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Diphenyl (2-chlorobenzylamido)-phosphate

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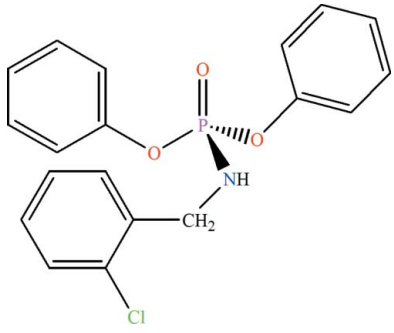
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.086; data-to-parameter ratio = 17.3.

In the title compound, $C_{19}H_{17}ClNO_3P$, the P atom exhibits a distorted tetrahedral configuration. In the crystal, pairs of intermolecular N—H···O(P) hydrogen bonds form centrosymmetric dimers.

Related literature

For related structures, see: Pourayoubi & Zargaran (2010); Pourayoubi *et al.* (2010).



Experimental

Crystal data

$C_{19}H_{17}ClNO_3P$ M_r = 373.76
Triclinic, $P\overline{1}$
a = 8.6178 (5) Å
b = 9.5901 (6) Å
c = 12.1543 (7) Å
α = 107.609 (1)°
β = 93.882 (1)°
γ = 110.036 (1)°
V = 882.86 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 100 K
0.40 × 0.35 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.881, T_{max} = 0.923
13560 measured reflections
3982 independent reflections
3681 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.086
S = 1.02
3982 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.80 (2)	2.08 (2)	2.8703 (15)	172.2 (19)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5173).

References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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supporting information

Acta Cryst. (2011). E**67**, o5 [https://doi.org/10.1107/S160053681004955X]

Diphenyl (2-chlorobenzylamido)phosphate

Mehrdad Pourayoubi, Poorya Zargaran, Arnold L. Rheingold and James A. Golen

S1. Comment

In our previous works, the crystal structures of some amidophosphoric acid ester compounds having the $P(O)(OC_6H_5)_2$ phosphoester moiety have been reported (Pourayoubi *et al.*, 2010; Pourayoubi & Zargaran, 2010). Herein, we report the synthesis and crystal structure of the title amidophosphoric acid ester.

The molecular structure of the title compound is shown in Fig. 1. The P atom has a distorted tetrahedral configuration with the bond angles in the range of 98.03 (5)° [O2–P1–O3] to 116.37 (6)° [O1–P1–O2]. In the crystal structure, pairs of intermolecular N-H···O(P) hydrogen bonds form centrosymmetric dimers.

S2. Experimental

To a solution of $(C_6H_5O)_2P(O)Cl$ in chloroform, a solution of 2-chlorobenzylamine (1:2 mole ratio) in chloroform was added at 273 K. After 4 h stirring, the solvent was removed and product was washed with distilled water and recrystallized from CH_3CN at room temperature. IR (KBr, cm^{-1}): 3206.6, 3065.7, 2909.2, 2715.0, 1947.0, 1591.1, 1486.9, 1456.4, 1257.1, 1198.0, 1131.5, 1016.5, 940.3, 756.3, 686.0.

S3. Refinement

Data corrected for absorption using *SADABS* (Bruker, 2009) and structure solved by direct methods. All non-hydrogen atoms refined as anisotropic by Fourier full matrix least squares. Hydrogen atoms H1N found from a Fourier difference map and allowed to refine while all other hydrogen atoms were placed in calculated positions with appropriate riding models.

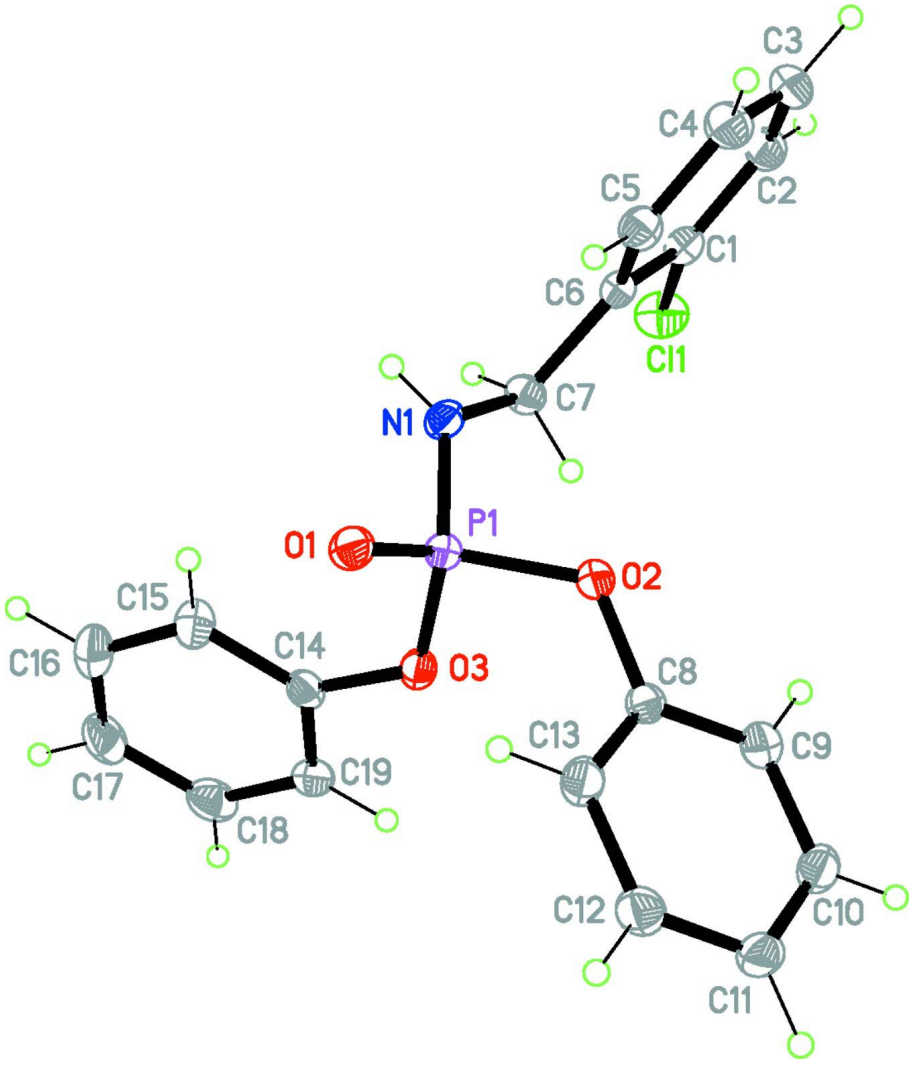

Figure 1

The molecular structure of the title compound. Ellipsoids are given at the 50% probability level.

Diphenyl (2-chlorobenzylamido)phosphate

Crystal data

$C_{19}H_{17}ClNO_3P$
$M_r = 373.76$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.6178\ (5)$ Å
$b = 9.5901\ (6)$ Å
$c = 12.1543\ (7)$ Å
$\alpha = 107.609\ (1)°$
$\beta = 93.882\ (1)°$
$\gamma = 110.036\ (1)°$
$V = 882.86\ (9)$ Å^3
$Z = 2$
$F(000) = 388$
$D_x = 1.406$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9988 reflections
$\theta = 2.4–28.0°$
$\mu = 0.33$ mm^{-1}
$T = 100$ K
Block, colourless
0.40 × 0.35 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.881, T_{max} = 0.923
13560 measured reflections
3982 independent reflections
3681 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°, θ_{min} = 2.4°
h = −11→10
k = −11→12
l = −15→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.086
S = 1.02
3982 reflections
230 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.4993P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	1.12274 (4)	0.65811 (4)	0.77058 (3)	0.02847 (10)
P1	0.49017 (4)	0.15695 (4)	0.67814 (3)	0.01573 (9)
O1	0.35884 (12)	0.01133 (11)	0.59500 (8)	0.0201 (2)
O2	0.44078 (12)	0.30686 (11)	0.72227 (8)	0.01837 (19)
O3	0.53917 (12)	0.14595 (11)	0.80359 (8)	0.0184 (2)
N1	0.65627 (15)	0.21809 (13)	0.62570 (10)	0.0194 (2)
C1	0.97088 (17)	0.61883 (16)	0.65109 (11)	0.0190 (3)
C2	1.00000 (19)	0.72582 (16)	0.59229 (13)	0.0244 (3)
H2A	1.1015	0.8170	0.6159	0.029*
C3	0.8788 (2)	0.69789 (18)	0.49836 (13)	0.0276 (3)
H3A	0.8965	0.7708	0.4577	0.033*
C4	0.7317 (2)	0.56346 (18)	0.46398 (13)	0.0262 (3)
H4A	0.6485	0.5443	0.3999	0.031*
C5	0.70658 (17)	0.45701 (16)	0.52345 (12)	0.0207 (3)
H5A	0.6058	0.3650	0.4989	0.025*

C6	0.82539 (16)	0.48181 (15)	0.61804 (11)	0.0167 (2)
C7	0.80201 (17)	0.36547 (15)	0.68278 (11)	0.0188 (3)
H7A	0.9043	0.3410	0.6882	0.023*
H7B	0.7890	0.4157	0.7639	0.023*
C8	0.32185 (16)	0.31673 (15)	0.79467 (11)	0.0170 (3)
C9	0.35893 (18)	0.46128 (16)	0.88220 (12)	0.0206 (3)
H9A	0.4608	0.5481	0.8920	0.025*
C10	0.24451 (19)	0.47717 (17)	0.95549 (12)	0.0232 (3)
H10A	0.2686	0.5755	1.0163	0.028*
C11	0.09537 (19)	0.35066 (17)	0.94059 (13)	0.0235 (3)
H11A	0.0181	0.3619	0.9915	0.028*
C12	0.05959 (18)	0.20772 (17)	0.85097 (13)	0.0241 (3)
H12A	−0.0430	0.1213	0.8402	0.029*
C13	0.17259 (18)	0.18976 (16)	0.77676 (12)	0.0215 (3)
H13A	0.1478	0.0921	0.7149	0.026*
C14	0.60296 (16)	0.03684 (15)	0.82152 (12)	0.0177 (3)
C15	0.60994 (18)	−0.08832 (16)	0.73025 (13)	0.0233 (3)
H15A	0.5699	−0.1047	0.6508	0.028*
C16	0.67701 (19)	−0.18925 (17)	0.75786 (14)	0.0277 (3)
H16A	0.6826	−0.2756	0.6965	0.033*
C17	0.7356 (2)	−0.16551 (18)	0.87353 (15)	0.0287 (3)
H17A	0.7817	−0.2348	0.8913	0.034*
C18	0.7269 (2)	−0.03992 (19)	0.96360 (14)	0.0278 (3)
H18A	0.7667	−0.0235	1.0431	0.033*
C19	0.65988 (18)	0.06176 (16)	0.93767 (12)	0.0214 (3)
H19A	0.6533	0.1475	0.9991	0.026*
H1N	0.661 (2)	0.160 (2)	0.5644 (18)	0.031 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02121 (18)	0.0312 (2)	0.02217 (17)	0.00205 (14)	−0.00251 (13)	0.00503 (14)
P1	0.01745 (17)	0.01436 (16)	0.01548 (16)	0.00594 (13)	0.00454 (12)	0.00518 (12)
O1	0.0195 (5)	0.0182 (5)	0.0192 (4)	0.0047 (4)	0.0049 (4)	0.0043 (4)
O2	0.0209 (5)	0.0169 (4)	0.0210 (4)	0.0091 (4)	0.0081 (4)	0.0086 (4)
O3	0.0242 (5)	0.0170 (4)	0.0164 (4)	0.0107 (4)	0.0044 (4)	0.0056 (3)
N1	0.0207 (6)	0.0157 (5)	0.0184 (5)	0.0048 (4)	0.0074 (5)	0.0029 (4)
C1	0.0186 (6)	0.0199 (6)	0.0170 (6)	0.0080 (5)	0.0041 (5)	0.0036 (5)
C2	0.0262 (7)	0.0184 (6)	0.0290 (7)	0.0072 (6)	0.0116 (6)	0.0088 (5)
C3	0.0369 (8)	0.0268 (7)	0.0302 (7)	0.0179 (7)	0.0142 (7)	0.0170 (6)
C4	0.0302 (8)	0.0322 (8)	0.0235 (7)	0.0178 (6)	0.0053 (6)	0.0128 (6)
C5	0.0198 (6)	0.0218 (6)	0.0201 (6)	0.0088 (5)	0.0032 (5)	0.0059 (5)
C6	0.0181 (6)	0.0166 (6)	0.0166 (6)	0.0086 (5)	0.0054 (5)	0.0049 (5)
C7	0.0178 (6)	0.0189 (6)	0.0188 (6)	0.0054 (5)	0.0025 (5)	0.0072 (5)
C8	0.0190 (6)	0.0198 (6)	0.0173 (6)	0.0109 (5)	0.0048 (5)	0.0091 (5)
C9	0.0211 (7)	0.0179 (6)	0.0223 (6)	0.0072 (5)	0.0035 (5)	0.0068 (5)
C10	0.0289 (7)	0.0217 (7)	0.0209 (6)	0.0132 (6)	0.0057 (6)	0.0056 (5)
C11	0.0259 (7)	0.0295 (7)	0.0249 (7)	0.0171 (6)	0.0107 (6)	0.0140 (6)

C12	0.0199 (7)	0.0238 (7)	0.0315 (7)	0.0085 (5)	0.0084 (6)	0.0125 (6)
C13	0.0217 (7)	0.0185 (6)	0.0238 (7)	0.0085 (5)	0.0050 (5)	0.0056 (5)
C14	0.0161 (6)	0.0162 (6)	0.0225 (6)	0.0057 (5)	0.0050 (5)	0.0092 (5)
C15	0.0257 (7)	0.0201 (6)	0.0231 (7)	0.0103 (6)	0.0016 (5)	0.0049 (5)
C16	0.0281 (8)	0.0198 (7)	0.0352 (8)	0.0120 (6)	0.0052 (6)	0.0066 (6)
C17	0.0272 (8)	0.0265 (7)	0.0410 (9)	0.0138 (6)	0.0075 (7)	0.0193 (7)
C18	0.0297 (8)	0.0339 (8)	0.0279 (7)	0.0142 (6)	0.0068 (6)	0.0190 (6)
C19	0.0230 (7)	0.0229 (7)	0.0214 (6)	0.0094 (5)	0.0080 (5)	0.0102 (5)

Geometric parameters (Å, º)

Cl1—C1	1.7429 (14)	C8—C9	1.3834 (18)
P1—O1	1.4699 (10)	C8—C13	1.3831 (19)
P1—O2	1.5872 (9)	C9—C10	1.389 (2)
P1—O3	1.5984 (9)	C9—H9A	0.9500
P1—N1	1.6042 (12)	C10—C11	1.387 (2)
O2—C8	1.4043 (15)	C10—H10A	0.9500
O3—C14	1.4002 (15)	C11—C12	1.386 (2)
N1—C7	1.4591 (17)	C11—H11A	0.9500
N1—H1N	0.80 (2)	C12—C13	1.390 (2)
C1—C2	1.3850 (19)	C12—H12A	0.9500
C1—C6	1.3942 (18)	C13—H13A	0.9500
C2—C3	1.389 (2)	C14—C19	1.3829 (19)
C2—H2A	0.9500	C14—C15	1.3884 (19)
C3—C4	1.388 (2)	C15—C16	1.392 (2)
C3—H3A	0.9500	C15—H15A	0.9500
C4—C5	1.388 (2)	C16—C17	1.384 (2)
C4—H4A	0.9500	C16—H16A	0.9500
C5—C6	1.3910 (18)	C17—C18	1.389 (2)
C5—H5A	0.9500	C17—H17A	0.9500
C6—C7	1.5175 (17)	C18—C19	1.389 (2)
C7—H7A	0.9900	C18—H18A	0.9500
C7—H7B	0.9900	C19—H19A	0.9500
O1—P1—O2	116.37 (6)	C9—C8—O2	116.66 (12)
O1—P1—O3	113.92 (5)	C13—C8—O2	121.59 (12)
O2—P1—O3	98.03 (5)	C8—C9—C10	118.77 (13)
O1—P1—N1	113.05 (6)	C8—C9—H9A	120.6
O2—P1—N1	104.00 (6)	C10—C9—H9A	120.6
O3—P1—N1	110.13 (6)	C11—C10—C9	120.55 (13)
C8—O2—P1	123.42 (8)	C11—C10—H10A	119.7
C14—O3—P1	124.66 (8)	C9—C10—H10A	119.7
C7—N1—P1	125.99 (9)	C12—C11—C10	119.65 (13)
C7—N1—H1N	117.4 (14)	C12—C11—H11A	120.2
P1—N1—H1N	116.5 (14)	C10—C11—H11A	120.2
C2—C1—C6	122.43 (13)	C11—C12—C13	120.56 (14)
C2—C1—Cl1	118.35 (11)	C11—C12—H12A	119.7
C6—C1—Cl1	119.22 (10)	C13—C12—H12A	119.7

C1—C2—C3	119.07 (13)	C8—C13—C12	118.73 (13)
C1—C2—H2A	120.5	C8—C13—H13A	120.6
C3—C2—H2A	120.5	C12—C13—H13A	120.6
C4—C3—C2	119.95 (13)	C19—C14—C15	121.49 (12)
C4—C3—H3A	120.0	C19—C14—O3	115.36 (11)
C2—C3—H3A	120.0	C15—C14—O3	123.15 (12)
C3—C4—C5	119.83 (14)	C14—C15—C16	118.46 (13)
C3—C4—H4A	120.1	C14—C15—H15A	120.8
C5—C4—H4A	120.1	C16—C15—H15A	120.8
C4—C5—C6	121.60 (13)	C17—C16—C15	120.86 (14)
C4—C5—H5A	119.2	C17—C16—H16A	119.6
C6—C5—H5A	119.2	C15—C16—H16A	119.6
C5—C6—C1	117.10 (12)	C16—C17—C18	119.76 (13)
C5—C6—C7	122.47 (12)	C16—C17—H17A	120.1
C1—C6—C7	120.42 (12)	C18—C17—H17A	120.1
N1—C7—C6	113.03 (11)	C19—C18—C17	120.15 (14)
N1—C7—H7A	109.0	C19—C18—H18A	119.9
C6—C7—H7A	109.0	C17—C18—H18A	119.9
N1—C7—H7B	109.0	C14—C19—C18	119.28 (13)
C6—C7—H7B	109.0	C14—C19—H19A	120.4
H7A—C7—H7B	107.8	C18—C19—H19A	120.4
C9—C8—C13	121.71 (13)		

O1—P1—O2—C8	67.62 (11)	C1—C6—C7—N1	−171.04 (11)
O3—P1—O2—C8	−54.18 (10)	P1—O2—C8—C9	140.36 (10)
N1—P1—O2—C8	−167.34 (10)	P1—O2—C8—C13	−41.74 (16)
O1—P1—O3—C14	59.85 (11)	C13—C8—C9—C10	1.6 (2)
O2—P1—O3—C14	−176.56 (10)	O2—C8—C9—C10	179.53 (11)
N1—P1—O3—C14	−68.38 (11)	C8—C9—C10—C11	−0.4 (2)
O1—P1—N1—C7	174.57 (10)	C9—C10—C11—C12	−0.7 (2)
O2—P1—N1—C7	47.45 (12)	C10—C11—C12—C13	0.6 (2)
O3—P1—N1—C7	−56.72 (12)	C9—C8—C13—C12	−1.7 (2)
C6—C1—C2—C3	1.3 (2)	O2—C8—C13—C12	−179.47 (12)
Cl1—C1—C2—C3	−178.73 (11)	C11—C12—C13—C8	0.5 (2)
C1—C2—C3—C4	−0.6 (2)	P1—O3—C14—C19	171.04 (10)
C2—C3—C4—C5	−0.2 (2)	P1—O3—C14—C15	−8.87 (18)
C3—C4—C5—C6	0.4 (2)	C19—C14—C15—C16	−0.4 (2)
C4—C5—C6—C1	0.29 (19)	O3—C14—C15—C16	179.53 (13)
C4—C5—C6—C7	−178.79 (12)	C14—C15—C16—C17	−0.1 (2)
C2—C1—C6—C5	−1.16 (19)	C15—C16—C17—C18	0.4 (2)
Cl1—C1—C6—C5	178.91 (10)	C16—C17—C18—C19	−0.2 (2)
C2—C1—C6—C7	177.94 (12)	C15—C14—C19—C18	0.5 (2)
Cl1—C1—C6—C7	−1.99 (17)	O3—C14—C19—C18	−179.36 (12)
P1—N1—C7—C6	−113.96 (12)	C17—C18—C19—C14	−0.3 (2)
C5—C6—C7—N1	8.01 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.80 (2)	2.08 (2)	2.8703 (15)	172.2 (19)

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.