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Crystal structure of (*n*-butyl)[2-(2,6-dimethoxyphenyl)-6-methylphenyl](2-methoxyphenyl)-phosphonium chloride monohydrate

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The title hydrated salt, $C_{26}H_{32}O_3P^+ \cdot Cl^- \cdot H_2O$, contains four different substituents (H, alkyl, aryl, and biaryl) on the P atom. The P—H hydrogen atom of the phosphonium ion was located in a difference Fourier map and refined without imposing additional restraints. In the crystal, the Cl^- ions and water molecules are linked by pairs of $O_{\text{water}}-H \cdots Cl^-$ hydrogen bonds and further linked to the phosphonium cation by $P-H^+ \cdots Cl^-$ and $C_{Ar/OMe}-H \cdots O_{\text{water}}$ hydrogen bonds to form an infinite one-dimensional chain along the [010] direction.

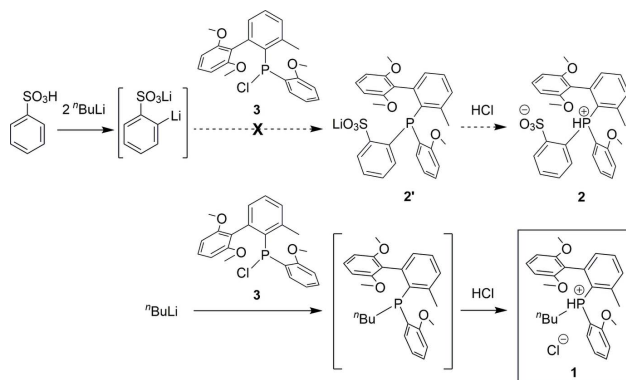
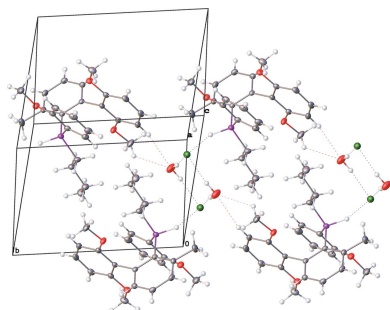
Keywords: crystal structure; phosphonium salt; hydrogen bonding

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1. Chemical context

Palladium(II) alkyl complexes that contain *ortho*-phosphino-arenesulfonate ligands ($[PO]^-$) exhibit unique behavior in olefin polymerization (Nakamura *et al.*, 2009; Ito & Nozaki, 2010; Nakamura *et al.*, 2013). One of the main drawbacks of traditional (PO)Pd alkyl catalysts is that they produce polyethylene with only low-to-moderate molecular weight (Drent *et al.*, 2002; Vela *et al.*, 2007). Studies have shown that incorporating bulky substituents on phosphorous in the $[PO]^-$ ligand is an effective strategy to increase the molecular weight of the produced polymer (Skupov *et al.*, 2007; Shen & Jordan, 2009; Ota *et al.*, 2014). Therefore we were interested in developing the new $[PO]^-$ ligand **2** that contains bulky *P*-substituents (see Scheme). We attempted to prepare **2** by the reaction of (2-[2,6-(OMe)₂-Ph]-6-Me-Ph)(2-OMe-Ph)PCl (**3**) with *in situ*-generated dilithiated benzenesulfonate to generate **2'**, followed by acidification with HCl. However, this procedure did not afford **2** but rather produced [(2-[2,6-(OMe)₂-Ph]-6-Me-Ph)(2-OMe-Ph)(*n*-Bu)PH]Cl (**1**) in low yield after workup, which crystallizes as the monohydrate **1**·H₂O (**I**). **1** likely formed by the reaction of **3** with the slight excess of *n*-BuLi present in the dilithiated benzenesulfonate solution. Here we report the crystal structure of **I**.



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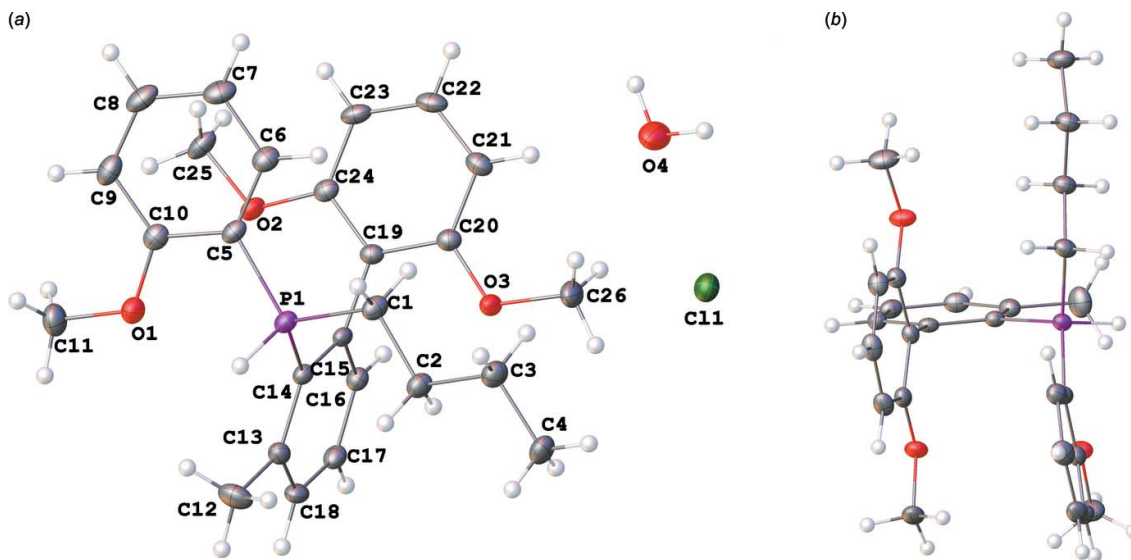


Figure 1

(a) The molecular structure of **I** drawn with the 50% probability ellipsoids and showing the atom-labelling scheme. (b) A different view of **I** with H₂O and Cl[−] moieties omitted for clarity.

2. Structural commentary

Crystals of **1**·H₂O (**I**) suitable for X-ray diffraction analysis were obtained by recrystallization from wet CH₂Cl₂/Et₂O (Fig. 1a). The P—C bond lengths are almost equal for the alkyl, aryl, and biaryl substituents [1.7994 (14), 1.7824 (14), and 1.8043 (13) Å, respectively]. The C—P—H angles are also very similar [106.2 (7), 104.9 (7), and 107.5 (7)° for the alkyl, aryl, and biaryl substituents, respectively]. The aryl rings in the biaryl unit are essentially perpendicular to each other, with the angle between the mean planes passing through the six-membered rings being 88.60 (6)°. This conformation minimizes steric interactions between the *ortho*-methoxy groups and the *ortho*-hydrogens on the two rings. The mean planes

passing through 2,6-dimethoxyphenyl ring and the C-atoms of the 2-methoxyphenyl and *n*-butyl groups are almost parallel to each other [the angle is 10.36 (5)°, Fig. 1b]. The P—H hydrogen atom was located in a difference Fourier map and refined without additional restraints. The refined P—H bond length of 1.313 (16) Å is similar to those previously reported (Burke *et al.*, 2000; Zhu *et al.*, 2007; Wucher *et al.*, 2013).

3. Supramolecular features

The P—H⁺, Cl[−], and water molecule are involved in intermolecular hydrogen bonding (Fig. 2, Table 1). Two Cl[−] ions and two water molecules form a rhombus (Fig. 3) in which the O...Cl distances are almost equal [3.1717 (13) and

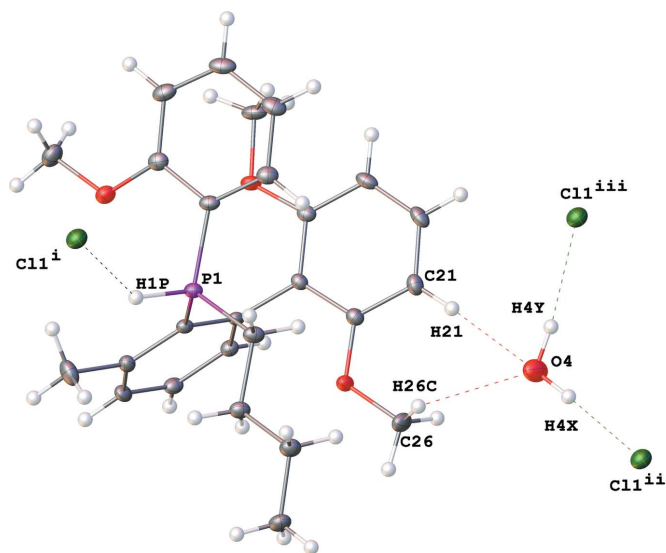


Figure 2

Hydrogen bonds in **I**. [Symmetry codes: (i) $x - 1, y + 1, z$; (ii) $-x + 1, -y, -z + 1$; (iii) $x - 1, y, z$.]

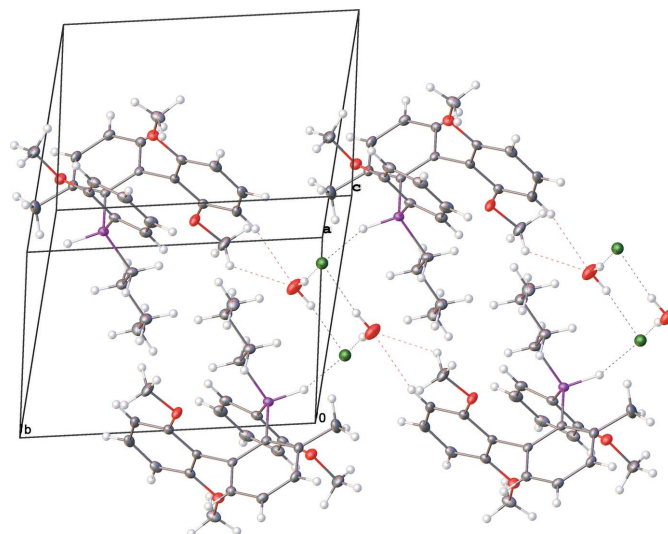


Figure 3

A fragment of the crystal packing of **I**.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$P1-H1P\cdots Cl1^i$	1.313 (16)	2.523 (16)	3.5798 (5)	135.5 (10)
$C21-H21\cdots O4$	0.95	2.53	3.4594 (19)	167
$C26-H26C\cdots O4$	0.98	2.53	3.2250 (19)	128
$O4-H4X\cdots Cl1^{ii}$	0.93 (2)	2.24 (2)	3.1717 (13)	173 (2)
$O4-H4Y\cdots Cl1^{iii}$	0.94 (2)	2.25 (2)	3.1841 (13)	173 (2)

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

3.1841 (13) Å]. The Cl^- ions are further engaged in $P-H^+\cdots Cl^-$ hydrogen bonds [2.523 (16) Å], and the water molecules are also involved in $C_{Ar}/OMe-H\cdots O_{water}$ contacts [2.243 (16) and 2.254 (16) Å], forming infinite chains along the [010] direction (Fig. 3). The involvement of the P–H hydrogen atom in hydrogen bonding stands in contrast to what has been observed in some related structures. For example, in the structures of triphenylphosphonium perchlorate (Zhu *et al.*, 2007) and tris(*ortho*-tolyl)phosphonium tetrachloroborate (Burke *et al.*, 2000), there is no evidence for involvement of the P–H hydrogen atom in hydrogen bonding.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.36, last update May 2015; Groom & Allen, 2014) revealed that structures of phosphonium salts having different alkyl/aryl/biaryl substituents on phosphorous are rare [CSD refcodes: BZMNPB (Böhme *et al.*, 1975), EDOSOF (Schiemenz *et al.*, 2002), SUXFUN (Dziuba *et al.*, 2010)]. To the best of our knowledge **1** is the first example of a crystallographically characterized phosphonium salt having four different substituents at phosphorous. Moreover, there are only three other examples of structures of protonated phosphonium aryl/biaryl salts [CSD refcodes: WEMSIQ (Carre *et al.*, 1997), OCOWUY (Karaçar *et al.*, 2001), TOMZIF (Wang *et al.*, 2008)].

5. Synthesis and crystallization

(2-{2,6-(OMe)₂-Ph}-6-Me-Ph)(2-OMe-Ph)PCl (**3**) was synthesized by a modification of a previously reported procedure (Neuwald *et al.*, 2013). The reaction of **3** with *in situ*-generated dilithiated benzenesulfonate was attempted to synthesize **2'** (see Scheme). However ³¹P and ESI-MS of the reaction mixture showed that **2'** was not formed. The reaction mixture was acidified with aqueous HCl and extracted with Et₂O. After removal of volatiles from the Et₂O fraction under vacuum, a yellow oil and white crystals (low yield) were obtained. Recrystallization of the white crystals from wet CH₂Cl₂/Et₂O yielded crystals of [(2-{2,6-(OMe)₂-Ph}-6-Me-Ph)(2-OMe-Ph)-(n-Bu)PH]Cl·H₂O (**1**·H₂O, **1**), which was identified by X-ray crystallography analysis.

Table 2

Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{32}O_3P^+\cdot Cl^-\cdot H_2O$
M_r	476.95
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	9.6920 (6), 10.2790 (6), 12.4154 (8)
α, β, γ (°)	96.836 (2), 98.481 (2), 94.188 (2)
V (Å ³)	1209.47 (13)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.25
Crystal size (mm)	0.22 × 0.15 × 0.14
Data collection	
Diffractometer	Bruker D8 Venture PHOTON 100 CMOS
Absorption correction	Numerical (SADABS; Bruker, 2014)
T_{min}, T_{max}	0.959, 0.987
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33225, 6228, 5241
R_{int}	0.028
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.677
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.038, 0.096, 1.04
No. of reflections	6228
No. of parameters	339
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.51, -0.18

Computer programs: APEX2 and SAINT (Bruker, 2012), SHELXT2014 (Sheldrick, 2015a), SHELXL2014 (Sheldrick, 2015b), OLEX2 (Dolomanov *et al.*, 2009), Mercury (Macrae *et al.*, 2008), and publCIF (Westrip, 2010).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Most of the carbon-bound H atoms were included in idealized positions for structure factor calculations [$C-H = 0.95-0.98$ Å, $U_{iso}(H)$ set to $1.2-1.5U_{eq}(C)$]. The P–H hydrogen atom and the H atoms of the butyl group were located in a difference Fourier map and refined without additional restraints. The H atoms bound to oxygen atom O4 were also located in the difference Fourier map but were restrained to be at 0.96 Å from O4 (within 0.02 Å) with their thermal parameters set to $1.5U_{eq}$ of O4.

Acknowledgements

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supporting information

Acta Cryst. (2016). E72, 174–177 [doi:10.1107/S2056989015024780]

Crystal structure of (*n*-butyl)[2-(2,6-dimethoxyphenyl)-6-methylphenyl](2-methoxyphenyl)phosphonium chloride monohydrate

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXT2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(*n*-Butyl)[2-(2,6-dimethoxyphenyl)-6-methylphenyl](2-methoxyphenyl)phosphonium chloride monohydrate

Crystal data

$C_{26}H_{32}O_3P^+Cl^- \cdot H_2O$

$M_r = 476.95$

Triclinic, $P\bar{1}$

$a = 9.6920$ (6) Å

$b = 10.2790$ (6) Å

$c = 12.4154$ (8) Å

$\alpha = 96.836$ (2)°

$\beta = 98.481$ (2)°

$\gamma = 94.188$ (2)°

$V = 1209.47$ (13) Å³

$Z = 2$

$F(000) = 508$

$D_x = 1.310$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9958 reflections

$\theta = 2.4$ – 28.7°

$\mu = 0.25$ mm⁻¹

$T = 100$ K

Block, colorless

$0.22 \times 0.15 \times 0.14$ mm

Data collection

Bruker D8 Venture PHOTON 100 CMOS
diffractometer

Radiation source: INCOATEC I μ S micro-focus
source

Mirrors monochromator

Detector resolution: 10.4167 pixels mm⁻¹

ω and ϕ scans

Absorption correction: numerical
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.959$, $T_{\max} = 0.987$

33225 measured reflections

6228 independent reflections

5241 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 28.8^\circ$, $\theta_{\min} = 2.1^\circ$

$h = -13 \rightarrow 13$

$k = -13 \rightarrow 13$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.096$

$S = 1.04$

6228 reflections

339 parameters

2 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.6272P]$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$$

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.99543 (4)	0.06475 (3)	0.71011 (3)	0.02320 (9)
P1	0.22865 (3)	0.81030 (3)	0.70834 (3)	0.01537 (9)
H1P	0.1976 (17)	0.9280 (16)	0.6876 (14)	0.021 (4)*
O1	0.28474 (11)	0.98104 (10)	0.90415 (8)	0.0218 (2)
O2	0.41677 (10)	0.67819 (10)	0.96813 (8)	0.0191 (2)
O3	0.39185 (11)	0.49203 (9)	0.60356 (8)	0.0202 (2)
C1	0.14332 (15)	0.69712 (15)	0.59177 (11)	0.0191 (3)
H1A	0.1528 (17)	0.6081 (17)	0.6062 (14)	0.021 (4)*
H1B	0.0437 (19)	0.7148 (17)	0.5838 (14)	0.025 (4)*
C2	0.20590 (15)	0.71909 (15)	0.48809 (11)	0.0200 (3)
H2A	0.3053 (18)	0.7053 (16)	0.4998 (14)	0.020 (4)*
H2B	0.1977 (17)	0.8097 (17)	0.4745 (14)	0.021 (4)*
C3	0.13321 (16)	0.62469 (15)	0.38878 (12)	0.0228 (3)
H3A	0.035 (2)	0.6419 (17)	0.3743 (15)	0.028 (5)*
H3B	0.1359 (19)	0.5340 (19)	0.4054 (15)	0.029 (5)*
C4	0.20372 (19)	0.63906 (17)	0.28846 (13)	0.0266 (3)
H4A	0.300 (2)	0.6220 (18)	0.3021 (15)	0.027 (5)*
H4B	0.157 (2)	0.581 (2)	0.2247 (17)	0.038 (5)*
H4C	0.201 (2)	0.725 (2)	0.2709 (16)	0.034 (5)*
C5	0.15371 (14)	0.78501 (14)	0.82781 (11)	0.0165 (3)
C6	0.05961 (14)	0.67784 (15)	0.83277 (12)	0.0201 (3)
H6	0.0367	0.6102	0.7721	0.024*
C7	−0.00070 (15)	0.67025 (16)	0.92696 (13)	0.0239 (3)
H7	−0.0642	0.5969	0.9314	0.029*
C8	0.03227 (15)	0.77041 (16)	1.01452 (12)	0.0245 (3)
H8	−0.0104	0.7652	1.0782	0.029*
C9	0.12588 (15)	0.87774 (15)	1.01127 (12)	0.0225 (3)
H9	0.1471	0.9456	1.0718	0.027*
C10	0.18838 (14)	0.88450 (14)	0.91790 (11)	0.0186 (3)
C11	0.32790 (17)	1.08374 (15)	0.99388 (13)	0.0269 (3)
H11A	0.2459	1.1266	1.0125	0.040*
H11B	0.3947	1.1486	0.9728	0.040*
H11C	0.3727	1.0463	1.0577	0.040*
C12	0.43520 (17)	1.03920 (15)	0.66031 (14)	0.0288 (3)
H12A	0.5106	1.1035	0.6504	0.043*
H12B	0.3830	1.0780	0.7160	0.043*

H12C	0.3718	1.0145	0.5905	0.043*
C13	0.49738 (14)	0.91817 (13)	0.69739 (11)	0.0173 (3)
C14	0.41672 (13)	0.81018 (13)	0.72460 (10)	0.0141 (2)
C15	0.48062 (13)	0.70142 (12)	0.76063 (10)	0.0138 (2)
C16	0.62572 (14)	0.70164 (13)	0.76972 (11)	0.0165 (3)
H16	0.6703	0.6293	0.7952	0.020*
C17	0.70582 (14)	0.80644 (14)	0.74193 (11)	0.0185 (3)
H17	0.8045	0.8052	0.7476	0.022*
C18	0.64171 (15)	0.91265 (14)	0.70595 (11)	0.0192 (3)
H18	0.6974	0.9836	0.6866	0.023*
C19	0.39855 (13)	0.58266 (13)	0.78584 (11)	0.0145 (3)
C20	0.35655 (14)	0.47546 (13)	0.70350 (11)	0.0166 (3)
C21	0.28231 (14)	0.36210 (13)	0.72404 (12)	0.0197 (3)
H21	0.2515	0.2913	0.6671	0.024*
C22	0.25470 (14)	0.35547 (14)	0.82964 (13)	0.0212 (3)
H22	0.2044	0.2786	0.8446	0.025*
C23	0.29803 (14)	0.45739 (14)	0.91409 (12)	0.0202 (3)
H23	0.2791	0.4499	0.9861	0.024*
C24	0.36994 (14)	0.57144 (13)	0.89184 (11)	0.0164 (3)
C25	0.38278 (16)	0.67481 (16)	1.07612 (12)	0.0233 (3)
H25A	0.2811	0.6580	1.0716	0.035*
H25B	0.4148	0.7595	1.1211	0.035*
H25C	0.4291	0.6046	1.1097	0.035*
C26	0.36073 (17)	0.38149 (15)	0.51867 (12)	0.0267 (3)
H26A	0.4082	0.3066	0.5429	0.040*
H26B	0.3934	0.4048	0.4518	0.040*
H26C	0.2593	0.3577	0.5034	0.040*
O4	0.12871 (13)	0.13559 (12)	0.50244 (11)	0.0373 (3)
H4X	0.097 (2)	0.072 (2)	0.4420 (16)	0.056*
H4Y	0.085 (2)	0.108 (2)	0.5594 (16)	0.056*

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

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02306 (18)	0.02389 (18)	0.02263 (18)	0.00422 (13)	0.00472 (13)	0.00038 (13)
P1	0.01430 (16)	0.01926 (17)	0.01294 (17)	0.00220 (13)	0.00277 (12)	0.00249 (13)
O1	0.0250 (5)	0.0225 (5)	0.0166 (5)	−0.0003 (4)	0.0035 (4)	−0.0008 (4)
O2	0.0213 (5)	0.0242 (5)	0.0124 (5)	0.0012 (4)	0.0050 (4)	0.0025 (4)
O3	0.0268 (5)	0.0180 (5)	0.0148 (5)	−0.0052 (4)	0.0061 (4)	−0.0008 (4)
C1	0.0161 (6)	0.0259 (7)	0.0145 (6)	0.0002 (5)	0.0012 (5)	0.0024 (5)
C2	0.0201 (7)	0.0246 (7)	0.0150 (7)	0.0006 (6)	0.0029 (5)	0.0020 (5)
C3	0.0250 (8)	0.0263 (8)	0.0167 (7)	0.0005 (6)	0.0029 (6)	0.0021 (6)
C4	0.0359 (9)	0.0280 (8)	0.0164 (7)	0.0028 (7)	0.0079 (6)	0.0002 (6)
C5	0.0138 (6)	0.0232 (7)	0.0138 (6)	0.0049 (5)	0.0038 (5)	0.0038 (5)
C6	0.0148 (6)	0.0265 (7)	0.0192 (7)	0.0026 (5)	0.0027 (5)	0.0034 (6)
C7	0.0148 (6)	0.0341 (8)	0.0250 (8)	0.0013 (6)	0.0059 (5)	0.0094 (6)
C8	0.0177 (7)	0.0401 (9)	0.0192 (7)	0.0092 (6)	0.0076 (5)	0.0086 (6)
C9	0.0211 (7)	0.0301 (8)	0.0172 (7)	0.0100 (6)	0.0041 (5)	0.0013 (6)

C10	0.0169 (6)	0.0223 (7)	0.0172 (7)	0.0058 (5)	0.0015 (5)	0.0042 (5)
C11	0.0322 (8)	0.0238 (7)	0.0219 (7)	0.0016 (6)	0.0004 (6)	−0.0036 (6)
C12	0.0285 (8)	0.0207 (7)	0.0391 (9)	0.0007 (6)	0.0052 (7)	0.0133 (7)
C13	0.0210 (7)	0.0156 (6)	0.0151 (6)	−0.0004 (5)	0.0032 (5)	0.0018 (5)
C14	0.0137 (6)	0.0167 (6)	0.0118 (6)	−0.0001 (5)	0.0030 (5)	0.0006 (5)
C15	0.0155 (6)	0.0155 (6)	0.0098 (6)	−0.0016 (5)	0.0031 (5)	0.0001 (5)
C16	0.0161 (6)	0.0180 (6)	0.0155 (6)	0.0026 (5)	0.0032 (5)	0.0013 (5)
C17	0.0151 (6)	0.0222 (7)	0.0176 (7)	−0.0010 (5)	0.0048 (5)	−0.0009 (5)
C18	0.0204 (7)	0.0184 (6)	0.0182 (7)	−0.0058 (5)	0.0058 (5)	0.0012 (5)
C19	0.0123 (6)	0.0161 (6)	0.0159 (6)	0.0013 (5)	0.0024 (5)	0.0047 (5)
C20	0.0146 (6)	0.0181 (6)	0.0173 (6)	0.0011 (5)	0.0026 (5)	0.0041 (5)
C21	0.0168 (6)	0.0163 (6)	0.0257 (7)	−0.0007 (5)	0.0021 (5)	0.0045 (5)
C22	0.0154 (6)	0.0210 (7)	0.0296 (8)	−0.0004 (5)	0.0056 (5)	0.0113 (6)
C23	0.0166 (6)	0.0268 (7)	0.0208 (7)	0.0035 (5)	0.0071 (5)	0.0114 (6)
C24	0.0134 (6)	0.0200 (6)	0.0171 (6)	0.0041 (5)	0.0035 (5)	0.0050 (5)
C25	0.0236 (7)	0.0348 (8)	0.0147 (7)	0.0091 (6)	0.0079 (5)	0.0059 (6)
C26	0.0329 (8)	0.0233 (7)	0.0209 (7)	−0.0081 (6)	0.0074 (6)	−0.0062 (6)
O4	0.0384 (7)	0.0375 (7)	0.0322 (7)	−0.0162 (5)	0.0104 (5)	−0.0042 (5)

Geometric parameters (Å, °)

P1—C5	1.7824 (14)	C11—H11B	0.9800
P1—C1	1.7994 (14)	C11—H11C	0.9800
P1—C14	1.8043 (13)	C12—C13	1.512 (2)
P1—H1P	1.313 (16)	C12—H12A	0.9800
O1—C10	1.3557 (17)	C12—H12B	0.9800
O1—C11	1.4313 (17)	C12—H12C	0.9800
O2—C24	1.3639 (17)	C13—C18	1.3930 (19)
O2—C25	1.4307 (16)	C13—C14	1.4135 (18)
O3—C20	1.3611 (16)	C14—C15	1.4042 (18)
O3—C26	1.4367 (17)	C15—C16	1.3940 (18)
C1—C2	1.5347 (19)	C15—C19	1.4979 (17)
C1—H1A	0.962 (17)	C16—C17	1.3884 (19)
C1—H1B	0.988 (18)	C16—H16	0.9500
C2—C3	1.522 (2)	C17—C18	1.382 (2)
C2—H2A	0.976 (17)	C17—H17	0.9500
C2—H2B	0.973 (17)	C18—H18	0.9500
C3—C4	1.523 (2)	C19—C24	1.4005 (18)
C3—H3A	0.975 (19)	C19—C20	1.4030 (18)
C3—H3B	0.980 (19)	C20—C21	1.3939 (19)
C4—H4A	0.952 (19)	C21—C22	1.385 (2)
C4—H4B	0.96 (2)	C21—H21	0.9500
C4—H4C	0.94 (2)	C22—C23	1.385 (2)
C5—C6	1.391 (2)	C22—H22	0.9500
C5—C10	1.4058 (19)	C23—C24	1.3967 (19)
C6—C7	1.390 (2)	C23—H23	0.9500
C6—H6	0.9500	C25—H25A	0.9800
C7—C8	1.388 (2)	C25—H25B	0.9800

C7—H7	0.9500	C25—H25C	0.9800
C8—C9	1.385 (2)	C26—H26A	0.9800
C8—H8	0.9500	C26—H26B	0.9800
C9—C10	1.3913 (19)	C26—H26C	0.9800
C9—H9	0.9500	O4—H4X	0.933 (16)
C11—H11A	0.9800	O4—H4Y	0.935 (16)
C5—P1—C1	110.72 (7)	H11B—C11—H11C	109.5
C5—P1—C14	115.02 (6)	C13—C12—H12A	109.5
C1—P1—C14	111.73 (6)	C13—C12—H12B	109.5
C5—P1—H1P	104.9 (7)	H12A—C12—H12B	109.5
C1—P1—H1P	106.2 (7)	C13—C12—H12C	109.5
C14—P1—H1P	107.5 (7)	H12A—C12—H12C	109.5
C10—O1—C11	117.40 (11)	H12B—C12—H12C	109.5
C24—O2—C25	117.30 (11)	C18—C13—C14	118.04 (12)
C20—O3—C26	117.23 (11)	C18—C13—C12	118.55 (12)
C2—C1—P1	111.17 (10)	C14—C13—C12	123.41 (12)
C2—C1—H1A	109.2 (10)	C15—C14—C13	120.89 (12)
P1—C1—H1A	110.0 (10)	C15—C14—P1	119.76 (10)
C2—C1—H1B	111.4 (10)	C13—C14—P1	119.31 (10)
P1—C1—H1B	105.0 (10)	C16—C15—C14	118.88 (12)
H1A—C1—H1B	110.1 (14)	C16—C15—C19	118.49 (12)
C3—C2—C1	111.50 (12)	C14—C15—C19	122.59 (11)
C3—C2—H2A	108.6 (10)	C17—C16—C15	120.72 (13)
C1—C2—H2A	109.4 (10)	C17—C16—H16	119.6
C3—C2—H2B	110.2 (10)	C15—C16—H16	119.6
C1—C2—H2B	109.0 (10)	C18—C17—C16	119.89 (13)
H2A—C2—H2B	108.0 (14)	C18—C17—H17	120.1
C2—C3—C4	111.23 (12)	C16—C17—H17	120.1
C2—C3—H3A	108.7 (11)	C17—C18—C13	121.55 (12)
C4—C3—H3A	110.7 (11)	C17—C18—H18	119.2
C2—C3—H3B	109.5 (11)	C13—C18—H18	119.2
C4—C3—H3B	109.1 (11)	C24—C19—C20	118.43 (12)
H3A—C3—H3B	107.5 (15)	C24—C19—C15	121.77 (12)
C3—C4—H4A	111.1 (11)	C20—C19—C15	119.66 (11)
C3—C4—H4B	111.9 (12)	O3—C20—C21	123.42 (12)
H4A—C4—H4B	108.7 (16)	O3—C20—C19	115.07 (11)
C3—C4—H4C	110.1 (12)	C21—C20—C19	121.51 (13)
H4A—C4—H4C	107.5 (16)	C22—C21—C20	118.23 (13)
H4B—C4—H4C	107.3 (16)	C22—C21—H21	120.9
C6—C5—C10	120.16 (12)	C20—C21—H21	120.9
C6—C5—P1	123.45 (11)	C21—C22—C23	122.09 (13)
C10—C5—P1	116.29 (10)	C21—C22—H22	119.0
C7—C6—C5	119.61 (13)	C23—C22—H22	119.0
C7—C6—H6	120.2	C22—C23—C24	119.05 (13)
C5—C6—H6	120.2	C22—C23—H23	120.5
C8—C7—C6	119.68 (14)	C24—C23—H23	120.5
C8—C7—H7	120.2	O2—C24—C23	124.22 (12)

C6—C7—H7	120.2	O2—C24—C19	115.16 (12)
C9—C8—C7	121.58 (13)	C23—C24—C19	120.62 (13)
C9—C8—H8	119.2	O2—C25—H25A	109.5
C7—C8—H8	119.2	O2—C25—H25B	109.5
C8—C9—C10	118.89 (14)	H25A—C25—H25B	109.5
C8—C9—H9	120.6	O2—C25—H25C	109.5
C10—C9—H9	120.6	H25A—C25—H25C	109.5
O1—C10—C9	125.57 (13)	H25B—C25—H25C	109.5
O1—C10—C5	114.38 (12)	O3—C26—H26A	109.5
C9—C10—C5	120.05 (13)	O3—C26—H26B	109.5
O1—C11—H11A	109.5	H26A—C26—H26B	109.5
O1—C11—H11B	109.5	O3—C26—H26C	109.5
H11A—C11—H11B	109.5	H26A—C26—H26C	109.5
O1—C11—H11C	109.5	H26B—C26—H26C	109.5
H11A—C11—H11C	109.5	H4X—O4—H4Y	105 (2)
C5—P1—C1—C2	179.34 (10)	C13—C14—C15—C19	−177.42 (12)
C14—P1—C1—C2	−51.05 (12)	P1—C14—C15—C19	0.53 (17)
P1—C1—C2—C3	−179.38 (10)	C14—C15—C16—C17	−1.07 (19)
C1—C2—C3—C4	−175.19 (13)	C19—C15—C16—C17	176.72 (12)
C1—P1—C5—C6	10.21 (14)	C15—C16—C17—C18	0.7 (2)
C14—P1—C5—C6	−117.62 (12)	C16—C17—C18—C13	0.5 (2)
C1—P1—C5—C10	−166.26 (10)	C14—C13—C18—C17	−1.2 (2)
C14—P1—C5—C10	65.91 (12)	C12—C13—C18—C17	178.38 (13)
C10—C5—C6—C7	0.5 (2)	C16—C15—C19—C24	90.26 (16)
P1—C5—C6—C7	−175.84 (11)	C14—C15—C19—C24	−92.04 (16)
C5—C6—C7—C8	0.8 (2)	C16—C15—C19—C20	−85.45 (16)
C6—C7—C8—C9	−0.9 (2)	C14—C15—C19—C20	92.25 (16)
C7—C8—C9—C10	−0.2 (2)	C26—O3—C20—C21	−5.6 (2)
C11—O1—C10—C9	1.2 (2)	C26—O3—C20—C19	175.38 (12)
C11—O1—C10—C5	−178.31 (12)	C24—C19—C20—O3	−178.00 (11)
C8—C9—C10—O1	−177.92 (13)	C15—C19—C20—O3	−2.15 (18)
C8—C9—C10—C5	1.5 (2)	C24—C19—C20—C21	2.9 (2)
C6—C5—C10—O1	177.84 (12)	C15—C19—C20—C21	178.79 (12)
P1—C5—C10—O1	−5.57 (16)	O3—C20—C21—C22	178.79 (12)
C6—C5—C10—C9	−1.7 (2)	C19—C20—C21—C22	−2.2 (2)
P1—C5—C10—C9	174.93 (10)	C20—C21—C22—C23	0.2 (2)
C18—C13—C14—C15	0.86 (19)	C21—C22—C23—C24	1.0 (2)
C12—C13—C14—C15	−178.75 (13)	C25—O2—C24—C23	−3.54 (19)
C18—C13—C14—P1	−177.10 (10)	C25—O2—C24—C19	176.32 (11)
C12—C13—C14—P1	3.30 (18)	C22—C23—C24—O2	179.57 (12)
C5—P1—C14—C15	55.82 (12)	C22—C23—C24—C19	−0.3 (2)
C1—P1—C14—C15	−71.50 (12)	C20—C19—C24—O2	178.49 (11)
C5—P1—C14—C13	−126.20 (11)	C15—C19—C24—O2	2.72 (18)
C1—P1—C14—C13	106.48 (11)	C20—C19—C24—C23	−1.65 (19)
C13—C14—C15—C16	0.27 (19)	C15—C19—C24—C23	−177.41 (12)
P1—C14—C15—C16	178.22 (10)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
P1—H1P $\cdots$ Cl1 ⁱ	1.313 (16)	2.523 (16)	3.5798 (5)	135.5 (10)
C21—H21 $\cdots$ O4	0.95	2.53	3.4594 (19)	167
C26—H26C $\cdots$ O4	0.98	2.53	3.2250 (19)	128
O4—H4X $\cdots$ Cl1 ⁱⁱ	0.93 (2)	2.24 (2)	3.1717 (13)	173 (2)
O4—H4Y $\cdots$ Cl1 ⁱⁱⁱ	0.94 (2)	2.25 (2)	3.1841 (13)	173 (2)

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