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Two closely related {4-[(*N*-substituted amino)-(diethoxyphosphoryl)methyl]phenyl}boronic acids

Rui Zhang, Yundi Zhang, Chunhua Ge,* Jinpeng Miao and Xiangdong Zhang*

College of Chemistry, Liaoning University, Chongshanzhonglu 66, Shenyang, Liaoning 110036, People's Republic of China. *Correspondence e-mail: chhge@lnu.edu.cn, xd623@sina.com

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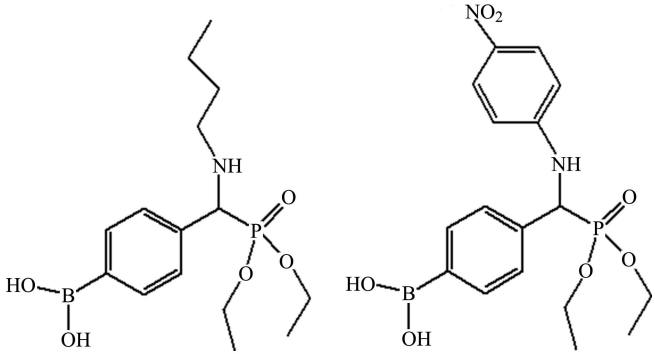
Organic phosphonic acids and organic phosphonic acid esters have been of much interest due to their applications in the fields of medicine, agriculture and industrial chemistry. Boronic acids can act as synthetic intermediates and building blocks and are used in sensing, protein manipulation, therapeutics, biological labelling and separation. The additional introduction of an aminophosphonic acid group into a boronic acid may give new opportunities for application. To study the structure of such multifunctional compounds, we prepared two new derivatives which can be easily converted to the corresponding phosphonic acids. In the title compounds, {4-[(butylamino)(diethoxyphosphoryl)methyl]phenyl}boronic acid monohydrate, $C_{15}H_{27}BNO_5P\cdot H_2O$, (I), and {4-[(diethoxyphosphoryl)(4-nitroanilino)methyl]phenyl}boronic acid, $C_{17}H_{22}BN_2O_7P$, (II), three different substituents are attached to a central C—H group, namely 4-boronophenyl, diethoxyphosphoryl and amine. Compound (I) crystallizes as a monohydrate and O_B—H···N hydrogen bonds link neighbouring molecules into chains along the [001] direction. The solvent water molecule connects two such chains running in opposite directions. Compound (II) crystallizes as an ansolvate and classical hydrogen bonds result in a layer structure in the (001) plane.

1. Introduction

In recent decades, organic phosphonic acids and organic phosphonic acid esters have been of much interest due to their applications in the fields of medicine, agriculture and industrial chemistry (Bandekar & Dhadke, 1998; Jia *et al.*, 1988; Kao *et al.*, 2006; Stallmach *et al.*, 1994; Stock *et al.*, 2005; Wu *et al.*, 2013).



(I) (II)

The formation of C—P bonds and the structural characterization of the resulting compounds has also attracted attention (Gaikwad *et al.*, 2011; Angelova *et al.*, 1992; Oleksyszyn & Subotkowska, 1980; Zhang *et al.*, 2005). Phosphonic acid esters are also used in the rapidly developing area of biochemistry. The synthesis of oligonucleotide analogues with an achiral phosphonic acid ester backbone has been carried

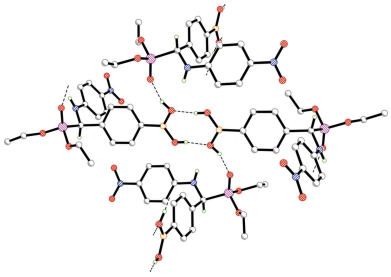


Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{15}H_{27}BNO_5P{\cdot}H_2O$	$C_{17}H_{22}BN_2O_7P$
M_r	361.17	408.14
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, *Pbcn*
Temperature (K)	295	173
a, b, c (Å)	8.4370 (2), 23.5403 (4), 10.3281 (2)	10.2590 (8), 14.5721 (14), 27.577 (3)
α, β, γ (°)	90, 98.1871 (7), 90	90, 90, 90
V (Å^3)	2030.35 (7)	4122.6 (6)
Z	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.16	0.17
Crystal size (mm)	0.20 × 0.10 × 0.10	0.20 × 0.20 × 0.10
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	73764, 3569, 3198	15530, 3726, 2312
R_{int}	0.036	0.056
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.596	0.603
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.067, 0.172, 1.09	0.060, 0.197, 1.09
No. of reflections	3569	3726
No. of parameters	235	265
No. of restraints	15	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.38	0.61, −0.33

Computer programs: *APEX2* (Bruker, 2010), *SAINT* (Bruker, 2010), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *SHELXTL* (Bruker, 2010).

out (Peyman *et al.*, 1996). Aminophosphonic acid and its derivatives are commonly known as potential enzyme inhibitors (Beers *et al.*, 1996; Pawełczak *et al.*, 1998; Vovk *et al.*, 2008).

Boronic acids can act as synthetic intermediates and building blocks and are used in sensing, protein manipulation, therapeutics, biological labelling and separation (Kubo *et al.*, 2015; Lacina *et al.*, 2014; Li *et al.*, 2014; Ma *et al.*, 2013; Pan *et al.*, 2013; Sun *et al.*, 2016; Zhang *et al.*, 2015). Boronic acid-modified lipid nanocapsules can be used as a platform for highly efficient inhibitors for the hepatitis C virus (Khanal *et al.*, 2015).

It has been recognized that the additional introduction of an aminophosphonic acid group into a boronic acid may give new opportunities for application. Synthetic (Młynarz *et al.*, 2011) and property studies (Piergies *et al.*, 2012; Proniewicz *et al.*, 2013) have been performed. Work in this area is in its infancy.

To study the structure of such multifunctional compounds, we prepared two new derivatives which can be easily converted to the corresponding phosphonic acids, namely {4-[(butylamino)(diethoxyphosphoryl)methyl]phenyl}boronic acid monohydrate, (I), and {4-[(diethoxyphosphoryl)(4-nitroanilino)methyl]phenyl}boronic acid, (II).

2. Experimental

2.1. Synthesis and crystallization

2.1.1. Preparation and spectroscopic data for (I). A mixture of 4-boronobenzaldehyde (3.0 g, 20 mmol), *n*-butylamine (1.5 g, 20 mmol) and absolute ethanol (30 ml) was refluxed for 12 h. Diethyl phosphate (2.8 g, 20 mmol) was then added dropwise. The resulting solution was refluxed for another 24 h. The volatiles were removed under reduced pressure, resulting in a yellow residue. This crude product was recrystallized from a 1:1 (*v*/*v*) water–ethanol mixture to give (I) (yield: 89.3%; m.p. 322–326 K). IR (KBr, cm^{-1}): 3325, 2959, 1610, 1563, 1441, 1409, 1369, 1228, 1056, and 1020. ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.010 (1H, *s*, O*H*), 7.748, 7.722 (2H, *d*, *J* = 7.7 Hz, Ar*H*), 7.357 (2H, *d*, *J* = 6.2 Hz, Ar*H*), 4.025 (1H, *m*, C*H*), 3.805 (4H, *m*, CH_2), 2.356 (2H, *m*, CH_2), 2.175 (1H, *s*, N*H*), 1.282 (7H, *m*, CH_2CH_3), 1.044–0.817 (6H, *t*, *J* = 7.1 Hz, CH_3).

2.1.2. Preparation and spectroscopic data for (II). Compound (II) was synthesized in a similar manner to (I), except that 4-nitroaniline was used instead of *n*-butylamine (yield: 76.5%; m.p. 461–463 K). IR (KBr, cm^{-1}): 3415, 3304, 2981, 1842, 1599, 1505, 1414, 1369, 1279, 1047 and 1012. ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.068 (2H, *s*, O*H*), 7.973 (2H, *d*, *J* = 8.9 Hz, Ar*H*), 7.782 (2H, *d*, *J* = 7.6 Hz, Ar*H*), 7.536 (2H, *d*, *J* = 7.5 Hz, Ar*H*), 6.975 (2H, *d*, *J* = 8.9 Hz, Ar*H*), 5.324 (1H, *dd*, *J* = 23.9 Hz, *J* = 9.4 Hz), 4.171–3.686 (4H, *m*, CH_2), 3.424 (1H, *d*, *J* = 1.7 Hz, N*H*), 1.186–1.082 (6H, *t*, *J* = 7.0 Hz, CH_3).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. In (I), carbon-bound H atoms were placed in calculated positions and refined using a riding model, with methyl C—H = 0.96 Å, secondary C—H = 0.97 Å, tertiary C—H = 0.98 Å and aromatic C—H = 0.93 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl or 1.2U_{eq}(C) for secondary, tertiary and aromatic H atoms. In (II), carbon-

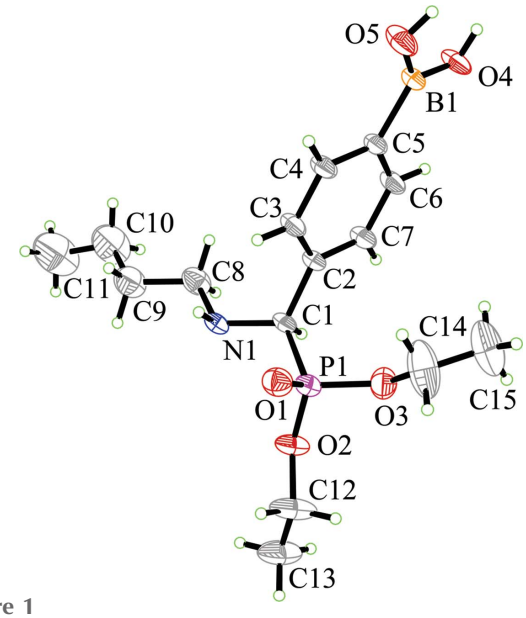


Figure 1
The asymmetric unit of (I), with the cocrystallized water molecule omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level.

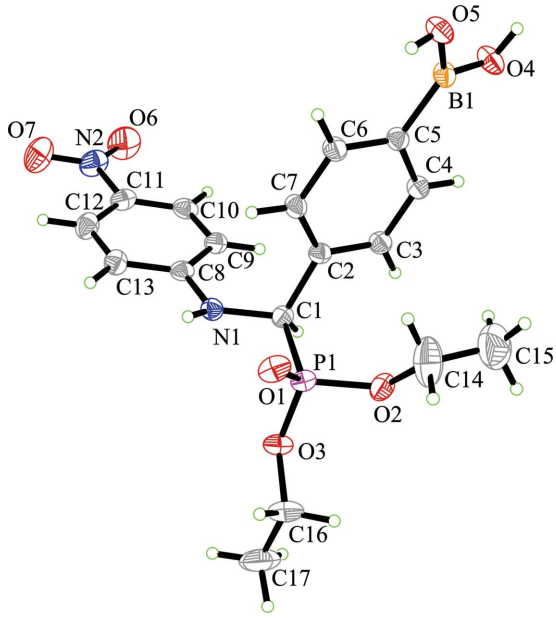


Figure 3
A view of the molecule of (II), shown with 30% probability displacement ellipsoids.

bound H atoms were placed in calculated positions and refined using a riding model, with methyl C—H = 0.98 Å, secondary C—H = 0.99 Å, tertiary C—H = 1.00 Å and aromatic C—H = 0.95 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl or 1.2U_{eq}(C) for secondary, tertiary and aromatic H atoms. H atoms bonded to O or N atoms were located in difference Fourier maps and were refined isotropically, with the isotropic displacement parameters coupled to the anisotropic displacement parameters of the parent N atoms or O atoms, *i.e.* U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O). Tentative free refinements of their positional coordinates resulted in an unsatisfactory wide range of *D*—H distances; the bond lengths were therefore restrained to 0.89 (2) Å for N—H and to 0.82 (2) Å for O—H.

For (I), distance restraints of 1.50 (2) Å were employed for the C—C bonds in the phosphonic ester moiety and for the terminal C—C bond in the butyl group, because unresolved disorder was causing shorter than normal apparent distances between the average positions for these atoms.. For the latter bond, restraints for similar displacement parameters and rigid-bond restraints were imposed to ensure physically reasonable displacement parameters.

3. Results and discussion

3.1. Structure analysis

The asymmetric unit of (I) comprises a single target molecule (Fig. 1) and a cocrystallized water molecule. Hydrogen bonds between the borate group and the amine N atom link the molecules into a one-dimensional chain parallel to the crystallographic *c* axis. O—H···O contacts between two water molecules and two of these chains about a centre of inversion give rise to a ladder-shaped polymer (Fig. 2) based on an $R_4^4(8)$ graph set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995). Details of the hydrogen-bond interactions are given in Table 2.

In contrast to (I), compound (II) is an ansolvate in which the asymmetric unit corresponds to a single molecule (Fig. 3). An $R_2^2(8)$ hydrogen-bond motif links molecules into dimers

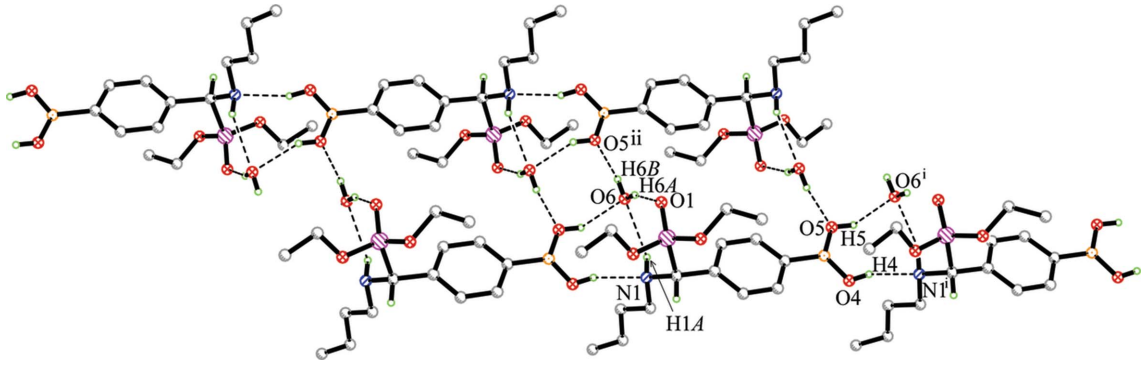


Figure 2
The double-stranded one-dimensional supramolecular ribbon in (I). Only H atoms involved in short contacts are shown. Symmetry codes are as given in Table 2.

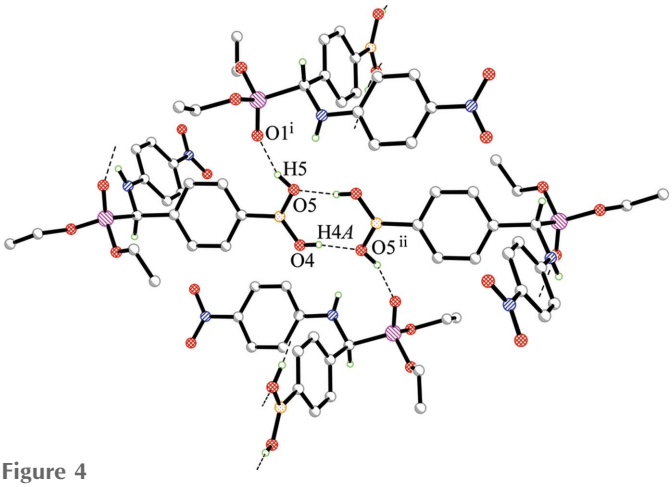


Figure 4
The hydrogen bonding in (II). Only H atoms involved in short contacts are shown. Symmetry codes are as given in Table 3.

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···N1^{i}	0.82 (2)	2.07 (2)	2.872 (3)	165 (4)
O5—H5···O6^{i}	0.80 (2)	2.00 (2)	2.787 (3)	170 (5)
N1—H1*A*···O6	0.89 (2)	2.36 (2)	3.159 (4)	149 (3)
O6—H6*A*···O1	0.83 (2)	1.95 (2)	2.754 (4)	164 (5)
O6—H6*B*···O5^{ii}	0.81 (2)	1.98 (2)	2.775 (4)	168 (5)

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

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O1^{i}	0.82 (2)	1.85 (2)	2.657 (4)	166 (5)
O4—H4*A*···O5^{ii}	0.83 (2)	1.88 (2)	2.702 (4)	176 (5)

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

(Fig. 4). Taking into account all hydrogen bonds, a layer structure is generated in the (001) plane. Weaker C—H···O interactions further connect these sheets into a three-dimensional network. Details of the hydrogen-bond interactions are given in Table 3.

In both (I) and (II), the central C—H group is bonded to a 4-boronophenyl group and a diethoxyphosphoryl group; the third substituent is a butylamino group in the case of (I) and a 4-nitroanilino group in the case of (II). Geometric parameters in these compounds are comparable to those of the previously reported ethyl [(*n*-butylammonio)(2-hydroxyphenyl)methyl]-phosphonate (Zhang *et al.*, 2007).

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supporting information

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Two closely related {4-[(*N*-substituted amino)(diethoxyphosphoryl)methyl]-phenyl}boronic acids

Rui Zhang, Yundi Zhang, Chunhua Ge, Jinpeng Miao and Xiangdong Zhang

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT* (Bruker, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Bruker, 2010). Software used to prepare material for publication: *SHELXL2016* (Sheldrick, 2015) for (1); *SHELXTL* (Bruker, 2010) for (2).

(1) {4-[(Butylamino)(diethoxyphosphoryl)methyl]phenyl}boronic acid monohydrate

Crystal data

$C_{15}H_{27}BNO_5P\cdot H_2O$
$M_r = 361.17$
Monoclinic, $P2_1/c$
$a = 8.4370\ (2)$ Å
$b = 23.5403\ (4)$ Å
$c = 10.3281\ (2)$ Å
$\beta = 98.1871\ (7)°$
$V = 2030.35\ (7)$ Å^3
$Z = 4$
$F(000) = 776$
$D_x = 1.182$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1321 reflections
$\theta = 2.2–25.0°$
$\mu = 0.16$ mm^{-1}
$T = 295$ K
Sheet, white
0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
73764 measured reflections
3569 independent reflections
3198 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 25.1°$, $\theta_{min} = 3.0°$
$h = -10\rightarrow9$
$k = -28\rightarrow28$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.172$
$S = 1.09$
3569 reflections
235 parameters
15 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 2.5299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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	0.05753 (10)	0.40870 (3)	0.71854 (7)	0.0481 (3)
O1	0.1336 (3)	0.46466 (10)	0.7185 (2)	0.0611 (6)
O2	−0.0129 (3)	0.38463 (11)	0.5818 (2)	0.0665 (7)
O3	−0.0862 (3)	0.40558 (11)	0.7994 (2)	0.0670 (7)
O4	0.2651 (3)	0.34135 (10)	1.42919 (19)	0.0626 (7)
H4	0.277 (5)	0.3502 (18)	1.507 (2)	0.094*
O5	0.4082 (4)	0.42696 (12)	1.4122 (2)	0.0715 (7)
H5	0.410 (6)	0.438 (2)	1.485 (2)	0.107*
B1	0.3262 (4)	0.38153 (15)	1.3577 (3)	0.0459 (8)
N1	0.3284 (3)	0.35052 (10)	0.7090 (2)	0.0436 (6)
H1A	0.383 (3)	0.3827 (10)	0.725 (3)	0.052*
C1	0.1922 (3)	0.35215 (12)	0.7835 (2)	0.0419 (6)
H1	0.134086	0.316227	0.767298	0.050*
C2	0.2342 (3)	0.35877 (12)	0.9314 (2)	0.0406 (6)
C5	0.2975 (3)	0.37367 (12)	1.2048 (2)	0.0418 (6)
C3	0.3349 (4)	0.40162 (13)	0.9851 (3)	0.0486 (7)
H3	0.382282	0.425729	0.930449	0.058*
C4	0.3660 (4)	0.40905 (13)	1.1197 (3)	0.0492 (7)
H4A	0.433818	0.438198	1.153717	0.059*
C6	0.1986 (4)	0.33071 (13)	1.1486 (3)	0.0510 (7)
H6	0.151848	0.306181	1.202778	0.061*
C7	0.1671 (4)	0.32304 (13)	1.0147 (3)	0.0497 (7)
H7	0.100297	0.293629	0.980550	0.060*
C8	0.4360 (5)	0.30250 (15)	0.7458 (3)	0.0662 (9)
H8A	0.376131	0.267351	0.730837	0.079*
H8B	0.476126	0.304861	0.838410	0.079*
C9	0.5732 (6)	0.3013 (2)	0.6707 (4)	0.0936 (15)
H9A	0.635529	0.335676	0.689549	0.112*
H9B	0.532184	0.301411	0.578058	0.112*
C10	0.6810 (9)	0.2514 (3)	0.6992 (6)	0.151 (3)
H10A	0.746084	0.258244	0.783024	0.181*
H10B	0.614097	0.218779	0.710277	0.181*
C11	0.7818 (11)	0.2363 (3)	0.6134 (7)	0.182 (4)
H11A	0.720401	0.223469	0.533343	0.273*
H11B	0.850853	0.206307	0.650418	0.273*
H11C	0.845195	0.268535	0.595891	0.273*
C12	−0.1070 (7)	0.4194 (2)	0.4847 (4)	0.1016 (17)
H12A	−0.198092	0.434697	0.520682	0.122*
H12B	−0.042565	0.451007	0.461969	0.122*

C13	−0.1621 (7)	0.3875 (2)	0.3701 (4)	0.1058 (17)
H13A	−0.072158	0.371401	0.335712	0.159*
H13B	−0.220208	0.411959	0.305696	0.159*
H13C	−0.231169	0.357533	0.391441	0.159*
C14	−0.1265 (9)	0.4492 (2)	0.8822 (7)	0.139 (3)
H14A	−0.028179	0.466011	0.925846	0.167*
H14B	−0.183683	0.478541	0.828409	0.167*
C15	−0.2186 (9)	0.4332 (3)	0.9763 (6)	0.140 (3)
H15A	−0.312922	0.413941	0.935302	0.210*
H15B	−0.249024	0.466268	1.021131	0.210*
H15C	−0.157815	0.408122	1.037812	0.210*
O6	0.4407 (3)	0.47579 (13)	0.6604 (3)	0.0710 (7)
H6A	0.348 (3)	0.479 (2)	0.675 (5)	0.106*
H6B	0.475 (6)	0.5065 (12)	0.644 (5)	0.106*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0524 (5)	0.0569 (5)	0.0339 (4)	−0.0004 (4)	0.0020 (3)	0.0014 (3)
O1	0.0670 (14)	0.0561 (13)	0.0593 (14)	−0.0023 (11)	0.0060 (11)	0.0070 (10)
O2	0.0814 (16)	0.0733 (15)	0.0381 (11)	0.0075 (13)	−0.0150 (11)	0.0008 (10)
O3	0.0584 (14)	0.0775 (16)	0.0677 (15)	−0.0042 (12)	0.0177 (12)	−0.0089 (12)
O4	0.0901 (17)	0.0735 (15)	0.0245 (10)	−0.0178 (13)	0.0091 (11)	0.0012 (10)
O5	0.0982 (19)	0.0794 (17)	0.0393 (12)	−0.0287 (14)	0.0180 (13)	−0.0160 (11)
B1	0.0512 (19)	0.059 (2)	0.0276 (15)	0.0022 (15)	0.0075 (13)	0.0005 (14)
N1	0.0575 (15)	0.0476 (13)	0.0259 (11)	0.0006 (11)	0.0069 (10)	−0.0005 (9)
C1	0.0546 (16)	0.0478 (15)	0.0229 (12)	−0.0047 (12)	0.0035 (11)	−0.0003 (11)
C2	0.0498 (15)	0.0467 (15)	0.0249 (12)	−0.0004 (12)	0.0042 (11)	0.0007 (11)
C5	0.0490 (15)	0.0507 (16)	0.0259 (13)	0.0035 (12)	0.0060 (11)	0.0011 (11)
C3	0.0625 (18)	0.0567 (17)	0.0274 (13)	−0.0145 (14)	0.0087 (12)	0.0027 (12)
C4	0.0615 (18)	0.0577 (18)	0.0281 (13)	−0.0118 (14)	0.0053 (12)	−0.0026 (12)
C6	0.0667 (19)	0.0574 (18)	0.0299 (14)	−0.0109 (15)	0.0102 (13)	0.0075 (12)
C7	0.0641 (19)	0.0531 (17)	0.0312 (14)	−0.0148 (14)	0.0043 (13)	0.0014 (12)
C8	0.086 (3)	0.060 (2)	0.0538 (19)	0.0168 (18)	0.0159 (18)	0.0056 (16)
C9	0.101 (3)	0.110 (3)	0.074 (3)	0.047 (3)	0.026 (2)	0.011 (2)
C10	0.160 (6)	0.180 (6)	0.122 (5)	0.114 (5)	0.054 (4)	0.041 (4)
C11	0.232 (9)	0.186 (7)	0.137 (6)	0.130 (7)	0.060 (6)	0.014 (5)
C12	0.140 (4)	0.093 (3)	0.057 (2)	0.027 (3)	−0.040 (3)	0.003 (2)
C13	0.127 (4)	0.115 (4)	0.062 (2)	0.021 (3)	−0.037 (3)	−0.011 (2)
C14	0.181 (6)	0.100 (4)	0.163 (6)	−0.010 (4)	0.115 (5)	−0.041 (4)
C15	0.189 (7)	0.114 (4)	0.140 (5)	−0.003 (4)	0.106 (5)	−0.013 (4)
O6	0.0797 (18)	0.0816 (18)	0.0535 (14)	−0.0191 (15)	0.0156 (13)	0.0030 (13)

Geometric parameters (Å, º)

P1—O1	1.465 (2)	C8—C9	1.483 (5)
P1—O2	1.559 (2)	C8—H8A	0.9700
P1—O3	1.569 (2)	C8—H8B	0.9700

P1—C1	1.816 (3)	C9—C10	1.488 (6)
O2—C12	1.441 (4)	C9—H9A	0.9700
O3—C14	1.409 (5)	C9—H9B	0.9700
O4—B1	1.347 (4)	C10—C11	1.360 (7)
O4—H4	0.818 (19)	C10—H10A	0.9700
O5—B1	1.352 (4)	C10—H10B	0.9700
O5—H5	0.795 (19)	C11—H11A	0.9600
B1—C5	1.574 (4)	C11—H11B	0.9600
N1—C8	1.466 (4)	C11—H11C	0.9600
N1—C1	1.471 (4)	C12—C13	1.423 (5)
N1—H1A	0.890 (18)	C12—H12A	0.9700
C1—C2	1.526 (3)	C12—H12B	0.9700
C1—H1	0.9800	C13—H13A	0.9600
C2—C7	1.381 (4)	C13—H13B	0.9600
C2—C3	1.383 (4)	C13—H13C	0.9600
C5—C6	1.385 (4)	C14—C15	1.380 (7)
C5—C4	1.395 (4)	C14—H14A	0.9700
C3—C4	1.388 (4)	C14—H14B	0.9700
C3—H3	0.9300	C15—H15A	0.9600
C4—H4A	0.9300	C15—H15B	0.9600
C6—C7	1.382 (4)	C15—H15C	0.9600
C6—H6	0.9300	O6—H6A	0.825 (19)
C7—H7	0.9300	O6—H6B	0.807 (19)

O1—P1—O2	115.92 (14)	H8A—C8—H8B	107.9
O1—P1—O3	114.45 (14)	C8—C9—C10	114.4 (4)
O2—P1—O3	103.97 (14)	C8—C9—H9A	108.7
O1—P1—C1	114.09 (14)	C10—C9—H9A	108.7
O2—P1—C1	101.88 (13)	C8—C9—H9B	108.7
O3—P1—C1	105.05 (13)	C10—C9—H9B	108.7
C12—O2—P1	121.7 (2)	H9A—C9—H9B	107.6
C14—O3—P1	124.1 (3)	C11—C10—C9	119.9 (6)
B1—O4—H4	111 (3)	C11—C10—H10A	107.4
B1—O5—H5	126 (4)	C9—C10—H10A	107.4
O4—B1—O5	122.7 (3)	C11—C10—H10B	107.4
O4—B1—C5	116.8 (3)	C9—C10—H10B	107.4
O5—B1—C5	120.5 (3)	H10A—C10—H10B	106.9
C8—N1—C1	112.7 (2)	C10—C11—H11A	109.5
C8—N1—H1A	109 (2)	C10—C11—H11B	109.5
C1—N1—H1A	108 (2)	H11A—C11—H11B	109.5
N1—C1—C2	116.0 (2)	C10—C11—H11C	109.5
N1—C1—P1	108.69 (18)	H11A—C11—H11C	109.5
C2—C1—P1	109.78 (19)	H11B—C11—H11C	109.5
N1—C1—H1	107.3	C13—C12—O2	111.2 (4)
C2—C1—H1	107.3	C13—C12—H12A	109.4
P1—C1—H1	107.3	O2—C12—H12A	109.4
C7—C2—C3	118.5 (2)	C13—C12—H12B	109.4
C7—C2—C1	120.3 (2)	O2—C12—H12B	109.4

C3—C2—C1	121.2 (2)	H12A—C12—H12B	108.0
C6—C5—C4	116.8 (2)	C12—C13—H13A	109.5
C6—C5—B1	120.2 (3)	C12—C13—H13B	109.5
C4—C5—B1	123.0 (3)	H13A—C13—H13B	109.5
C2—C3—C4	120.8 (3)	C12—C13—H13C	109.5
C2—C3—H3	119.6	H13A—C13—H13C	109.5
C4—C3—H3	119.6	H13B—C13—H13C	109.5
C3—C4—C5	121.3 (3)	C15—C14—O3	115.8 (5)
C3—C4—H4A	119.3	C15—C14—H14A	108.3
C5—C4—H4A	119.3	O3—C14—H14A	108.3
C7—C6—C5	122.2 (3)	C15—C14—H14B	108.3
C7—C6—H6	118.9	O3—C14—H14B	108.3
C5—C6—H6	118.9	H14A—C14—H14B	107.4
C2—C7—C6	120.4 (3)	C14—C15—H15A	109.5
C2—C7—H7	119.8	C14—C15—H15B	109.5
C6—C7—H7	119.8	H15A—C15—H15B	109.5
N1—C8—C9	112.4 (3)	C14—C15—H15C	109.5
N1—C8—H8A	109.1	H15A—C15—H15C	109.5
C9—C8—H8A	109.1	H15B—C15—H15C	109.5
N1—C8—H8B	109.1	H6A—O6—H6B	109 (5)
C9—C8—H8B	109.1		

O1—P1—O2—C12	−44.9 (4)	O5—B1—C5—C6	−172.8 (3)
O3—P1—O2—C12	81.6 (4)	O4—B1—C5—C4	−174.0 (3)
C1—P1—O2—C12	−169.4 (4)	O5—B1—C5—C4	6.2 (5)
O1—P1—O3—C14	−9.3 (5)	C7—C2—C3—C4	−0.9 (5)
O2—P1—O3—C14	−136.8 (5)	C1—C2—C3—C4	176.8 (3)
C1—P1—O3—C14	116.6 (5)	C2—C3—C4—C5	0.2 (5)
C8—N1—C1—C2	61.4 (3)	C6—C5—C4—C3	0.5 (5)
C8—N1—C1—P1	−174.3 (2)	B1—C5—C4—C3	−178.4 (3)
O1—P1—C1—N1	−56.1 (2)	C4—C5—C6—C7	−0.5 (5)
O2—P1—C1—N1	69.6 (2)	B1—C5—C6—C7	178.5 (3)
O3—P1—C1—N1	177.75 (18)	C3—C2—C7—C6	0.9 (5)
O1—P1—C1—C2	71.8 (2)	C1—C2—C7—C6	−176.8 (3)
O2—P1—C1—C2	−162.6 (2)	C5—C6—C7—C2	−0.2 (5)
O3—P1—C1—C2	−54.4 (2)	C1—N1—C8—C9	−179.8 (3)
N1—C1—C2—C7	−130.9 (3)	N1—C8—C9—C10	−176.7 (5)
P1—C1—C2—C7	105.4 (3)	C8—C9—C10—C11	161.2 (8)
N1—C1—C2—C3	51.4 (4)	P1—O2—C12—C13	−178.0 (4)
P1—C1—C2—C3	−72.2 (3)	P1—O3—C14—C15	−159.5 (6)
O4—B1—C5—C6	7.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···N1^{i}	0.82 (2)	2.07 (2)	2.872 (3)	165 (4)
O5—H5···O6^{i}	0.80 (2)	2.00 (2)	2.787 (3)	170 (5)
N1—H1*A*···O6	0.89 (2)	2.36 (2)	3.159 (4)	149 (3)

O6—H6*A*···O1	0.83 (2)	1.95 (2)	2.754 (4)	164 (5)
O6—H6*B*···O5^{ii}	0.81 (2)	1.98 (2)	2.775 (4)	168 (5)

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

(2) {4-[(Diethoxyphosphoryl)(4-nitroanilino)methyl]phenyl}boronic acid

Crystal data

$C_{17}H_{22}BN_2O_7P$
M_r = 408.14
Orthorhombic, *Pbcn*
a = 10.2590 (8) Å
b = 14.5721 (14) Å
c = 27.577 (3) Å
V = 4122.6 (6) Å^3
Z = 8
F(000) = 1712
D_x = 1.315 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 3802 reflections
θ = 3.2–25.3°
μ = 0.17 mm^{-1}
T = 173 K
Sheet, white
0.20 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
15530 measured reflections
3726 independent reflections
2312 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 25.4°, θ_{min} = 3.2°
h = −12→11
k = −17→16
l = −33→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.197
S = 1.09
3726 reflections
265 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 5.3769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: SHELXL2016 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (6)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.29978 (9)	0.26468 (7)	0.11014 (4)	0.0374 (3)
O1	0.2977 (3)	0.2135 (2)	0.06424 (9)	0.0493 (8)
O2	0.2926 (3)	0.37114 (19)	0.10325 (10)	0.0499 (7)
O3	0.1866 (2)	0.2417 (2)	0.14604 (10)	0.0464 (7)
O4	0.9308 (3)	0.4994 (2)	0.06307 (10)	0.0504 (8)
H4A	0.980 (4)	0.528 (3)	0.0449 (16)	0.076*

O5	0.9167 (3)	0.40026 (19)	−0.00333 (10)	0.0465 (7)
H5	0.877 (4)	0.360 (3)	−0.0180 (16)	0.070*
O6	0.9018 (4)	0.0213 (3)	0.29736 (15)	0.0873 (12)
O7	0.8391 (4)	−0.1102 (3)	0.26851 (14)	0.0847 (12)
N1	0.4546 (3)	0.1441 (2)	0.15380 (11)	0.0367 (7)
H1A	0.425 (4)	0.106 (2)	0.1318 (12)	0.044*
N2	0.8307 (4)	−0.0258 (3)	0.27132 (15)	0.0630 (11)
B1	0.8743 (4)	0.4260 (3)	0.04156 (15)	0.0388 (10)
C1	0.4439 (3)	0.2426 (2)	0.14655 (13)	0.0342 (8)
H1	0.432820	0.272842	0.178838	0.041*
C2	0.5610 (3)	0.2859 (3)	0.12075 (12)	0.0335 (8)
C3	0.5982 (4)	0.3751 (3)	0.13295 (13)	0.0376 (9)
H3	0.554918	0.406402	0.158492	0.045*
C4	0.6987 (4)	0.4182 (3)	0.10778 (13)	0.0362 (8)
H4	0.723602	0.478739	0.116592	0.043*
C5	0.7636 (4)	0.3740 (2)	0.06975 (13)	0.0362 (9)
C6	0.7260 (4)	0.2845 (3)	0.05856 (14)	0.0414 (9)
H6	0.769276	0.252873	0.033096	0.050*
C7	0.6264 (4)	0.2402 (3)	0.08377 (13)	0.0387 (9)
H7	0.603298	0.178985	0.075717	0.046*
C8	0.5481 (3)	0.1044 (3)	0.18368 (13)	0.0366 (9)
C9	0.6302 (4)	0.1570 (3)	0.21332 (13)	0.0400 (9)
H9	0.623234	0.222027	0.213320	0.048*
C10	0.7215 (4)	0.1140 (3)	0.24265 (14)	0.0437 (10)
H10	0.776478	0.149409	0.263115	0.052*
C11	0.7320 (4)	0.0200 (3)	0.24194 (14)	0.0449 (10)
C12	0.6501 (4)	−0.0334 (3)	0.21345 (15)	0.0509 (11)
H12	0.657407	−0.098356	0.213849	0.061*
C13	0.5581 (4)	0.0087 (3)	0.18455 (14)	0.0434 (10)
H13	0.501241	−0.027400	0.165172	0.052*
C14	0.3223 (8)	0.4153 (4)	0.0563 (2)	0.108 (3)
H14A	0.392259	0.380674	0.039650	0.130*
H14B	0.243855	0.414037	0.035396	0.130*
C15	0.3628 (8)	0.5076 (5)	0.0632 (3)	0.134 (3)
H15A	0.435422	0.509329	0.086279	0.201*
H15B	0.289839	0.543691	0.076042	0.201*
H15C	0.390894	0.533576	0.032157	0.201*
C16	0.0515 (4)	0.2502 (4)	0.12892 (19)	0.0640 (14)
H16A	0.034646	0.313934	0.118133	0.077*
H16B	0.036776	0.208619	0.101074	0.077*
C17	−0.0359 (5)	0.2264 (5)	0.1685 (2)	0.099 (2)
H17A	−0.015490	0.264218	0.196847	0.149*
H17B	−0.024920	0.161455	0.176680	0.149*
H17C	−0.126215	0.237538	0.158477	0.149*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0325 (5)	0.0438 (6)	0.0358 (6)	0.0042 (4)	0.0008 (4)	−0.0015 (4)
O1	0.0459 (17)	0.0640 (19)	0.0380 (15)	0.0097 (14)	−0.0052 (13)	−0.0144 (13)
O2	0.0522 (17)	0.0427 (17)	0.0547 (17)	0.0116 (13)	−0.0008 (14)	0.0035 (14)
O3	0.0302 (14)	0.0646 (19)	0.0445 (16)	0.0011 (13)	0.0044 (12)	0.0017 (14)
O4	0.063 (2)	0.0469 (17)	0.0413 (16)	−0.0219 (15)	0.0120 (14)	−0.0077 (13)
O5	0.0535 (18)	0.0457 (17)	0.0401 (16)	−0.0159 (13)	0.0113 (13)	−0.0071 (13)
O6	0.076 (3)	0.098 (3)	0.088 (3)	0.012 (2)	−0.037 (2)	0.012 (2)
O7	0.106 (3)	0.070 (3)	0.078 (3)	0.031 (2)	−0.015 (2)	0.024 (2)
N1	0.0347 (17)	0.0366 (19)	0.0387 (18)	−0.0018 (14)	−0.0031 (14)	0.0014 (14)
N2	0.062 (3)	0.072 (3)	0.055 (2)	0.019 (2)	0.000 (2)	0.024 (2)
B1	0.049 (3)	0.034 (2)	0.033 (2)	−0.002 (2)	0.005 (2)	−0.0002 (18)
C1	0.036 (2)	0.035 (2)	0.0312 (18)	0.0030 (16)	0.0026 (15)	0.0017 (15)
C2	0.0305 (19)	0.039 (2)	0.0311 (19)	0.0014 (15)	−0.0003 (15)	0.0027 (15)
C3	0.038 (2)	0.038 (2)	0.036 (2)	−0.0011 (17)	0.0034 (16)	−0.0028 (16)
C4	0.040 (2)	0.035 (2)	0.0330 (19)	−0.0021 (16)	0.0021 (16)	−0.0033 (16)
C5	0.039 (2)	0.035 (2)	0.0341 (19)	−0.0011 (16)	0.0029 (16)	0.0015 (16)
C6	0.047 (2)	0.039 (2)	0.038 (2)	−0.0028 (17)	0.0114 (18)	−0.0047 (17)
C7	0.043 (2)	0.032 (2)	0.040 (2)	−0.0046 (17)	0.0074 (17)	−0.0054 (17)
C8	0.032 (2)	0.045 (2)	0.0328 (19)	0.0022 (16)	0.0060 (16)	0.0045 (16)
C9	0.036 (2)	0.045 (2)	0.040 (2)	0.0009 (17)	0.0012 (17)	0.0050 (17)
C10	0.037 (2)	0.056 (3)	0.038 (2)	−0.0014 (18)	0.0050 (17)	0.0101 (19)
C11	0.041 (2)	0.057 (3)	0.037 (2)	0.0106 (19)	0.0039 (17)	0.0120 (19)
C12	0.064 (3)	0.044 (2)	0.046 (2)	0.010 (2)	0.010 (2)	0.0083 (19)
C13	0.052 (2)	0.042 (2)	0.037 (2)	0.0011 (19)	0.0039 (18)	0.0042 (18)
C14	0.194 (8)	0.056 (4)	0.074 (4)	−0.007 (4)	−0.028 (5)	0.020 (3)
C15	0.126 (6)	0.103 (6)	0.172 (8)	−0.006 (5)	−0.027 (6)	0.066 (6)
C16	0.030 (2)	0.089 (4)	0.073 (3)	0.005 (2)	−0.003 (2)	−0.004 (3)
C17	0.036 (3)	0.140 (6)	0.121 (5)	0.017 (3)	0.018 (3)	0.046 (5)

Geometric parameters (Å, º)

P1—O1	1.469 (3)	C6—C7	1.394 (5)
P1—O3	1.563 (3)	C6—H6	0.9500
P1—O2	1.565 (3)	C7—H7	0.9500
P1—C1	1.816 (4)	C8—C13	1.397 (5)
O2—C14	1.478 (6)	C8—C9	1.402 (5)
O3—C16	1.469 (5)	C9—C10	1.387 (5)
O4—B1	1.354 (5)	C9—H9	0.9500
O4—H4A	0.825 (19)	C10—C11	1.375 (6)
O5—B1	1.365 (5)	C10—H10	0.9500
O5—H5	0.823 (19)	C11—C12	1.389 (6)
O6—N2	1.232 (5)	C12—C13	1.380 (6)
O7—N2	1.236 (5)	C12—H12	0.9500
N1—C8	1.391 (5)	C13—H13	0.9500
N1—C1	1.453 (5)	C14—C15	1.421 (9)

N1—H1A	0.880 (19)	C14—H14A	0.9900
N2—C11	1.458 (5)	C14—H14B	0.9900
B1—C5	1.571 (6)	C15—H15A	0.9800
C1—C2	1.532 (5)	C15—H15B	0.9800
C1—H1	1.0000	C15—H15C	0.9800
C2—C7	1.390 (5)	C16—C17	1.454 (7)
C2—C3	1.395 (5)	C16—H16A	0.9900
C3—C4	1.393 (5)	C16—H16B	0.9900
C3—H3	0.9500	C17—H17A	0.9800
C4—C5	1.400 (5)	C17—H17B	0.9800
C4—H4	0.9500	C17—H17C	0.9800
C5—C6	1.395 (5)		

O1—P1—O3	115.22 (17)	N1—C8—C13	118.4 (3)
O1—P1—O2	113.46 (17)	N1—C8—C9	122.1 (3)
O3—P1—O2	104.75 (16)	C13—C8—C9	119.5 (3)
O1—P1—C1	113.45 (16)	C10—C9—C8	119.9 (4)
O3—P1—C1	102.52 (15)	C10—C9—H9	120.1
O2—P1—C1	106.34 (16)	C8—C9—H9	120.1
C14—O2—P1	121.9 (3)	C11—C10—C9	119.7 (4)
C16—O3—P1	118.6 (3)	C11—C10—H10	120.2
B1—O4—H4A	113 (4)	C9—C10—H10	120.2
B1—O5—H5	119 (3)	C10—C11—C12	121.3 (4)
C8—N1—C1	123.0 (3)	C10—C11—N2	120.2 (4)
C8—N1—H1A	113 (3)	C12—C11—N2	118.6 (4)
C1—N1—H1A	120 (3)	C13—C12—C11	119.5 (4)
O6—N2—O7	123.3 (4)	C13—C12—H12	120.3
O6—N2—C11	118.7 (4)	C11—C12—H12	120.3
O7—N2—C11	118.0 (5)	C12—C13—C8	120.2 (4)
O4—B1—O5	118.6 (4)	C12—C13—H13	119.9
O4—B1—C5	118.3 (3)	C8—C13—H13	119.9
O5—B1—C5	123.1 (4)	C15—C14—O2	110.8 (6)
N1—C1—C2	114.3 (3)	C15—C14—H14A	109.5
N1—C1—P1	108.2 (2)	O2—C14—H14A	109.5
C2—C1—P1	108.0 (2)	C15—C14—H14B	109.5
N1—C1—H1	108.7	O2—C14—H14B	109.5
C2—C1—H1	108.7	H14A—C14—H14B	108.1
P1—C1—H1	108.7	C14—C15—H15A	109.5
C7—C2—C3	119.4 (3)	C14—C15—H15B	109.5
C7—C2—C1	121.4 (3)	H15A—C15—H15B	109.5
C3—C2—C1	119.1 (3)	C14—C15—H15C	109.5
C4—C3—C2	120.2 (3)	H15A—C15—H15C	109.5
C4—C3—H3	119.9	H15B—C15—H15C	109.5
C2—C3—H3	119.9	C17—C16—O3	108.7 (4)
C3—C4—C5	121.2 (3)	C17—C16—H16A	110.0
C3—C4—H4	119.4	O3—C16—H16A	110.0
C5—C4—H4	119.4	C17—C16—H16B	110.0
C6—C5—C4	117.7 (3)	O3—C16—H16B	110.0

C6—C5—B1	122.8 (3)	H16A—C16—H16B	108.3
C4—C5—B1	119.5 (3)	C16—C17—H17A	109.5
C7—C6—C5	121.7 (3)	C16—C17—H17B	109.5
C7—C6—H6	119.2	H17A—C17—H17B	109.5
C5—C6—H6	119.2	C16—C17—H17C	109.5
C2—C7—C6	119.9 (4)	H17A—C17—H17C	109.5
C2—C7—H7	120.1	H17B—C17—H17C	109.5
C6—C7—H7	120.1		

O1—P1—O2—C14	−17.9 (5)	O4—B1—C5—C4	19.6 (6)
O3—P1—O2—C14	−144.4 (4)	O5—B1—C5—C4	−160.9 (4)
C1—P1—O2—C14	107.5 (4)	C4—C5—C6—C7	0.6 (6)
O1—P1—O3—C16	−55.4 (4)	B1—C5—C6—C7	−179.2 (4)
O2—P1—O3—C16	70.0 (3)	C3—C2—C7—C6	−1.6 (6)
C1—P1—O3—C16	−179.1 (3)	C1—C2—C7—C6	175.0 (3)
C8—N1—C1—C2	65.4 (4)	C5—C6—C7—C2	0.8 (6)
C8—N1—C1—P1	−174.3 (3)	C1—N1—C8—C13	−174.0 (3)
O1—P1—C1—N1	−54.5 (3)	C1—N1—C8—C9	7.2 (5)
O3—P1—C1—N1	70.4 (3)	N1—C8—C9—C10	179.7 (3)
O2—P1—C1—N1	−179.9 (2)	C13—C8—C9—C10	0.9 (5)
O1—P1—C1—C2	69.7 (3)	C8—C9—C10—C11	0.8 (6)
O3—P1—C1—C2	−165.4 (2)	C9—C10—C11—C12	−2.0 (6)
O2—P1—C1—C2	−55.7 (3)	C9—C10—C11—N2	177.8 (3)
N1—C1—C2—C7	36.7 (5)	O6—N2—C11—C10	1.6 (6)
P1—C1—C2—C7	−83.7 (4)	O7—N2—C11—C10	−178.0 (4)
N1—C1—C2—C3	−146.7 (3)	O6—N2—C11—C12	−178.6 (4)
P1—C1—C2—C3	92.8 (3)	O7—N2—C11—C12	1.8 (6)
C7—C2—C3—C4	1.0 (6)	C10—C11—C12—C13	1.3 (6)
C1—C2—C3—C4	−175.7 (3)	N2—C11—C12—C13	−178.5 (4)
C2—C3—C4—C5	0.5 (6)	C11—C12—C13—C8	0.5 (6)
C3—C4—C5—C6	−1.3 (5)	N1—C8—C13—C12	179.6 (3)
C3—C4—C5—B1	178.6 (4)	C9—C8—C13—C12	−1.6 (6)
O4—B1—C5—C6	−160.5 (4)	P1—O2—C14—C15	−155.3 (5)
O5—B1—C5—C6	18.9 (6)	P1—O3—C16—C17	−179.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O1^{i}	0.82 (2)	1.85 (2)	2.657 (4)	166 (5)
O4—H4*A*···O5^{ii}	0.83 (2)	1.88 (2)	2.702 (4)	176 (5)

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