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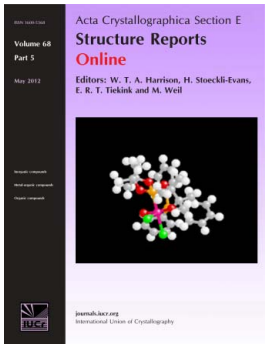
# *N*,*N*′-Dibenzyl-*N*″-(2-chloro-2,2-difluoroacetyl)phosphoric triamide

**Mehrdad Pourayoubi, Mojtaba Keikha, Jerry P. Jasinski and Amanda C. Keeley**

*Acta Cryst.* (2012). E**68**, o3009





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# *N*,*N*′-Dibenzyl-*N*″-(2-chloro-2,2-difluoroacetyl)phosphoric triamide

**Mehrdad Pourayoubi,[a]* Mojtaba Keikha,[a] Jerry P. Jasinski[b] and Amanda C. Keeley[b]**

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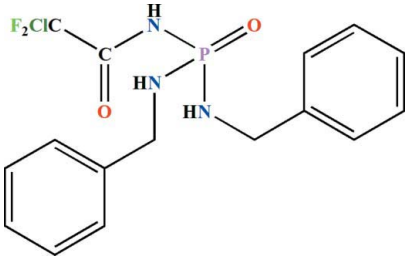
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.051; $wR$ factor = 0.122; data-to-parameter ratio = 20.8.

In the title molecule, $C_{16}H_{17}ClF_2N_3O_2P$, the N—H unit of the C(═O)NHP(═O) fragment adopts a *syn* orientation with respect to the P═O group. The two F atoms and the Cl atom of the $ClF_2C$ group are disordered over two sets of sites with refined occupancies of 0.605 (6) and 0.395 (6). In the crystal, molecules are linked *via* N—H···O═C hydrogen bonds and the (N—H···)(N—H···)O═P group into chains along [010].

## Related literature

For related structures with a P(═O)[NHC(═O)$CClF_2$] fragment, and for reference values of P═O, C═O and P—N bond lengths and P—N—C bond angles, see: Pourayoubi *et al.* (2011); Raissi Shabari *et al.* (2011); Pourayoubi & Saneei (2011). For the double hydrogen-bond acceptor capability of the phosphoryl O atom in phosphoramidates, see: Pourayoubi *et al.* (2012). For the synthesis of the starting material, $CClF_2C$(═O)NHP(═O)$Cl_2$, see: Iriarte *et al.* (2008).




## Experimental

### *Crystal data*

$C_{16}H_{17}ClF_2N_3O_2P$
$M_r$ = 387.75
Monoclinic, $P2_1$
$a$ = 12.9734 (5) Å
$b$ = 4.9900 (2) Å
$c$ = 13.7750 (4) Å
$\beta$ = 96.482 (3)°
$V$ = 886.06 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.34 mm$^{-1}$
$T$ = 173 K
0.35 × 0.22 × 0.12 mm

### *Data collection*

Oxford Xcalibur (Eos, Gemini) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{min}$ = 0.890, $T_{max}$ = 0.960
9633 measured reflections
5460 independent reflections
4909 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.122
$S$ = 1.09
5460 reflections
263 parameters
18 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.71 e Å$^{-3}$
Absolute structure: Flack (1983), with 2216 Friedel pairs
Flack parameter: 0.06 (11)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1N···O1$^{i}$ | 0.88 (2) | 2.30 (3) | 3.092 (3) | 151 (3) |
| N2—H2N···O1$^{i}$ | 0.86 (2) | 2.05 (2) | 2.867 (3) | 158 (3) |
| N3—H3N···O2$^{ii}$ | 0.86 (2) | 2.01 (2) | 2.854 (3) | 166 (3) |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); 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).

MP thanks Ferdowsi University of Mashhad for a Research University Grant (No. 15144/2). JPJ acknowledges the NSF-MRI program (grant No. CHE1039027) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5506).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o3009 [doi:10.1107/S1600536812039712]

## *N*,*N*′-Dibenzyl-*N*′′-(2-chloro-2,2-difluoroacetyl)phosphoric triamide

**Mehrdad Pourayoubi, Mojtaba Keikha, Jerry P. Jasinski and Amanda C. Keeley**

### Comment

In the previous studies, the structures of some compounds with a P(O)[NHC(O)CClF$_2$] fragment have been investigated; for example, [4-CH$_3$-C$_6$H$_4$NH]$_2$P(O)[NHC(O)CClF$_2$] (Pourayoubi, Tarahhomi *et al.*, 2011), [(C$_6$H$_5$CH$_2$)(CH$_3$)N]$_2$P(O)[NHC(O)CClF$_2$] (Raissi Shabari *et al.*, 2011) and [(CH$_3$)$_2$CHNH]$_2$P(O)[NHC(O)CClF$_2$] (Pourayoubi & Saneei, 2011). Here, the structure determination of the title compound (Fig. 1) is reported.

Atoms F1, F2 and Cl1 were refined as disordered over two sets of sites with occupancies of 0.605 (6) and 0.395 (6). The N—H unit of the C(O)NHP(O) fragment adopts a *syn* orientation with respect to the phosphoryl group. The P atom is bonded in a distorted tetrahedral environment as has been noted for other phosphoric triamides. The P═O, C═O and P—N bond lengths and P—N—C bond angles are within the expected values (Pourayoubi, Tarahhomi *et al.*, 2011; Raissi Shabari *et al.*, 2011; Pourayoubi & Saneei, 2011).

In the crystal, the O atom of P═O group acts as a double-hydrogen bond acceptor (Pourayoubi *et al.*, 2012) and molecules are linked by N—H···O═C hydrogen bonds and (N—H···)$_2$O═P group, into a linear arrangement along the *b* axis (Fig. 2).

### Experimental

ClF$_2$CC(O)NHP(O)Cl$_2$ was prepared according to the literature method reported by Iriarte *et al.* (2008).

To a solution of ClF$_2$CC(O)NHP(O)Cl$_2$ (0.473 g, 1.92 mmol) in dry chloroform (25 ml), a solution of benzylamine (0.823 g, 7.68 mmol) in the same solvent (5 ml) was added at 273 K. After 6 h stirring, the solvent was removed and the product was washed with distilled water and recrystallized from CH$_3$CN at room temperature.

### Refinement

H atoms H1N, H2N and H3N were located in a difference Fourier map and were refined with $U_{iso}$(H) = 1.2$U_{eq}$(N), giving N—H distances of 0.88 (2) or 0.86 (2) Å. The other H atoms were placed in calculated positions with 0.95 Å for CH, 0.99 Å for CH$_2$ and with $U_{iso}$(H) = 1.2$U_{eq}$(C). F atoms F1 and F2 and chlorine Cl1 are disordered over two sets of sites with occupancies of 0.605 (6) and 0.395 (6).

### Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); 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* (Sheldrick, 2008) and *enCIFer* (Allen *et al.*, 2004).

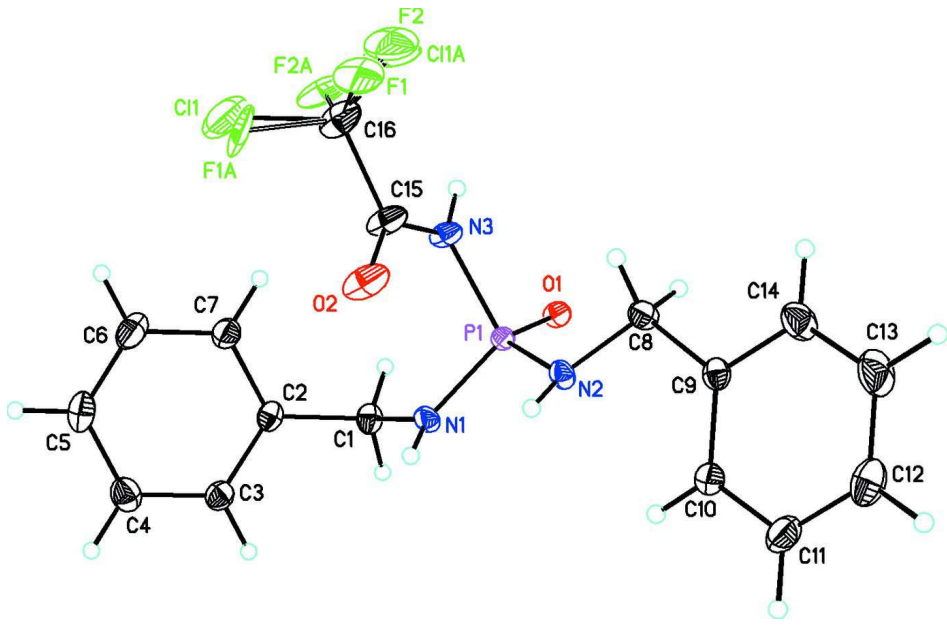


**Figure 1**

The molecular structure of the title compound. Displacement ellipsoids are given at the 30% probability level and H atoms are drawn as small spheres of arbitrary radii. The atoms of the minor component of disorder are labeled with suffix ′A′.

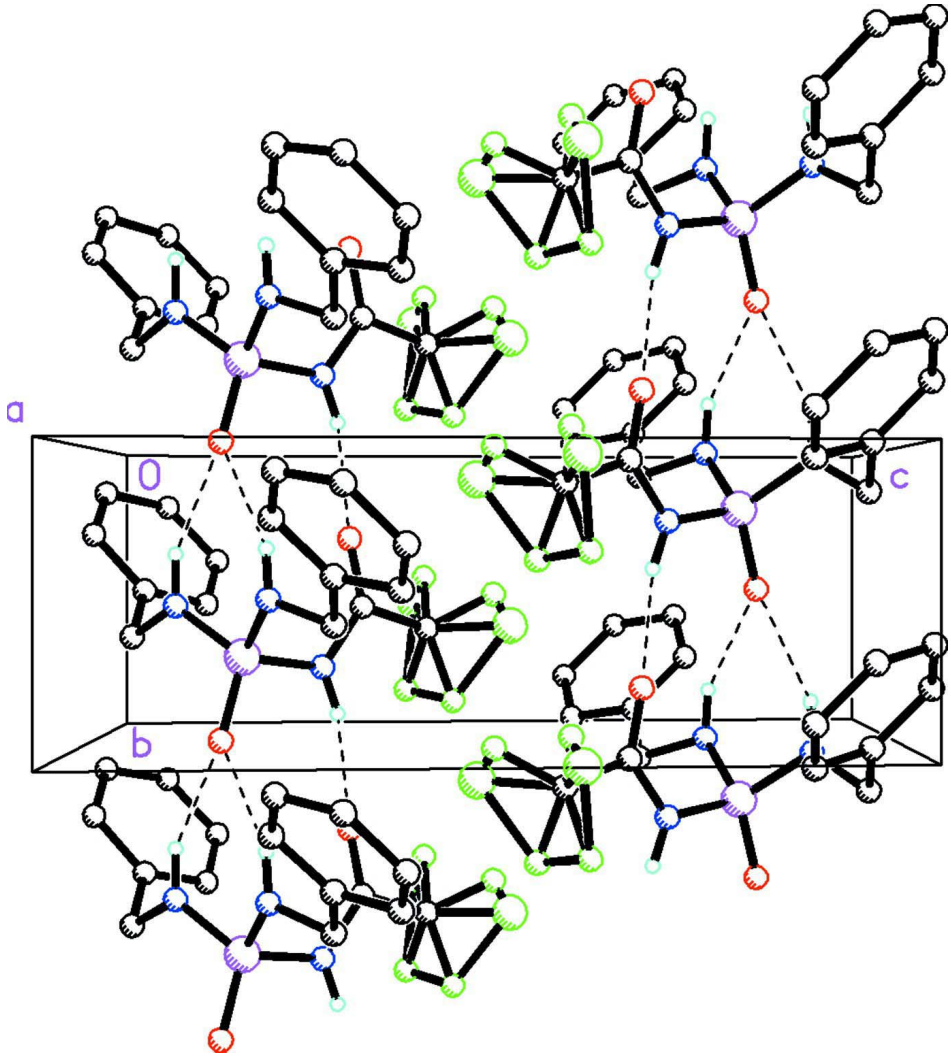


**Figure 2**

Crystal packing of title compound viewed approximately along the *a* axis. The N—H···O hydrogen bonds are shown by dashed lines. H atoms not involved in hydrogen bonding have been removed for clarity.

### *N,N′*-Dibenzyl-*N″*-(2-chloro-2,2-difluoroacetyl)phosphoric triamide

*Crystal data*

$C_{16}H_{17}ClF_2N_3O_2P$
$M_r$ = 387.75
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a$ = 12.9734 (5) Å
$b$ = 4.9900 (2) Å
$c$ = 13.7750 (4) Å
$\beta$ = 96.482 (3)°
$V$ = 886.06 (6) Å$^3$
$Z$ = 2
$F$(000) = 400
$D_x$ = 1.453 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3176 reflections
$\theta$ = 3.3–32.2°
$\mu$ = 0.34 mm$^{-1}$
$T$ = 173 K
Block, colourless
0.35 × 0.22 × 0.12 mm

*Data collection*

Oxford Xcalibur (Eos, Gemini) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1500 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{min}$ = 0.890, $T_{max}$ = 0.960
9633 measured reflections
5460 independent reflections
4909 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025
$\theta_{max}$ = 32.2°, $\theta_{min}$ = 3.3°
$h = -18 \rightarrow 19$
$k = -7 \rightarrow 6$
$l = -9 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.122
$S$ = 1.09
5460 reflections
263 parameters
18 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.3968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.71 e Å$^{-3}$
Absolute structure: Flack (1983), with 2216 Friedel pairs
Flack parameter: 0.06 (11)

*Special details*

**Experimental**. IR (KBr, $\nu$, cm$^{-1}$): 3253, 1718, 1457, 1419, 1282, 1215, 1139, 1073, 977, 873, 735 and 688.

**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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| P1 | 0.51645 (5) | 0.70594 (13) | 0.19236 (4) | 0.01939 (13) | |
| Cl1 | 0.2161 (2) | 0.5360 (8) | 0.3959 (2) | 0.0795 (10) | 0.605 (6) |
| F1 | 0.3784 (5) | 0.5030 (15) | 0.4995 (4) | 0.086 (2) | 0.605 (6) |
| F2 | 0.3555 (5) | 0.8763 (7) | 0.4479 (3) | 0.073 (2) | 0.605 (6) |
| Cl1A | 0.3973 (4) | 0.6102 (13) | 0.5203 (3) | 0.0829 (18) | 0.395 (6) |
| F1A | 0.2518 (6) | 0.4471 (17) | 0.4089 (7) | 0.066 (3) | 0.395 (6) |
| F2A | 0.2775 (5) | 0.8461 (12) | 0.3861 (5) | 0.058 (2) | 0.395 (6) |
| O1 | 0.54403 (15) | 0.9827 (4) | 0.16821 (14) | 0.0261 (4) | |
| O2 | 0.4173 (2) | 0.3092 (5) | 0.3189 (2) | 0.0528 (8) | |
| N1 | 0.45241 (18) | 0.5364 (5) | 0.10546 (18) | 0.0266 (5) | |
| H1N | 0.460 (3) | 0.362 (5) | 0.106 (3) | 0.032* | |
| N2 | 0.61194 (18) | 0.5089 (5) | 0.22963 (16) | 0.0241 (4) | |
| H2N | 0.601 (3) | 0.340 (5) | 0.226 (2) | 0.029* | |
| N3 | 0.4401 (2) | 0.7487 (5) | 0.28612 (18) | 0.0288 (5) | |

| | | | | |
|---|---|---|---|---|
| H3N | 0.428 (3) | 0.909 (5) | 0.305 (2) | 0.035* |
| C1 | 0.3635 (2) | 0.6492 (6) | 0.0436 (2) | 0.0294 (6) |
| H1B | 0.3792 | 0.6516 | −0.0251 | 0.035* |
| H1A | 0.3527 | 0.8367 | 0.0637 | 0.035* |
| C2 | 0.2652 (2) | 0.4925 (6) | 0.04980 (19) | 0.0270 (5) |
| C3 | 0.2351 (2) | 0.2941 (6) | −0.0179 (2) | 0.0309 (6) |
| H3A | 0.2759 | 0.2598 | −0.0696 | 0.037* |
| C4 | 0.1463 (3) | 0.1456 (7) | −0.0111 (3) | 0.0391 (7) |
| H4A | 0.1267 | 0.0093 | −0.0577 | 0.047* |
| C5 | 0.0860 (2) | 0.1951 (9) | 0.0635 (2) | 0.0432 (7) |
| H5A | 0.0247 | 0.0937 | 0.0679 | 0.052* |
| C6 | 0.1150 (3) | 0.3920 (9) | 0.1315 (3) | 0.0457 (9) |
| H6A | 0.0742 | 0.4238 | 0.1835 | 0.055* |
| C7 | 0.2035 (2) | 0.5440 (8) | 0.1241 (2) | 0.0364 (7) |
| H7A | 0.2219 | 0.6832 | 0.1698 | 0.044* |
| C8 | 0.6884 (2) | 0.5876 (6) | 0.3112 (2) | 0.0286 (6) |
| H8A | 0.6577 | 0.5637 | 0.3733 | 0.034* |
| H8B | 0.7050 | 0.7800 | 0.3048 | 0.034* |
| C9 | 0.7871 (2) | 0.4274 (6) | 0.31541 (19) | 0.0263 (5) |
| C10 | 0.8070 (2) | 0.2410 (7) | 0.2450 (2) | 0.0303 (6) |
| H10A | 0.7574 | 0.2120 | 0.1899 | 0.036* |
| C11 | 0.8993 (3) | 0.0966 (8) | 0.2550 (3) | 0.0401 (7) |
| H11A | 0.9121 | −0.0311 | 0.2067 | 0.048* |
| C12 | 0.9720 (3) | 0.1367 (8) | 0.3341 (3) | 0.0453 (9) |
| H12A | 1.0345 | 0.0358 | 0.3409 | 0.054* |
| C13 | 0.9537 (3) | 0.3244 (9) | 0.4035 (3) | 0.0505 (10) |
| H13A | 1.0045 | 0.3561 | 0.4575 | 0.061* |
| C14 | 0.8624 (3) | 0.4658 (8) | 0.3949 (2) | 0.0403 (7) |
| H14A | 0.8502 | 0.5920 | 0.4439 | 0.048* |
| C15 | 0.4032 (3) | 0.5428 (6) | 0.3344 (3) | 0.0405 (8) |
| C16 | 0.3359 (2) | 0.6241 (6) | 0.4143 (2) | 0.0490 (8) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0211 (3) | 0.0136 (2) | 0.0242 (3) | −0.0009 (3) | 0.00582 (19) | −0.0014 (2) |
| Cl1 | 0.0438 (12) | 0.139 (3) | 0.0597 (12) | 0.0070 (14) | 0.0255 (10) | −0.0081 (14) |
| F1 | 0.076 (4) | 0.169 (7) | 0.0121 (19) | −0.017 (4) | 0.0062 (19) | −0.016 (3) |
| F2 | 0.139 (5) | 0.036 (2) | 0.056 (3) | −0.013 (3) | 0.066 (3) | −0.0126 (18) |
| Cl1A | 0.076 (3) | 0.148 (5) | 0.0257 (15) | 0.005 (3) | 0.0091 (13) | −0.0213 (17) |
| F1A | 0.028 (3) | 0.079 (6) | 0.098 (6) | −0.017 (4) | 0.036 (4) | 0.007 (5) |
| F2A | 0.081 (5) | 0.043 (3) | 0.060 (4) | 0.032 (3) | 0.047 (4) | 0.012 (3) |
| O1 | 0.0319 (10) | 0.0160 (9) | 0.0322 (9) | −0.0018 (7) | 0.0107 (7) | 0.0004 (7) |
| O2 | 0.0767 (19) | 0.0144 (10) | 0.0769 (18) | 0.0017 (12) | 0.0504 (16) | 0.0025 (11) |
| N1 | 0.0229 (10) | 0.0199 (11) | 0.0361 (11) | 0.0025 (9) | −0.0006 (8) | −0.0079 (9) |
| N2 | 0.0240 (10) | 0.0158 (10) | 0.0316 (11) | −0.0011 (8) | −0.0008 (8) | −0.0015 (9) |
| N3 | 0.0405 (13) | 0.0129 (12) | 0.0366 (11) | 0.0009 (9) | 0.0198 (10) | −0.0012 (8) |
| C1 | 0.0263 (12) | 0.0335 (18) | 0.0281 (12) | −0.0024 (10) | 0.0009 (9) | 0.0050 (10) |
| C2 | 0.0218 (12) | 0.0308 (14) | 0.0281 (12) | 0.0024 (10) | 0.0014 (9) | 0.0067 (10) |
| C3 | 0.0275 (13) | 0.0332 (16) | 0.0321 (13) | 0.0026 (11) | 0.0033 (11) | 0.0008 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.0366 (16) | 0.0320 (19) | 0.0477 (17) | −0.0011 (12) | 0.0000 (13) | −0.0013 (12) |
| C5 | 0.0271 (13) | 0.0458 (19) | 0.0568 (18) | −0.0050 (17) | 0.0047 (12) | 0.008 (2) |
| C6 | 0.0327 (17) | 0.064 (3) | 0.0427 (17) | −0.0004 (17) | 0.0129 (13) | 0.0049 (17) |
| C7 | 0.0287 (14) | 0.049 (2) | 0.0314 (13) | −0.0020 (14) | 0.0042 (11) | −0.0040 (13) |
| C8 | 0.0296 (13) | 0.0269 (14) | 0.0284 (12) | 0.0019 (11) | −0.0006 (10) | −0.0036 (10) |
| C9 | 0.0252 (12) | 0.0265 (13) | 0.0270 (12) | −0.0032 (10) | 0.0025 (9) | 0.0035 (10) |
| C10 | 0.0260 (12) | 0.0340 (17) | 0.0309 (12) | −0.0004 (12) | 0.0030 (9) | −0.0017 (11) |
| C11 | 0.0298 (15) | 0.0427 (19) | 0.0494 (18) | 0.0062 (14) | 0.0113 (13) | −0.0021 (15) |
| C12 | 0.0297 (15) | 0.052 (2) | 0.0533 (19) | 0.0088 (14) | 0.0011 (13) | 0.0121 (16) |
| C13 | 0.0378 (19) | 0.062 (3) | 0.0474 (19) | 0.0040 (17) | −0.0156 (15) | 0.0013 (18) |
| C14 | 0.0393 (17) | 0.0429 (19) | 0.0356 (15) | 0.0020 (15) | −0.0096 (12) | −0.0027 (14) |
| C15 | 0.060 (2) | 0.0165 (13) | 0.0516 (18) | 0.0013 (13) | 0.0366 (16) | 0.0015 (12) |
| C16 | 0.0641 (16) | 0.0245 (15) | 0.066 (2) | −0.0012 (15) | 0.0394 (15) | 0.0048 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| P1—O1 | 1.474 (2) | C3—H3A | 0.9500 |
| P1—N1 | 1.617 (2) | C4—C5 | 1.382 (5) |
| P1—N2 | 1.619 (2) | C4—H4A | 0.9500 |
| P1—N3 | 1.728 (2) | C5—C6 | 1.381 (6) |
| Cl1—C16 | 1.607 (3) | C5—H5A | 0.9500 |
| F1—C16 | 1.379 (5) | C6—C7 | 1.390 (5) |
| F2—C16 | 1.355 (4) | C6—H6A | 0.9500 |
| Cl1A—C16 | 1.585 (4) | C7—H7A | 0.9500 |
| F1A—C16 | 1.399 (5) | C8—C9 | 1.505 (4) |
| F2A—C16 | 1.373 (4) | C8—H8A | 0.9900 |
| O2—C15 | 1.203 (4) | C8—H8B | 0.9900 |
| N1—C1 | 1.467 (4) | C9—C10 | 1.388 (4) |
| N1—H1N | 0.88 (2) | C9—C14 | 1.396 (4) |
| N2—C8 | 1.466 (3) | C10—C11 | 1.391 (4) |
| N2—H2N | 0.86 (2) | C10—H10A | 0.9500 |
| N3—C15 | 1.341 (4) | C11—C12 | 1.373 (5) |
| N3—H3N | 0.86 (2) | C11—H11A | 0.9500 |
| C1—C2 | 1.507 (4) | C12—C13 | 1.378 (6) |
| C1—H1B | 0.9900 | C12—H12A | 0.9500 |
| C1—H1A | 0.9900 | C13—C14 | 1.373 (5) |
| C2—C3 | 1.386 (4) | C13—H13A | 0.9500 |
| C2—C7 | 1.393 (4) | C14—H14A | 0.9500 |
| C3—C4 | 1.382 (4) | C15—C16 | 1.534 (4) |
| | | | |
| O1—P1—N1 | 116.13 (13) | C9—C8—H8B | 109.0 |
| O1—P1—N2 | 116.36 (12) | H8A—C8—H8B | 107.8 |
| N1—P1—N2 | 103.17 (12) | C10—C9—C14 | 118.1 (3) |
| O1—P1—N3 | 103.15 (11) | C10—C9—C8 | 123.6 (2) |
| N1—P1—N3 | 109.14 (13) | C14—C9—C8 | 118.3 (3) |
| N2—P1—N3 | 108.72 (12) | C9—C10—C11 | 120.2 (3) |
| C1—N1—P1 | 122.3 (2) | C9—C10—H10A | 119.9 |
| C1—N1—H1N | 118 (2) | C11—C10—H10A | 119.9 |
| P1—N1—H1N | 117 (2) | C12—C11—C10 | 120.7 (3) |
| C8—N2—P1 | 120.50 (19) | C12—C11—H11A | 119.7 |

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| C8—N2—H2N | 114 (2) | C10—C11—H11A | 119.7 |
| P1—N2—H2N | 118 (2) | C11—C12—C13 | 119.5 (3) |
| C15—N3—P1 | 122.91 (19) | C11—C12—H12A | 120.2 |
| C15—N3—H3N | 119 (2) | C13—C12—H12A | 120.2 |
| P1—N3—H3N | 118 (2) | C14—C13—C12 | 120.3 (3) |
| N1—C1—C2 | 112.5 (2) | C14—C13—H13A | 119.9 |
| N1—C1—H1B | 109.1 | C12—C13—H13A | 119.9 |
| C2—C1—H1B | 109.1 | C13—C14—C9 | 121.2 (3) |
| N1—C1—H1A | 109.1 | C13—C14—H14A | 119.4 |
| C2—C1—H1A | 109.1 | C9—C14—H14A | 119.4 |
| H1B—C1—H1A | 107.8 | O2—C15—N3 | 125.7 (3) |
| C3—C2—C7 | 118.9 (3) | O2—C15—C16 | 119.6 (3) |
| C3—C2—C1 | 120.6 (2) | N3—C15—C16 | 114.6 (2) |
| C7—C2—C1 | 120.5 (3) | F2—C16—F2A | 54.8 (4) |
| C4—C3—C2 | 120.8 (3) | F2—C16—F1 | 94.3 (4) |
| C4—C3—H3A | 119.6 | F2A—C16—F1 | 138.5 (4) |
| C2—C3—H3A | 119.6 | F2—C16—F1A | 136.0 (5) |
| C5—C4—C3 | 120.1 (3) | F2A—C16—F1A | 95.3 (5) |
| C5—C4—H4A | 120.0 | F1—C16—F1A | 90.2 (5) |
| C3—C4—H4A | 120.0 | F2—C16—C15 | 113.0 (3) |
| C6—C5—C4 | 119.9 (3) | F2A—C16—C15 | 110.6 (3) |
| C6—C5—H5A | 120.1 | F1—C16—C15 | 106.8 (4) |
| C4—C5—H5A | 120.1 | F1A—C16—C15 | 107.3 (5) |
| C5—C6—C7 | 120.2 (3) | F2—C16—Cl1A | 70.7 (4) |
| C5—C6—H6A | 119.9 | F2A—C16—Cl1A | 120.0 (4) |
| C7—C6—H6A | 119.9 | F1A—C16—Cl1A | 109.2 (5) |
| C6—C7—C2 | 120.1 (3) | C15—C16—Cl1A | 112.5 (3) |
| C6—C7—H7A | 119.9 | F2—C16—Cl1 | 116.7 (4) |
| C2—C7—H7A | 119.9 | F2A—C16—Cl1 | 71.6 (4) |
| N2—C8—C9 | 112.7 (2) | F1—C16—Cl1 | 107.5 (4) |
| N2—C8—H8A | 109.0 | C15—C16—Cl1 | 115.7 (3) |
| C9—C8—H8A | 109.0 | Cl1A—C16—Cl1 | 120.6 (3) |
| N2—C8—H8B | 109.0 | | |

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| O1—P1—N1—C1 | −45.6 (3) | C14—C9—C10—C11 | 0.6 (5) |
| N2—P1—N1—C1 | −174.1 (2) | C8—C9—C10—C11 | −178.7 (3) |
| N3—P1—N1—C1 | 70.4 (2) | C9—C10—C11—C12 | −0.3 (5) |
| O1—P1—N2—C8 | 53.1 (2) | C10—C11—C12—C13 | −0.8 (6) |
| N1—P1—N2—C8 | −178.5 (2) | C11—C12—C13—C14 | 1.5 (6) |
| N3—P1—N2—C8 | −62.8 (2) | C12—C13—C14—C9 | −1.2 (6) |
| O1—P1—N3—C15 | −175.7 (3) | C10—C9—C14—C13 | 0.2 (5) |
| N1—P1—N3—C15 | 60.2 (3) | C8—C9—C14—C13 | 179.5 (3) |
| N2—P1—N3—C15 | −51.6 (3) | P1—N3—C15—O2 | −0.6 (6) |
| P1—N1—C1—C2 | −120.2 (2) | P1—N3—C15—C16 | −179.3 (2) |
| N1—C1—C2—C3 | −94.8 (3) | O2—C15—C16—F2 | 157.9 (5) |
| N1—C1—C2—C7 | 84.9 (3) | N3—C15—C16—F2 | −23.3 (5) |
| C7—C2—C3—C4 | −1.2 (4) | O2—C15—C16—F2A | −142.8 (5) |
| C1—C2—C3—C4 | 178.4 (3) | N3—C15—C16—F2A | 36.0 (6) |
| C2—C3—C4—C5 | 0.4 (5) | O2—C15—C16—F1 | 55.6 (6) |

| | | | |
|---|---|---|---|
| C3—C4—C5—C6 | −0.4 (5) | N3—C15—C16—F1 | −125.6 (4) |
| C4—C5—C6—C7 | 1.2 (6) | O2—C15—C16—F1A | −40.0 (6) |
| C5—C6—C7—C2 | −2.0 (6) | N3—C15—C16—F1A | 138.8 (5) |
| C3—C2—C7—C6 | 2.0 (5) | O2—C15—C16—Cl1A | 80.1 (5) |
| C1—C2—C7—C6 | −177.6 (3) | N3—C15—C16—Cl1A | −101.1 (4) |
| P1—N2—C8—C9 | −162.00 (19) | O2—C15—C16—Cl1 | −64.0 (5) |
| N2—C8—C9—C10 | 5.0 (4) | N3—C15—C16—Cl1 | 114.8 (4) |
| N2—C8—C9—C14 | −174.2 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O1$^{i}$ | 0.88 (2) | 2.30 (3) | 3.092 (3) | 151 (3) |
| N2—H2*N*···O1$^{i}$ | 0.86 (2) | 2.05 (2) | 2.867 (3) | 158 (3) |
| N3—H3*N*···O2$^{ii}$ | 0.86 (2) | 2.01 (2) | 2.854 (3) | 166 (3) |

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*, *y*+1, *z*.