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N,*N*′-Bis(2-chlorobenzyl)-*N*″-(2,2,2-trichloroacetyl)phosphoric triamide

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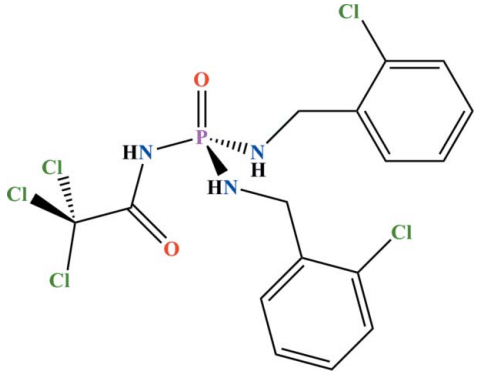
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.053; wR factor = 0.129; data-to-parameter ratio = 18.9.

The P atom in the title compound, $C_{16}H_{15}Cl_5N_3O_2P$, exhibits a tetrahedral coordination geometry and the phosphoryl and carbonyl groups are *anti* with respect to one another. The dihedral angle between the benzene rings is 44.90 (15)°. One of the 2-chlorobenzylamido fragments is disordered over two sets of sites with occupancies of 0.8823 (17) and 0.1177 (17). In the crystal, adjacent molecules are linked *via* N—H···O(P) and N—H···O(C) hydrogen bonds into an extended chain running parallel to the *a* axis.

Related literature

For details of compounds having a C(O)NHP(O) skeleton, see: Toghraee *et al.* (2011). For bond lengths in related structures, see: Pourayoubi *et al.* (2011); Rudd *et al.* (1996).



Experimental

Crystal data

$C_{16}H_{15}Cl_5N_3O_2P$
M_r = 489.53
Triclinic, $P\overline{1}$
a = 9.9789 (2) Å
b = 10.6058 (3) Å
c = 10.8386 (3) Å
α = 75.8920 (13)°
β = 72.2250 (15)°
γ = 69.6050 (15)°
V = 1011.66 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 173 K
0.18 × 0.14 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer with APEXII CCD detector
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.867, T_{max} = 0.909
8947 measured reflections
4638 independent reflections
4083 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.129
S = 1.05
4638 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.88	1.94	2.804 (3)	168
N2—H2···O1^{ii}	0.88	2.38	3.106 (3)	141
N2′—H2′···O1	0.88	2.42	3.025 (3)	126
N3—H3···O1^{ii}	0.88	2.34	3.129 (3)	149

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

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: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5176).

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supporting information

Acta Cryst. (2011). E**67**, o2046 [doi:10.1107/S1600536811027681]

N,*N*′-Bis(2-chlorobenzyl)-*N*′′-(2,2,2-trichloroacetyl)phosphoric triamide

Mehrdad Pourayoubi, Hassan Fadaei and Masood Parvez

S1. Comment

The structure determination of the title compound, P(O)[NHC(O)CCl_3][$NHCH_2C_6H_4$(2-Cl)]$_2$ (Fig. 1), was performed as a part of a project in our laboratory on the synthesis of new phosphoramidate compounds having a C(O)NHP(O) skeleton (Toghraee *et al.*, 2011). Single crystals of the title compound were obtained from a solution of CH_3OH after slow evaporation at room temperature.

The P═O (1.472 (2) Å) and C═O (1.212 (3) Å) bond lengths are standard for this category of compounds (Pourayoubi *et al.*, 2011). The P atom has a distorted tetrahedral configuration (Fig. 1) as has been noted for other phosphoric triamides and their chalco-derivatives (Rudd *et al.*, 1996). The bond angles at the P atom vary in the range 103.26 (13)-116.10 (12)°. The P—N2 and P—N3 bonds (with lengths of 1.621 (2) Å and 1.622 (2) Å) are shorter than the P—N1 bond (1.715 (2) Å). As can be expected the C1—N1 bond distance (1.339 (3) Å) is shorter than the other C—N bond distances. The N—H unit of C(O)NHP(O) moiety and the phosphoryl group have a *syn* orientation with respect to each other. One of the 2-chlorobenzylamido fragments is disordered over two sites with occupancies of 0.8823 (17) and 0.1177 (17).

In the crystal, each molecule is hydrogen-bonded to two adjacent molecules through $N_{C(O)NHP(O)}$—H···O(P) and N—H···O(C) hydrogen bonds forming linear chains parallel to [100] (Table 1).

S2. Experimental

The reaction of phosphorus pentachloride (35.6 mmol) and 2,2,2-trichloroacetamide (35.6 mmol) in dry CCl_4 (25 ml) at 353 K (3 h) and then treatment with formic acid 85% (35.6 mmol) at 271 K leads to the formation of $CCl_3C(O)NHP(O)Cl_2$ as a white solid.

To a solution of $CCl_3C(O)NHP(O)Cl_2$ (2.5 mmol) in dry $CHCl_3$ (30 ml), a solution of 2-chlorobenzylamine (10 mmol) in dry $CHCl_3$ (10 ml) was added dropwise at 271 K. After 4 h stirring, the solvent was evaporated at room temperature. The solid was washed with distilled water and recrystallized from CH_3OH.

S3. Refinement

The H-atoms were included at geometrically idealized positions with distances N—H = 0.88 Å and C—H = 0.95 and 0.99 Å for aryl and methylene type H-atoms, respectively. The chlorobenzyl group attached to N2 was disordered with its atoms located over two sites with site occupancy factors 0.8823 (17) and 0.1177 (17). The H-atoms were assigned U_{iso} = 1.2 times U_{eq} of the parent atoms (C/N). The highest electron density peak in the final difference map was located close to a Cl atom and was essentially meaningless.

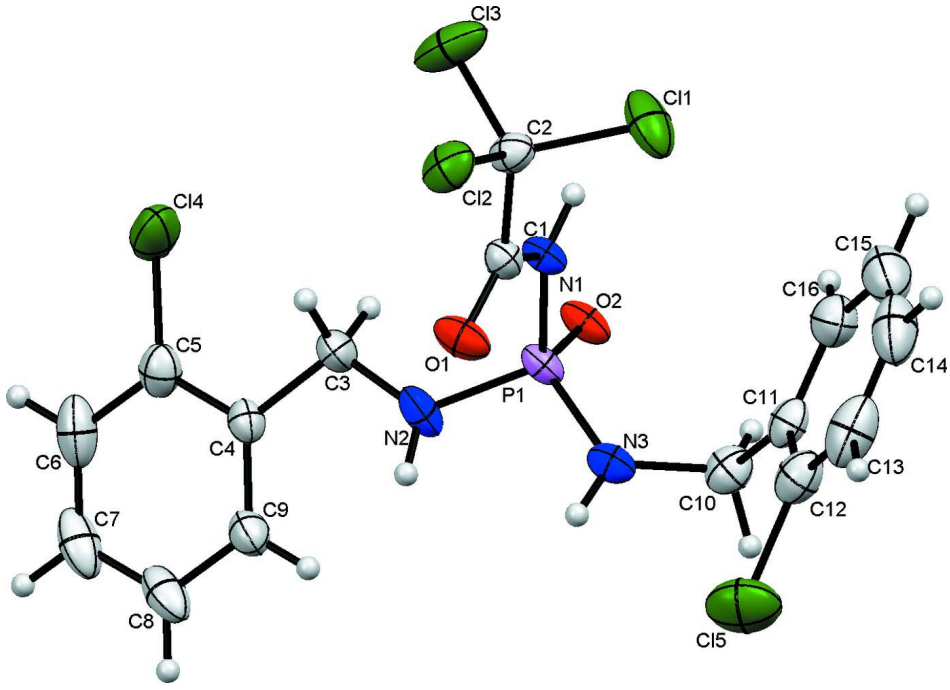

Figure 1

The molecular structure of the title compound with ellipsoids shown at the 50% probability level. The minor disorder component is not shown.

N,*N*′-Bis(2-chlorobenzyl)-*N*′′-(2,2,2- trichloroacetyl)phosphoric triamide

Crystal data

$C_{16}H_{15}Cl_5N_3O_2P$
$M_r = 489.53$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.9789\ (2)$ Å
$b = 10.6058\ (3)$ Å
$c = 10.8386\ (3)$ Å
$\alpha = 75.8920\ (13)°$
$\beta = 72.2250\ (15)°$
$\gamma = 69.6050\ (15)°$
$V = 1011.66\ (4)$ Å^3
$Z = 2$
$F(000) = 496$
$D_x = 1.607$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4567 reflections
$\theta = 1.0–27.5°$
$\mu = 0.81$ mm^{-1}
$T = 173$ K
Prism, colorless
0.18 × 0.14 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer with APEXII CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.867$, $T_{max} = 0.909$
8947 measured reflections
4638 independent reflections
4083 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.129$
$S = 1.05$
4638 reflections
245 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 2.0234P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.42$ e Å^{-3}
$\Delta\rho_{min} = -0.68$ e Å^{-3}

Special details

Experimental. IR (KBr, cm^{-1}): 3371, 3061, 2875, 1692, 1448, 1257, 1224, 1076, 1043, 885, 837, 751, 675.
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)
Cl1	0.58102 (9)	−0.04802 (11)	1.36183 (8)	0.0547 (3)	
Cl2	0.87488 (8)	−0.22348 (7)	1.35952 (8)	0.03998 (19)	
Cl3	0.68228 (12)	−0.30242 (9)	1.26210 (11)	0.0590 (3)	
P1	0.72798 (7)	0.04068 (7)	0.90391 (6)	0.02507 (16)	
O1	0.9079 (2)	−0.0722 (2)	1.1030 (2)	0.0386 (5)	
O2	0.60042 (19)	0.0572 (2)	0.85370 (18)	0.0317 (4)	
N1	0.6903 (2)	−0.0446 (2)	1.0605 (2)	0.0262 (4)	
H1	0.6044	−0.0605	1.0925	0.031*	
C1	0.7866 (3)	−0.0882 (3)	1.1360 (3)	0.0259 (5)	
C2	0.7338 (3)	−0.1636 (3)	1.2751 (3)	0.0301 (5)	
N2	0.8865 (2)	−0.0463 (3)	0.8250 (2)	0.0352 (6)	
H2	0.9630	−0.0151	0.8031	0.042*	0.8823 (17)
C3	0.9024 (3)	−0.1740 (3)	0.7909 (3)	0.0335 (7)	0.8823 (17)
H3A	0.8074	−0.1713	0.7779	0.040*	0.8823 (17)
H3B	0.9247	−0.2470	0.8649	0.040*	0.8823 (17)
C4	1.0236 (2)	−0.2094 (2)	0.66667 (18)	0.0269 (7)	0.8823 (17)
C5	1.0496 (2)	−0.3322 (2)	0.6261 (2)	0.0362 (7)	0.8823 (17)
C6	1.1578 (3)	−0.3675 (2)	0.5135 (2)	0.0476 (10)	0.8823 (17)
H6	1.1755	−0.4515	0.4858	0.057*	0.8823 (17)
C7	1.2401 (3)	−0.2800 (3)	0.4415 (2)	0.0501 (11)	0.8823 (17)
H7	1.3141	−0.3041	0.3645	0.060*	0.8823 (17)
C8	1.2141 (2)	−0.1572 (2)	0.4820 (2)	0.0467 (9)	0.8823 (17)
H8	1.2704	−0.0973	0.4328	0.056*	0.8823 (17)
C9	1.1059 (2)	−0.12185 (18)	0.5946 (2)	0.0342 (7)	0.8823 (17)

H9	1.0882	−0.0379	0.6223	0.041*	0.8823 (17)
Cl4	0.94994 (12)	−0.44175 (10)	0.71412 (13)	0.0605 (3)	0.8823 (17)
H2′	0.9162	−0.1096	0.8886	0.042*	0.1177 (17)
C3′	0.993 (3)	−0.079 (2)	0.730 (2)	0.0335 (7)	0.1177 (17)
H3′1	1.0801	−0.0997	0.7645	0.040*	0.1177 (17)
H3′2	0.9892	0.0079	0.6686	0.040*	0.1177 (17)
C4′	1.036 (2)	−0.1806 (18)	0.6432 (18)	0.0269 (7)	0.1177 (17)
C5′	1.148 (2)	−0.1813 (19)	0.529 (2)	0.0362 (7)	0.1177 (17)
C6′	1.199 (2)	−0.289 (2)	0.4586 (18)	0.0476 (10)	0.1177 (17)
H6′	1.2753	−0.2892	0.3808	0.057*	0.1177 (17)
C7′	1.137 (2)	−0.395 (2)	0.501 (2)	0.0501 (11)	0.1177 (17)
H7′	1.1711	−0.4687	0.4529	0.060*	0.1177 (17)
C8′	1.024 (2)	−0.3946 (17)	0.6152 (19)	0.0467 (9)	0.1177 (17)
H8′	0.9819	−0.4675	0.6445	0.056*	0.1177 (17)
C9′	0.9738 (19)	−0.287 (2)	0.6861 (15)	0.0342 (7)	0.1177 (17)
H9′	0.8970	−0.2867	0.7638	0.041*	0.1177 (17)
Cl4′	1.2278 (9)	−0.0556 (8)	0.4817 (10)	0.0605 (3)	0.1177 (17)
N3	0.7555 (2)	0.1823 (2)	0.9050 (2)	0.0309 (5)	
H3	0.8465	0.1849	0.8889	0.037*	
C10	0.6334 (3)	0.3062 (3)	0.9322 (3)	0.0356 (6)	
H10A	0.5478	0.3027	0.9065	0.043*	
H10B	0.6632	0.3860	0.8773	0.043*	
C11	0.5868 (3)	0.3256 (3)	1.0740 (3)	0.0322 (6)	
C12	0.6657 (4)	0.3724 (3)	1.1291 (3)	0.0409 (7)	
C13	0.6224 (5)	0.3866 (4)	1.2614 (4)	0.0529 (9)	
H13	0.6780	0.4188	1.2971	0.064*	
C14	0.4988 (5)	0.3535 (4)	1.3393 (4)	0.0584 (10)	
H14	0.4689	0.3624	1.4296	0.070*	
C15	0.4189 (4)	0.3081 (4)	1.2880 (4)	0.0559 (9)	
H15	0.3331	0.2856	1.3424	0.067*	
C16	0.4620 (4)	0.2945 (3)	1.1571 (3)	0.0437 (7)	
H16	0.4047	0.2629	1.1228	0.052*	
Cl5	0.82320 (12)	0.41376 (12)	1.03083 (12)	0.0672 (3)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0395 (4)	0.0749 (6)	0.0293 (4)	0.0045 (4)	−0.0024 (3)	−0.0105 (4)
Cl2	0.0424 (4)	0.0350 (4)	0.0463 (4)	−0.0098 (3)	−0.0260 (3)	0.0039 (3)
Cl3	0.0763 (6)	0.0416 (5)	0.0793 (7)	−0.0360 (4)	−0.0472 (5)	0.0180 (4)
P1	0.0178 (3)	0.0380 (4)	0.0204 (3)	−0.0100 (3)	−0.0013 (2)	−0.0080 (3)
O1	0.0246 (10)	0.0590 (14)	0.0358 (11)	−0.0193 (9)	−0.0083 (8)	−0.0023 (10)
O2	0.0199 (9)	0.0517 (12)	0.0251 (9)	−0.0139 (8)	−0.0036 (7)	−0.0063 (8)
N1	0.0195 (10)	0.0363 (12)	0.0235 (10)	−0.0118 (9)	−0.0025 (8)	−0.0042 (9)
C1	0.0224 (12)	0.0282 (13)	0.0269 (13)	−0.0077 (10)	−0.0050 (10)	−0.0049 (10)
C2	0.0302 (13)	0.0271 (13)	0.0333 (14)	−0.0094 (10)	−0.0119 (11)	0.0013 (10)
N2	0.0201 (10)	0.0533 (15)	0.0354 (13)	−0.0106 (10)	0.0012 (9)	−0.0232 (11)
C3	0.0285 (15)	0.0314 (16)	0.0339 (16)	−0.0083 (12)	0.0032 (12)	−0.0078 (13)

C4	0.0245 (13)	0.0277 (16)	0.0247 (15)	−0.0021 (12)	−0.0062 (11)	−0.0053 (12)
C5	0.0303 (16)	0.0358 (17)	0.0444 (19)	−0.0038 (13)	−0.0142 (14)	−0.0117 (14)
C6	0.039 (2)	0.052 (2)	0.050 (2)	0.0103 (17)	−0.0210 (17)	−0.0285 (19)
C7	0.040 (2)	0.068 (3)	0.0244 (17)	0.0100 (19)	−0.0069 (15)	−0.0146 (17)
C8	0.0312 (17)	0.061 (2)	0.0269 (17)	0.0004 (16)	−0.0008 (14)	0.0019 (16)
C9	0.0285 (15)	0.0358 (17)	0.0307 (16)	−0.0046 (13)	−0.0026 (12)	−0.0050 (13)
Cl4	0.0475 (5)	0.0380 (5)	0.0988 (9)	−0.0148 (4)	−0.0086 (5)	−0.0240 (5)
N2′	0.0201 (10)	0.0533 (15)	0.0354 (13)	−0.0106 (10)	0.0012 (9)	−0.0232 (11)
C3′	0.0285 (15)	0.0314 (16)	0.0339 (16)	−0.0083 (12)	0.0032 (12)	−0.0078 (13)
C4′	0.0245 (13)	0.0277 (16)	0.0247 (15)	−0.0021 (12)	−0.0062 (11)	−0.0053 (12)
C5′	0.0303 (16)	0.0358 (17)	0.0444 (19)	−0.0038 (13)	−0.0142 (14)	−0.0117 (14)
C6′	0.039 (2)	0.052 (2)	0.050 (2)	0.0103 (17)	−0.0210 (17)	−0.0285 (19)
C7′	0.040 (2)	0.068 (3)	0.0244 (17)	0.0100 (19)	−0.0069 (15)	−0.0146 (17)
C8′	0.0312 (17)	0.061 (2)	0.0269 (17)	0.0004 (16)	−0.0008 (14)	0.0019 (16)
C9′	0.0285 (15)	0.0358 (17)	0.0307 (16)	−0.0046 (13)	−0.0026 (12)	−0.0050 (13)
Cl4′	0.0475 (5)	0.0380 (5)	0.0988 (9)	−0.0148 (4)	−0.0086 (5)	−0.0240 (5)
N3	0.0251 (11)	0.0398 (13)	0.0293 (12)	−0.0138 (10)	−0.0051 (9)	−0.0035 (10)
C10	0.0399 (15)	0.0336 (15)	0.0342 (15)	−0.0088 (12)	−0.0169 (12)	0.0002 (11)
C11	0.0369 (14)	0.0232 (13)	0.0353 (15)	−0.0020 (11)	−0.0174 (12)	−0.0017 (11)
C12	0.0452 (17)	0.0320 (15)	0.0484 (18)	−0.0072 (13)	−0.0219 (14)	−0.0041 (13)
C13	0.073 (3)	0.0379 (18)	0.054 (2)	0.0001 (16)	−0.038 (2)	−0.0127 (15)
C14	0.063 (2)	0.050 (2)	0.0396 (19)	0.0104 (18)	−0.0097 (17)	−0.0114 (16)
C15	0.048 (2)	0.053 (2)	0.048 (2)	−0.0016 (16)	−0.0025 (16)	−0.0043 (17)
C16	0.0402 (17)	0.0381 (17)	0.0478 (19)	−0.0070 (13)	−0.0101 (14)	−0.0053 (14)
Cl5	0.0632 (6)	0.0785 (7)	0.0774 (7)	−0.0424 (5)	−0.0238 (5)	−0.0030 (5)

Geometric parameters (Å, º)

Cl1—C2	1.759 (3)	C3′—H3′2	0.9900
Cl2—C2	1.761 (3)	C4′—C5′	1.3900
Cl3—C2	1.768 (3)	C4′—C9′	1.3900
P1—O2	1.4724 (18)	C5′—C6′	1.3900
P1—N2	1.621 (2)	C5′—Cl4′	1.682 (17)
P1—N3	1.622 (2)	C6′—C7′	1.3900
P1—N1	1.715 (2)	C6′—H6′	0.9500
O1—C1	1.212 (3)	C7′—C8′	1.3900
N1—C1	1.339 (3)	C7′—H7′	0.9500
N1—H1	0.8800	C8′—C9′	1.3900
C1—C2	1.553 (4)	C8′—H8′	0.9500
N2—C3	1.434 (4)	C9′—H9′	0.9500
N2—H2	0.8800	N3—C10	1.467 (4)
C3—C4	1.534 (3)	N3—H3	0.8800
C3—H3A	0.9900	C10—C11	1.507 (4)
C3—H3B	0.9900	C10—H10A	0.9900
C4—C5	1.3900	C10—H10B	0.9900
C4—C9	1.3900	C11—C12	1.388 (4)
C5—C6	1.3900	C11—C16	1.389 (4)
C5—Cl4	1.713 (2)	C12—C13	1.395 (5)

C6—C7	1.3900	C12—Cl5	1.740 (4)
C6—H6	0.9500	C13—C14	1.371 (6)
C7—C8	1.3900	C13—H13	0.9500
C7—H7	0.9500	C14—C15	1.359 (6)
C8—C9	1.3900	C14—H14	0.9500
C8—H8	0.9500	C15—C16	1.380 (5)
C9—H9	0.9500	C15—H15	0.9500
C3′—C4′	1.47 (3)	C16—H16	0.9500
C3′—H3′1	0.9900		

O2—P1—N2	116.10 (12)	H3′1—C3′—H3′2	105.4
O2—P1—N3	114.51 (12)	C5′—C4′—C9′	120.0
N2—P1—N3	103.26 (13)	C5′—C4′—C3′	121.6 (16)
O2—P1—N1	104.46 (11)	C9′—C4′—C3′	118.0 (16)
N2—P1—N1	108.13 (13)	C4′—C5′—C6′	120.0
N3—P1—N1	110.31 (11)	C4′—C5′—Cl4′	119.8 (12)
C1—N1—P1	122.56 (18)	C6′—C5′—Cl4′	120.1 (12)
C1—N1—H1	118.7	C5′—C6′—C7′	120.0
P1—N1—H1	118.7	C5′—C6′—H6′	120.0
O1—C1—N1	124.8 (2)	C7′—C6′—H6′	120.0
O1—C1—C2	119.8 (2)	C8′—C7′—C6′	120.0
N1—C1—C2	115.5 (2)	C8′—C7′—H7′	120.0
C1—C2—Cl1	108.21 (18)	C6′—C7′—H7′	120.0
C1—C2—Cl2	110.50 (18)	C7′—C8′—C9′	120.0
Cl1—C2—Cl2	109.24 (16)	C7′—C8′—H8′	120.0
C1—C2—Cl3	109.63 (19)	C9′—C8′—H8′	120.0
Cl1—C2—Cl3	109.77 (15)	C8′—C9′—C4′	120.0
Cl2—C2—Cl3	109.47 (15)	C8′—C9′—H9′	120.0
C3—N2—P1	120.0 (2)	C4′—C9′—H9′	120.0
C3—N2—H2	120.0	C10—N3—P1	122.06 (19)
P1—N2—H2	120.0	C10—N3—H3	119.0
N2—C3—C4	113.7 (2)	P1—N3—H3	119.0
N2—C3—H3A	108.8	N3—C10—C11	113.5 (2)
C4—C3—H3A	108.8	N3—C10—H10A	108.9
N2—C3—H3B	108.8	C11—C10—H10A	108.9
C4—C3—H3B	108.8	N3—C10—H10B	108.9
H3A—C3—H3B	107.7	C11—C10—H10B	108.9
C5—C4—C9	120.0	H10A—C10—H10B	107.7
C5—C4—C3	118.28 (18)	C12—C11—C16	116.5 (3)
C9—C4—C3	121.71 (18)	C12—C11—C10	122.8 (3)
C4—C5—C6	120.0	C16—C11—C10	120.7 (3)
C4—C5—Cl4	120.65 (14)	C11—C12—C13	121.7 (3)
C6—C5—Cl4	119.35 (14)	C11—C12—Cl5	119.1 (3)
C7—C6—C5	120.0	C13—C12—Cl5	119.1 (3)
C7—C6—H6	120.0	C14—C13—C12	119.3 (3)
C5—C6—H6	120.0	C14—C13—H13	120.3
C6—C7—C8	120.0	C12—C13—H13	120.3
C6—C7—H7	120.0	C15—C14—C13	120.3 (4)

C8—C7—H7	120.0	C15—C14—H14	119.9
C9—C8—C7	120.0	C13—C14—H14	119.9
C9—C8—H8	120.0	C14—C15—C16	120.1 (4)
C7—C8—H8	120.0	C14—C15—H15	119.9
C8—C9—C4	120.0	C16—C15—H15	119.9
C8—C9—H9	120.0	C15—C16—C11	121.9 (3)
C4—C9—H9	120.0	C15—C16—H16	119.0
C4′—C3′—H3′1	103.9	C11—C16—H16	119.0
C4′—C3′—H3′2	103.9		

O2—P1—N1—C1	174.8 (2)	C9′—C4′—C5′—C6′	0.0
N2—P1—N1—C1	50.6 (2)	C3′—C4′—C5′—C6′	172 (2)
N3—P1—N1—C1	−61.6 (2)	C9′—C4′—C5′—Cl4′	−177.3 (15)
P1—N1—C1—O1	0.2 (4)	C3′—C4′—C5′—Cl4′	−5 (2)
P1—N1—C1—C2	−179.98 (18)	C4′—C5′—C6′—C7′	0.0
O1—C1—C2—Cl1	116.0 (3)	Cl4′—C5′—C6′—C7′	177.2 (16)
N1—C1—C2—Cl1	−63.9 (3)	C5′—C6′—C7′—C8′	0.0
O1—C1—C2—Cl2	−3.6 (3)	C6′—C7′—C8′—C9′	0.0
N1—C1—C2—Cl2	176.56 (19)	C7′—C8′—C9′—C4′	0.0
O1—C1—C2—Cl3	−124.3 (2)	C5′—C4′—C9′—C8′	0.0
N1—C1—C2—Cl3	55.8 (3)	C3′—C4′—C9′—C8′	−172 (2)
O2—P1—N2—C3	−47.0 (3)	O2—P1—N3—C10	34.9 (2)
N3—P1—N2—C3	−173.2 (2)	N2—P1—N3—C10	162.0 (2)
N1—P1—N2—C3	69.9 (3)	N1—P1—N3—C10	−82.6 (2)
P1—N2—C3—C4	151.5 (2)	P1—N3—C10—C11	94.5 (3)
N2—C3—C4—C5	179.0 (2)	N3—C10—C11—C12	77.3 (3)
N2—C3—C4—C9	−1.5 (4)	N3—C10—C11—C16	−101.5 (3)
C9—C4—C5—C6	0.0	C16—C11—C12—C13	0.4 (4)
C3—C4—C5—C6	179.5 (2)	C10—C11—C12—C13	−178.5 (3)
C9—C4—C5—Cl4	179.67 (18)	C16—C11—C12—Cl5	−179.9 (2)
C3—C4—C5—Cl4	−0.8 (2)	C10—C11—C12—Cl5	1.2 (4)
C4—C5—C6—C7	0.0	C11—C12—C13—C14	0.0 (5)
Cl4—C5—C6—C7	−179.67 (18)	Cl5—C12—C13—C14	−179.7 (3)
C5—C6—C7—C8	0.0	C12—C13—C14—C15	−0.3 (5)
C6—C7—C8—C9	0.0	C13—C14—C15—C16	0.2 (6)
C7—C8—C9—C4	0.0	C14—C15—C16—C11	0.2 (5)
C5—C4—C9—C8	0.0	C12—C11—C16—C15	−0.5 (5)
C3—C4—C9—C8	−179.5 (3)	C10—C11—C16—C15	178.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.88	1.94	2.804 (3)	168
N2—H2···O1^{ii}	0.88	2.38	3.106 (3)	141
N2′—H2′···O1	0.88	2.42	3.025 (3)	126
N3—H3···O1^{ii}	0.88	2.34	3.129 (3)	149

Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*+2, −*y*, −*z*+2.