

# Crystal structure of bis[ $\mu$ -2-(diisopropylphosphoryl)propan-2-olato- $\kappa^3 O^1, O^2: O^1$ ]bis[chloridooxido-vanadium(IV)]

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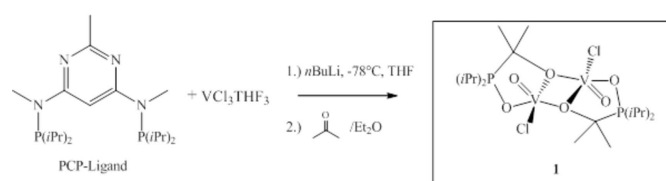
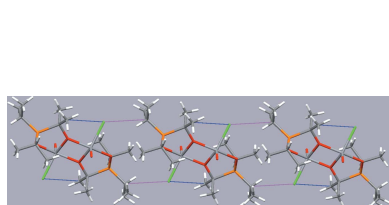
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**Keywords:** crystal structure; binuclear centrosymmetric complex; vanadium; square-pyramidal coordination geometry.**CCDC reference:** 1477727**Supporting information:** this article has supporting information at journals.iucr.org/e

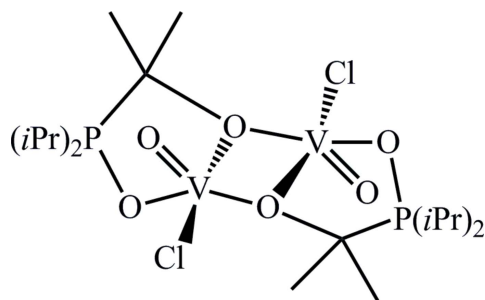
The dinuclear molecule of the title complex,  $[\text{VOCl}\{\mu\text{-OC}(\text{Me})_2\text{P}(\text{iPr})_2\text{-}\kappa^2\text{O}\}]_2$  or  $[\text{V}_2(\text{C}_9\text{H}_{20}\text{O}_2\text{P})_2\text{Cl}_2\text{O}_2]$ , which was obtained due to an unexpected oxidation reaction, is centrosymmetric, with the inversion centre located in the middle of the central  $\text{V}_2\text{O}_2$  core. These core O atoms arise from the symmetry-related 2-(diisopropylphosphoryl)propan-2-olate dianions. The  $\text{V}^{\text{IV}}$  atom is additionally bonded to one terminal Cl ligand, the second O atom of the dianion and double bonded to a vanadyl O atom, leading to an overall distorted square-pyramidal  $\text{VO}_4\text{Cl}$  coordination polyhedron with the vanadyl O atom as the apex. An intramolecular  $\text{C-H}\cdots\text{Cl}$  contact helps to establish the molecular configuration. In the crystal, molecules are stacked in rows parallel to  $[001]$  and are linked by  $\text{C-H}\cdots\text{Cl}$  contacts to form chains running in the same direction.

## 1. Chemical context

Tridentate pincer ligands play an important role in coordination chemistry and have found various applications, for example in the fields of catalysis, synthetic chemistry or molecular recognition (Szabo & Wendt, 2014). Whereas a plethora of second- and third-row transition metal complexes with pincer ligands of various types (*e.g.* PNP- or PCP-coordinating) has been reported in recent years, investigations with respect to first-row transition metals are scarce (Murugesan & Kirchner, 2016). During a current project to prepare the first vanadium pincer complexes (Mastalir *et al.*, 2016), we also attempted to synthesize a vanadium(III) PCP-complex according to the reaction scheme presented in Fig. 1. However, during the course of crystallization using a diffusion method in the presence of traces of water and/or oxygen, a variety of side-reactions took place. Those included oxidation of vanadium(III) to vanadium(IV) and of phosphorus, cleavage of the P–N bond and the formation of a P–C bond. As a result, the vanadium(IV) title complex  $[\text{VOCl}\{\mu\text{-OC}(\text{Me})_2\text{P}(\text{iPr})_2\text{-}\kappa^2\text{O}\}]_2$ , (1), was obtained instead. Its crystal structure is reported in this communication.



**Figure 1**  
Schematic representation of the attempted formation of a vanadium(III) complex with the PCP ligand.



## 2. Structural commentary

The dinuclear molecular complex of (1) is centrosymmetric, containing a rhombic  $V_2O_2$  core [V—O—V angle  $105.36(8)^\circ$ , O—V—O angle  $74.64(7)^\circ$ ]. The  $V^{IV}$  atom adopts a distorted square-pyramidal geometry with atoms O1, O2, O2<sup>i</sup> and Cl1 forming the basal plane and vanadyl atom O3 the apex [for symmetry operator (i), see Fig. 2]. The  $V^{IV}$  atom is displaced by  $0.6157(5)$  Å from the least-squares plane towards the apex. The Addison  $\tau$ -parameter (Addison *et al.*, 1984), as calculated by  $-0.01667 \cdot (139.45) + 0.01667 \cdot (148.82) = 0.156$ , also points to this coordination (a value of 0 refers to an ideal square-pyramidal, a value of 1 to an ideal trigonal-bipyramidal coordination). The V=O double-bond length of  $1.586(2)$  Å is in the typical range of those reported in similar dimeric oxido-chlorido-vanadium(IV) complexes containing alkoxide bridges (Cui *et al.*, 2010; Crans *et al.*, 1991; Foulon *et al.*, 1993; Janas *et al.*, 1997; Rosenthal, 2009).

## 3. Supramolecular features

In the crystal, the molecules are stacked into rows along [001] (Fig. 3). An intramolecular C—H...Cl contact [ $3.425(3)$  Å] involving one methyl H atom of the isopropyl moiety is present. A similar intermolecular contact [ $3.578(3)$  Å]

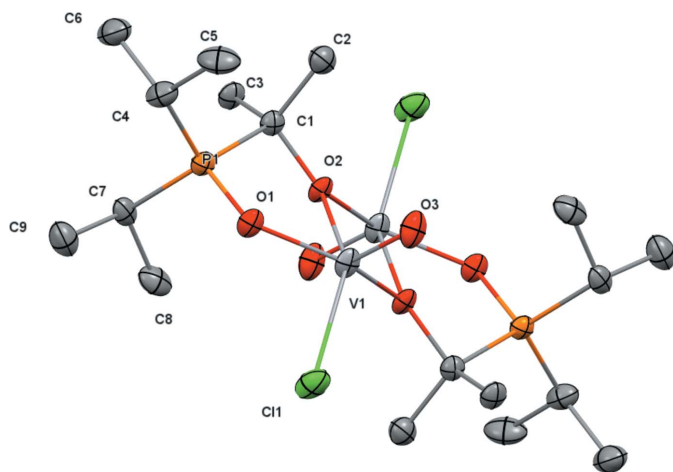


Figure 2

The molecular structure of the binuclear complex with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are generated by symmetry code  $(-x + 1, -y, -z + 1)$ .

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C3—H2C3...Cl1 <sup>i</sup>	0.96	2.68	3.425 (3)	135
C4—H1C4...Cl1 <sup>ii</sup>	0.96	2.77	3.578 (3)	142

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

between the Cl atom of one and the secondary H atom of the isopropyl moiety of an adjacent molecule leads to the formation of hydrogen-bonded chains along the stacking direction (Fig. 4). Numerical details of these interactions are given in Table 1.

## 4. Database survey

A search in the Cambridge Structural Database (Groom *et al.*, 2016) for structures of compounds containing the  $V_2O_2$  core and vanadium atoms additionally bonded to one Cl atom and double-bonded to one vanadyl O atom revealed 22 entries. In all these structures the coordination environment of the vanadium atoms is similar to that of the title structure.

## 5. Synthesis and crystallization

General. All manipulations were performed under an inert atmosphere of argon by using Schlenk techniques or in a MBraun inert-gas glovebox. The solvents were purified according to standard procedures.  $VCl_3(THF)_3$  was purchased from Sigma–Aldrich and used without further purification.

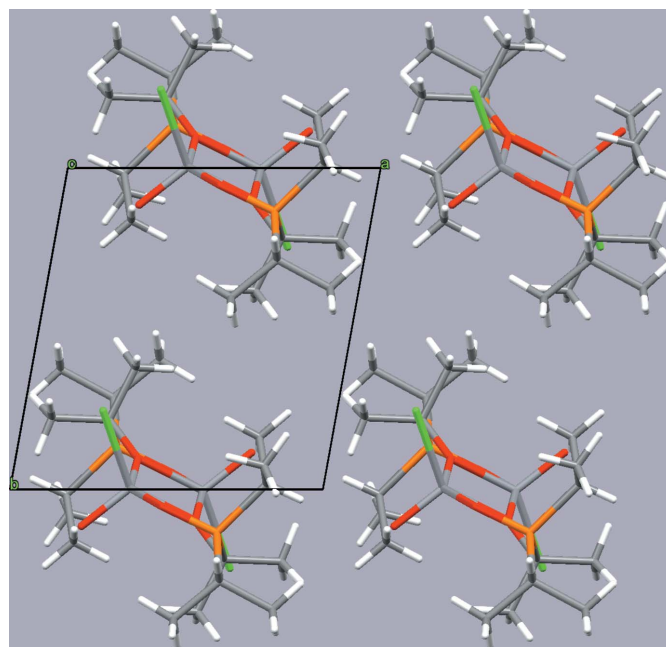


Figure 3

A projection of the crystal structure along [001] showing the stacking of molecules of (1) in this direction.

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$[\text{V}_2(\text{C}_9\text{H}_{20}\text{O}_2\text{P})_2\text{Cl}_2\text{O}_2]$
$M_r$	587.2
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
$a, b, c$ (Å)	8.0592 (17), 8.611 (2), 10.170 (2)
$\alpha, \beta, \gamma$ (°)	104.148 (7), 96.778 (6), 98.132 (6)
$V$ (Å <sup>3</sup> )	668.9 (3)
$Z$	1
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	1.05
Crystal size (mm)	0.38 × 0.18 × 0.01
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2015)
$T_{\min}$ , $T_{\max}$	0.80, 0.99
No. of measured, independent and observed [ $I > 3\sigma(I)$ ] reflections	13875, 3233, 2231
$R_{\text{int}}$	0.053
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.661
Refinement	
$R[F > 3\sigma(F)]$ , $wR(F)$ , $S$	0.040, 0.044, 1.53
No. of reflections	3233
No. of parameters	136
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.59, -0.31

Computer programs: APEX2 and SAINT-Plus (Bruker, 2015), SUPERFLIP (Palatinus & Chapuis, 2007), JANA2006 (Petříček *et al.*, 2014), Mercury (Macrae *et al.*, 2006) and publCIF (Westrip, 2010).

The synthesis of the PCP ligand employed was described in detail by Murugesan *et al.* (2014).

The oxido-vanadium complex (1) was formed during an attempt to synthesize a V<sup>III</sup> PCP complex (Fig. 1).  $\text{VCl}_3(\text{THF})_3$  (75 mg, 0.20 mmol) and the PCP ligand (85 mg, 0.22 mmol) were stirred in 7 ml THF for 30 min and cooled to 195 K. Upon addition of 0.22 mmol *n*-BuLi (2.5 M solution in *n*-hexane), the mixture was allowed to reach room temperature and was stirred for another two h. The colour changed from orange to violet. After evaporation of the solvent, the remaining solids were redissolved in 5 ml acetone and filtrated over celite. The clear violet solution was layered with 10 ml

diethyl ether and was left to stand for two days. Pale violet crystals, mostly with a needle-like form, suitable for X-ray analysis were isolated. IR spectrum (Perkin–Elmer 400 FIR FTIR spectrometer, equipped with a Pike Technologies GladiATR using a diamond crystal plate):  $\nu(\text{V}=\text{O})$  996 cm<sup>-1</sup> (for the full spectrum see Supporting information).

## 6. Refinement

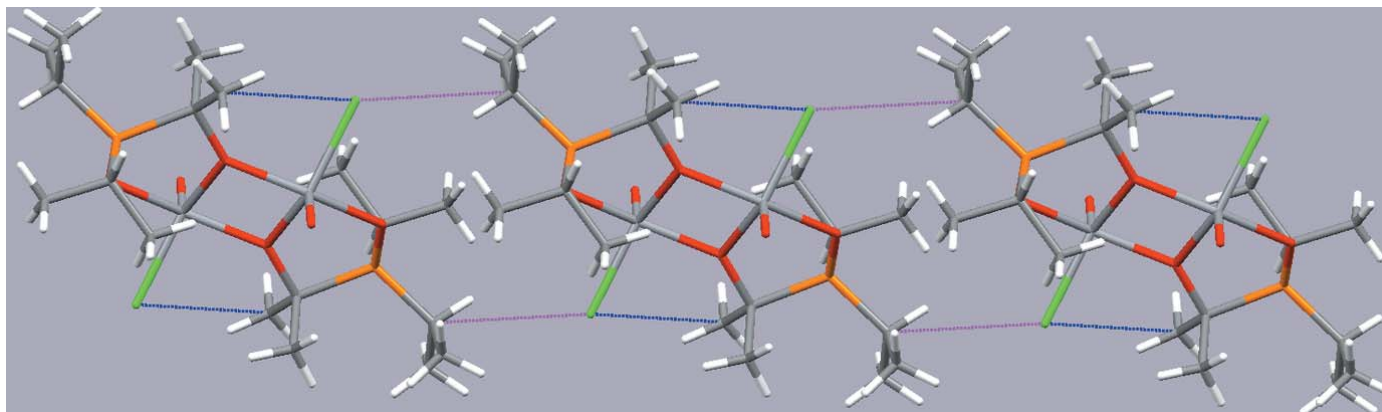
Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in calculated positions and were refined in the riding-atom approximation, with C–H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

## Acknowledgements

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**Figure 4**

A hydrogen-bonded chain of molecules extending parallel to [001]. Intramolecular C–H...Cl contacts are given as blue dotted lines and intermolecular C–H...Cl contacts as red dotted lines.

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## supporting information

*Acta Cryst.* (2016). E72, 785-788 [https://doi.org/10.1107/S2056989016007362]

## Crystal structure of bis[ $\mu$ -2-(diisopropylphosphoryl)propan-2-olato- $\kappa^3O^1, O^2:O^1$ ]bis[chloridooxidovanadium(IV)]

Mathias Glatz, Berthold Stöger, Matthias Weil and Karl Kirchner

### Computing details

Data collection: *APEX2* (Bruker, 2015); cell refinement: *SAINTE-Plus* (Bruker, 2015); data reduction: *SAINTE-Plus* (Bruker, 2015); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2014); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### Bis[ $\mu$ -2-(diisopropylphosphoryl)propan-2-olato- $\kappa^3O^1, O^2:O^1$ ]bis[chloridooxidovanadium(IV)]

#### Crystal data

$[V_2(C_9H_{20}O_2P)_2Cl_2O_2]$

$M_r = 587.2$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 8.0592$  (17) Å

$b = 8.611$  (2) Å

$c = 10.170$  (2) Å

$\alpha = 104.148$  (7)°

$\beta = 96.778$  (6)°

$\gamma = 98.132$  (6)°

$V = 668.9$  (3) Å<sup>3</sup>

$Z = 1$

$F(000) = 306$

$D_x = 1.458$  Mg m<sup>-3</sup>

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

Cell parameters from 4741 reflections

$\theta = 2.5$ – $25.5$ °

$\mu = 1.05$  mm<sup>-1</sup>

$T = 100$  K

Plate, translucent pale violet

$0.38 \times 0.18 \times 0.01$  mm

#### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: X-ray tube

Graphite monochromator

$\omega$ - and  $\phi$ -scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2015)

$T_{\min} = 0.80$ ,  $T_{\max} = 0.99$

13875 measured reflections

3233 independent reflections

2231 reflections with  $I > 3\sigma(I)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 28.0$ °,  $\theta_{\min} = 2.1$ °

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -13 \rightarrow 13$

#### Refinement

Refinement on  $F$

$R[F > 3\sigma(F)] = 0.040$

$wR(F) = 0.044$

$S = 1.53$

3233 reflections

136 parameters

0 restraints

80 constraints

H-atom parameters constrained

Weighting scheme based on measured s.u.'s  $w =$   
 $1/(\sigma^2(F) + 0.0001F^2)$

$(\Delta/\sigma)_{\max} = 0.009$

$\Delta\rho_{\max} = 0.59$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
V1	0.38304 (5)	-0.00427 (6)	0.36112 (5)	0.01926 (17)
Cl1	0.25262 (9)	-0.24490 (9)	0.20308 (7)	0.0341 (3)
P1	0.68528 (8)	0.13495 (8)	0.24674 (7)	0.0173 (2)
O1	0.49776 (19)	0.0539 (2)	0.21289 (17)	0.0192 (6)
O2	0.60943 (19)	0.0969 (2)	0.47621 (17)	0.0174 (6)
O3	0.2494 (2)	0.1146 (2)	0.38388 (19)	0.0281 (7)
C1	0.7236 (3)	0.2170 (3)	0.4368 (2)	0.0173 (9)
C2	0.6686 (3)	0.3802 (3)	0.4827 (3)	0.0256 (10)
C3	0.9074 (3)	0.2240 (3)	0.4969 (3)	0.0213 (9)
C4	0.7223 (3)	0.2850 (3)	0.1523 (3)	0.0260 (10)
C5	0.5959 (4)	0.4029 (3)	0.1606 (3)	0.0369 (12)
C6	0.9071 (4)	0.3734 (3)	0.1821 (3)	0.0342 (11)
C7	0.8172 (3)	-0.0168 (3)	0.1968 (3)	0.0215 (9)
C8	0.7671 (3)	-0.1627 (3)	0.2529 (3)	0.0289 (11)
C9	0.7981 (4)	-0.0735 (4)	0.0400 (3)	0.0337 (11)
H1c2	0.745608	0.462229	0.460631	0.0307*
H2c2	0.556075	0.374592	0.436426	0.0307*
H3c2	0.669143	0.407097	0.580109	0.0307*
H1c3	0.981032	0.288554	0.455184	0.0256*
H2c3	0.924596	0.272033	0.594195	0.0256*
H3c3	0.932653	0.115831	0.479036	0.0256*
H1c4	0.700955	0.222399	0.058035	0.0312*
H1c5	0.483119	0.343833	0.152296	0.0443*
H2c5	0.623875	0.483795	0.247252	0.0443*
H3c5	0.601381	0.454885	0.0874	0.0443*
H1c6	0.981535	0.294962	0.173736	0.041*
H2c6	0.924906	0.436542	0.117545	0.041*
H3c6	0.930691	0.444049	0.273627	0.041*
H1c7	0.933264	0.031353	0.234338	0.0257*
H1c8	0.771966	-0.126298	0.350545	0.0347*
H2c8	0.653646	-0.215951	0.210932	0.0347*
H3c8	0.844026	-0.237513	0.232403	0.0347*
H1c9	0.851769	0.012298	0.006097	0.0404*
H2c9	0.850708	-0.167368	0.014034	0.0404*
H3c9	0.679713	-0.1011	0.001481	0.0404*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
V1	0.0077 (2)	0.0305 (3)	0.0189 (2)	0.00292 (19)	-0.00264 (18)	0.0079 (2)
Cl1	0.0313 (4)	0.0409 (5)	0.0202 (4)	-0.0142 (3)	-0.0024 (3)	0.0041 (3)
P1	0.0116 (3)	0.0196 (4)	0.0192 (4)	0.0018 (3)	-0.0006 (3)	0.0044 (3)
O1	0.0098 (9)	0.0280 (11)	0.0191 (9)	0.0024 (8)	-0.0036 (7)	0.0082 (8)
O2	0.0074 (8)	0.0242 (10)	0.0193 (9)	-0.0004 (7)	-0.0018 (7)	0.0070 (8)
O3	0.0147 (10)	0.0439 (13)	0.0314 (11)	0.0116 (9)	0.0038 (8)	0.0169 (10)

C1	0.0133 (13)	0.0208 (15)	0.0166 (13)	0.0028 (11)	-0.0006 (10)	0.0045 (11)
C2	0.0190 (14)	0.0269 (16)	0.0287 (16)	0.0041 (12)	0.0021 (12)	0.0039 (13)
C3	0.0123 (13)	0.0274 (16)	0.0206 (14)	-0.0008 (11)	-0.0014 (11)	0.0040 (12)
C4	0.0270 (15)	0.0247 (16)	0.0244 (15)	-0.0028 (13)	0.0001 (12)	0.0093 (13)
C5	0.0474 (19)	0.0229 (17)	0.0393 (19)	0.0034 (15)	-0.0047 (16)	0.0134 (14)
C6	0.0380 (17)	0.0282 (17)	0.0328 (18)	-0.0055 (14)	0.0060 (15)	0.0078 (15)
C7	0.0118 (13)	0.0256 (16)	0.0228 (14)	0.0054 (11)	-0.0007 (11)	-0.0008 (12)
C8	0.0236 (15)	0.0246 (16)	0.0357 (17)	0.0110 (13)	-0.0030 (13)	0.0023 (14)
C9	0.0290 (17)	0.0390 (19)	0.0289 (16)	0.0106 (15)	0.0038 (14)	-0.0013 (14)

*Geometric parameters (Å, °)*

V1—C11	2.3105 (13)	C4—C5	1.532 (4)
V1—O1	1.986 (2)	C4—C6	1.533 (4)
V1—O2	2.0014 (17)	C4—H1c4	0.96
V1—O2 <sup>i</sup>	2.003 (2)	C5—H1c5	0.96
V1—O3	1.586 (2)	C5—H2c5	0.96
P1—O1	1.5333 (17)	C5—H3c5	0.96
P1—C1	1.861 (3)	C6—H1c6	0.96
P1—C4	1.802 (3)	C6—H2c6	0.96
P1—C7	1.814 (3)	C6—H3c6	0.96
O2—C1	1.448 (3)	C7—C8	1.525 (4)
C1—C2	1.514 (4)	C7—C9	1.532 (4)
C1—C3	1.522 (3)	C7—H1c7	0.96
C2—H1c2	0.96	C8—H1c8	0.96
C2—H2c2	0.96	C8—H2c8	0.96
C2—H3c2	0.96	C8—H3c8	0.96
C3—H1c3	0.96	C9—H1c9	0.96
C3—H2c3	0.96	C9—H2c9	0.96
C3—H3c3	0.96	C9—H3c9	0.96
C11—V1—O1	87.75 (5)	H2c3—C3—H3c3	109.47
C11—V1—O2	139.45 (6)	P1—C4—C5	114.9 (2)
C11—V1—O2 <sup>i</sup>	95.33 (5)	P1—C4—C6	112.9 (2)
C11—V1—O3	108.79 (6)	P1—C4—H1c4	103.96
O1—V1—O2	82.92 (7)	C5—C4—C6	112.4 (2)
O1—V1—O2 <sup>i</sup>	148.82 (7)	C5—C4—H1c4	104.59
O1—V1—O3	103.91 (10)	C6—C4—H1c4	107.07
O2—V1—O2 <sup>i</sup>	74.64 (7)	C4—C5—H1c5	109.47
O2—V1—O3	111.77 (8)	C4—C5—H2c5	109.47
O2 <sup>i</sup> —V1—O3	104.38 (9)	C4—C5—H3c5	109.47
O1—P1—C1	104.13 (10)	H1c5—C5—H2c5	109.47
O1—P1—C4	109.68 (11)	H1c5—C5—H3c5	109.47
O1—P1—C7	109.71 (10)	H2c5—C5—H3c5	109.47
C1—P1—C4	114.97 (12)	C4—C6—H1c6	109.47
C1—P1—C7	109.83 (12)	C4—C6—H2c6	109.47
C4—P1—C7	108.40 (13)	C4—C6—H3c6	109.47
V1—O1—P1	118.99 (10)	H1c6—C6—H2c6	109.47

V1—O2—V1 <sup>i</sup>	105.36 (8)	H1c6—C6—H3c6	109.47
V1—O2—C1	120.68 (14)	H2c6—C6—H3c6	109.47
V1 <sup>i</sup> —O2—C1	133.87 (13)	P1—C7—C8	110.47 (19)
P1—C1—O2	100.94 (13)	P1—C7—C9	110.0 (2)
P1—C1—C2	112.56 (19)	P1—C7—H1c7	108.43
P1—C1—C3	111.43 (17)	C8—C7—C9	109.4 (2)
O2—C1—C2	108.3 (2)	C8—C7—H1c7	109.02
O2—C1—C3	111.6 (2)	C9—C7—H1c7	109.51
C2—C1—C3	111.50 (18)	C7—C8—H1c8	109.47
C1—C2—H1c2	109.47	C7—C8—H2c8	109.47
C1—C2—H2c2	109.47	C7—C8—H3c8	109.47
C1—C2—H3c2	109.47	H1c8—C8—H2c8	109.47
H1c2—C2—H2c2	109.47	H1c8—C8—H3c8	109.47
H1c2—C2—H3c2	109.47	H2c8—C8—H3c8	109.47
H2c2—C2—H3c2	109.47	C7—C9—H1c9	109.47
C1—C3—H1c3	109.47	C7—C9—H2c9	109.47
C1—C3—H2c3	109.47	C7—C9—H3c9	109.47
C1—C3—H3c3	109.47	H1c9—C9—H2c9	109.47
H1c3—C3—H2c3	109.47	H1c9—C9—H3c9	109.47
H1c3—C3—H3c3	109.47	H2c9—C9—H3c9	109.47

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

#### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

<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>
C3—H2C3 $\cdots$ C11 <sup>i</sup>	0.96	2.68	3.425 (3)	135
C4—H1C4 $\cdots$ C11 <sup>ii</sup>	0.96	2.77	3.578 (3)	142

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