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Crystal structure of diethyl {2,2,2-trichloro-1-[2-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-4-methylpentanamido]ethyl}phosphonate

V. S. Brovarets,[a] O. V. Golovchenko,[a] E. B. Rusanov[b] and J. A. Rusanova[c]*

[a]Institute of Bioorganic Chemistry and Petrochemistry, National Academy of, Sciences of Ukraine, 1 Murmanska St., Kyiv 02660, Ukraine, [b]Institute of Organic Chemistry, National Academy of Sciences of Ukraine, 5, Murmanska St., Kyiv 02660, Ukraine, and [c]Department of Chemistry, Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska Street, Kyiv 01601, Ukraine. *Correspondence e-mail: rusanova.j@gmail.com

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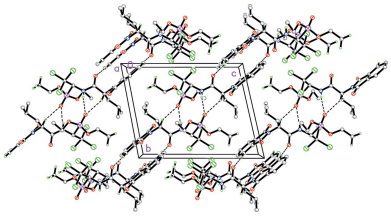
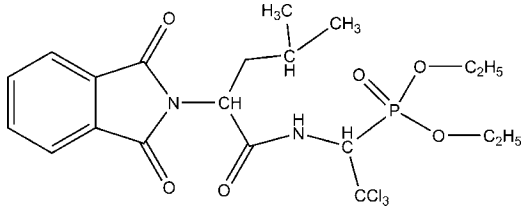
In the title phosphorylated compound, $C_{20}H_{26}Cl_3N_2O_6P$, the phthalimide unit is essentially planar (r.m.s. deviation = 0.0129 Å) and the O atoms of this unit deviate from the mean plane by 0.080 (3) and 0.041 (3) Å. In the crystal, pairs of molecules are linked by N—H···O and weak C—H···O hydrogen bonds involving the same acceptor atom, forming inversion dimers. In addition, π–π stacking interactions between the phthalimide groups, with a centroid–centroid distance of 3.7736 (13) Å, and further weak C—H···O hydrogen bonds connect the inversion dimers into columns along [0$\bar{1}$1].

1. Chemical context

In early 1950, J. W. Cornforth noted that oxazole derivatives rarely occur in nature and were therefore not promising as new biologically active substances. Studies performed mostly during recent decades have shown that the oxazole ring occurs in a multitude of natural products and it has been widely employed as a component of biologically active compounds in medicinal chemistry (Jin *et al.*, 2006). Various bacteria and marine organisms produce numerous antibiotics belonging to the oxazole series (Chamberlin *et al.*, 1977; Bertram *et al.*, 2001; Jansen *et al.*,1992; Moody & Bagley, 1998). Today, numerous oxazole-based synthetic bioregulators with strong antimicrobial, cytostatic, immune stimulating, neuroleptic, analgesic, and other kinds of biological activity are known (Turchi *et al.*, 1986; Palmer *et al.*, 2003). In particular, 5-amino-1,3-oxazole and its derivatives are well recognized for their potent and diverse bioregulation activity. Here we present the crystal structure of the title compound, which is an intermediate product of synthesis of phosphorylated 5-amino-1,3-oxazol-4-ylphosphonic acid derivatives.





2. Structural commentary

The molecular structure of the title compound is illustrated in Fig. 1. The phthalimide unit (N1/C1–C8) is essentially planar

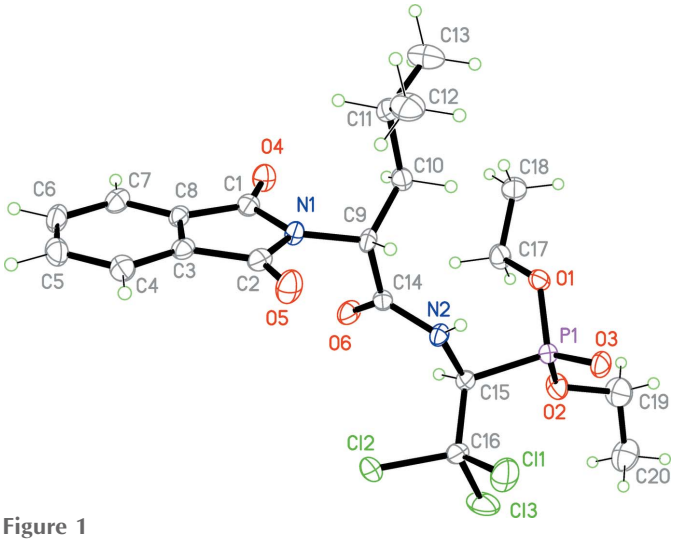


Figure 1
The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

with an r.m.s. deviation of 0.0129 Å. The oxygen atoms O4 and O5 deviate from the mean plane by 0.080 (3) and 0.041 (3) Å, respectively. In the five-membered 3-pyrroline ring, the C—C bond lengths are equivalent [C1—C8 = 1.487 (3) and C2—C3 = 1.486 (3) Å] and the C—N bond lengths differ slightly [N1—C1 = 1.417 (3) and N1—C2 = 1.398 (3) Å], while the corresponding bond angles are not equal [C1—N1—C9 = 127.97 (18) and C2—N1—C9 = 120.53 (17)°] possibly due to the steric influence of the isobutyl group. The mean C—C bond length in the C3–C8 phenyl ring is 1.387 Å. All bond lengths and angles are within normal ranges (Ng, 1992; Feeder & Jones, 1996).

In the acetamide moiety, the lone pair of atom N2 is conjugated with the π-system of the C=O group. Thus, the sum of nitrogen valency angles is 359.3° and the C14—N2 bond length of 1.356 (3) Å is intermediate between that for a double and a single bond (1.28 and 1.45 Å, respectively; Allen *et al.*, 1987). The C15—N2 bond has a typical value for a single bond at 1.442 (3) Å.

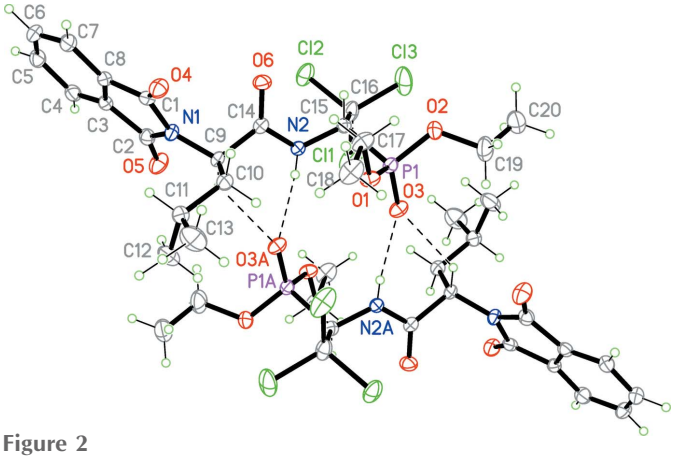


Figure 2
An inversion pair of the title compound, showing the intermolecular N—H···O and C—H···O hydrogen bonds [symmetry code: (A) $1-x, 1-y, 1-z$].

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O3^{i}	0.76 (2)	2.09 (2)	2.846 (3)	170 (2)
C9—H9···O3^{i}	1.00	2.47	3.265 (3)	136
C7—H7···O6^{ii}	0.95	2.46	3.393 (3)	169

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

The P—O distances in the phosphonate group show typical values for double [P1=O3 1.4616 (15) Å] and single (with bridging O1 and O2) bonds. The P1—O1 and P1—O2 bonds are equivalent within experimental error with values of 1.5670 (15) and 1.5664 (16) Å, respectively. The C15—P1—O1 and C15—P1—O2 bond angles are equivalent [103.45 (9) and 102.73 (9)°, respectively], while angles O1—P1—O3 and O2—P1—O3 are not [109.82 (9) and 116.77 (9)°], which is probably due to molecular packing effects.

The CCl_3 group has typical values for the C—Cl distances (the mean C—Cl bond lengths is 1.773 Å). In general, all bonding parameters and the dimensions of the angles in the title complex are in good agreement with those encountered in related complexes (Bhatti *et al.*, 2010).

3. Supramolecular features

In the crystal, pairs of molecules are linked by N2—H1···O3^{i} and C9—H9···O3^{i} hydrogen bonds (Table 1, Fig. 2) involving the same acceptor atom, forming inversion dimers. In addition, π–π stacking interactions between the C3–C8 benzene rings of the phthalimide units connect the dimers into columns along [0$\bar{1}$1] with a centroid–centroid distance of 3.7736 (13) Å for $Cg\cdots Cg(2-x, -y, 2-z)$. Further weak C—H···O hydrogen bonds occur within these columns (Fig. 3).

4. Database survey

A search of the Cambridge Structural Database (Version 5.38; last update November 2016; Groom *et al.*, 2016) for related

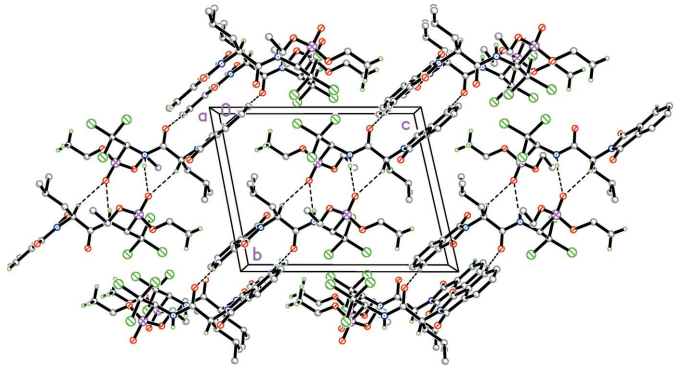


Figure 3
The crystal packing of the title compound viewed along the *a* axis. Intermolecular N—H···O and C—H···O hydrogen bonds are shown as dashed lines. Only selected H atoms are shown.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{26}Cl_3N_2O_6P$
M_r	527.75
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, b, c (Å)	8.4601 (2), 10.9425 (3), 13.5321 (4)
α, β, γ (°)	78.188 (2), 88.644 (2), 75.442 (2)
V (Å^3)	1186.32 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.49
Crystal size (mm)	0.24 × 0.19 × 0.08
Data collection	
Diffractometer	Bruker SMART APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.87, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16619, 4423, 3306
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.090, 1.08
No. of reflections	4423
No. of parameters	293
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.44

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

compounds with a phthalimide fragment gave 77 hits including the closely related structures of the 2-[2-(1,3-dioxoisoindolin-2-yl)-acetamido]acetic acid, (*S*)-4-fluoro-*N*-methyl-2-(1,3-dioxoisoindolin-2-yl)pent-4-enamide dichloromethane solvate and (*S*)-4-carbamoyl-4-(1,3-dioxoisoindolin-2-yl)butanoic acid (Bhatti *et al.*, 2010; Shendage *et al.*, 2004; Otogawa *et al.*, 2015). All bond lengths and angles in these related compounds are similar to those in the title compound. Differences in the values of the O—P—O bond angles in the phosphonate group and C—N—C angles around the pthalimide nitrogen appear to be due to molecular packing and steric effects.

5. Synthesis and crystallization

The general procedure for the preparation of the title compound was previously described by Lukashuk *et al.* (2015). A mixture of 2-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-3-methyl-*N*-(1,2,2,2-tetrachloroethyl)butanamide (0.14 mol), triethyl phosphite (30 mL, 0.17 mol), and dry dioxane (150 mL) was refluxed for 3 h. Colourless crystals suitable for single-crystal X-ray analysis were formed after slowly cooling to room temperature. The analytically pure title compound was obtained by solvent evaporation under reduced pressure to dryness (yield 58.84 g, 80% as a yellow oil). Analysis calculated for $C_{19}H_{26}Cl_3N_2O_6P$: C, 44.42; H, 4.71; Cl, 20.70; N, 5.45; P, 6.03%; found: C, 44.55; H, 4.86; Cl, 20.82; N, 5.53; P, 6.19%.

The NMR spectra [^{1}H (500 MHz), ^{31}P (202 MHz), ^{13}C (125 MHz); *s*, singlet; *br*, broad; *d*, doublet; *m*, multiplet] were obtained on a Bruker Avance DRX-500 instrument in a solution of DMSO-d_6, relative to internal TMS or external 85% phosphoric acid. ^{1}H NMR: 9.32 ($\frac{1}{2}$H, *d*, *J* = 9.3 Hz, NH), 9.22 ($\frac{1}{2}$H, *d*, *J* = 9.3 Hz, NH), 7.92–7.88 (4H, *m*, aromatic), 5.29–5.21 (1H, *m*, CHP), 4.68–4.61 (1H, *m*, CH), 4.10–4.00 (4H, *m*, 2OCH$_2$CH$_3$), 2.97–2.90 (1H, *m*, CH), 1.22–1.15 (6H, *m*, 2OCH$_2$CH$_3$), 1.11–1.05 (3H, *m*, CH$_3$), 0.88–0.79 (3H, *m*, CH$_3$). ^{13}C NMR: 168.61 (*d*, *J* = 4.5 Hz, C═O), 167.41 (*d*, *J* = 4.5 Hz, C═O), 134.41, 134.36, 130.55, 130.53, 122.85, 122.80 (aromatic), 96.21 (*d*, *J* = 14.5 Hz, CCl$_3$), 96.06 (*d*, *J* = 14.5 Hz, CCl$_3$), 62.30 (*d*, *J* = 6.5 Hz, OCH$_2$CH$_3$), 62.07 (*d*, *J* = 6.5 Hz, OCH$_2$CH$_3$), 60.31 (*d*, *J* = 158.8 Hz, CP), 60.26 (*d*, *J* = 158.6 Hz, CP), 59.31, 59.22 (CH), 25.92, 25.84 (CH), 18.55, 18.50 (CH$_3$), 18.49, 18.36 (CH$_3$), 14.97 (*d*, *J* = 6.0 Hz, OCH$_2$CH$_3$), 14.86 (*d*, *J* = 6.0 Hz, OCH$_2$CH$_3$). ^{31}P NMR: 14.4. 14.2.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All C—H hydrogen atoms were placed in calculated positions (C—H = 0.98–1.00Å) and refined in the riding-model approximation with U_{iso}(H) = 1.2–1.5U_{eq}(H). The H atom bonded to atom N2 was located in a difference-Fourier map and refined isotropically.

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Crystal structure of diethyl {2,2,2-trichloro-1-[2-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-4-methylpentanamido]ethyl}phosphonate

V. S. Brovarets, O. V. Golovchenko, E. B. Rusanov and J. A. Rusanova

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Diethyl {2,2,2-trichloro-1-[2-(1,3-dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-4-methylpentanamido]ethyl}phosphonate

Crystal data

$C_{20}H_{26}Cl_3N_2O_6P$
$M_r = 527.75$
Triclinic, $P\overline{1}$
$a = 8.4601$ (2) Å
$b = 10.9425$ (3) Å
$c = 13.5321$ (4) Å
$\alpha = 78.188$ (2)°
$\beta = 88.644$ (2)°
$\gamma = 75.442$ (2)°
$V = 1186.32$ (6) Å^3
$Z = 2$
$F(000) = 548$
$D_x = 1.477$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2780 reflections
$\theta = 3.4$–$45.8°$
$\mu = 0.49$ mm^{-1}
$T = 173$ K
Plate, colourless
0.24 × 0.19 × 0.08 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.87$, $T_{max} = 0.96$
16619 measured reflections
4423 independent reflections
3306 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.2°$
$h = -10 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.090$
$S = 1.08$
4423 reflections
293 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.38432 (7)	0.33488 (6)	0.45621 (4)	0.01836 (15)
CL1	0.79937 (8)	0.30683 (7)	0.43532 (5)	0.0447 (2)
CL2	0.85600 (7)	0.07099 (6)	0.58507 (4)	0.03428 (18)
CL3	0.69011 (8)	0.09144 (7)	0.39715 (5)	0.0491 (2)
O1	0.24551 (17)	0.37482 (14)	0.53081 (11)	0.0219 (4)
O2	0.33202 (19)	0.24320 (15)	0.39667 (11)	0.0267 (4)
O3	0.42397 (18)	0.45000 (14)	0.39643 (11)	0.0247 (4)
O4	0.44032 (19)	0.15069 (15)	0.97521 (11)	0.0257 (4)
O5	0.86366 (19)	0.32359 (16)	0.85338 (12)	0.0310 (4)
O6	0.54932 (19)	0.11923 (15)	0.74528 (11)	0.0262 (4)
N1	0.6311 (2)	0.25003 (17)	0.88866 (13)	0.0187 (4)
N2	0.5722 (2)	0.2870 (2)	0.62148 (13)	0.0182 (4)
C1	0.5759 (3)	0.1669 (2)	0.96882 (16)	0.0203 (5)
C2	0.7905 (3)	0.2556 (2)	0.90771 (16)	0.0215 (5)
C3	0.8451 (3)	0.1656 (2)	1.00570 (15)	0.0194 (5)
C4	0.9935 (3)	0.1328 (2)	1.05637 (16)	0.0232 (5)
H4	1.080114	0.170025	1.030700	0.028*
C5	1.0114 (3)	0.0426 (2)	1.14710 (17)	0.0275 (6)
H5	1.112112	0.017187	1.184424	0.033*
C6	0.8833 (3)	−0.0105 (2)	1.18346 (17)	0.0275 (6)
H6	0.898632	−0.071669	1.245561	0.033*
C7	0.7331 (3)	0.0230 (2)	1.13198 (16)	0.0248 (6)
H7	0.645930	−0.013498	1.157508	0.030*
C8	0.7172 (3)	0.1123 (2)	1.04154 (15)	0.0184 (5)
C9	0.5453 (3)	0.3201 (2)	0.79368 (15)	0.0188 (5)
H9	0.599930	0.389770	0.763816	0.023*
C10	0.3643 (3)	0.3834 (2)	0.80426 (16)	0.0219 (5)
H10A	0.320062	0.436484	0.737746	0.026*
H10B	0.305581	0.314522	0.821981	0.026*
C11	0.3267 (3)	0.4684 (2)	0.88262 (17)	0.0252 (6)
H11	0.361031	0.412380	0.950673	0.030*
C12	0.4201 (3)	0.5725 (2)	0.86449 (19)	0.0360 (7)
H12A	0.537536	0.532229	0.864970	0.054*
H12B	0.386046	0.630024	0.798851	0.054*
H12C	0.396886	0.622302	0.917967	0.054*
C13	0.1436 (3)	0.5270 (3)	0.8824 (2)	0.0418 (7)
H13A	0.086390	0.458013	0.894246	0.063*
H13B	0.118619	0.576531	0.936024	0.063*
H13C	0.107779	0.584253	0.816908	0.063*

C14	0.5585 (3)	0.2307 (2)	0.71927 (15)	0.0180 (5)
C15	0.5510 (3)	0.2253 (2)	0.54002 (15)	0.0178 (5)
H15	0.512342	0.147068	0.570548	0.021*
C16	0.7154 (3)	0.1776 (2)	0.49071 (16)	0.0269 (6)
C17	0.1635 (3)	0.2852 (2)	0.59219 (18)	0.0275 (6)
H17A	0.238670	0.226629	0.646535	0.033*
H17B	0.127708	0.232492	0.550083	0.033*
C18	0.0190 (3)	0.3634 (2)	0.63664 (18)	0.0342 (6)
H18A	−0.039138	0.305605	0.678612	0.051*
H18B	0.055967	0.414975	0.678234	0.051*
H18C	−0.054538	0.420814	0.582181	0.051*
C19	0.2312 (3)	0.2932 (3)	0.30282 (19)	0.0380 (7)
H19A	0.227578	0.385415	0.277641	0.046*
H19B	0.118185	0.285406	0.315925	0.046*
C20	0.3037 (3)	0.2176 (3)	0.22655 (19)	0.0418 (7)
H20A	0.237677	0.249970	0.163708	0.063*
H20B	0.415264	0.226244	0.213626	0.063*
H20C	0.306207	0.126542	0.251793	0.063*
H1	0.577 (3)	0.357 (2)	0.6099 (17)	0.017 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0179 (3)	0.0187 (3)	0.0166 (3)	−0.0024 (3)	−0.0006 (2)	−0.0022 (2)
CL1	0.0242 (4)	0.0651 (5)	0.0359 (4)	−0.0130 (3)	0.0039 (3)	0.0116 (3)
CL2	0.0240 (3)	0.0416 (4)	0.0269 (3)	0.0092 (3)	−0.0043 (2)	−0.0045 (3)
CL3	0.0398 (4)	0.0655 (5)	0.0372 (4)	0.0148 (4)	−0.0066 (3)	−0.0338 (4)
O1	0.0190 (8)	0.0186 (9)	0.0250 (8)	−0.0024 (7)	0.0047 (7)	−0.0012 (7)
O2	0.0290 (9)	0.0254 (9)	0.0255 (9)	−0.0042 (8)	−0.0092 (7)	−0.0071 (7)
O3	0.0270 (9)	0.0225 (9)	0.0217 (8)	−0.0073 (8)	0.0016 (7)	0.0035 (7)
O4	0.0248 (10)	0.0273 (10)	0.0250 (9)	−0.0107 (8)	−0.0007 (7)	−0.0001 (7)
O5	0.0290 (10)	0.0377 (11)	0.0250 (9)	−0.0150 (9)	0.0010 (7)	0.0046 (8)
O6	0.0407 (11)	0.0176 (9)	0.0199 (8)	−0.0081 (8)	0.0048 (7)	−0.0024 (7)
N1	0.0197 (10)	0.0210 (11)	0.0146 (9)	−0.0060 (9)	−0.0013 (7)	−0.0007 (8)
N2	0.0240 (11)	0.0159 (12)	0.0146 (10)	−0.0062 (10)	−0.0001 (8)	−0.0015 (8)
C1	0.0270 (14)	0.0144 (12)	0.0200 (12)	−0.0052 (11)	0.0004 (10)	−0.0044 (10)
C2	0.0240 (13)	0.0225 (13)	0.0191 (12)	−0.0061 (11)	0.0027 (10)	−0.0061 (10)
C3	0.0249 (13)	0.0168 (12)	0.0172 (11)	−0.0051 (11)	0.0020 (9)	−0.0057 (9)
C4	0.0241 (13)	0.0225 (13)	0.0246 (13)	−0.0064 (11)	−0.0021 (10)	−0.0072 (10)
C5	0.0293 (14)	0.0254 (14)	0.0262 (13)	−0.0015 (12)	−0.0088 (11)	−0.0070 (11)
C6	0.0384 (15)	0.0223 (14)	0.0179 (12)	−0.0038 (12)	−0.0045 (11)	0.0003 (10)
C7	0.0318 (14)	0.0239 (14)	0.0199 (12)	−0.0100 (11)	0.0031 (10)	−0.0041 (10)
C8	0.0237 (13)	0.0161 (12)	0.0154 (11)	−0.0045 (10)	0.0007 (9)	−0.0036 (9)
C9	0.0248 (13)	0.0175 (12)	0.0128 (11)	−0.0055 (10)	−0.0022 (9)	0.0008 (9)
C10	0.0226 (13)	0.0207 (13)	0.0210 (12)	−0.0023 (10)	−0.0027 (9)	−0.0042 (10)
C11	0.0324 (14)	0.0224 (14)	0.0197 (12)	−0.0042 (12)	0.0003 (10)	−0.0054 (10)
C12	0.0431 (17)	0.0289 (15)	0.0406 (16)	−0.0090 (13)	0.0021 (13)	−0.0177 (12)
C13	0.0337 (16)	0.0431 (17)	0.0513 (18)	−0.0047 (14)	0.0105 (13)	−0.0228 (14)

C14	0.0166 (12)	0.0189 (13)	0.0170 (11)	−0.0032 (10)	0.0007 (9)	−0.0016 (10)
C15	0.0194 (12)	0.0176 (12)	0.0158 (11)	−0.0036 (10)	0.0005 (9)	−0.0037 (9)
C16	0.0218 (13)	0.0369 (15)	0.0180 (12)	−0.0002 (11)	0.0011 (10)	−0.0056 (11)
C17	0.0210 (13)	0.0277 (14)	0.0289 (13)	−0.0060 (11)	0.0038 (10)	0.0046 (11)
C18	0.0225 (14)	0.0422 (17)	0.0309 (14)	−0.0018 (12)	0.0076 (11)	−0.0004 (12)
C19	0.0327 (15)	0.0481 (18)	0.0337 (15)	−0.0071 (13)	−0.0117 (12)	−0.0123 (13)
C20	0.0566 (19)	0.0424 (18)	0.0311 (15)	−0.0186 (15)	−0.0055 (13)	−0.0096 (13)

Geometric parameters (Å, º)

P1—O3	1.4616 (15)	C9—C14	1.526 (3)
P1—O2	1.5564 (16)	C9—C10	1.531 (3)
P1—O1	1.5670 (15)	C9—H9	1.0000
P1—C15	1.839 (2)	C10—C11	1.526 (3)
CL1—C16	1.764 (3)	C10—H10A	0.9900
CL2—C16	1.780 (2)	C10—H10B	0.9900
CL3—C16	1.772 (2)	C11—C12	1.520 (3)
O1—C17	1.454 (3)	C11—C13	1.520 (3)
O2—C19	1.474 (3)	C11—H11	1.0000
O4—C1	1.202 (3)	C12—H12A	0.9800
O5—C2	1.209 (3)	C12—H12B	0.9800
O6—C14	1.220 (2)	C12—H12C	0.9800
N1—C2	1.398 (3)	C13—H13A	0.9800
N1—C1	1.417 (3)	C13—H13B	0.9800
N1—C9	1.459 (2)	C13—H13C	0.9800
N2—C14	1.356 (3)	C15—C16	1.545 (3)
N2—C15	1.442 (3)	C15—H15	1.0000
N2—H1	0.76 (2)	C17—C18	1.496 (3)
C1—C8	1.487 (3)	C17—H17A	0.9900
C2—C3	1.486 (3)	C17—H17B	0.9900
C3—C4	1.374 (3)	C18—H18A	0.9800
C3—C8	1.387 (3)	C18—H18B	0.9800
C4—C5	1.394 (3)	C18—H18C	0.9800
C4—H4	0.9500	C19—C20	1.480 (4)
C5—C6	1.389 (3)	C19—H19A	0.9900
C5—H5	0.9500	C19—H19B	0.9900
C6—C7	1.392 (3)	C20—H20A	0.9800
C6—H6	0.9500	C20—H20B	0.9800
C7—C8	1.387 (3)	C20—H20C	0.9800
C7—H7	0.9500		

O3—P1—O2	116.77 (9)	C13—C11—H11	108.1
O3—P1—O1	109.82 (9)	C10—C11—H11	108.1
O2—P1—O1	107.96 (9)	C11—C12—H12A	109.5
O3—P1—C15	115.02 (10)	C11—C12—H12B	109.5
O2—P1—C15	102.73 (9)	H12A—C12—H12B	109.5
O1—P1—C15	103.45 (9)	C11—C12—H12C	109.5
C17—O1—P1	123.88 (14)	H12A—C12—H12C	109.5

C19—O2—P1	121.74 (15)	H12B—C12—H12C	109.5
C2—N1—C1	111.45 (18)	C11—C13—H13A	109.5
C2—N1—C9	120.53 (17)	C11—C13—H13B	109.5
C1—N1—C9	127.97 (18)	H13A—C13—H13B	109.5
C14—N2—C15	121.5 (2)	C11—C13—H13C	109.5
C14—N2—H1	118.0 (17)	H13A—C13—H13C	109.5
C15—N2—H1	119.8 (17)	H13B—C13—H13C	109.5
O4—C1—N1	125.4 (2)	O6—C14—N2	122.8 (2)
O4—C1—C8	129.3 (2)	O6—C14—C9	122.46 (19)
N1—C1—C8	105.22 (18)	N2—C14—C9	114.63 (19)
O5—C2—N1	124.7 (2)	N2—C15—C16	111.14 (18)
O5—C2—C3	128.9 (2)	N2—C15—P1	107.70 (14)
N1—C2—C3	106.43 (19)	C16—C15—P1	116.87 (14)
C4—C3—C8	122.5 (2)	N2—C15—H15	106.9
C4—C3—C2	129.6 (2)	C16—C15—H15	106.9
C8—C3—C2	107.91 (19)	P1—C15—H15	106.9
C3—C4—C5	117.0 (2)	C15—C16—CL1	111.57 (16)
C3—C4—H4	121.5	C15—C16—CL3	110.61 (16)
C5—C4—H4	121.5	CL1—C16—CL3	109.04 (12)
C6—C5—C4	120.6 (2)	C15—C16—CL2	108.95 (14)
C6—C5—H5	119.7	CL1—C16—CL2	108.53 (13)
C4—C5—H5	119.7	CL3—C16—CL2	108.04 (13)
C5—C6—C7	122.2 (2)	O1—C17—C18	107.44 (19)
C5—C6—H6	118.9	O1—C17—H17A	110.2
C7—C6—H6	118.9	C18—C17—H17A	110.2
C8—C7—C6	116.6 (2)	O1—C17—H17B	110.2
C8—C7—H7	121.7	C18—C17—H17B	110.2
C6—C7—H7	121.7	H17A—C17—H17B	108.5
C7—C8—C3	121.0 (2)	C17—C18—H18A	109.5
C7—C8—C1	130.0 (2)	C17—C18—H18B	109.5
C3—C8—C1	108.93 (18)	H18A—C18—H18B	109.5
N1—C9—C14	110.41 (17)	C17—C18—H18C	109.5
N1—C9—C10	114.38 (17)	H18A—C18—H18C	109.5
C14—C9—C10	108.24 (17)	H18B—C18—H18C	109.5
N1—C9—H9	107.9	O2—C19—C20	108.7 (2)
C14—C9—H9	107.9	O2—C19—H19A	109.9
C10—C9—H9	107.9	C20—C19—H19A	109.9
C11—C10—C9	115.53 (18)	O2—C19—H19B	109.9
C11—C10—H10A	108.4	C20—C19—H19B	109.9
C9—C10—H10A	108.4	H19A—C19—H19B	108.3
C11—C10—H10B	108.4	C19—C20—H20A	109.5
C9—C10—H10B	108.4	C19—C20—H20B	109.5
H10A—C10—H10B	107.5	H20A—C20—H20B	109.5
C12—C11—C13	111.1 (2)	C19—C20—H20C	109.5
C12—C11—C10	111.88 (19)	H20A—C20—H20C	109.5
C13—C11—C10	109.45 (19)	H20B—C20—H20C	109.5
C12—C11—H11	108.1		

O3—P1—O1—C17	172.13 (16)	N1—C1—C8—C3	1.9 (2)
O2—P1—O1—C17	43.80 (18)	C2—N1—C9—C14	97.2 (2)
C15—P1—O1—C17	−64.61 (18)	C1—N1—C9—C14	−79.8 (3)
O3—P1—O2—C19	−37.1 (2)	C2—N1—C9—C10	−140.4 (2)
O1—P1—O2—C19	87.18 (18)	C1—N1—C9—C10	42.6 (3)
C15—P1—O2—C19	−163.92 (17)	N1—C9—C10—C11	51.7 (3)
C2—N1—C1—O4	174.9 (2)	C14—C9—C10—C11	175.29 (19)
C9—N1—C1—O4	−7.8 (4)	C9—C10—C11—C12	54.9 (3)
C2—N1—C1—C8	−2.6 (2)	C9—C10—C11—C13	178.5 (2)
C9—N1—C1—C8	174.64 (19)	C15—N2—C14—O6	9.9 (3)
C1—N1—C2—O5	−176.7 (2)	C15—N2—C14—C9	−166.63 (18)
C9—N1—C2—O5	5.8 (3)	N1—C9—C14—O6	39.6 (3)
C1—N1—C2—C3	2.2 (2)	C10—C9—C14—O6	−86.3 (2)
C9—N1—C2—C3	−175.22 (17)	N1—C9—C14—N2	−143.88 (19)
O5—C2—C3—C4	−3.4 (4)	C10—C9—C14—N2	90.2 (2)
N1—C2—C3—C4	177.7 (2)	C14—N2—C15—C16	−108.9 (2)
O5—C2—C3—C8	178.0 (2)	C14—N2—C15—P1	121.90 (19)
N1—C2—C3—C8	−0.9 (2)	O3—P1—C15—N2	71.35 (17)
C8—C3—C4—C5	−0.2 (3)	O2—P1—C15—N2	−160.69 (14)
C2—C3—C4—C5	−178.7 (2)	O1—P1—C15—N2	−48.41 (16)
C3—C4—C5—C6	−0.1 (3)	O3—P1—C15—C16	−54.52 (19)
C4—C5—C6—C7	0.1 (4)	O2—P1—C15—C16	73.43 (18)
C5—C6—C7—C8	0.3 (3)	O1—P1—C15—C16	−174.29 (16)
C6—C7—C8—C3	−0.6 (3)	N2—C15—C16—CL1	−62.8 (2)
C6—C7—C8—C1	179.4 (2)	P1—C15—C16—CL1	61.4 (2)
C4—C3—C8—C7	0.6 (3)	N2—C15—C16—CL3	175.64 (15)
C2—C3—C8—C7	179.4 (2)	P1—C15—C16—CL3	−60.2 (2)
C4—C3—C8—C1	−179.39 (19)	N2—C15—C16—CL2	57.0 (2)
C2—C3—C8—C1	−0.6 (2)	P1—C15—C16—CL2	−178.83 (11)
O4—C1—C8—C7	4.5 (4)	P1—O1—C17—C18	−169.00 (15)
N1—C1—C8—C7	−178.1 (2)	P1—O2—C19—C20	133.89 (19)
O4—C1—C8—C3	−175.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O3^{i}	0.76 (2)	2.09 (2)	2.846 (3)	170 (2)
C9—H9···O3^{i}	1.00	2.47	3.265 (3)	136
C7—H7···O6^{ii}	0.95	2.46	3.393 (3)	169

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