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N,*N*′-Di-*tert*-butyl-*N*″-(2-chloroacetyl)-phosphoric triamide

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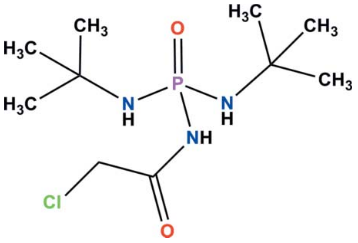
Key indicators: single-crystal X-ray study; *T* = 173 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.043; *wR* factor = 0.103; data-to-parameter ratio = 20.2.

The P atom in the title molecule, $C_{10}H_{23}ClN_3O_2P$, has a distorted tetrahedral coordination. In the C(O)NHP(O) unit, which has *syn*-oriented phosphoryl and N—H groups, the P—N bond of 1.703 (2) Å is longer and the O—P—N angle of 103.86 (7)° is contracted compared with the respective values in the two P(O)NHC$(CH_3)_3$ units [P—N = 1.632 (2) and 1.624 (2) Å; O—P—N = 116.80 (8) and 115.32 (8)°]. In the crystal, each molecule is hydrogen bonded to two adjacent molecules *via* N—H⋯O hydrogen bonds, forming a linear sequence of alternating $R_2^2(8)$ and $R_2^2(12)/R_2^1(6)$-fused rings along [010]. The O atom of the carbonyl group acts as a double H-atom acceptor.

Related literature

For compounds containing a C(O)NHP(O) skeleton and related bond lengths and angles, see: Pourayoubi *et al.* (2011). For the graph-set description of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For double hydrogen-bond acceptors, see: Steiner (2002).



Experimental

Crystal data

$C_{10}H_{23}ClN_3O_2P$
M_r = 283.73
Monoclinic, *C*2/*c*
a = 16.4781 (5) Å
b = 9.8872 (2) Å
c = 19.6509 (6) Å
β = 111.5747 (12)°
V = 2977.26 (14) Å^3
Z = 8
Mo *K*α radiation
μ = 0.36 mm^{-1}
T = 173 K
0.16 × 0.14 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer with APEXII CCD
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min}$ = 0.945, $T_{\max}$ = 0.958
6472 measured reflections
3410 independent reflections
2937 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.103
S = 1.10
3410 reflections
169 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.32 e Å^{-3}
$\Delta\rho_{\min}$ = −0.32 e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*N*⋯O2^{i}	0.87 (1)	1.92 (1)	2.778 (2)	175 (2)
N2—H2*N*⋯O1^{ii}	0.85 (1)	2.56 (1)	3.337 (2)	153 (2)
N3—H3*N*⋯O1^{ii}	0.86 (1)	2.12 (1)	2.979 (2)	175 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z$; (ii) $-x+\frac{3}{2}, -y-\frac{1}{2}, -z$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2026).

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supporting information

Acta Cryst. (2011). E**67**, o2848 [doi:10.1107/S1600536811040773]

N,*N*′-Di-*tert*-butyl-*N*′′-(2-chloroacetyl)phosphoric triamide

Mehrdad Pourayoubi, Behrouz Elahi and Masood Parvez

S1. Comment

The structure of the title molecule, $P(O)[NHC(O)CH_2Cl][NHC(CH_3)_3]_2$ (Fig. 1), was determined as a part of the work on the synthesis of new phosphoramidate compounds containing a P(O)NHC(O) moiety (Pourayoubi *et al.*, 2011).

Single crystals were obtained from a solution of $CHCl_3$ after slow evaporation at room temperature. The P═O and C═O bond lengths and the P—N—C bond angles are within the expected values for this category of molecules (Pourayoubi *et al.*, 2011). The P atom has a distorted tetrahedral configuration, as has been noted for the other phosphoric triamides. As expected, the P—N2 and P—N3 bonds are shorter than the P—N1 bond. In the C(O)NHP(O) moiety, the phosphoryl group has a *syn* orientation with respect to the N—H unit. The two other N—H units have an *anti* configuration relative to the P═O group.

In the crystal packing, each molecule is H-bonded to two neighbouring molecules through the $N_{C(O)NHP(O)}$—H···(O)P hydrogen bonds and the $[N—H]_2$···(O)C grouping, forming a sequence of alternated $R_2^2(8)$ and $R_2^2(12)R_2^1(6)$ rings (for graph-set definition in hydrogen bond motifs, see: Bernstein *et al.*, 1995) in a linear arrangement parallel to the *y* axis, Fig. 2 and Table 1. The carbonyl oxygen atom acts as a double hydrogen bond acceptor (Steiner, 2002).

S2. Experimental

Synthesis of $CH_2ClC(O)NHP(O)Cl_2$: The reaction of phosphorus pentachloride (33.9 mmol) and 2-chloroacetamide (33.9 mmol) in dry benzene (40 ml) at 353 K (3 h) and then the treatment of formic acid (33.9 mmol) at ice-bath temperature (2.5 h), then removing the solvent in vacuum, leads to the formation of $CH_2ClC(O)NHP(O)Cl_2$ as solid product.

Synthesis of the title molecule: To a solution of $CH_2ClC(O)NHP(O)Cl_2$ (2.42 mmol) in $CHCl_3$ (20 ml), a solution of *tert*-butylamine (9.68 mmol) in $CHCl_3$ (10 ml) was added dropwise at 273 K. After 4 h of stirring, the solvent was evaporated in vacuum. The obtained solid was washed with distilled water. Single crystals were obtained from a solution of the title molecule in $CHCl_3$ after slow evaporation at room temperature. IR (KBr, cm^{-1}): 3376, 3324, 3104, 2971, 2913, 1692, 1486, 1396, 1186, 1023, 880, 818.

S3. Refinement

H atoms were positioned geometrically with C—H = 0.99 and 0.98 Å for methylene and methyl H atoms, respectively, and with constrained distances N—H = 0.87 (1) Å in a riding model on their parent atoms with $U_{iso}(H)$ = 1.2 times $U_{eq}(N/C)$; the positions of H atoms of the methyl groups were rotationally optimized. Few low angle reflections were omitted as they were located behind the beam stop.

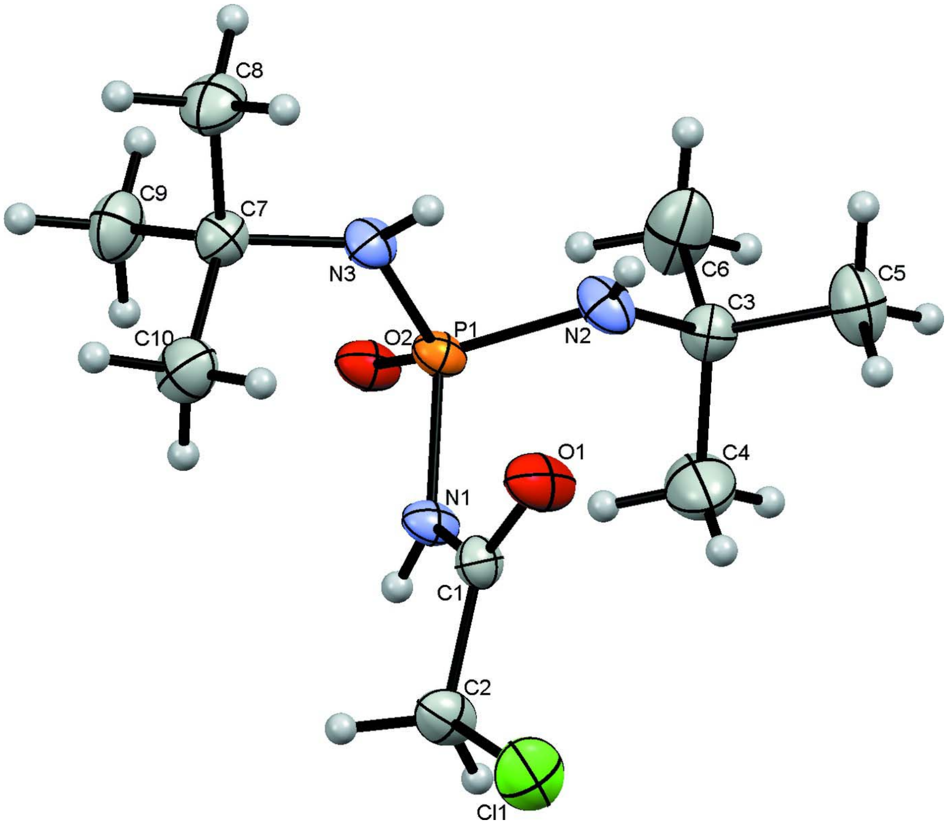

Figure 1

An *ORTEP* drawing of the title molecule with labeling. Displacement ellipsoids are given at 50% probability level.

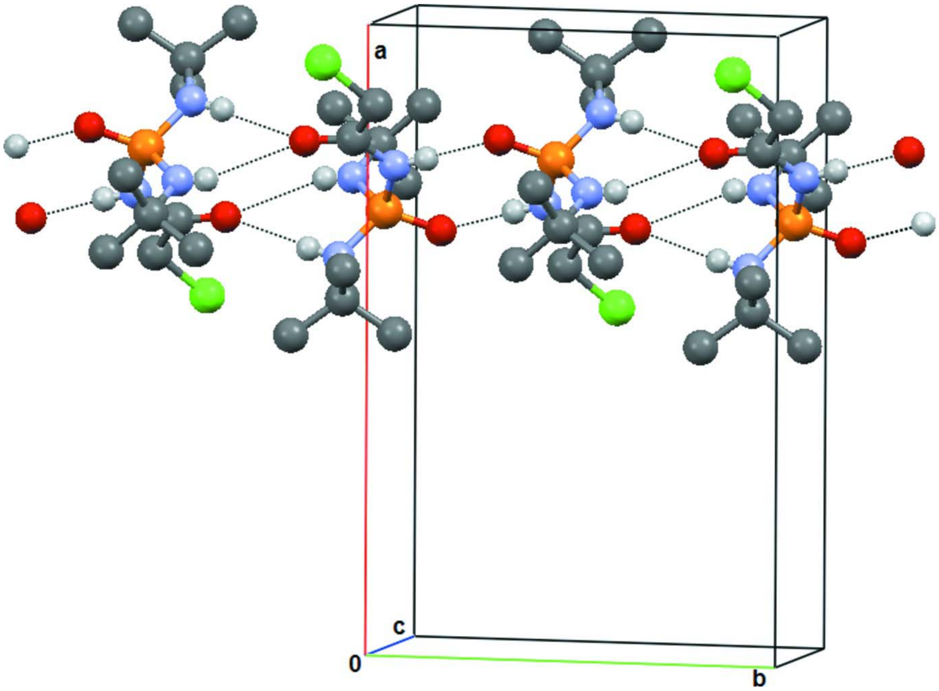

Figure 2

Part of the crystal packing with hydrogen bonds shown as dotted lines. Only H atoms involved in hydrogen bonds are shown.

N,*N*′-Di-*tert*-butyl-*N*′′-(2-chloroacetyl)phosphoric triamide

Crystal data

$C_{10}H_{23}ClN_3O_2P$	$F(000) = 1216$
$M_r = 283.73$	$D_x = 1.266$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 3520 reflections
$a = 16.4781$ (5) Å	$\theta = 1.0$–27.5°
$b = 9.8872$ (2) Å	$\mu = 0.36$ mm^{-1}
$c = 19.6509$ (6) Å	$T = 173$ K
$\beta = 111.5747$ (12)°	Prism, colorless
$V = 2977.26$ (14) Å^3	0.16 × 0.14 × 0.12 mm
$Z = 8$	

Data collection

Nonius KappaCCD diffractometer with APEXII CCD	6472 measured reflections
Radiation source: fine-focus sealed tube	3410 independent reflections
Graphite monochromator	2937 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.026$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
$T_{min} = 0.945$, $T_{max} = 0.958$	$h = -21 \rightarrow 21$
	$k = -11 \rightarrow 12$
	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.103$
$S = 1.10$
3410 reflections
169 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 4.5534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.94023 (4)	−0.09873 (6)	−0.09379 (3)	0.04413 (16)
P1	0.70496 (3)	0.03761 (4)	0.02879 (3)	0.02208 (13)
O1	0.81276 (10)	−0.15073 (13)	−0.02559 (9)	0.0381 (4)
O2	0.67546 (9)	0.17456 (12)	0.03962 (7)	0.0287 (3)
N1	0.77781 (10)	0.06685 (14)	−0.01251 (8)	0.0233 (3)
H1N	0.7907 (13)	0.1494 (12)	−0.0191 (11)	0.028*
N2	0.75351 (11)	−0.04883 (15)	0.10356 (8)	0.0284 (4)
H2N	0.7503 (14)	−0.1342 (10)	0.0981 (12)	0.034*
N3	0.63044 (10)	−0.06675 (15)	−0.02058 (9)	0.0248 (3)
H3N	0.6432 (13)	−0.1496 (11)	−0.0079 (11)	0.030*
C1	0.81982 (11)	−0.03053 (17)	−0.03486 (10)	0.0235 (4)
C2	0.87490 (13)	0.02675 (19)	−0.07542 (11)	0.0311 (4)
H2A	0.9129	0.0993	−0.0456	0.037*
H2B	0.8361	0.0672	−0.1221	0.037*
C3	0.80542 (12)	0.0073 (2)	0.17664 (10)	0.0280 (4)
C4	0.86795 (17)	0.1160 (3)	0.17114 (14)	0.0516 (6)
H4A	0.9015	0.1511	0.2201	0.062*
H4B	0.8347	0.1899	0.1400	0.062*
H4C	0.9079	0.0772	0.1498	0.062*
C5	0.85793 (16)	−0.1082 (2)	0.22316 (12)	0.0427 (5)
H5A	0.8914	−0.0751	0.2726	0.051*
H5B	0.8981	−0.1440	0.2011	0.051*
H5C	0.8182	−0.1801	0.2258	0.051*
C6	0.74387 (16)	0.0653 (3)	0.21120 (13)	0.0521 (7)

H6A	0.7780	0.1017	0.2597	0.063*
H6B	0.7052	−0.0064	0.2160	0.063*
H6C	0.7088	0.1377	0.1801	0.063*
C7	0.56724 (12)	−0.04828 (19)	−0.09668 (10)	0.0270 (4)
C8	0.50741 (14)	−0.1718 (2)	−0.11260 (13)	0.0392 (5)
H8A	0.4649	−0.1667	−0.1629	0.047*
H8B	0.4765	−0.1737	−0.0785	0.047*
H8C	0.5424	−0.2542	−0.1067	0.047*
C9	0.51410 (14)	0.0797 (2)	−0.10192 (12)	0.0381 (5)
H9A	0.4705	0.0884	−0.1516	0.046*
H9B	0.5530	0.1584	−0.0907	0.046*
H9C	0.4846	0.0747	−0.0669	0.046*
C10	0.61356 (14)	−0.0416 (2)	−0.15144 (11)	0.0380 (5)
H10A	0.5701	−0.0439	−0.2014	0.046*
H10B	0.6530	−0.1191	−0.1439	0.046*
H10C	0.6472	0.0425	−0.1440	0.046*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0473 (3)	0.0452 (3)	0.0496 (3)	0.0194 (3)	0.0291 (3)	0.0033 (2)
P1	0.0271 (2)	0.0159 (2)	0.0265 (2)	−0.00014 (17)	0.01385 (19)	0.00075 (17)
O1	0.0427 (8)	0.0170 (7)	0.0628 (10)	0.0058 (6)	0.0291 (8)	0.0069 (6)
O2	0.0354 (7)	0.0170 (6)	0.0402 (8)	0.0001 (5)	0.0216 (6)	−0.0002 (5)
N1	0.0280 (8)	0.0140 (7)	0.0319 (8)	−0.0010 (6)	0.0157 (6)	0.0008 (6)
N2	0.0405 (9)	0.0180 (7)	0.0265 (8)	−0.0030 (7)	0.0122 (7)	−0.0004 (6)
N3	0.0278 (8)	0.0151 (7)	0.0313 (8)	−0.0003 (6)	0.0107 (7)	0.0012 (6)
C1	0.0246 (8)	0.0190 (9)	0.0266 (9)	0.0035 (7)	0.0090 (7)	0.0023 (7)
C2	0.0355 (10)	0.0263 (10)	0.0390 (11)	0.0072 (8)	0.0224 (9)	0.0028 (8)
C3	0.0292 (9)	0.0277 (10)	0.0274 (9)	0.0009 (8)	0.0106 (8)	−0.0018 (8)
C4	0.0477 (14)	0.0489 (14)	0.0491 (14)	−0.0168 (12)	0.0070 (11)	0.0031 (11)
C5	0.0546 (14)	0.0398 (12)	0.0302 (10)	0.0109 (11)	0.0115 (10)	0.0018 (9)
C6	0.0453 (13)	0.0766 (18)	0.0349 (12)	0.0185 (13)	0.0153 (10)	−0.0099 (12)
C7	0.0263 (9)	0.0257 (9)	0.0286 (9)	0.0006 (7)	0.0096 (7)	−0.0014 (7)
C8	0.0342 (11)	0.0327 (11)	0.0460 (12)	−0.0070 (9)	0.0092 (9)	−0.0028 (9)
C9	0.0351 (11)	0.0327 (11)	0.0420 (12)	0.0090 (9)	0.0086 (9)	0.0019 (9)
C10	0.0357 (11)	0.0500 (13)	0.0278 (10)	−0.0014 (10)	0.0110 (9)	−0.0052 (9)

Geometric parameters (Å, º)

Cl1—C2	1.7647 (19)	C4—H4C	0.9800
P1—O2	1.4803 (13)	C5—H5A	0.9800
P1—N3	1.6241 (16)	C5—H5B	0.9800
P1—N2	1.6324 (16)	C5—H5C	0.9800
P1—N1	1.7030 (15)	C6—H6A	0.9800
O1—C1	1.214 (2)	C6—H6B	0.9800
N1—C1	1.350 (2)	C6—H6C	0.9800
N1—H1N	0.866 (9)	C7—C9	1.520 (3)

N2—C3	1.483 (2)	C7—C8	1.528 (3)
N2—H2N	0.850 (9)	C7—C10	1.533 (3)
N3—C7	1.486 (2)	C8—H8A	0.9800
N3—H3N	0.859 (9)	C8—H8B	0.9800
C1—C2	1.520 (2)	C8—H8C	0.9800
C2—H2A	0.9900	C9—H9A	0.9800
C2—H2B	0.9900	C9—H9B	0.9800
C3—C5	1.519 (3)	C9—H9C	0.9800
C3—C4	1.520 (3)	C10—H10A	0.9800
C3—C6	1.525 (3)	C10—H10B	0.9800
C4—H4A	0.9800	C10—H10C	0.9800
C4—H4B	0.9800		

O2—P1—N3	116.80 (8)	C3—C5—H5A	109.5
O2—P1—N2	115.32 (8)	C3—C5—H5B	109.5
N3—P1—N2	102.57 (8)	H5A—C5—H5B	109.5
O2—P1—N1	103.86 (7)	C3—C5—H5C	109.5
N3—P1—N1	109.50 (8)	H5A—C5—H5C	109.5
N2—P1—N1	108.65 (8)	H5B—C5—H5C	109.5
C1—N1—P1	124.72 (12)	C3—C6—H6A	109.5
C1—N1—H1N	116.1 (14)	C3—C6—H6B	109.5
P1—N1—H1N	119.1 (14)	H6A—C6—H6B	109.5
C3—N2—P1	126.36 (13)	C3—C6—H6C	109.5
C3—N2—H2N	118.8 (15)	H6A—C6—H6C	109.5
P1—N2—H2N	114.8 (15)	H6B—C6—H6C	109.5
C7—N3—P1	128.25 (12)	N3—C7—C9	110.09 (15)
C7—N3—H3N	114.5 (14)	N3—C7—C8	105.57 (15)
P1—N3—H3N	112.5 (14)	C9—C7—C8	109.82 (17)
O1—C1—N1	124.08 (17)	N3—C7—C10	111.48 (15)
O1—C1—C2	123.48 (16)	C9—C7—C10	109.86 (17)
N1—C1—C2	112.41 (15)	C8—C7—C10	109.94 (17)
C1—C2—Cl1	111.84 (13)	C7—C8—H8A	109.5
C1—C2—H2A	109.2	C7—C8—H8B	109.5
Cl1—C2—H2A	109.2	H8A—C8—H8B	109.5
C1—C2—H2B	109.2	C7—C8—H8C	109.5
Cl1—C2—H2B	109.2	H8A—C8—H8C	109.5
H2A—C2—H2B	107.9	H8B—C8—H8C	109.5
N2—C3—C5	107.42 (16)	C7—C9—H9A	109.5
N2—C3—C4	111.14 (17)	C7—C9—H9B	109.5
C5—C3—C4	108.87 (18)	H9A—C9—H9B	109.5
N2—C3—C6	109.32 (16)	C7—C9—H9C	109.5
C5—C3—C6	109.85 (18)	H9A—C9—H9C	109.5
C4—C3—C6	110.2 (2)	H9B—C9—H9C	109.5
C3—C4—H4A	109.5	C7—C10—H10A	109.5
C3—C4—H4B	109.5	C7—C10—H10B	109.5
H4A—C4—H4B	109.5	H10A—C10—H10B	109.5
C3—C4—H4C	109.5	C7—C10—H10C	109.5
H4A—C4—H4C	109.5	H10A—C10—H10C	109.5

H4B—C4—H4C	109.5	H10B—C10—H10C	109.5

O2—P1—N1—C1	−177.92 (15)	P1—N1—C1—C2	175.25 (13)
N3—P1—N1—C1	−52.46 (17)	O1—C1—C2—Cl1	−9.6 (3)
N2—P1—N1—C1	58.83 (17)	N1—C1—C2—Cl1	172.32 (13)
O2—P1—N2—C3	−29.69 (19)	P1—N2—C3—C5	−164.41 (15)
N3—P1—N2—C3	−157.76 (15)	P1—N2—C3—C4	−45.4 (2)
N1—P1—N2—C3	86.38 (16)	P1—N2—C3—C6	76.4 (2)
O2—P1—N3—C7	55.94 (17)	P1—N3—C7—C9	−56.2 (2)
N2—P1—N3—C7	−176.93 (15)	P1—N3—C7—C8	−174.63 (14)
N1—P1—N3—C7	−61.68 (16)	P1—N3—C7—C10	66.0 (2)
P1—N1—C1—O1	−2.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.87 (1)	1.92 (1)	2.778 (2)	175 (2)
N2—H2*N*···O1^{ii}	0.85 (1)	2.56 (1)	3.337 (2)	153 (2)
N3—H3*N*···O1^{ii}	0.86 (1)	2.12 (1)	2.979 (2)	175 (2)

Symmetry codes: (i) $-x+3/2, -y+1/2, -z$; (ii) $-x+3/2, -y-1/2, -z$.