

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# *N,N,N',N'*-Tetraethyl-*N''*-(2-fluorobenzoyl)phosphoric triamide

 Atekeh Tarahhomi,<sup>a</sup> Mehrdad Pourayoubi,<sup>a\*</sup> Arnold L. Rheingold<sup>b</sup> and James A. Golen<sup>b</sup>
<sup>a</sup>Department of Chemistry, Ferdowsi University of Mashhad, Mashhad 91779, Iran, and

<sup>b</sup>Department of Chemistry, University of California, San Diego, 9500 Gilman Drive, La Jolla, CA 92093, USA

Correspondence e-mail: mehrdad\_pourayoubi@yahoo.com

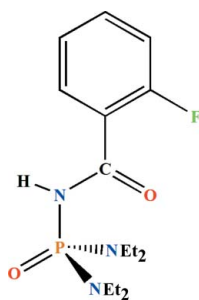
Received 6 August 2011; accepted 12 September 2011

 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.047;  $wR$  factor = 0.122; data-to-parameter ratio = 15.9.

In the title compound,  $\text{C}_{15}\text{H}_{25}\text{FN}_3\text{O}_2\text{P}$ , the phosphoryl group is in an *anti* and *syn* orientation to the  $\text{C}=\text{O}$  and  $\text{N}-\text{H}$  groups, respectively. The P atom is in a distorted tetrahedral environment. One of the ethyl groups is disordered over two sets of sites with refined occupancies of 0.755 (6) and 0.245 (6). In addition, the F atom was refined as disordered with occupancies fixed at 0.9 and 0.1. This disorder corresponds to a rotation of approximately  $180^\circ$  of the fluorobenzene ring about its connecting  $\text{C}-\text{C}$  bond. In the crystal, pairs of intermolecular  $\text{N}-\text{H}\cdots\text{O}(\text{=P})$  hydrogen bonds form centrosymmetric dimers.

## Related literature

For background to phosphoric triamide compounds containing a  $\text{C}(\text{=O})\text{NHP}(\text{=O})$  skeleton, see: Pourayoubi *et al.* (2011a,b); Tarahhomi *et al.* (2011). For the synthesis of the starting material 2-F- $\text{C}_6\text{H}_4\text{C}(\text{=O})\text{NHP}(\text{=O})\text{Cl}_2$ , see: Pourayoubi *et al.* (2011a). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



## Experimental

### Crystal data

 $\text{C}_{15}\text{H}_{25}\text{FN}_3\text{O}_2\text{P}$ 
 $M_r = 329.35$ 

 Monoclinic,  $P2_1/c$ 
 $a = 10.9296$  (9) Å

 $b = 12.2221$  (10) Å

 $c = 12.3423$  (10) Å

 $\beta = 91.443$  (1) $^\circ$ 
 $V = 1648.2$  (2) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.19$  mm<sup>-1</sup>
 $T = 100$  K

 $0.45 \times 0.40 \times 0.35$  mm

### Data collection

Bruker APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.920$ ,  $T_{\max} = 0.937$ 

11336 measured reflections

3741 independent reflections

 3056 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.051$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$ 
 $wR(F^2) = 0.122$ 
 $S = 1.08$ 

3741 reflections

236 parameters

6 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.55$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>
**Table 1**

 Hydrogen-bond geometry (Å,  $^\circ$ ).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.86 (2)	1.93 (2)	2.7714 (19)	167 (2)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and enCIFer (Allen *et al.*, 2004).

Support of this investigation by Ferdowsi University of Mashhad is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5308).

## References

- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). SADABS, SAINT and APEX2. Bruker AXS Inc., Madison, Wisconsin, USA.
- Pourayoubi, M., Tarahhomi, A., Rheingold, A. L. & Golen, J. A. (2011a). *Acta Cryst.* **E67**, o934.
- Pourayoubi, M., Tarahhomi, A., Saneei, A., Rheingold, A. L. & Golen, J. A. (2011b). *Acta Cryst.* **C67**, o265–o272.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tarahhomi, A., Pourayoubi, M., Rheingold, A. L. & Golen, J. A. (2011). *Struct. Chem.* **22**, 201–210.

## supporting information

*Acta Cryst.* (2011). E67, o2643 [https://doi.org/10.1107/S1600536811036944]

***N,N,N',N'*-Tetraethyl-*N''*-(2-fluorobenzoyl)phosphoric triamide****Atekeh Tarahhomi, Mehrdad Pourayoubi, Arnold L. Rheingold and James A. Golen****S1. Comment**

Following our previous work on phosphoric triamides containing an XC(O)NHP(O) moiety,  $X = 2\text{-F-C}_6\text{H}_4$  (Pourayoubi *et al.*, 2011*a*),  $4\text{-F-C}_6\text{H}_4$  (Tarahhomi *et al.*, 2011) and  $2,6\text{-F}_2\text{-C}_6\text{H}_3$  (Pourayoubi *et al.*, 2011*b*), herein, we report the synthesis and crystal structure of the title compound,  $\text{P}(\text{O})[2\text{-F-C}_6\text{H}_4\text{C}(\text{O})\text{NH}][\text{N}(\text{C}_2\text{H}_5)_2]_2$  (Fig. 1).

One of the  $-\text{CH}_2\text{CH}_3$  groups in the diethylamido substituent N2 is disordered over two sets of sites with refined occupancies of 0.755 (6) and 0.245 (6). The fluorine atom of the aromatic ring is disordered over two sets of sites with occupancies of 0.9 and 0.1.

In the  $\text{C}(\text{=O})\text{NHP}(\text{=O})$  skeleton of the title phosphoric triamide, the phosphoryl group adopts the *anti* orientation with respect to the carbonyl group; whereas it is in a *syn* position relative to the  $\text{N-H}$  unit. The tetrahedral environment at the P atom is distorted as has been noted for the other phosphoric triamides. The  $\text{P=O}$ ,  $\text{C=O}$  and  $\text{P-N}$  bond lengths are within the expected values (Pourayoubi *et al.* (2011*b*) and Tarahhomi *et al.* (2011)).

In the crystal, pairs of intermolecular  $\text{N-H}\cdots\text{O}(\text{P})$  hydrogen bonds form a hydrogen-bonded dimer with  $R_2^2(8)$  graph-set notation (Bernstein *et al.*, 1995).

**S2. Experimental**

$2\text{-F-C}_6\text{H}_4\text{C}(\text{O})\text{NHP}(\text{O})\text{Cl}_2$  was prepared according to the literature method reported by Pourayoubi *et al.* (2011*a*).

The title compound was synthesized from the reaction of 0.40 g (1.56 mmol)  $2\text{-F-C}_6\text{H}_4\text{C}(\text{O})\text{NHP}(\text{O})\text{Cl}_2$  with 0.456 g (6.24 mmol) of diethyl amine in dry chloroform (30 ml). The amine was added dropwise to a solution of  $2\text{-F-C}_6\text{H}_4\text{C}(\text{O})\text{NHP}(\text{O})\text{Cl}_2$  at 273 K, with continuous stirring. After 17 h, the solvent was evaporated; the obtained solid was washed with distilled water and dissolved in a mixture of  $\text{CH}_3\text{OH}/\text{DMF}$  [4:1 (v/v)]. Single crystals were obtained from this solution after a few days at room temperature. IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3061 (NH), 2970, 2886, 1675 ( $\text{C=O}$ ), 1457, 1196, 1048, 946, 766.

**S3. Refinement**

Atoms C8 and C9 are disordered over two sets of sites with refined occupancies of 0.755 (6) and 0.245 (6). The fluorine atom is disordered over two positions and refined with the occupancy F1 and F1A fixed at 0.9 and 0.1 and the corresponding hydrogen atoms H1A and H5A were also treated as disordered. Hydrogen H1N was found in a Fourier difference map and refined with a  $\text{N1-H1N}$  distance was set at 0.87 (0.02) Å and allowed to refine with  $U_{\text{iso}}$  at 1.2 of parent N atom. All other hydrogen atoms were placed in calculated positions with  $\text{C-H}$  distances for  $\text{CH}_2$  of 0.99 Å,  $\text{CH}_3$  of 0.98 Å and  $\text{C}(\text{Ar})\text{H}$  of 0.95 Å and with  $U_{\text{iso}}$  of 1.20 (or 1.5 for methyl H atoms) that of the parent C atom.

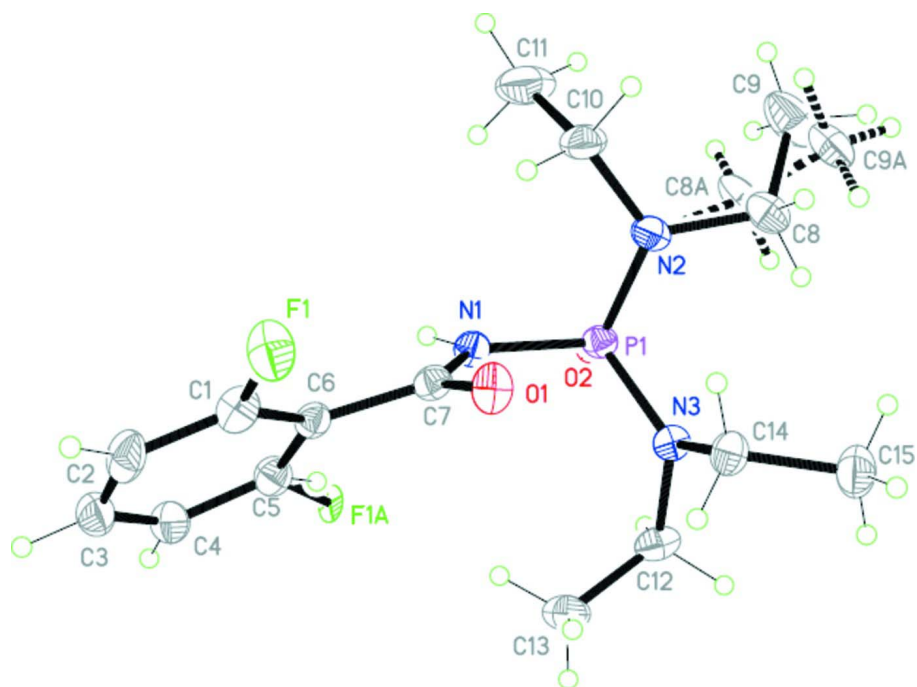


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are given at 50% probability level and H atoms are drawn as small spheres of arbitrary radii. Disordered atoms are labeled with the suffix 'A'.

#### *N,N,N',N'*-Tetraethyl-*N''*-(2-fluorobenzoyl)phosphoric triamide

##### Crystal data

$C_{15}H_{25}FN_3O_2P$

$M_r = 329.35$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 10.9296\ (9)\ \text{\AA}$

$b = 12.2221\ (10)\ \text{\AA}$

$c = 12.3423\ (10)\ \text{\AA}$

$\beta = 91.443\ (1)^\circ$

$V = 1648.2\ (2)\ \text{\AA}^3$

$Z = 4$

$F(000) = 704$

$D_x = 1.327\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 6938 reflections

$\theta = 2.5\text{--}27.9^\circ$

$\mu = 0.19\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Block, colourless

$0.45 \times 0.40 \times 0.35\ \text{mm}$

##### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.920$ ,  $T_{\max} = 0.937$

11336 measured reflections

3741 independent reflections

3056 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\max} = 28.0^\circ$ ,  $\theta_{\min} = 1.9^\circ$

$h = -14 \rightarrow 7$

$k = -16 \rightarrow 14$

$l = -15 \rightarrow 16$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.047$  $wR(F^2) = 0.122$  $S = 1.08$ 

3741 reflections

236 parameters

6 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.7763P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$ *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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
P1	0.62104 (4)	0.64819 (4)	0.52994 (4)	0.01901 (14)	
F1	0.88953 (13)	0.41423 (11)	0.83105 (10)	0.0338 (3)	0.90
F1A	0.7449 (9)	0.3460 (7)	0.4748 (4)	0.019 (2)	0.10
O1	0.83305 (12)	0.57884 (11)	0.68306 (10)	0.0259 (3)	
O2	0.52742 (11)	0.62160 (11)	0.44464 (10)	0.0230 (3)	
N1	0.66071 (13)	0.52777 (13)	0.58794 (12)	0.0199 (3)	
H1N	0.6116 (17)	0.4741 (15)	0.5750 (16)	0.024*	
N2	0.57927 (14)	0.72748 (13)	0.62914 (12)	0.0260 (4)	
N3	0.73894 (13)	0.70810 (12)	0.47587 (11)	0.0201 (3)	
C1	0.86735 (17)	0.34892 (16)	0.74688 (15)	0.0254 (4)	
H1A	0.8811	0.3968	0.8066	0.030*	0.10
C2	0.91255 (17)	0.24343 (17)	0.75327 (17)	0.0303 (5)	
H2	0.9557	0.2194	0.8167	0.036*	
C3	0.89453 (18)	0.17367 (17)	0.66716 (17)	0.0300 (4)	
H3	0.9265	0.1014	0.6704	0.036*	
C4	0.82999 (18)	0.20843 (16)	0.57575 (16)	0.0289 (4)	
H4	0.8176	0.1603	0.5161	0.035*	
C5	0.78342 (17)	0.31405 (16)	0.57156 (15)	0.0243 (4)	
H5A	0.7374	0.3366	0.5092	0.029*	0.90
C6	0.80224 (15)	0.38782 (15)	0.65610 (14)	0.0200 (4)	
C7	0.76718 (16)	0.50656 (15)	0.64574 (13)	0.0203 (4)	
C8	0.5579 (3)	0.8451 (2)	0.6092 (2)	0.0279 (7)	0.755 (6)
H8A	0.5829	0.8629	0.5347	0.034*	0.755 (6)
H8B	0.6103	0.8880	0.6601	0.034*	0.755 (6)

C9	0.4260 (3)	0.8792 (3)	0.6222 (3)	0.0463 (10)	0.755 (6)
H9A	0.4164	0.9562	0.6016	0.069*	0.755 (6)
H9B	0.4033	0.8698	0.6980	0.069*	0.755 (6)
H9C	0.3728	0.8338	0.5756	0.069*	0.755 (6)
C8A	0.4916 (9)	0.8158 (7)	0.5807 (7)	0.032 (2)	0.245 (6)
H8AA	0.5086	0.8280	0.5032	0.038*	0.245 (6)
H8AB	0.4054	0.7922	0.5867	0.038*	0.245 (6)
C9A	0.5141 (8)	0.9196 (7)	0.6449 (7)	0.033 (3)	0.245 (6)
H9AA	0.4629	0.9785	0.6147	0.049*	0.245 (6)
H9AB	0.6005	0.9403	0.6409	0.049*	0.245 (6)
H9AC	0.4935	0.9073	0.7208	0.049*	0.245 (6)
C10	0.54246 (19)	0.68425 (18)	0.73515 (15)	0.0297 (4)	
H10A	0.5370	0.7459	0.7868	0.036*	
H10B	0.6071	0.6341	0.7629	0.036*	
C11	0.4206 (2)	0.6232 (2)	0.7324 (2)	0.0485 (6)	
H11A	0.3562	0.6713	0.7029	0.073*	
H11B	0.4002	0.6010	0.8061	0.073*	
H11C	0.4271	0.5582	0.6865	0.073*	
C12	0.78030 (17)	0.66718 (17)	0.37025 (15)	0.0260 (4)	
H12A	0.8211	0.7276	0.3318	0.031*	
H12B	0.7077	0.6450	0.3259	0.031*	
C13	0.86787 (19)	0.57083 (18)	0.37903 (18)	0.0358 (5)	
H13A	0.9365	0.5897	0.4281	0.054*	
H13B	0.8989	0.5536	0.3072	0.054*	
H13C	0.8247	0.5071	0.4074	0.054*	
C14	0.82939 (17)	0.77616 (15)	0.53521 (15)	0.0241 (4)	
H14A	0.9105	0.7403	0.5329	0.029*	
H14B	0.8062	0.7812	0.6121	0.029*	
C15	0.8391 (2)	0.89111 (17)	0.48869 (18)	0.0343 (5)	
H15A	0.8554	0.8867	0.4111	0.051*	
H15B	0.9060	0.9304	0.5259	0.051*	
H15C	0.7620	0.9303	0.4991	0.051*	

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
P1	0.0162 (2)	0.0221 (3)	0.0187 (2)	0.00183 (17)	0.00024 (16)	0.00116 (17)
F1	0.0485 (8)	0.0294 (7)	0.0229 (6)	0.0034 (6)	-0.0118 (6)	-0.0017 (5)
F1A	0.025 (5)	0.014 (5)	0.018 (5)	0.003 (4)	-0.003 (4)	0.003 (4)
O1	0.0268 (7)	0.0235 (7)	0.0271 (7)	-0.0039 (6)	-0.0082 (5)	0.0007 (5)
O2	0.0172 (6)	0.0280 (7)	0.0236 (6)	-0.0007 (5)	-0.0028 (5)	0.0052 (5)
N1	0.0158 (7)	0.0218 (8)	0.0220 (7)	-0.0009 (6)	-0.0011 (5)	0.0005 (6)
N2	0.0284 (8)	0.0253 (8)	0.0245 (8)	0.0068 (7)	0.0064 (6)	0.0005 (6)
N3	0.0192 (7)	0.0224 (8)	0.0186 (7)	-0.0011 (6)	-0.0006 (5)	-0.0006 (6)
C1	0.0255 (9)	0.0283 (10)	0.0221 (9)	-0.0044 (8)	-0.0043 (7)	0.0018 (7)
C2	0.0247 (9)	0.0319 (11)	0.0338 (11)	0.0001 (8)	-0.0069 (8)	0.0140 (9)
C3	0.0236 (10)	0.0250 (10)	0.0414 (12)	0.0022 (8)	0.0037 (8)	0.0071 (9)
C4	0.0309 (10)	0.0237 (10)	0.0323 (10)	-0.0007 (8)	0.0043 (8)	-0.0004 (8)

C5	0.0254 (9)	0.0253 (10)	0.0222 (9)	0.0001 (8)	-0.0007 (7)	0.0025 (7)
C6	0.0152 (8)	0.0223 (9)	0.0223 (9)	-0.0007 (7)	0.0006 (6)	0.0043 (7)
C7	0.0198 (8)	0.0241 (9)	0.0170 (8)	-0.0002 (7)	0.0011 (6)	0.0021 (7)
C8	0.0256 (16)	0.0286 (15)	0.0294 (15)	0.0069 (12)	-0.0018 (12)	-0.0034 (11)
C9	0.0295 (17)	0.0396 (19)	0.069 (2)	0.0107 (14)	-0.0061 (14)	-0.0157 (16)
C8A	0.036 (5)	0.033 (5)	0.026 (4)	0.020 (4)	-0.017 (4)	-0.011 (4)
C9A	0.038 (5)	0.029 (5)	0.031 (4)	0.013 (4)	-0.003 (4)	-0.007 (4)
C10	0.0339 (11)	0.0328 (11)	0.0229 (10)	-0.0025 (9)	0.0099 (8)	-0.0048 (8)
C11	0.0439 (14)	0.0531 (16)	0.0498 (14)	-0.0144 (12)	0.0241 (11)	-0.0154 (12)
C12	0.0223 (9)	0.0356 (11)	0.0203 (9)	-0.0060 (8)	0.0034 (7)	-0.0037 (8)
C13	0.0316 (11)	0.0344 (12)	0.0420 (12)	-0.0005 (9)	0.0155 (9)	-0.0062 (9)
C14	0.0246 (9)	0.0221 (9)	0.0255 (9)	-0.0045 (8)	-0.0025 (7)	-0.0002 (7)
C15	0.0424 (12)	0.0231 (10)	0.0375 (11)	-0.0042 (9)	0.0012 (9)	0.0017 (9)

*Geometric parameters (Å, °)*

P1—O2	1.4855 (13)	C9—H9A	0.9800
P1—N2	1.6354 (16)	C9—H9B	0.9800
P1—N3	1.6387 (15)	C9—H9C	0.9800
P1—N1	1.6885 (16)	C8A—C9A	1.513 (11)
O1—C7	1.222 (2)	C8A—H8AA	0.9900
N1—C7	1.374 (2)	C8A—H8AB	0.9900
N1—H1N	0.860 (15)	C9A—H9AA	0.9800
N2—C8	1.476 (3)	C9A—H9AB	0.9800
N2—C10	1.476 (2)	C9A—H9AC	0.9800
N2—C8A	1.553 (7)	C10—C11	1.526 (3)
N3—C14	1.473 (2)	C10—H10A	0.9900
N3—C12	1.478 (2)	C10—H10B	0.9900
C1—C2	1.382 (3)	C11—H11A	0.9800
C1—C6	1.396 (2)	C11—H11B	0.9800
C1—H1A	0.9500	C11—H11C	0.9800
C2—C3	1.373 (3)	C12—C13	1.520 (3)
C2—H2	0.9500	C12—H12A	0.9900
C3—C4	1.382 (3)	C12—H12B	0.9900
C3—H3	0.9500	C13—H13A	0.9800
C4—C5	1.388 (3)	C13—H13B	0.9800
C4—H4	0.9500	C13—H13C	0.9800
C5—C6	1.390 (3)	C14—C15	1.522 (3)
C5—H5A	0.9500	C14—H14A	0.9900
C6—C7	1.506 (3)	C14—H14B	0.9900
C8—C9	1.514 (4)	C15—H15A	0.9800
C8—H8A	0.9900	C15—H15B	0.9800
C8—H8B	0.9900	C15—H15C	0.9800
O2—P1—N2	117.38 (8)	N2—C8A—H8AA	110.3
O2—P1—N3	110.11 (7)	C9A—C8A—H8AB	110.3
N2—P1—N3	106.10 (8)	N2—C8A—H8AB	110.3
O2—P1—N1	105.95 (8)	H8AA—C8A—H8AB	108.6

N2—P1—N1	105.82 (8)	C8A—C9A—H9AA	109.5
N3—P1—N1	111.47 (8)	C8A—C9A—H9AB	109.5
C7—N1—P1	126.13 (13)	H9AA—C9A—H9AB	109.5
C7—N1—H1N	118.0 (14)	C8A—C9A—H9AC	109.5
P1—N1—H1N	115.7 (14)	H9AA—C9A—H9AC	109.5
C8—N2—C10	116.79 (17)	H9AB—C9A—H9AC	109.5
C8—N2—C8A	33.4 (4)	N2—C10—C11	114.35 (18)
C10—N2—C8A	114.2 (4)	N2—C10—H10A	108.7
C8—N2—P1	119.88 (15)	C11—C10—H10A	108.7
C10—N2—P1	122.61 (14)	N2—C10—H10B	108.7
C8A—N2—P1	107.7 (3)	C11—C10—H10B	108.7
C14—N3—C12	114.42 (14)	H10A—C10—H10B	107.6
C14—N3—P1	125.08 (12)	C10—C11—H11A	109.5
C12—N3—P1	117.98 (12)	C10—C11—H11B	109.5
C2—C1—C6	122.54 (18)	H11A—C11—H11B	109.5
C2—C1—H1A	118.7	C10—C11—H11C	109.5
C6—C1—H1A	118.7	H11A—C11—H11C	109.5
C3—C2—C1	119.41 (18)	H11B—C11—H11C	109.5
C3—C2—H2	120.3	N3—C12—C13	113.92 (16)
C1—C2—H2	120.3	N3—C12—H12A	108.8
C2—C3—C4	120.08 (19)	C13—C12—H12A	108.8
C2—C3—H3	120.0	N3—C12—H12B	108.8
C4—C3—H3	120.0	C13—C12—H12B	108.8
C3—C4—C5	119.69 (19)	H12A—C12—H12B	107.7
C3—C4—H4	120.2	C12—C13—H13A	109.5
C5—C4—H4	120.2	C12—C13—H13B	109.5
C4—C5—C6	121.87 (17)	H13A—C13—H13B	109.5
C4—C5—H5A	119.1	C12—C13—H13C	109.5
C6—C5—H5A	119.1	H13A—C13—H13C	109.5
C5—C6—C1	116.39 (17)	H13B—C13—H13C	109.5
C5—C6—C7	121.92 (15)	N3—C14—C15	112.69 (16)
C1—C6—C7	121.34 (16)	N3—C14—H14A	109.1
O1—C7—N1	122.84 (17)	C15—C14—H14A	109.1
O1—C7—C6	121.22 (15)	N3—C14—H14B	109.1
N1—C7—C6	115.87 (15)	C15—C14—H14B	109.1
N2—C8—C9	113.5 (2)	H14A—C14—H14B	107.8
N2—C8—H8A	108.9	C14—C15—H15A	109.5
C9—C8—H8A	108.9	C14—C15—H15B	109.5
N2—C8—H8B	108.9	H15A—C15—H15B	109.5
C9—C8—H8B	108.9	C14—C15—H15C	109.5
H8A—C8—H8B	107.7	H15A—C15—H15C	109.5
C9A—C8A—N2	106.9 (6)	H15B—C15—H15C	109.5
C9A—C8A—H8AA	110.3		
O2—P1—N1—C7	158.75 (14)	C4—C5—C6—C7	171.39 (17)
N2—P1—N1—C7	-75.93 (16)	C2—C1—C6—C5	0.7 (3)
N3—P1—N1—C7	38.97 (17)	C2—C1—C6—C7	-172.59 (17)
O2—P1—N2—C8	-72.0 (2)	P1—N1—C7—O1	17.5 (3)

N3—P1—N2—C8	51.5 (2)	P1—N1—C7—C6	-159.56 (12)
N1—P1—N2—C8	170.05 (18)	C5—C6—C7—O1	-139.50 (18)
O2—P1—N2—C10	97.97 (16)	C1—C6—C7—O1	33.4 (3)
N3—P1—N2—C10	-138.49 (15)	C5—C6—C7—N1	37.6 (2)
N1—P1—N2—C10	-19.96 (17)	C1—C6—C7—N1	-149.49 (17)
O2—P1—N2—C8A	-37.8 (5)	C10—N2—C8—C9	-56.1 (3)
N3—P1—N2—C8A	85.7 (5)	C8A—N2—C8—C9	37.9 (6)
N1—P1—N2—C8A	-155.7 (5)	P1—N2—C8—C9	114.5 (2)
O2—P1—N3—C14	159.56 (14)	C8—N2—C8A—C9A	-28.3 (5)
N2—P1—N3—C14	31.58 (16)	C10—N2—C8A—C9A	74.1 (8)
N1—P1—N3—C14	-83.15 (16)	P1—N2—C8A—C9A	-146.0 (6)
O2—P1—N3—C12	-39.48 (15)	C8—N2—C10—C11	100.8 (2)
N2—P1—N3—C12	-167.46 (13)	C8A—N2—C10—C11	63.7 (5)
N1—P1—N3—C12	77.81 (15)	P1—N2—C10—C11	-69.5 (2)
C6—C1—C2—C3	0.7 (3)	C14—N3—C12—C13	77.6 (2)
C1—C2—C3—C4	-1.0 (3)	P1—N3—C12—C13	-85.37 (18)
C2—C3—C4—C5	-0.1 (3)	C12—N3—C14—C15	75.8 (2)
C3—C4—C5—C6	1.6 (3)	P1—N3—C14—C15	-122.62 (16)
C4—C5—C6—C1	-1.9 (3)		

*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>
N1—H1N $\cdots$ O2 <sup>i</sup>	0.86 (2)	1.93 (2)	2.7714 (19)	167 (2)

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