
H12A	0.4465	0.3391	0.2720	0.030*
C13	0.4683 (3)	0.2321 (2)	0.4577 (2)	0.0263 (5)
H13A	0.5547	0.2497	0.4485	0.032*
C14	0.4077 (3)	0.1535 (2)	0.5757 (2)	0.0267 (5)
H14A	0.4521	0.1177	0.6473	0.032*
C15	0.2827 (3)	0.1271 (2)	0.5896 (2)	0.0279 (5)
H15A	0.2415	0.0723	0.6702	0.034*
C16	0.2175 (3)	0.1812 (2)	0.4847 (2)	0.0250 (5)
H16A	0.1309	0.1635	0.4948	0.030*
C17	0.2317 (2)	0.7958 (2)	0.1275 (2)	0.0213 (4)
C18	0.3323 (3)	0.8782 (2)	0.1073 (2)	0.0242 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0166 (3)	0.0174 (3)	0.0179 (3)	-0.0063 (2)	-0.0047 (2)	-0.0021 (2)
Cl1	0.0383 (3)	0.0335 (3)	0.0279 (3)	-0.0217 (3)	-0.0053 (2)	-0.0007 (2)
F1	0.0315 (7)	0.0306 (7)	0.0366 (8)	-0.0135 (6)	-0.0154 (6)	-0.0052 (6)
F2	0.0362 (8)	0.0289 (7)	0.0408 (8)	-0.0132 (6)	-0.0024 (6)	-0.0182 (6)
O1	0.0184 (7)	0.0203 (7)	0.0230 (8)	-0.0061 (6)	-0.0055 (6)	-0.0059 (6)
O2	0.0209 (8)	0.0242 (8)	0.0371 (9)	-0.0052 (7)	-0.0030 (7)	-0.0108 (7)
N1	0.0179 (8)	0.0201 (9)	0.0189 (9)	-0.0076 (7)	-0.0057 (7)	-0.0004 (7)
N2	0.0197 (9)	0.0193 (9)	0.0186 (8)	-0.0077 (7)	-0.0038 (7)	0.0002 (7)
N3	0.0167 (8)	0.0188 (9)	0.0237 (9)	-0.0056 (7)	-0.0065 (7)	-0.0044 (7)
C1	0.0195 (11)	0.0406 (14)	0.0272 (12)	-0.0104 (10)	-0.0092 (9)	-0.0002 (10)

C2	0.0294 (11)	0.0194 (10)	0.0213 (11)	-0.0098 (9)	-0.0103 (9)	0.0008 (8)
C3	0.0211 (10)	0.0259 (11)	0.0195 (10)	-0.0124 (9)	-0.0057 (8)	-0.0013 (8)
C4	0.0208 (10)	0.0270 (11)	0.0239 (11)	-0.0105 (9)	-0.0070 (9)	-0.0005 (9)
C5	0.0277 (12)	0.0373 (13)	0.0210 (11)	-0.0146 (10)	-0.0114 (9)	0.0023 (9)
C6	0.0314 (12)	0.0449 (14)	0.0208 (11)	-0.0212 (11)	-0.0062 (9)	-0.0060 (10)
C7	0.0321 (12)	0.0300 (12)	0.0285 (12)	-0.0155 (10)	-0.0046 (10)	-0.0079 (10)
C8	0.0282 (11)	0.0242 (11)	0.0246 (11)	-0.0125 (9)	-0.0084 (9)	0.0001 (9)
C9	0.0250 (11)	0.0295 (12)	0.0173 (10)	-0.0114 (9)	-0.0016 (9)	-0.0023 (9)
C10	0.0275 (11)	0.0203 (10)	0.0243 (11)	-0.0116 (9)	-0.0102 (9)	0.0016 (8)
C11	0.0236 (10)	0.0171 (10)	0.0237 (11)	-0.0063 (8)	-0.0084 (9)	-0.0025 (8)
C12	0.0253 (11)	0.0253 (11)	0.0236 (11)	-0.0119 (9)	-0.0055 (9)	-0.0020 (9)
C13	0.0222 (11)	0.0258 (11)	0.0317 (12)	-0.0078 (9)	-0.0096 (9)	-0.0054 (9)
C14	0.0273 (11)	0.0249 (11)	0.0264 (11)	-0.0042 (9)	-0.0125 (9)	-0.0051 (9)
C15	0.0308 (12)	0.0236 (11)	0.0241 (11)	-0.0102 (10)	-0.0080 (9)	0.0026 (9)
C16	0.0242 (11)	0.0245 (11)	0.0268 (11)	-0.0116 (9)	-0.0092 (9)	0.0013 (9)
C17	0.0235 (11)	0.0205 (10)	0.0192 (10)	-0.0088 (9)	-0.0054 (8)	-0.0025 (8)
C18	0.0249 (11)	0.0205 (10)	0.0261 (11)	-0.0072 (9)	-0.0055 (9)	-0.0068 (9)

Geometric parameters (Å, °)

P1—O1	1.4769 (15)	C5—H5A	0.9500
P1—N2	1.6241 (18)	C6—C7	1.391 (3)
P1—N1	1.6308 (18)	C6—H6A	0.9500
P1—N3	1.7093 (18)	C7—C8	1.387 (3)
C11—C18	1.768 (2)	C7—H7A	0.9500
F1—C18	1.345 (3)	C8—H8A	0.9500
F2—C18	1.336 (2)	C9—H9A	0.9800
O2—C17	1.215 (3)	C9—H9B	0.9800
N1—C1	1.453 (3)	C9—H9C	0.9800
N1—C2	1.471 (3)	C10—C11	1.512 (3)
N2—C9	1.469 (3)	C10—H10A	0.9900
N2—C10	1.469 (3)	C10—H10B	0.9900
N3—C17	1.353 (3)	C11—C16	1.387 (3)
N3—H3N	0.8700	C11—C12	1.399 (3)
C1—H1A	0.9800	C12—C13	1.387 (3)
C1—H1B	0.9800	C12—H12A	0.9500
C1—H1C	0.9800	C13—C14	1.383 (3)
C2—C3	1.517 (3)	C13—H13A	0.9500
C2—H2A	0.9900	C14—C15	1.379 (3)
C2—H2B	0.9900	C14—H14A	0.9500
C3—C4	1.393 (3)	C15—C16	1.391 (3)
C3—C8	1.394 (3)	C15—H15A	0.9500
C4—C5	1.394 (3)	C16—H16A	0.9500
C4—H4A	0.9500	C17—C18	1.543 (3)
C5—C6	1.384 (4)		
O1—P1—N2	112.34 (9)	C7—C8—C3	120.0 (2)
O1—P1—N1	117.13 (9)	C7—C8—H8A	120.0
N2—P1—N1	107.02 (9)	C3—C8—H8A	120.0
O1—P1—N3	105.06 (9)	N2—C9—H9A	109.5

supplementary materials

N2—P1—N3	110.57 (9)	N2—C9—H9B	109.5
N1—P1—N3	104.38 (9)	H9A—C9—H9B	109.5
C1—N1—C2	114.36 (17)	N2—C9—H9C	109.5
C1—N1—P1	126.21 (15)	H9A—C9—H9C	109.5
C2—N1—P1	119.42 (14)	H9B—C9—H9C	109.5
C9—N2—C10	115.45 (17)	N2—C10—C11	113.28 (18)
C9—N2—P1	121.56 (14)	N2—C10—H10A	108.9
C10—N2—P1	122.42 (15)	C11—C10—H10A	108.9
C17—N3—P1	127.29 (15)	N2—C10—H10B	108.9
C17—N3—H3N	117.5	C11—C10—H10B	108.9
P1—N3—H3N	114.0	H10A—C10—H10B	107.7
N1—C1—H1A	109.5	C16—C11—C12	118.6 (2)
N1—C1—H1B	109.5	C16—C11—C10	120.9 (2)
H1A—C1—H1B	109.5	C12—C11—C10	120.59 (19)
N1—C1—H1C	109.5	C13—C12—C11	120.2 (2)
H1A—C1—H1C	109.5	C13—C12—H12A	119.9
H1B—C1—H1C	109.5	C11—C12—H12A	119.9
N1—C2—C3	114.05 (17)	C14—C13—C12	120.4 (2)
N1—C2—H2A	108.7	C14—C13—H13A	119.8
C3—C2—H2A	108.7	C12—C13—H13A	119.8
N1—C2—H2B	108.7	C15—C14—C13	120.1 (2)
C3—C2—H2B	108.7	C15—C14—H14A	120.0
H2A—C2—H2B	107.6	C13—C14—H14A	120.0
C4—C3—C8	119.4 (2)	C14—C15—C16	119.6 (2)
C4—C3—C2	119.3 (2)	C14—C15—H15A	120.2
C8—C3—C2	121.15 (19)	C16—C15—H15A	120.2
C3—C4—C5	120.3 (2)	C11—C16—C15	121.2 (2)
C3—C4—H4A	119.8	C11—C16—H16A	119.4
C5—C4—H4A	119.8	C15—C16—H16A	119.4
C6—C5—C4	120.1 (2)	O2—C17—N3	127.4 (2)
C6—C5—H5A	120.0	O2—C17—C18	118.39 (19)
C4—C5—H5A	120.0	N3—C17—C18	114.16 (18)
C5—C6—C7	119.7 (2)	F2—C18—F1	107.18 (18)
C5—C6—H6A	120.1	F2—C18—C17	110.49 (18)
C7—C6—H6A	120.1	F1—C18—C17	111.98 (17)
C8—C7—C6	120.5 (2)	F2—C18—C11	108.70 (15)
C8—C7—H7A	119.8	F1—C18—C11	109.06 (15)
C6—C7—H7A	119.8	C17—C18—C11	109.35 (15)
O1—P1—N1—C1	115.12 (19)	C6—C7—C8—C3	-0.1 (3)
N2—P1—N1—C1	-12.0 (2)	C4—C3—C8—C7	1.2 (3)
N3—P1—N1—C1	-129.24 (19)	C2—C3—C8—C7	-173.7 (2)
O1—P1—N1—C2	-64.27 (18)	C9—N2—C10—C11	-61.8 (2)
N2—P1—N1—C2	168.61 (15)	P1—N2—C10—C11	109.64 (19)
N3—P1—N1—C2	51.38 (17)	N2—C10—C11—C16	122.1 (2)
O1—P1—N2—C9	167.42 (16)	N2—C10—C11—C12	-57.4 (3)
N1—P1—N2—C9	-62.69 (18)	C16—C11—C12—C13	-0.2 (3)
N3—P1—N2—C9	50.40 (19)	C10—C11—C12—C13	179.2 (2)
O1—P1—N2—C10	-3.55 (19)	C11—C12—C13—C14	0.2 (3)
N1—P1—N2—C10	126.34 (17)	C12—C13—C14—C15	0.3 (3)

N3—P1—N2—C10	-120.57 (16)	C13—C14—C15—C16	-0.6 (3)
O1—P1—N3—C17	158.89 (18)	C12—C11—C16—C15	-0.1 (3)
N2—P1—N3—C17	-79.68 (19)	C10—C11—C16—C15	-179.6 (2)
N1—P1—N3—C17	35.1 (2)	C14—C15—C16—C11	0.6 (4)
C1—N1—C2—C3	-75.1 (2)	P1—N3—C17—O2	13.3 (3)
P1—N1—C2—C3	104.32 (19)	P1—N3—C17—C18	-164.20 (15)
N1—C2—C3—C4	143.87 (19)	O2—C17—C18—F2	17.7 (3)
N1—C2—C3—C8	-41.2 (3)	N3—C17—C18—F2	-164.59 (18)
C8—C3—C4—C5	-1.5 (3)	O2—C17—C18—F1	137.1 (2)
C2—C3—C4—C5	173.5 (2)	N3—C17—C18—F1	-45.2 (2)
C3—C4—C5—C6	0.7 (3)	O2—C17—C18—C11	-101.9 (2)
C4—C5—C6—C7	0.4 (3)	N3—C17—C18—C11	75.8 (2)
C5—C6—C7—C8	-0.6 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N3—H3N \cdots O1 ⁱ	0.87	1.91	2.772 (3)	174

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

Fig. 1

