

Dichlorobis(triphenylphosphine oxide- $\kappa$ O)nickel(II)Rodolfo Moreno-Fuquen,<sup>a\*</sup> Olga Cifuentes,<sup>a</sup> Jaime Valderrama Naranjo,<sup>b</sup> Luis Manuel Serratto<sup>a</sup> and Alan R. Kennedy<sup>c</sup><sup>a</sup>Departamento de Química, Facultad de Ciencias, Universidad del Valle, Apartado 25360, Santiago de Cali, Colombia,<sup>b</sup>Departamento de Física, Facultad de Ciencias, Universidad del Valle, Apartado 25360, Santiago de Cali, Colombia, and <sup>c</sup>Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland

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## Key indicators

Single-crystal X-ray study

T = 123 K

Mean  $\sigma$ (C–C) = 0.003 Å

R factor = 0.026

wR factor = 0.063

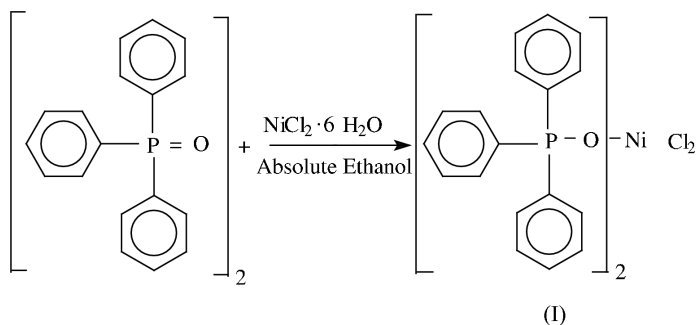
Data-to-parameter ratio = 18.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title nickel(II) complex,  $[\text{NiCl}_2(\text{C}_{18}\text{H}_{15}\text{OP})_2]$ , was prepared in order to determine the coordination of the Ni atom. The Ni atom lies on a twofold axis and is four-coordinated by two Cl atoms and two O atoms in a distorted tetrahedral coordination geometry. The Ni–Cl and Ni–O distances are 2.2151 (6) and 1.9662 (16) Å, respectively. The title compound is compared with the four-coordinated copper(II) analog; the two systems are isostructural.

## Comment

In the period 1950–1960, there was a suggestion, based on the valence-bond theory, that paramagnetic nickel(II) complexes should be planar (Ballhausen & Liehr, 1959). This suggestion was difficult to verify due to the rather rare occurrence of tetrahedrally coordinated nickel(II) ions. Later, during the 1960s, some  $[(\text{C}_6\text{H}_5)_3\text{PO}]_2\text{NiX}_2$  complexes ( $X = \text{Cl}, \text{Br}, \text{I}$ ) were prepared (Cotton & Goodgame, 1960). Their electronic spectra, IR spectra and magnetic moments were recorded and analyzed in order to demonstrate that the nickel ion, in each case, was tetrahedrally coordinated (Cotton & Goodgame, 1960). Unfortunately, the purity of the  $[(\text{C}_6\text{H}_5)_3\text{PO}]_2\text{NiCl}_2$  complex system was questioned and doubt of a tetrahedral array persisted. In order to elucidate the coordination of the nickel(II) ion in the  $[(\text{C}_6\text{H}_5)_3\text{PO}]_2\text{NiCl}_2$  system, its crystal structure determination was undertaken.



A perspective view of the title molecule, (I), showing the atomic numbering scheme is given in Fig. 1. The  $\text{Ni}^{\text{II}}$  ion has  $C_2$  site symmetry;  $C_2$  is parallel to the  $c$  axis. The analogous dichlorobis(triphenylphosphine oxide)copper(II) system, (II) (Bertrand & Kalyanaraman, 1971), was taken as a reference and its internal parameters were compared to the parameters of the title complex. Within the tetrahedral geometry around the central Ni atom, complex (II) shows Cu–Cl and Cu–O bond lengths of 2.170 (2) and 1.958 (4) Å, respectively, while (I) shows Ni–Cl and Ni–O bond lengths of 2.2151 (6) and 1.9662 (16) Å, respectively. The Cl–Cu–Cl and O–Cu–O

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bond angles in (II) are 102.2 (1) and 93.0 (1)°, while in (I) the Cl–Ni–Cl and O–Ni–O bond angles are 116.17 (3) and 95.91 (10)°, respectively. The two systems are isostructural. The triphenylphosphine portion of the complex does not show any unusual features and the other bond lengths and angles of these systems are within expected ranges.

## Experimental

Reagents and solvents for the synthesis were purchased from the Aldrich Chemical Co. and were used without additional purification. Crystals of (I) were obtained following the synthesis previously reported by Cotton & Goodgame (1960). The product of this synthesis was recrystallized by slow evaporation of an acetonitrile solution at room temperature, in contact with oxygen. The pale-blue crystals had a melting point of 463 (1) K.

### Crystal data

[NiCl <sub>2</sub> (C <sub>18</sub> H <sub>15</sub> OP) <sub>2</sub> ]	Mo K $\alpha$ radiation
$M_r = 686.15$	Cell parameters from 3709 reflections
Orthorhombic, <i>Fdd2</i>	$\theta = 1.0\text{--}27.5^\circ$
$a = 20.6356$ (3) Å	$\mu = 0.89$ mm <sup>-1</sup>
$b = 32.5388$ (6) Å	$T = 123$ (2) K
$c = 9.7240$ (1) Å	Prism, pale blue
$V = 6529.25$ (17) Å <sup>3</sup>	$0.35 \times 0.30 \times 0.15$ mm
$Z = 8$	
$D_x = 1.396$ Mg m <sup>-3</sup>	

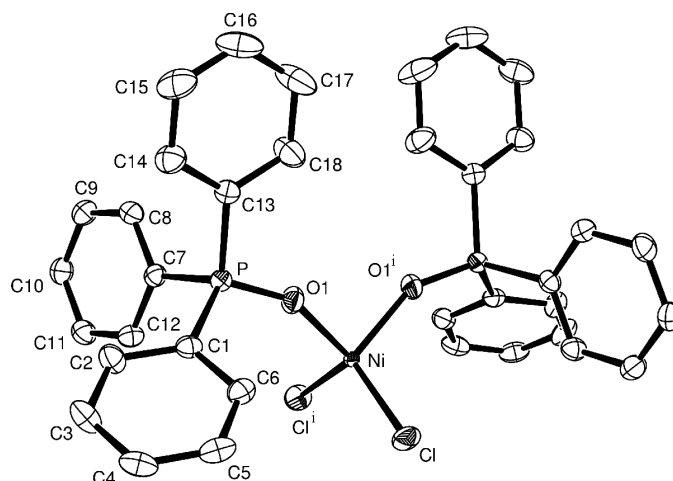
### Data collection

Nonius KappaCCD diffractometer	3686 independent reflections
$\omega$ scans	3468 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan ( <i>DENZO</i> ; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.021$
$T_{\text{min}} = 0.802$ , $T_{\text{max}} = 0.868$	$\theta_{\text{max}} = 27.5^\circ$
7169 measured reflections	$h = -26 \rightarrow 26$
	$k = -42 \rightarrow 42$
	$l = -12 \rightarrow 12$

### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.026$	$\Delta\rho_{\text{max}} = 0.45$ e Å <sup>-3</sup>
$wR(F^2) = 0.063$	$\Delta\rho_{\text{min}} = -0.29$ e Å <sup>-3</sup>
$S = 1.07$	Extinction correction: <i>SHELXL97</i>
3686 reflections	Extinction coefficient: 0.00057 (6)
195 parameters	Absolute structure: Flack (1983), 1705 Friedel pairs
H-atom parameters constrained	Flack parameter = 0.001 (1)
$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 7.7683P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

All H atoms were placed at geometrically idealized positions and were treated as riding atoms, with C–H = 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

An ORTEP-3 plot (Farrugia, 1997) of the title compound, with the atomic labeling scheme. Displacement ellipsoids are plotted at the 50% probability level. H atoms are shown as spheres of arbitrary radii. [Symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, z$ .]

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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## supporting information

*Acta Cryst.* (2004). E60, m1861–m1862 [https://doi.org/10.1107/S1600536804029125]

Dichlorobis(triphenylphosphine oxide- $\kappa$ O)nickel(II)

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Dichlorobis(triphenylphosphine oxide- $\kappa$ O)nickel(II)*Crystal data*

[NiCl<sub>2</sub>(C<sub>18</sub>H<sub>15</sub>OP)<sub>2</sub>]

$M_r = 686.15$

Orthorhombic, *Fdd2*

Hall symbol: F 2 -2d

$a = 20.6356$  (3) Å

$b = 32.5388$  (6) Å

$c = 9.7240$  (1) Å

$V = 6529.25$  (17) Å<sup>3</sup>

$Z = 8$

$F(000) = 2832$

$D_x = 1.396$  Mg m<sup>-3</sup>

Melting point: 463(1) K

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

Cell parameters from 3709 reflections

$\theta = 1.0$ – $27.5^\circ$

$\mu = 0.89$  mm<sup>-1</sup>

$T = 123$  K

Prism, pale blue

$0.35 \times 0.30 \times 0.15$  mm

*Data collection*

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(DENZO; Otwinowski & Minor, 1997)

$T_{\min} = 0.802$ ,  $T_{\max} = 0.868$

7169 measured reflections

3686 independent reflections

3468 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.3^\circ$

$h = -26 \rightarrow 26$

$k = -42 \rightarrow 42$

$l = -12 \rightarrow 12$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.063$

$S = 1.07$

3686 reflections

195 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 7.7683P]$

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

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

$\Delta\rho_{\max} = 0.45$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.29$  e Å<sup>-3</sup>

Extinction correction: SHELXL97

Extinction coefficient: 0.00057 (6)

Absolute structure: Flack (1983)

Absolute structure parameter: 0.001 (1)

*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni	0.2500	0.2500	0.0000	0.01108 (9)
P	0.16831 (3)	0.178349 (16)	0.18060 (7)	0.01842 (12)
Cl	0.32802 (3)	0.22015 (2)	-0.12042 (7)	0.03064 (14)
O1	0.21631 (7)	0.21054 (5)	0.13540 (17)	0.0254 (3)
C1	0.17605 (10)	0.13228 (7)	0.0797 (2)	0.0212 (4)
C2	0.12395 (11)	0.10513 (7)	0.0611 (3)	0.0269 (5)
H2	0.0837	0.1103	0.1052	0.032*
C3	0.13114 (12)	0.07063 (7)	-0.0219 (3)	0.0316 (5)
H3	0.0959	0.0522	-0.0342	0.038*
C4	0.18974 (12)	0.06317 (7)	-0.0865 (2)	0.0298 (5)
H4	0.1944	0.0399	-0.1447	0.036*
C5	0.24159 (12)	0.08943 (8)	-0.0666 (2)	0.0293 (5)
H5	0.2819	0.0838	-0.1097	0.035*
C6	0.23510 (10)	0.12386 (7)	0.0153 (2)	0.0247 (5)
H6	0.2709	0.1418	0.0279	0.030*
C7	0.08511 (10)	0.19434 (6)	0.1652 (2)	0.0199 (4)
C8	0.04882 (11)	0.20515 (7)	0.2806 (2)	0.0246 (5)
H8	0.0677	0.2040	0.3696	0.030*
C9	-0.01545 (10)	0.21768 (7)	0.2646 (3)	0.0290 (5)
H9	-0.0402	0.2251	0.3431	0.035*
C10	-0.04314 (10)	0.21945 (7)	0.1362 (3)	0.0269 (5)
H10	-0.0870	0.2278	0.1267	0.032*
C11	-0.00720 (10)	0.20909 (7)	0.0201 (2)	0.0262 (5)
H11	-0.0264	0.2105	-0.0685	0.031*
C12	0.05715 (11)	0.19661 (7)	0.0343 (2)	0.0239 (5)
H12	0.0819	0.1897	-0.0447	0.029*
C13	0.18248 (10)	0.16712 (7)	0.3592 (2)	0.0221 (4)
C14	0.15370 (12)	0.13430 (7)	0.4272 (3)	0.0304 (5)
H14	0.1273	0.1154	0.3780	0.036*
C15	0.16364 (14)	0.12914 (9)	0.5678 (3)	0.0394 (6)
H15	0.1441	0.1066	0.6142	0.047*
C16	0.20120 (16)	0.15620 (9)	0.6391 (3)	0.0447 (7)
H16	0.2068	0.1528	0.7355	0.054*
C17	0.23141 (18)	0.18867 (9)	0.5724 (3)	0.0477 (8)
H17	0.2582	0.2071	0.6226	0.057*
C18	0.22245 (14)	0.19416 (8)	0.4323 (3)	0.0361 (6)
H18	0.2434	0.2162	0.3861	0.043*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ni	0.00926 (14)	0.01456 (16)	0.00942 (15)	0.00033 (14)	0.000	0.000
P	0.0187 (2)	0.0189 (3)	0.0177 (3)	-0.0013 (2)	0.0003 (2)	0.0005 (2)
Cl	0.0214 (2)	0.0456 (3)	0.0249 (3)	0.0083 (2)	0.0051 (2)	-0.0055 (2)
O1	0.0229 (7)	0.0261 (8)	0.0272 (8)	-0.0050 (7)	0.0003 (7)	0.0044 (7)
C1	0.0232 (10)	0.0235 (10)	0.0168 (10)	0.0023 (9)	-0.0013 (8)	0.0015 (9)
C2	0.0220 (10)	0.0282 (12)	0.0306 (12)	0.0009 (9)	-0.0037 (9)	-0.0056 (10)
C3	0.0309 (11)	0.0286 (12)	0.0354 (14)	0.0012 (10)	-0.0123 (10)	-0.0086 (11)
C4	0.0425 (13)	0.0263 (12)	0.0205 (12)	0.0100 (10)	-0.0046 (10)	-0.0052 (9)
C5	0.0361 (12)	0.0308 (13)	0.0210 (11)	0.0102 (10)	0.0065 (10)	0.0032 (10)
C6	0.0248 (11)	0.0272 (11)	0.0221 (12)	0.0016 (8)	0.0016 (10)	0.0029 (10)
C7	0.0212 (10)	0.0175 (10)	0.0210 (11)	-0.0013 (8)	0.0022 (8)	-0.0001 (8)
C8	0.0240 (11)	0.0290 (12)	0.0208 (12)	-0.0011 (9)	-0.0021 (8)	-0.0011 (9)
C9	0.0219 (11)	0.0363 (13)	0.0286 (13)	0.0001 (9)	0.0065 (10)	-0.0039 (11)
C10	0.0200 (10)	0.0294 (12)	0.0312 (12)	0.0003 (9)	0.0004 (9)	0.0004 (10)
C11	0.0254 (11)	0.0287 (12)	0.0245 (12)	-0.0003 (9)	-0.0047 (9)	0.0001 (9)
C12	0.0254 (11)	0.0253 (11)	0.0209 (11)	0.0024 (9)	0.0026 (8)	-0.0016 (9)
C13	0.0231 (10)	0.0225 (11)	0.0205 (11)	0.0006 (9)	-0.0008 (9)	0.0000 (8)
C14	0.0372 (13)	0.0260 (12)	0.0280 (12)	-0.0017 (10)	0.0016 (11)	0.0033 (10)
C15	0.0572 (18)	0.0311 (14)	0.0298 (14)	0.0028 (12)	0.0059 (13)	0.0122 (11)
C16	0.0708 (19)	0.0422 (16)	0.0212 (12)	0.0152 (14)	-0.0080 (14)	0.0024 (12)
C17	0.069 (2)	0.0421 (17)	0.0315 (15)	-0.0025 (15)	-0.0210 (15)	-0.0056 (13)
C18	0.0475 (15)	0.0296 (13)	0.0311 (13)	-0.0093 (12)	-0.0115 (12)	0.0016 (11)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Ni—O1 <sup>i</sup>	1.9661 (15)	C8—C9	1.396 (3)
Ni—O1	1.9661 (15)	C8—H8	0.9500
Ni—Cl <sup>i</sup>	2.2152 (5)	C9—C10	1.374 (4)
Ni—Cl	2.2152 (5)	C9—H9	0.9500
P—O1	1.5072 (15)	C10—C11	1.392 (3)
P—C13	1.799 (2)	C10—H10	0.9500
P—C1	1.799 (2)	C11—C12	1.396 (3)
P—C7	1.800 (2)	C11—H11	0.9500
C1—C6	1.397 (3)	C12—H12	0.9500
C1—C2	1.403 (3)	C13—C14	1.389 (3)
C2—C3	1.390 (3)	C13—C18	1.400 (3)
C2—H2	0.9500	C14—C15	1.392 (4)
C3—C4	1.384 (4)	C14—H14	0.9500
C3—H3	0.9500	C15—C16	1.363 (4)
C4—C5	1.383 (4)	C15—H15	0.9500
C4—H4	0.9500	C16—C17	1.388 (4)
C5—C6	1.381 (3)	C16—H16	0.9500
C5—H5	0.9500	C17—C18	1.386 (4)
C6—H6	0.9500	C17—H17	0.9500
C7—C8	1.394 (3)	C18—H18	0.9500

C7—C12	1.399 (3)		
O1 <sup>i</sup> —Ni—O1	95.92 (10)	C7—C8—C9	119.6 (2)
O1 <sup>i</sup> —Ni—Cl <sup>i</sup>	108.95 (5)	C7—C8—H8	120.2
O1—Ni—Cl <sup>i</sup>	112.54 (5)	C9—C8—H8	120.2
O1 <sup>i</sup> —Ni—Cl	112.54 (5)	C10—C9—C8	120.6 (2)
O1—Ni—Cl	108.95 (5)	C10—C9—H9	119.7
Cl <sup>i</sup> —Ni—Cl	116.17 (3)	C8—C9—H9	119.7
O1—P—C13	108.42 (10)	C9—C10—C11	120.3 (2)
O1—P—C1	111.20 (10)	C9—C10—H10	119.8
C13—P—C1	110.06 (10)	C11—C10—H10	119.8
O1—P—C7	113.68 (9)	C10—C11—C12	119.8 (2)
C13—P—C7	107.11 (10)	C10—C11—H11	120.1
C1—P—C7	106.27 (10)	C12—C11—H11	120.1
P—O1—Ni	151.84 (10)	C11—C12—C7	119.9 (2)
C6—C1—C2	119.1 (2)	C11—C12—H12	120.1
C6—C1—P	119.05 (17)	C7—C12—H12	120.1
C2—C1—P	121.80 (17)	C14—C13—C18	119.6 (2)
C3—C2—C1	120.1 (2)	C14—C13—P	123.11 (18)
C3—C2—H2	120.0	C18—C13—P	117.28 (18)
C1—C2—H2	120.0	C13—C14—C15	119.8 (2)
C4—C3—C2	119.9 (2)	C13—C14—H14	120.1
C4—C3—H3	120.1	C15—C14—H14	120.1
C2—C3—H3	120.1	C16—C15—C14	120.4 (3)
C5—C4—C3	120.3 (2)	C16—C15—H15	119.8
C5—C4—H4	119.9	C14—C15—H15	119.8
C3—C4—H4	119.9	C15—C16—C17	120.6 (3)
C6—C5—C4	120.5 (2)	C15—C16—H16	119.7
C6—C5—H5	119.8	C17—C16—H16	119.7
C4—C5—H5	119.8	C18—C17—C16	119.8 (3)
C5—C6—C1	120.1 (2)	C18—C17—H17	120.1
C5—C6—H6	119.9	C16—C17—H17	120.1
C1—C6—H6	119.9	C17—C18—C13	119.8 (3)
C8—C7—C12	119.81 (19)	C17—C18—H18	120.1
C8—C7—P	121.21 (16)	C13—C18—H18	120.1
C12—C7—P	118.96 (16)		
C13—P—O1—Ni	-171.8 (2)	C13—P—C7—C12	-166.62 (17)
C1—P—O1—Ni	67.1 (2)	C1—P—C7—C12	-49.0 (2)
C7—P—O1—Ni	-52.8 (2)	C12—C7—C8—C9	0.8 (3)
O1 <sup>i</sup> —Ni—O1—P	148.4 (3)	P—C7—C8—C9	179.55 (17)
Cl <sup>i</sup> —Ni—O1—P	35.1 (2)	C7—C8—C9—C10	0.0 (4)
Cl—Ni—O1—P	-95.3 (2)	C8—C9—C10—C11	-0.6 (4)
O1—P—C1—C6	24.3 (2)	C9—C10—C11—C12	0.4 (4)
C13—P—C1—C6	-95.91 (19)	C10—C11—C12—C7	0.4 (3)
C7—P—C1—C6	148.45 (18)	C8—C7—C12—C11	-1.0 (3)
O1—P—C1—C2	-154.12 (18)	P—C7—C12—C11	-179.76 (17)
C13—P—C1—C2	85.7 (2)	O1—P—C13—C14	-169.46 (19)

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C7—P—C1—C2	-29.9 (2)	C1—P—C13—C14	-47.6 (2)
C6—C1—C2—C3	-0.8 (3)	C7—P—C13—C14	67.5 (2)
P—C1—C2—C3	177.60 (18)	O1—P—C13—C18	13.3 (2)
C1—C2—C3—C4	-0.2 (3)	C1—P—C13—C18	135.08 (19)
C2—C3—C4—C5	1.3 (4)	C7—P—C13—C18	-109.8 (2)
C3—C4—C5—C6	-1.5 (4)	C18—C13—C14—C15	1.5 (4)
C4—C5—C6—C1	0.4 (3)	P—C13—C14—C15	-175.8 (2)
C2—C1—C6—C5	0.7 (3)	C13—C14—C15—C16	0.3 (4)
P—C1—C6—C5	-177.74 (18)	C14—C15—C16—C17	-1.6 (5)
O1—P—C7—C8	-105.11 (19)	C15—C16—C17—C18	1.2 (5)
C13—P—C7—C8	14.6 (2)	C16—C17—C18—C13	0.6 (5)
C1—P—C7—C8	132.25 (18)	C14—C13—C18—C17	-1.9 (4)
O1—P—C7—C12	73.65 (19)	P—C13—C18—C17	175.5 (3)

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Symmetry code: (i)  $-x+1/2, -y+1/2, z$ .