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Diethyl [hydroxy(2-nitrophenyl)methyl]-phosphonate

Cai-bao Chen,* Wei-wei Jin and Xin-yong Li

Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: cbbbccn@yahoo.com.cn

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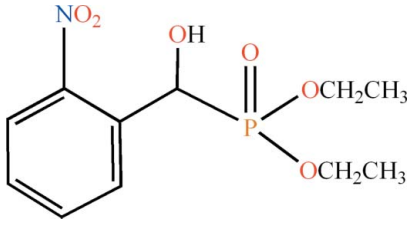
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.052; wR factor = 0.157; data-to-parameter ratio = 14.5.

In the title molecule, $C_{11}H_{16}NO_6P$, the nitro group is twisted out of the mean plane of the benzene ring at 29.91 (3)°. The two ethyl groups are disordered between two orientations in the ratios 0.784 (7)/0.216 (7) and 0.733 (6)/0.267 (6). Intermolecular O—H···O hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For general background, see: Allen *et al.* (1978); Hirschmann *et al.* (1994).



Experimental

Crystal data

$C_{11}H_{16}NO_6P$
M_r = 289.22
Triclinic, $P\overline{1}$
a = 7.5659 (13) Å
b = 8.3844 (15) Å
c = 12.557 (2) Å
α = 73.356 (3)°
β = 87.391 (3)°
γ = 64.432 (3)°
V = 685.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 291 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
6168 measured reflections
2800 independent reflections
2381 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.157
S = 1.05
2800 reflections
193 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O4^{i}	0.82 (1)	1.857 (11)	2.671 (3)	174 (4)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

We thank Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2361).

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supporting information

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Diethyl [hydroxy(2-nitrophenyl)methyl]phosphonate

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S1. Comment

Phosphonates, especially enantiomerically pure forms, are particularly important in connection with their remarkable biological activities. They have been used as enzyme inhibitors, antibacterial agents, anti-HIV agents, botryticides, and haptens for catalytic antibodies (Allen *et al.*, 1978; Hirschmann *et al.*, 1994). In this regard, the preparation of various optically active phosphonates with a diversity of structures is highly desirable for drug discovery and medicinal chemistry. The title compound (I) was obtained in the reaction of diphenylphosphite with an aromatic aldehyde in the presence of triethylamine.

In (I) (Fig. 1), the nitro group is twisted out of the mean plane of benzene ring at 29.91 (3)°. In the crystal (Fig. 2), intermolecular O—H···O hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers (Fig. 2).

S2. Experimental

To a solution of 2-nitrobenzylaldehyde(1 mmol) in tetrahydrofuran(0.6 ml) was added diphenyl phosphite(1 mmol) at 0°C. After 15 minutes, triethylamine (0.1 ml) was added, and the reaction mixture was stirred for 2 h at 0°C. The resulting solution was washed with saturated $NaHCO_3$ solution, extracted with dichloromethane and dried over $MgSO_4$. The solution was filtered and purified by column chroatography on silica gel, using ehtyl acetate and petroleum as eluant to afford the title compound. Crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a chloroform and methanol solution in ratio of 100:1 at 293 K.

S3. Refinement

C-bound H atoms were initially located in difference maps and then constrained to their ideal positions (C–H = 0.93–0.98 Å), and refined as riding with U_{iso}(H)=1.2–1.5Ueq(C). The hydroxy atom H3A was located on difference map and refined with bond restraint O–H = 0.82 (1) Å, and with the U_{iso}(H) =1.5U_{eq}(O). Two ethyl groups were treated as disordered between two orientations with the refined occupancies of 0.786 (7)/0.214 (7) [C8—C9/C8′-C9′] and 0.727 (6)/0.273 (6) [C10—C11/C10′-C11′], respectively.

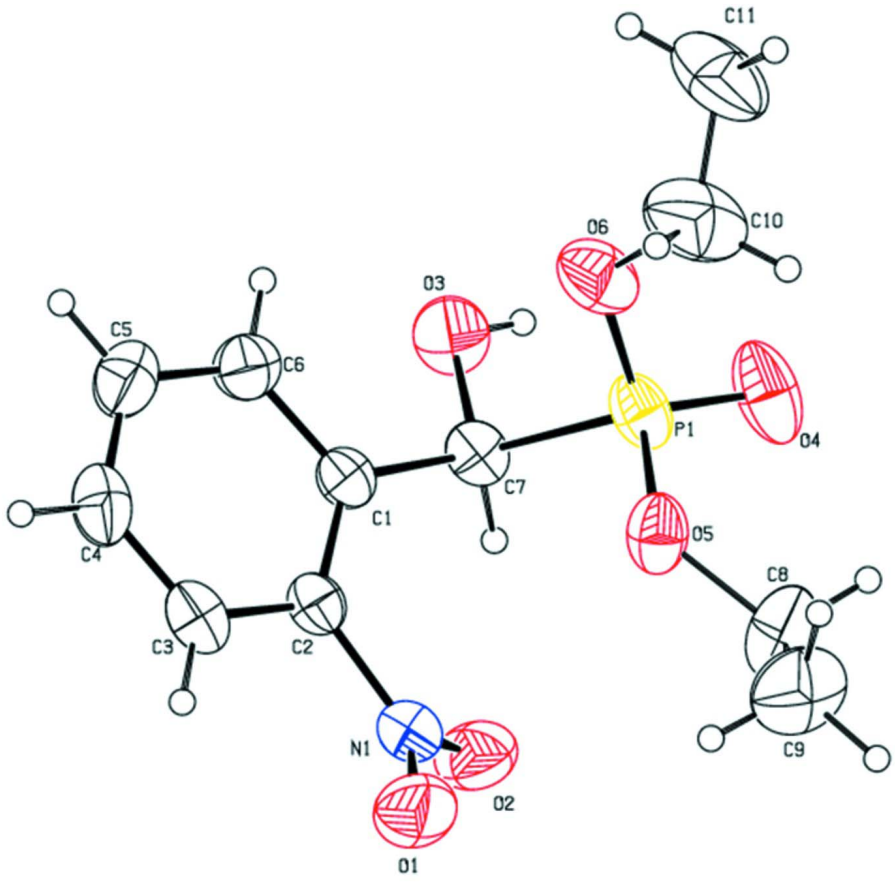

Figure 1

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary radius. The minor parts of disordered ethyl groups are omitted.

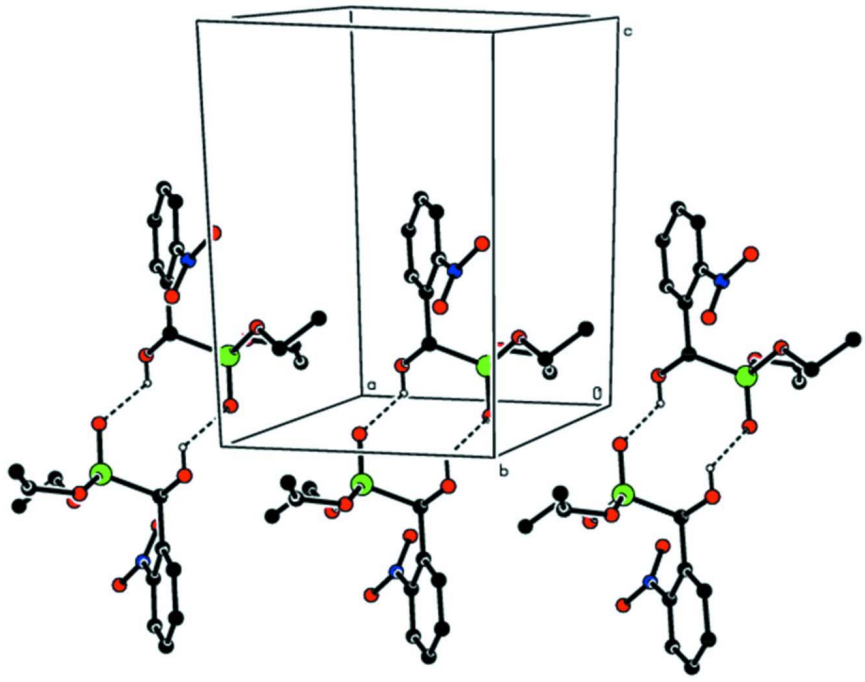

Figure 2

A portion of crystal packing showing the hydrogen-bonded (dashed lines) dimers in (I). H atoms not invloved in hydrogen bonds have been omitted for clarity.

Diethyl [hydroxy(2-nitrophenyl)methyl]phosphonate

Crystal data

$C_{11}H_{16}NO_6P$
$M_r = 289.22$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.5659$ (13) Å
$b = 8.3844$ (15) Å
$c = 12.557$ (2) Å
$\alpha = 73.356$ (3)°
$\beta = 87.391$ (3)°
$\gamma = 64.432$ (3)°
$V = 685.6$ (2) Å^3
$Z = 2$
$F(000) = 304$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3014 reflections
$\theta = 2.8$–$28.0°$
$\mu = 0.22$ mm^{-1}
$T = 291$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
6168 measured reflections
2800 independent reflections
2381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 26.5°$, $\theta_{min} = 1.7°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.157$
$S = 1.05$
2800 reflections
193 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0955P)^2 + 0.1438P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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^ > σ(*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}	Occ. (<1)
P1	0.33556 (9)	0.33802 (9)	0.13960 (4)	0.0648 (2)	
C1	0.6327 (3)	0.2053 (3)	0.31083 (16)	0.0521 (4)	
C2	0.5564 (3)	0.2596 (3)	0.40485 (16)	0.0548 (5)	
C3	0.6201 (4)	0.1419 (3)	0.51225 (18)	0.0691 (6)	
H3	0.5640	0.1819	0.5728	0.083*	
C4	0.7655 (4)	−0.0332 (3)	0.5291 (2)	0.0760 (6)	
H4	0.8103	−0.1125	0.6012	0.091*	
C5	0.8460 (4)	−0.0924 (3)	0.4386 (2)	0.0782 (7)	
H5	0.9455	−0.2115	0.4495	0.094*	
C6	0.7784 (3)	0.0256 (3)	0.3323 (2)	0.0676 (5)	
H6	0.8328	−0.0171	0.2723	0.081*	
N1	0.4048 (3)	0.4469 (3)	0.39566 (16)	0.0673 (5)	
C7	0.5651 (3)	0.3239 (3)	0.19053 (16)	0.0574 (5)	
H7	0.5481	0.4491	0.1839	0.069*	
C8	0.0059 (6)	0.5738 (7)	0.1961 (3)	0.0961 (13)	0.784 (7)
H8A	−0.0469	0.6001	0.1208	0.115*	0.784 (7)
H8B	0.0354	0.6744	0.1987	0.115*	0.784 (7)
C9	−0.1387 (9)	0.5585 (11)	0.2750 (7)	0.1132 (17)	0.784 (7)
H9A	−0.1731	0.4637	0.2688	0.170*	0.784 (7)
H9B	−0.2543	0.6746	0.2584	0.170*	0.784 (7)
H9C	−0.0833	0.5271	0.3496	0.170*	0.784 (7)
C10	0.2216 (8)	0.0840 (9)	0.1406 (5)	0.1275 (19)	0.733 (6)
H10A	0.1865	0.0309	0.2129	0.153*	0.733 (6)

H10B	0.1072	0.1975	0.1039	0.153*	0.733 (6)
C11	0.2598 (15)	−0.0343 (12)	0.0801 (5)	0.133 (2)	0.733 (6)
H11A	0.2744	0.0237	0.0046	0.199*	0.733 (6)
H11B	0.1531	−0.0677	0.0811	0.199*	0.733 (6)
H11C	0.3792	−0.1436	0.1115	0.199*	0.733 (6)
C8′	−0.024 (2)	0.471 (3)	0.2069 (13)	0.0961 (13)	0.216 (7)
H8C	−0.0615	0.5126	0.1274	0.115*	0.216 (7)
H8D	−0.0636	0.3735	0.2412	0.115*	0.216 (7)
C9′	−0.112 (4)	0.604 (4)	0.247 (3)	0.1132 (17)	0.216 (7)
H9D	−0.1637	0.5624	0.3148	0.170*	0.216 (7)
H9E	−0.2174	0.7016	0.1941	0.170*	0.216 (7)
H9F	−0.0209	0.6482	0.2619	0.170*	0.216 (7)
C10′	0.340 (2)	0.050 (2)	0.0838 (15)	0.1275 (19)	0.267 (6)
H10C	0.2555	0.1460	0.0194	0.153*	0.267 (6)
H10D	0.4623	−0.0245	0.0579	0.153*	0.267 (6)
C11′	0.254 (5)	−0.055 (4)	0.1332 (16)	0.133 (2)	0.267 (6)
H11D	0.3195	−0.1279	0.2064	0.199*	0.267 (6)
H11E	0.2613	−0.1348	0.0901	0.199*	0.267 (6)
H11F	0.1188	0.0221	0.1394	0.199*	0.267 (6)
O1	0.2928 (3)	0.4664 (3)	0.46827 (17)	0.0988 (6)	
O2	0.3979 (3)	0.5760 (2)	0.31795 (16)	0.0925 (6)	
O3	0.7091 (3)	0.2488 (3)	0.12000 (14)	0.0823 (5)	
H3A	0.711 (6)	0.338 (3)	0.072 (2)	0.123*	
O4	0.2695 (3)	0.4578 (3)	0.02454 (13)	0.0976 (7)	
O5	0.1853 (2)	0.3993 (2)	0.22587 (12)	0.0713 (4)	
O6	0.3792 (3)	0.1322 (3)	0.15899 (16)	0.0909 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0644 (4)	0.0939 (5)	0.0463 (3)	−0.0471 (3)	0.0061 (2)	−0.0157 (3)
C1	0.0521 (10)	0.0583 (10)	0.0551 (10)	−0.0320 (8)	0.0044 (8)	−0.0172 (8)
C2	0.0544 (11)	0.0607 (11)	0.0555 (10)	−0.0299 (9)	0.0010 (8)	−0.0181 (8)
C3	0.0756 (15)	0.0867 (15)	0.0526 (11)	−0.0434 (13)	−0.0008 (10)	−0.0178 (10)
C4	0.0763 (15)	0.0768 (14)	0.0667 (14)	−0.0378 (13)	−0.0155 (11)	0.0005 (11)
C5	0.0650 (14)	0.0618 (12)	0.0967 (18)	−0.0248 (11)	−0.0065 (13)	−0.0101 (12)
C6	0.0627 (13)	0.0669 (12)	0.0765 (14)	−0.0292 (10)	0.0083 (10)	−0.0249 (11)
N1	0.0710 (12)	0.0710 (11)	0.0634 (11)	−0.0281 (9)	0.0009 (9)	−0.0288 (9)
C7	0.0570 (11)	0.0715 (12)	0.0523 (10)	−0.0357 (10)	0.0109 (8)	−0.0193 (9)
C8	0.069 (2)	0.089 (3)	0.091 (2)	−0.0168 (18)	0.0057 (17)	0.001 (2)
C9	0.070 (3)	0.132 (5)	0.152 (5)	−0.049 (2)	0.049 (3)	−0.061 (4)
C10	0.105 (4)	0.176 (5)	0.175 (5)	−0.095 (4)	0.042 (3)	−0.107 (4)
C11	0.192 (5)	0.146 (4)	0.114 (5)	−0.114 (4)	−0.006 (6)	−0.048 (5)
C8′	0.069 (2)	0.089 (3)	0.091 (2)	−0.0168 (18)	0.0057 (17)	0.001 (2)
C9′	0.070 (3)	0.132 (5)	0.152 (5)	−0.049 (2)	0.049 (3)	−0.061 (4)
C10′	0.105 (4)	0.176 (5)	0.175 (5)	−0.095 (4)	0.042 (3)	−0.107 (4)
C11′	0.192 (5)	0.146 (4)	0.114 (5)	−0.114 (4)	−0.006 (6)	−0.048 (5)
O1	0.0921 (14)	0.1054 (14)	0.0858 (12)	−0.0239 (11)	0.0255 (11)	−0.0433 (11)

O2	0.1164 (16)	0.0613 (9)	0.0886 (12)	−0.0296 (10)	0.0086 (11)	−0.0210 (9)
O3	0.0725 (11)	0.1066 (14)	0.0690 (10)	−0.0401 (10)	0.0278 (8)	−0.0296 (9)
O4	0.0928 (13)	0.1595 (19)	0.0495 (9)	−0.0782 (13)	0.0003 (8)	−0.0059 (10)
O5	0.0563 (9)	0.0873 (10)	0.0569 (8)	−0.0282 (8)	0.0041 (7)	−0.0067 (7)
O6	0.0959 (14)	0.1074 (14)	0.1021 (14)	−0.0641 (12)	0.0083 (10)	−0.0463 (11)

Geometric parameters (Å, º)

P1—O4	1.4653 (18)	C9—H9B	0.9600
P1—O6	1.556 (2)	C9—H9C	0.9600
P1—O5	1.5598 (16)	C10—C11	1.345 (9)
P1—C7	1.822 (2)	C10—O6	1.461 (5)
C1—C6	1.386 (3)	C10—H10A	0.9700
C1—C2	1.398 (3)	C10—H10B	0.9700
C1—C7	1.518 (3)	C11—H11A	0.9600
C2—C3	1.383 (3)	C11—H11B	0.9600
C2—N1	1.466 (3)	C11—H11C	0.9600
C3—C4	1.364 (4)	C8′—C9′	1.26 (3)
C3—H3	0.9300	C8′—O5	1.435 (15)
C4—C5	1.381 (4)	C8′—H8C	0.9700
C4—H4	0.9300	C8′—H8D	0.9700
C5—C6	1.375 (3)	C9′—H9D	0.9600
C5—H5	0.9300	C9′—H9E	0.9600
C6—H6	0.9300	C9′—H9F	0.9600
N1—O1	1.213 (3)	C10′—C11′	1.31 (3)
N1—O2	1.215 (3)	C10′—O6	1.426 (13)
C7—O3	1.417 (2)	C10′—H10C	0.9700
C7—H7	0.9800	C10′—H10D	0.9700
C8—O5	1.463 (4)	C11′—H11D	0.9600
C8—C9	1.465 (7)	C11′—H11E	0.9600
C8—H8A	0.9700	C11′—H11F	0.9600
C8—H8B	0.9700	O3—H3A	0.817 (10)
C9—H9A	0.9600		

O4—P1—O6	115.38 (12)	H8A—C8—H8B	108.3
O4—P1—O5	114.16 (11)	C11—C10—O6	116.7 (6)
O6—P1—O5	103.70 (10)	C11—C10—H10A	108.1
O4—P1—C7	112.67 (10)	O6—C10—H10A	108.1
O6—P1—C7	103.71 (10)	C11—C10—H10B	108.1
O5—P1—C7	106.13 (9)	O6—C10—H10B	108.1
C6—C1—C2	115.53 (19)	H10A—C10—H10B	107.3
C6—C1—C7	118.91 (18)	C9′—C8′—O5	111 (2)
C2—C1—C7	125.53 (17)	C9′—C8′—H8C	109.4
C3—C2—C1	122.48 (19)	O5—C8′—H8C	109.4
C3—C2—N1	115.65 (18)	C9′—C8′—H8D	109.4
C1—C2—N1	121.86 (17)	O5—C8′—H8D	109.4
C4—C3—C2	119.8 (2)	H8C—C8′—H8D	108.0
C4—C3—H3	120.1	C8′—C9′—H9D	109.5

C2—C3—H3	120.1	C8′—C9′—H9E	109.5
C3—C4—C5	119.7 (2)	H9D—C9′—H9E	109.5
C3—C4—H4	120.2	C8′—C9′—H9F	109.5
C5—C4—H4	120.2	H9D—C9′—H9F	109.5
C6—C5—C4	119.7 (2)	H9E—C9′—H9F	109.5
C6—C5—H5	120.1	C11′—C10′—O6	110.4 (15)
C4—C5—H5	120.1	C11′—C10′—H10C	109.6
C5—C6—C1	122.8 (2)	O6—C10′—H10C	109.6
C5—C6—H6	118.6	C11′—C10′—H10D	109.6
C1—C6—H6	118.6	O6—C10′—H10D	109.6
O1—N1—O2	122.7 (2)	H10C—C10′—H10D	108.1
O1—N1—C2	117.8 (2)	C10′—C11′—H11D	109.5
O2—N1—C2	119.50 (19)	C10′—C11′—H11E	109.5
O3—C7—C1	109.76 (17)	H11D—C11′—H11E	109.5
O3—C7—P1	106.87 (14)	C10′—C11′—H11F	109.5
C1—C7—P1	113.49 (13)	H11D—C11′—H11F	109.5
O3—C7—H7	108.9	H11E—C11′—H11F	109.5
C1—C7—H7	108.9	C7—O3—H3A	106 (3)
P1—C7—H7	108.9	C8′—O5—P1	125.3 (7)
O5—C8—C9	109.0 (4)	C8—O5—P1	122.36 (19)
O5—C8—H8A	109.9	C10′—O6—C10	45.2 (6)
C9—C8—H8A	109.9	C10′—O6—P1	128.7 (8)
O5—C8—H8B	109.9	C10—O6—P1	120.3 (3)
C9—C8—H8B	109.9		

C6—C1—C2—C3	−0.6 (3)	O6—P1—C7—C1	−56.38 (16)
C7—C1—C2—C3	177.55 (19)	O5—P1—C7—C1	52.54 (17)
C6—C1—C2—N1	178.54 (19)	C9′—C8′—O5—C8	−39.6 (17)
C7—C1—C2—N1	−3.3 (3)	C9′—C8′—O5—P1	−139.1 (16)
C1—C2—C3—C4	1.3 (3)	C9—C8—O5—C8′	49.1 (11)
N1—C2—C3—C4	−177.8 (2)	C9—C8—O5—P1	156.6 (4)
C2—C3—C4—C5	−0.9 (4)	O4—P1—O5—C8′	39.3 (10)
C3—C4—C5—C6	−0.2 (4)	O6—P1—O5—C8′	−87.1 (10)
C4—C5—C6—C1	1.0 (4)	C7—P1—O5—C8′	164.0 (10)
C2—C1—C6—C5	−0.6 (3)	O4—P1—O5—C8	−7.3 (3)
C7—C1—C6—C5	−178.8 (2)	O6—P1—O5—C8	−133.7 (3)
C3—C2—N1—O1	−29.3 (3)	C7—P1—O5—C8	117.4 (3)
C1—C2—N1—O1	151.6 (2)	C11′—C10′—O6—C10	−35.3 (18)
C3—C2—N1—O2	149.4 (2)	C11′—C10′—O6—P1	−131.9 (19)
C1—C2—N1—O2	−29.8 (3)	C11—C10—O6—C10′	16.3 (12)
C6—C1—C7—O3	−19.2 (2)	C11—C10—O6—P1	132.5 (6)
C2—C1—C7—O3	162.69 (18)	O4—P1—O6—C10′	−8.9 (8)
C6—C1—C7—P1	100.3 (2)	O5—P1—O6—C10′	116.7 (8)
C2—C1—C7—P1	−77.8 (2)	C7—P1—O6—C10′	−132.6 (8)
O4—P1—C7—O3	−60.70 (19)	O4—P1—O6—C10	−63.6 (3)
O6—P1—C7—O3	64.75 (16)	O5—P1—O6—C10	62.0 (3)
O5—P1—C7—O3	173.68 (13)	C7—P1—O6—C10	172.7 (3)
O4—P1—C7—C1	178.17 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O4^{i}	0.82 (1)	1.86 (1)	2.671 (3)	174 (4)

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