

# 5-Phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride

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Received 5 February 2022

Accepted 17 February 2022

Edited by B. Therrien, University of Neuchâtel, Switzerland

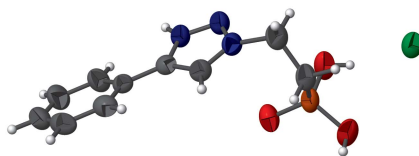
Keywords: phosphonate; triazole; hydrogen bonding; click chemistry; crystal structure.

CCDC reference: 2145106

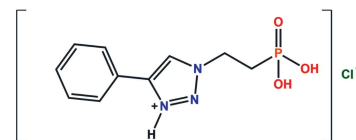
Structural data: full structural data are available from iucrdata.iucr.org

The new triazole-functionalized phosphonic acid 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride, C<sub>10</sub>H<sub>13</sub>N<sub>3</sub>O<sub>3</sub>P<sup>+</sup>·Cl<sup>-</sup> (PTEPHCl), was synthesized by the 'click' reaction of the alkyl azide diethyl-(2-azidoethyl)phosphonate with phenylacetylene to give the diethyl[2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethyl]phosphonate ester, which was then hydrolyzed under acidic conditions (HCl) to give the 'free' phosphonic acid. The use of HCl for the hydrolysis caused protonation of the triazole ring, rendering the compound cationic, charged-balanced by a Cl<sup>-</sup> anion. There are extensive hydrogen-bonding interactions in the structure of PTEPHCl, involving the phosphonic acid (–PO<sub>3</sub>H<sub>2</sub>) group, the triazolium ring and the Cl<sup>-</sup> anion.

## 3D view

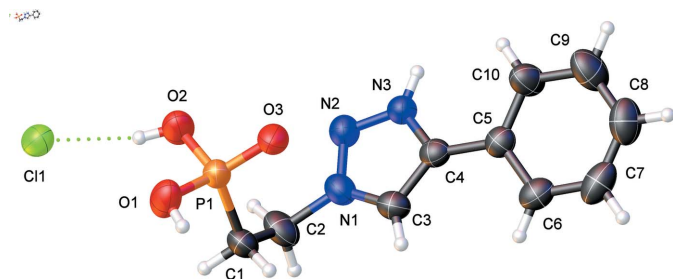


## Chemical scheme



## Structure description

The exponential growth of the field of MOFs and coordination polymers over the past few decades is partially due to the design, synthesis and functionalization of appropriate linkers (Zaręba, 2017). Although the field was initiated with compounds that were mainly based on polycarboxylate linkers, its continuous development currently embraces virtually all molecules that are able to bind to metals. Among the plethora of ligands, (poly)phosphonic acids stand out because they can construct networks with high thermal and hydrolytic stability (Clearfield & Demadis, 2012). The field of metal phosphonates also relies on the availability of proper phosphonate linkers that offer structural diversity and can produce metal phosphonate compounds with attractive properties. Most of the published work on new phosphonic acids is based on two synthetic methodologies: (i) the Arbuzov reaction (Babu *et al.*, 2017) and (ii) the Mannich-type (a.k.a. Moedritzer–Irani) reaction (Villemin *et al.*, 2021). The Arbuzov reaction can convert an organic halide to a phosphonic acid group, whereas Mannich-type reactions transform an amine group to an



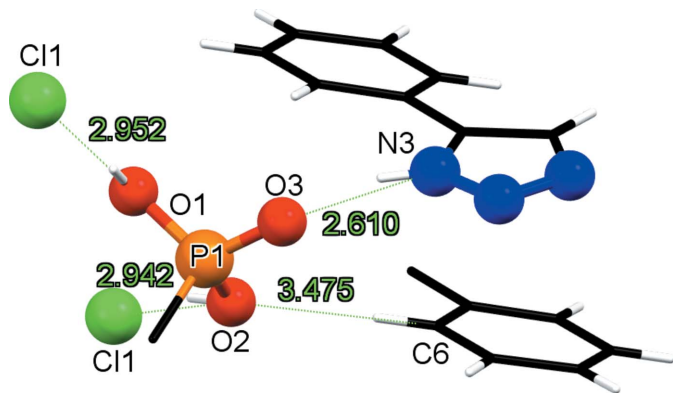
**Figure 1**  
Molecular structure of 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride with the atom-labeling scheme. Displacement ellipsoids are shown at the 50% probability level. Color code: P orange, O red, C black, N blue, Cl green, H white.

aminomethylenephosphonic group. Both synthetic strategies aim at introducing a phosphonic acid moiety to a pre-formed organic fragment. We recently initiated synthetic efforts that are based on ‘click’ chemistry. Specifically, the approach is based on a ‘reactive’ organic molecule that already contains a phosphonic acid group, but can undergo other transformations elsewhere on the backbone.

The reaction of an organic azide with an alkyne to give a triazole is a well-known process (Mukherjee *et al.*, 2013). Herein, this transformation was performed on an organic azide that already contains a phosphonate group on its backbone to yield a phosphonate-modified triazole. Specifically, an alkyl azide [diethyl-(2-azidoethyl)phosphonate] was reacted with an aromatic alkyne (phenylacetylene) to give diethyl[2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethyl]phosphonate ester. This ester was then hydrolyzed in acidic conditions to give 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride (PTEPHCl). In the present work, we report the crystal structure of the above-mentioned triazole-functionalized phosphonic acid PTEPHCl.

#### Molecular structure

Fig. 1 shows the molecular structure of 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride. Because HCl was used for the ester hydrolysis, the N3 atom of the triazole ring and the O1 and O2 atoms of the phosphonate group are protonated due to the synthesis of PTEPHCl at low pH, hence a chloride counter-ion (Cl1) is found in the structure.



**Figure 2**  
Hydrogen-bonding scheme of the phosphonic acid group in the structure of PTEPHCl.

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

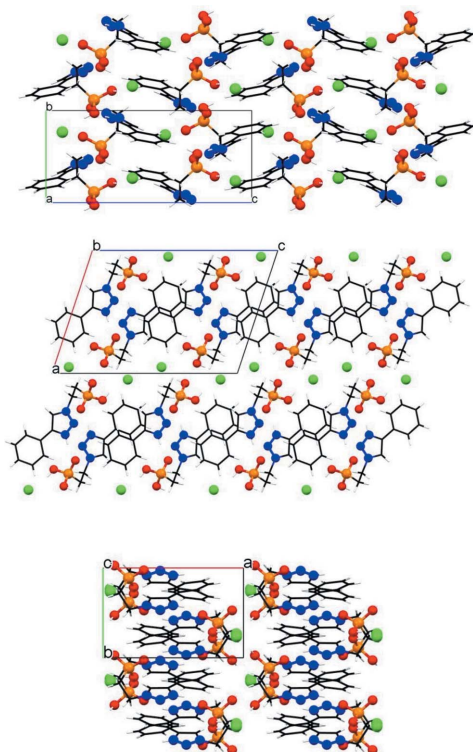
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots Cl1^i$	0.82	2.15	2.9521 (16)	167
$O2-H2\cdots Cl1$	0.82	2.16	2.9422 (17)	160
$N3-H3\cdots O3^{ii}$	0.86	1.78	2.610 (2)	162
$C6-H6\cdots O2^{iii}$	0.93	2.58	3.476 (3)	163

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

There are two ‘long’ P–O bonds [P1–O1 = 1.5526 (16) and P1–O2 = 1.5513 (16) Å] and one ‘short’ P–O bond [P1–O3 = 1.4805 (14) Å]. The ‘long’ P–O bonds correspond to the P–O–H moieties and the ‘short’ P–O bond corresponds to the phosphoryl P=O moiety. All P–O bond lengths have the expected values (Colodrero *et al.*, 2013). The bond lengths of the triazolium moiety [N1–N2 = 1.317 (2) Å, N1–C2 = 1.474 (3) Å, N1–C3 = 1.346 (3) Å, N2–N3 = 1.318 (2) Å, N3–C4 = 1.352 (2) Å] are very similar to those in 1,2,4-triazolium chloride (Bujak & Zaleski, 2002).

#### Hydrogen bonding

The phosphonic acid moiety forms four hydrogen-bonding interactions (Fig. 2 and Table 1). Specifically, each of the two P–O–H groups interacts with a different Cl<sup>−</sup> counter-ion, with contacts  $O1\cdots Cl1 = 2.9521$  (16) Å and  $O2\cdots Cl1 = 2.9422$  (17) Å. The phosphoryl P=O group forms a hydrogen bond with the N–H portion of the triazolium ring [ $O3\cdots N3$  2.610 (2) Å]. Finally, the benzene ring interacts with a phosphonate oxygen through a weak C–H $\cdots$ O contact at 3.476 (3) Å ( $C6\cdots O2$ ).



**Figure 3**  
Packing of 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride along the *a*- (upper), *b*- (middle), and *c*-axes (lower).

### $\pi$ - $\pi$ stacking interactions

There is only one type of very weak  $\pi$ - $\pi$  stacking interaction in the structure of 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride. The centroid-to-centroid distance is 4.0423 (15) Å, with the rings being 'shifted' from one another (slippage distance between the rings: 2.222 Å) and parallel.

### Crystal packing

Fig. 3 shows the packing along the three axes. The  $\pi$ - $\pi$  stacking interactions are parallel to the *b* axis. The chloride anions form corrugated sheets ['short' Cl...Cl distances at 4.9455 (12) Å and 'long' Cl...Cl distances at 6.4564 (9) Å] that are parallel to the *bc* plane.

## Synthesis and crystallization

### Reagents and materials

All starting materials were obtained from commercial sources and used without further purification. The reagents diethyl 2-bromoethylphosphonate (97%), phenylacetylene (98+%), copper sulfate pentahydrate (99%), zinc nitrate hexahydrate (98%) and ethylenediaminetetraacetic acid (98%) were from Alfa Aesar. Sodium azide and L-ascorbic acid were from Serva. Sodium sulfate was from Merck. Dichloromethane, tetrahydrofuran (THF), hydrochloric acid (37%) and nitric acid (70%) were from Scharlau. Ion-exchange-column deionized water was used.

### Synthesis of 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride (PTEPHCl)

Three distinct steps were followed for the syntheses of the ligand PTEP. The first step was the synthesis of diethyl-(2-azidoethyl)phosphonate, following a properly adapted published procedure (Sheikhi *et al.*, 2018). Specifically, sodium azide (10.6 g, 163.05 mmol) was added to a solution of diethyl-2-bromoethylphosphonate (10.4 g, 42.44 mmol) in water (50 mL). The reaction mixture was stirred at 338 K for 24 h. Then, extraction was carried out with dichloromethane (4 × 50 mL) and the organic phase was collected and dried over sodium sulfate. After filtration, a yellow oil was obtained, which is diethyl-(2-azidoethyl)phosphonate. The second step included the reaction of diethyl-(2-azidoethyl)phosphonate (3 mL, 2.07 mmol) with phenyl acetylene (895 µL, 1.035 mmol) in THF (67.5 mL), in the presence of copper sulfate (1.198 g 0.64 mmol) and L-ascorbic acid (0.218 g, 1.24 mmol) to produce diethyl[2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethyl]phosphonate ester. The reaction mixture was heated at 313 K under vigorous stirring for 48 h. After filtration, the filtrate was mixed with dichloromethane (50 mL) and an aqueous solution of the Cu<sup>2+</sup> chelant ethylenediaminetetraacetic acid (50 mL, 0.2 M) and the mixture was stirred for ~1 h. After extraction with dichloromethane (4 × 50 mL) and evaporation, diethyl[2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethyl]phosphonate ester was obtained in solid form. Finally, the latter (0.5 g) was hydrolyzed with 25 mL of H<sub>2</sub>O and 30 mL of HCl at 373 K for 48 h, giving 5-phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride in crystalline form (yield: 0.3 g, 60%). The crystal used for the data collection was handled

Table 2

Experimental details.

Crystal data	
Chemical formula	C <sub>10</sub> H <sub>13</sub> N <sub>3</sub> O <sub>3</sub> P <sup>+</sup> ·Cl <sup>-</sup>
<i>M<sub>r</sub></i>	289.65
Crystal system, space group	Monoclinic, <i>P</i> <sub>2</sub> / <i>c</i>
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.5857 (6), 7.0616 (4), 16.6118 (9)
$\beta$ (°)	108.222 (2)
<i>V</i> (Å <sup>3</sup> )	1290.92 (12)
<i>Z</i>	4
Radiation type	Cu <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	3.86
Crystal size (mm)	0.12 × 0.09 × 0.08
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2016)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.524, 0.753
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	11621, 2268, 2048
<i>R</i> <sub>int</sub>	0.046
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.596
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.042, 0.129, 1.11
No. of reflections	2268
No. of parameters	166
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.21, -0.34

Computer programs: *APEX3* (Bruker, 2019), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

under inert conditions. It was manipulated while immersed in a perfluoropolyether protecting oil and mounted on a MiTeGen Micromount<sup>™</sup>.

<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  8.51 (*s*, 1H), 7.93 (*d*, 2H), 7.67 (*m*, 3H), 4.82 (*m*, 2H), 2.52 (*m*, 2H). <sup>13</sup>C NMR (75.5 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  146.71, 131.27, 129.38, 128.28, 125.53, 121.87, 45.42, 30.45 (*d*, *J*<sub>CP</sub> = 134.5 Hz). <sup>31</sup>P NMR (121.5 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  20.17.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

### Funding information

Funding for this research was provided for the research project 'Innovative Materials and Applications' (INNOVAMAT, KA 10694) by the Special Account for Research Grants.

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## full crystallographic data

*IUCrData* (2022). 7, x220189 [https://doi.org/10.1107/S2414314622001894]

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## 5-Phenyl-3-(2-phosphonoethyl)-1,2,3-triazol-1-ium chloride

*Crystal data*

$C_{10}H_{13}N_3O_3P^+Cl^-$   
 $M_r = 289.65$   
 Monoclinic,  $P2_1/c$   
 $a = 11.5857$  (6) Å  
 $b = 7.0616$  (4) Å  
 $c = 16.6118$  (9) Å  
 $\beta = 108.222$  (2)°  
 $V = 1290.92$  (12) Å<sup>3</sup>  
 $Z = 4$

$F(000) = 600$   
 $D_x = 1.490$  Mg m<sup>-3</sup>  
 Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å  
 Cell parameters from 9685 reflections  
 $\theta = 4.0$ – $66.7$ °  
 $\mu = 3.86$  mm<sup>-1</sup>  
 $T = 298$  K  
 Plate, colourless  
 $0.12 \times 0.09 \times 0.08$  mm

*Data collection*

Bruker D8 Venture  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2016)  
 $T_{\min} = 0.524$ ,  $T_{\max} = 0.753$   
 11621 measured reflections

2268 independent reflections  
 2048 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.046$   
 $\theta_{\max} = 66.7$ °,  $\theta_{\min} = 4.0$ °  
 $h = -12 \rightarrow 13$   
 $k = -8 \rightarrow 8$   
 $l = -18 \rightarrow 19$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.129$   
 $S = 1.11$   
 2268 reflections  
 166 parameters  
 0 restraints  
 Hydrogen site location: inferred from  
 neighbouring sites

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0842P)^2 + 0.2113P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL2019/1  
 (Sheldrick 2015b),  
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0086 (14)

*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.

**Refinement.** All hydrogen atoms were located in difference Fourier maps and included as fixed contributions riding on attached atoms with isotropic thermal displacement parameters 1.2 or 1.5 times those of the respective atom.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.81725 (4)	0.61394 (8)	0.26679 (3)	0.0473 (2)
O1	0.91887 (15)	0.4728 (3)	0.26434 (10)	0.0660 (5)
H1	0.946479	0.421812	0.310754	0.099*
O2	0.78145 (14)	0.7154 (3)	0.17962 (10)	0.0663 (5)
H2	0.838534	0.711018	0.160392	0.099*
O3	0.70907 (13)	0.5261 (2)	0.28078 (9)	0.0570 (4)
N1	0.68960 (15)	0.9187 (2)	0.35631 (11)	0.0480 (4)
N2	0.59278 (15)	0.9687 (3)	0.29376 (10)	0.0497 (4)
N3	0.50156 (14)	0.9160 (2)	0.32010 (10)	0.0446 (4)
H3	0.426572	0.931250	0.290694	0.054*
C1	0.88515 (18)	0.7829 (3)	0.34778 (14)	0.0530 (5)
H1A	0.902609	0.720386	0.402270	0.064*
H1B	0.962183	0.821985	0.341559	0.064*
C2	0.8107 (2)	0.9598 (3)	0.34921 (17)	0.0626 (6)
H2A	0.855085	1.038465	0.396689	0.075*
H2B	0.801074	1.031479	0.297697	0.075*
C3	0.66201 (18)	0.8378 (3)	0.42134 (13)	0.0468 (5)
H3A	0.716089	0.792849	0.471680	0.056*
C4	0.53734 (17)	0.8355 (3)	0.39786 (12)	0.0406 (4)
C5	0.45429 (18)	0.7694 (2)	0.44224 (12)	0.0419 (5)
C6	0.4974 (2)	0.7322 (3)	0.52898 (13)	0.0509 (5)
H6	0.579280	0.748521	0.558805	0.061*
C7	0.4176 (3)	0.6708 (3)	0.57037 (15)	0.0634 (6)
H7	0.446686	0.644005	0.628029	0.076*
C8	0.2960 (3)	0.6489 (3)	0.52761 (18)	0.0678 (7)
H8	0.242943	0.611045	0.556500	0.081*
C9	0.2534 (2)	0.6829 (4)	0.44228 (18)	0.0727 (7)
H9	0.171424	0.665745	0.412999	0.087*
C10	0.3319 (2)	0.7427 (3)	0.39929 (16)	0.0573 (6)
H10	0.302340	0.765053	0.341264	0.069*
Cl1	0.94890 (5)	0.76568 (8)	0.07972 (3)	0.0583 (3)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
P1	0.0364 (3)	0.0693 (4)	0.0365 (4)	0.0015 (2)	0.0118 (2)	0.0013 (2)
O1	0.0610 (10)	0.0914 (12)	0.0490 (9)	0.0254 (8)	0.0223 (7)	0.0080 (8)
O2	0.0475 (9)	0.1068 (13)	0.0442 (9)	0.0135 (8)	0.0136 (7)	0.0171 (8)
O3	0.0468 (8)	0.0797 (10)	0.0457 (8)	-0.0157 (7)	0.0163 (6)	-0.0101 (7)
N1	0.0392 (9)	0.0508 (9)	0.0543 (10)	-0.0028 (7)	0.0150 (7)	-0.0041 (7)
N2	0.0458 (9)	0.0583 (10)	0.0469 (10)	0.0002 (7)	0.0174 (8)	0.0004 (7)
N3	0.0383 (8)	0.0560 (9)	0.0385 (9)	0.0008 (7)	0.0104 (6)	-0.0007 (7)
C1	0.0325 (10)	0.0742 (14)	0.0510 (12)	-0.0047 (9)	0.0110 (9)	-0.0007 (10)
C2	0.0422 (11)	0.0659 (14)	0.0816 (16)	-0.0130 (10)	0.0220 (11)	-0.0053 (12)
C3	0.0401 (10)	0.0492 (10)	0.0470 (11)	-0.0016 (8)	0.0076 (8)	-0.0018 (8)

C4	0.0419 (9)	0.0394 (9)	0.0384 (10)	0.0006 (7)	0.0097 (7)	-0.0037 (7)
C5	0.0470 (11)	0.0379 (9)	0.0414 (11)	0.0027 (7)	0.0145 (8)	-0.0006 (7)
C6	0.0610 (13)	0.0468 (11)	0.0419 (11)	0.0008 (9)	0.0118 (9)	-0.0026 (8)
C7	0.1016 (19)	0.0488 (12)	0.0472 (12)	0.0034 (12)	0.0339 (12)	0.0020 (9)
C8	0.0832 (18)	0.0559 (13)	0.0814 (18)	0.0002 (12)	0.0503 (15)	0.0100 (12)
C9	0.0525 (13)	0.0751 (16)	0.094 (2)	-0.0061 (12)	0.0289 (13)	0.0171 (14)
C10	0.0476 (12)	0.0685 (14)	0.0525 (13)	-0.0047 (9)	0.0110 (10)	0.0134 (10)
C11	0.0507 (4)	0.0795 (4)	0.0435 (4)	0.0062 (2)	0.0130 (3)	-0.0025 (2)

*Geometric parameters (Å, °)*

P1—O1	1.5526 (16)	C2—H2B	0.9700
P1—O2	1.5513 (16)	C3—H3A	0.9300
P1—O3	1.4805 (14)	C3—C4	1.373 (3)
P1—C1	1.786 (2)	C4—C5	1.460 (3)
O1—H1	0.8200	C5—C6	1.394 (3)
O2—H2	0.8200	C5—C10	1.386 (3)
N1—N2	1.317 (2)	C6—H6	0.9300
N1—C2	1.474 (3)	C6—C7	1.383 (3)
N1—C3	1.346 (3)	C7—H7	0.9300
N2—N3	1.318 (2)	C7—C8	1.373 (4)
N3—H3	0.8600	C8—H8	0.9300
N3—C4	1.352 (2)	C8—C9	1.368 (4)
C1—H1A	0.9700	C9—H9	0.9300
C1—H1B	0.9700	C9—C10	1.387 (3)
C1—C2	1.522 (3)	C10—H10	0.9300
C2—H2A	0.9700		
O1—P1—C1	106.83 (10)	H2A—C2—H2B	107.7
O2—P1—O1	104.80 (9)	N1—C3—H3A	127.2
O2—P1—C1	108.71 (11)	N1—C3—C4	105.65 (18)
O3—P1—O1	114.92 (11)	C4—C3—H3A	127.2
O3—P1—O2	110.35 (9)	N3—C4—C3	104.31 (17)
O3—P1—C1	110.89 (9)	N3—C4—C5	124.28 (17)
P1—O1—H1	109.5	C3—C4—C5	131.39 (18)
P1—O2—H2	109.5	C6—C5—C4	120.16 (19)
N2—N1—C2	118.78 (18)	C10—C5—C4	120.86 (18)
N2—N1—C3	112.94 (16)	C10—C5—C6	119.0 (2)
C3—N1—C2	128.26 (18)	C5—C6—H6	120.2
N1—N2—N3	103.64 (15)	C7—C6—C5	119.6 (2)
N2—N3—H3	123.3	C7—C6—H6	120.2
N2—N3—C4	113.45 (16)	C6—C7—H7	119.5
C4—N3—H3	123.3	C8—C7—C6	121.0 (2)
P1—C1—H1A	108.2	C8—C7—H7	119.5
P1—C1—H1B	108.2	C7—C8—H8	120.2
H1A—C1—H1B	107.4	C9—C8—C7	119.7 (2)
C2—C1—P1	116.22 (15)	C9—C8—H8	120.2
C2—C1—H1A	108.2	C8—C9—H9	119.8

C2—C1—H1B	108.2	C8—C9—C10	120.3 (2)
N1—C2—C1	113.42 (18)	C10—C9—H9	119.8
N1—C2—H2A	108.9	C5—C10—C9	120.4 (2)
N1—C2—H2B	108.9	C5—C10—H10	119.8
C1—C2—H2A	108.9	C9—C10—H10	119.8
C1—C2—H2B	108.9		
P1—C1—C2—N1	56.6 (3)	C2—N1—C3—C4	178.52 (19)
O1—P1—C1—C2	167.10 (17)	C3—N1—N2—N3	-0.6 (2)
O2—P1—C1—C2	54.50 (19)	C3—N1—C2—C1	65.1 (3)
O3—P1—C1—C2	-67.0 (2)	C3—C4—C5—C6	14.3 (3)
N1—N2—N3—C4	0.6 (2)	C3—C4—C5—C10	-165.7 (2)
N1—C3—C4—N3	0.0 (2)	C4—C5—C6—C7	179.59 (18)
N1—C3—C4—C5	-178.62 (18)	C4—C5—C10—C9	-178.9 (2)
N2—N1—C2—C1	-116.9 (2)	C5—C6—C7—C8	-1.1 (3)
N2—N1—C3—C4	0.4 (2)	C6—C5—C10—C9	1.0 (3)
N2—N3—C4—C3	-0.4 (2)	C6—C7—C8—C9	1.9 (4)
N2—N3—C4—C5	178.37 (17)	C7—C8—C9—C10	-1.2 (4)
N3—C4—C5—C6	-164.07 (18)	C8—C9—C10—C5	-0.2 (4)
N3—C4—C5—C10	15.9 (3)	C10—C5—C6—C7	-0.4 (3)
C2—N1—N2—N3	-178.94 (17)		

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...C11 <sup>i</sup>	0.82	2.15	2.9521 (16)	167
O2—H2...C11	0.82	2.16	2.9422 (17)	160
N3—H3...O3 <sup>ii</sup>	0.86	1.78	2.610 (2)	162
C6—H6...O2 <sup>iii</sup>	0.93	2.58	3.476 (3)	163

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