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# O-4-Chlorobenzoyl diphenylselenophosphate

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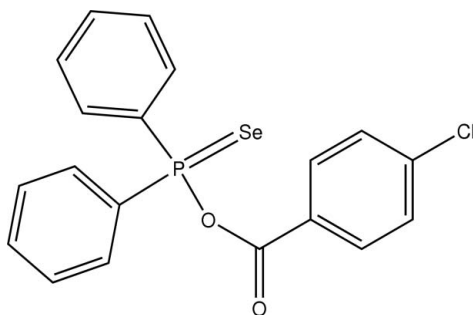
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 Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.162; data-to-parameter ratio = 19.2.

The title compound,  $\text{C}_{19}\text{H}_{14}\text{ClO}_2\text{PSe}$ , was obtained in the reaction of the diphenylmonoselenophosphinic acid ammonium salt with 4-chlorobenzoyl chloride. The dihedral angle between the P-bonded aromatic rings is  $72.64(14)^\circ$ . Packing of the molecules in the crystal is reinforced by  $\pi$ - $\pi$  stacking interactions between two inversion-related 4-chlorobenzene rings [centroid-centroid separation =  $4.189(2)$  Å] and a C-H...O interaction also occurs.

## Related literature

Syntheses of *O*-acyl monoselenophosphates have already been described by Rachon *et al.* (2005); Mielniczak & Łopusinski (2001). For a related *O*-acyl derivative, see Cholewinski *et al.* (2009). For related *O*-alkyl or *O*-aryl derivatives, see: Lepicard *et al.* (1969); Balakrishna *et al.* (2002, 2005); Mague *et al.* (2007).



## Experimental

### Crystal data

 $\text{C}_{19}\text{H}_{14}\text{ClO}_2\text{PSe}$ 
 $M_r = 419.68$ 

 Monoclinic,  $P2_1/c$   
 $a = 9.3390(5)$  Å  
 $b = 9.7132(5)$  Å  
 $c = 19.1353(15)$  Å  
 $\beta = 97.059(6)^\circ$   
 $V = 1722.64(19)$  Å<sup>3</sup>
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 2.44$  mm<sup>-1</sup>  
 $T = 120$  K  
 $0.46 \times 0.33 \times 0.26$  mm

### Data collection

 Oxford Diffraction KM-4-CCD diffractometer  
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)  
 $T_{\min} = 0.325$ ,  $T_{\max} = 0.53$ 

 14128 measured reflections  
 4158 independent reflections  
 3376 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.045$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.162$   
 $S = 1.16$   
 4158 reflections

 217 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.82$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.56$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> -H... <i>A</i>	<i>D</i> -H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> -H... <i>A</i>
C16-H16...O2 <sup>i</sup>	0.95	2.59	3.359 (6)	138

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZZ164).

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## supporting information

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## O-4-Chlorobenzoyl diphenylselenophosphinate

Grzegorz Cholewinski, Jaroslaw Chojnacki, Jerzy Pikies and Janusz Rachon

### S1. Comment

*O*-acyl monoselenophosphates were studied as part of a search for potential selenoacylating agents. *O*-4-chlorobenzoyl-diphenylmonoselenophosphinate was obtained in the reaction of diphenylmonoselenophosphinic acid ammonium salt with 4-chlorobenzoyl chloride (Rachon *et al.* 2005).

The title compound, C<sub>19</sub>H<sub>14</sub>ClO<sub>2</sub>PSe, together with *O*-pivaloyl-diphenylselenophosphinate, reported in our preceding paper (Cholewinski *et al.*, 2009) are the first reported X-ray diffraction structures of *O*-acyl derivatives of diphenylmonoselenophosphinic acid. Several other monoselenophosphinates, such as *O*-alkyl esters have been characterized by X-ray diffraction (Lepicard *et al.*, 1969; Balakrishna *et al.*, 2005; Balakrishna *et al.*, 2002; Mague *et al.*, 2007).

The P–Se bond length is in the usual range, while the P–O bond is longer than in *O*-aryl or *O*-alkyl derivatives, but very close to the value for the other *O*-acyl derivative, reported in our preceding paper (Cholewinski *et al.*, 2009). Unlike the pivaloyl analogue, no phenyl ring is close to being synplanar with the Se–P–C plane. The smallest Se–P–C–C torsion angle is relatively large at 25.7 (4)°. Noteworthy is that the carboxyl group is twisted in relation to the adjacent phenyl ring, forming a dihedral angle between the C1–C6 phenyl ring and the plane defined by O1/C7/O2 of 11.6 (7)°.

Packing of *O*-4-chlorobenzoyl-diphenylmonoselenophosphinate molecules in the crystal is reinforced by  $\pi$ – $\pi$  stacking interactions between two adjacent 4-chlorobenzene rings residing close to a symmetry centre. The centroid to centroid distance is 4.189 (2) Å, the dihedral angle between the planes is  $\alpha=0.0^\circ$  and the angle between the vector span on the centroids and the vector normal to the ring is  $\beta=35.08^\circ$ . No classical hydrogen bonds are present, however a weak C–H $\cdots$ O interaction link molecules into chains along the *a*-axis.

### S2. Experimental

*O*-4-chlorobenzoyl-diphenylmonoselenophosphinate was obtained in the reaction of diphenylmonoselenophosphinic acid ammonium salt (Mielniczak *et al.*, 2001) with 4-chlorobenzoyl chloride in 46% yield according to Rachon *et al.* (2005). The compound which was encoded in this paper as **2t**, melts at 105–107 °C. Relevant <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P NMR, MS and IR spectra were recorded and are consistent with the formula anticipated - see the supporting information for the article cited.

### S3. Refinement

Hydrogen atoms were placed in calculated positions and refined using a standard riding model. C–H bond lengths were set to 0.95 Å and  $U_{iso}(H)$  were set to 1.2 $U_{eq}(C)$  for aromatic C–H groups, respectively. The residual electron-density peak is 0.95 Å from Se1 and the deepest electron-density hole is 1.49 Å from Se1.

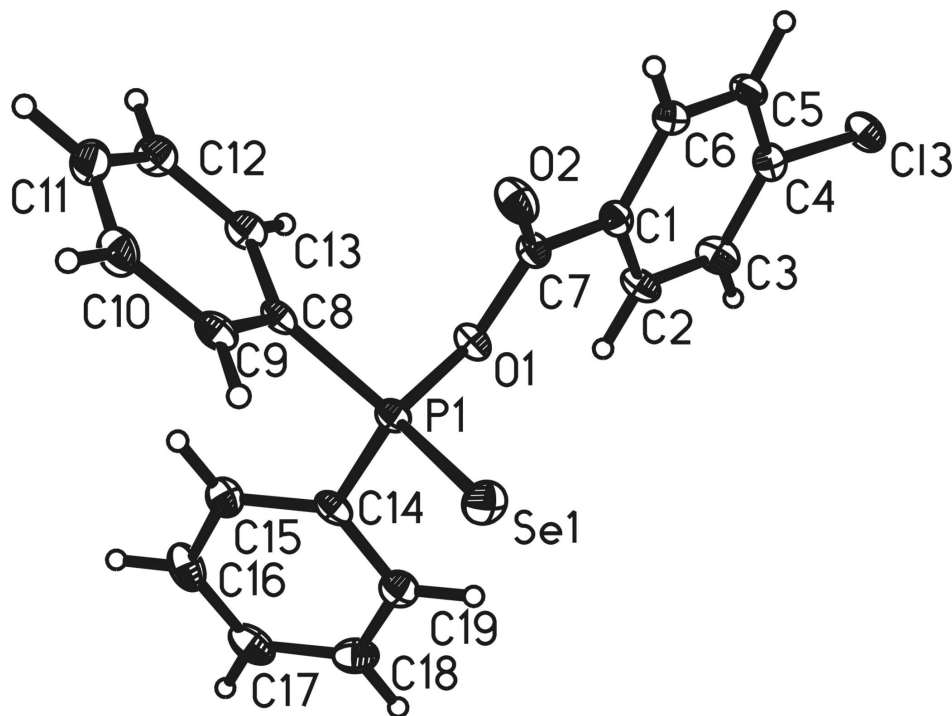


Figure 1

View of the title compound showing the atom-numbering scheme (50% probability displacement ellipsoids).

### O-4-Chlorobenzoyl diphenylselenophosphinate

#### Crystal data

$C_{19}H_{14}ClO_2PSe$   
 $M_r = 419.68$   
 Monoclinic,  $P2_1/c$   
 Hall symbol:  $-P\ 2_1/c$   
 $a = 9.3390$  (5) Å  
 $b = 9.7132$  (5) Å  
 $c = 19.1353$  (15) Å  
 $\beta = 97.059$  (6)°  
 $V = 1722.64$  (19) Å<sup>3</sup>  
 $Z = 4$

$F(000) = 840$   
 $D_x = 1.618$  Mg m<sup>-3</sup>  
 Melting point: 379(2) K  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
 Cell parameters from 9084 reflections  
 $\theta = 2.1$ – $32.4$ °  
 $\mu = 2.44$  mm<sup>-1</sup>  
 $T = 120$  K  
 Prism, colourless  
 $0.46 \times 0.33 \times 0.26$  mm

#### Data collection

Oxford Diffraction KM-4-CCD  
 diffractometer  
 Radiation source: enhance  
 Graphite monochromator  
 $\omega$  scans (0.75° width)  
 Absorption correction: multi-scan  
 (*CrysAlis RED*; Oxford Diffraction, 2008)  
 $T_{\min} = 0.325$ ,  $T_{\max} = 0.53$

14128 measured reflections  
 4158 independent reflections  
 3376 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.045$   
 $\theta_{\max} = 28^\circ$ ,  $\theta_{\min} = 2.1^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -12 \rightarrow 12$   
 $l = -21 \rightarrow 25$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.162$   
 $S = 1.16$   
 4158 reflections  
 217 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 5.5587P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.82 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.28670 (5)	1.03290 (5)	0.35818 (2)	0.02233 (16)
Cl3	0.57203 (12)	0.18536 (11)	0.48614 (6)	0.0244 (2)
P1	0.15102 (11)	0.87791 (11)	0.31430 (5)	0.0146 (2)
O1	0.2030 (3)	0.7199 (3)	0.33713 (15)	0.0179 (6)
O2	0.3944 (3)	0.7246 (4)	0.27566 (17)	0.0242 (7)
C1	0.3808 (4)	0.5449 (4)	0.3593 (2)	0.0167 (8)
C2	0.3182 (5)	0.5004 (5)	0.4180 (2)	0.0206 (9)
H2	0.2362	0.5467	0.4314	0.025*
C3	0.3764 (5)	0.3881 (5)	0.4567 (2)	0.0232 (9)
H3	0.3361	0.3584	0.4974	0.028*
C4	0.4933 (5)	0.3202 (4)	0.4353 (2)	0.0186 (8)
C5	0.5533 (5)	0.3590 (5)	0.3756 (2)	0.0197 (8)
H5	0.631	0.3083	0.3606	0.024*
C6	0.4977 (5)	0.4734 (5)	0.3380 (2)	0.0196 (8)
H6	0.5394	0.5031	0.2977	0.023*
C7	0.3321 (4)	0.6701 (5)	0.3192 (2)	0.0177 (8)
C8	0.1201 (4)	0.8755 (4)	0.2195 (2)	0.0136 (7)
C9	0.1340 (5)	0.9975 (4)	0.1825 (2)	0.0192 (8)
H9	0.1618	1.0799	0.2072	0.023*
C10	0.1069 (5)	0.9982 (5)	0.1093 (2)	0.0239 (9)
H10	0.1154	1.0814	0.0841	0.029*
C11	0.0675 (5)	0.8778 (5)	0.0733 (2)	0.0235 (9)
H11	0.0511	0.878	0.0233	0.028*
C12	0.0521 (5)	0.7567 (5)	0.1102 (2)	0.0214 (9)
H12	0.0237	0.6745	0.0852	0.026*
C13	0.0776 (4)	0.7549 (4)	0.1828 (2)	0.0186 (8)
H13	0.0663	0.6718	0.2078	0.022*
C14	-0.0242 (4)	0.8745 (4)	0.3454 (2)	0.0156 (8)

C15	-0.1503 (5)	0.8706 (5)	0.2982 (2)	0.0206 (9)
H15	-0.1454	0.8667	0.2489	0.025*
C16	-0.2835 (5)	0.8724 (5)	0.3239 (3)	0.0261 (10)
H16	-0.37	0.8687	0.2922	0.031*
C17	-0.2900 (5)	0.8795 (5)	0.3957 (3)	0.0259 (10)
H17	-0.3812	0.882	0.4128	0.031*
C18	-0.1647 (5)	0.8830 (5)	0.4427 (2)	0.0236 (9)
H18	-0.17	0.8872	0.4919	0.028*
C19	-0.0315 (5)	0.8803 (5)	0.4177 (2)	0.0204 (8)
H19	0.0547	0.8824	0.4497	0.024*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.0197 (2)	0.0211 (3)	0.0260 (3)	-0.00604 (17)	0.00179 (16)	-0.00448 (17)
Cl3	0.0239 (5)	0.0179 (5)	0.0317 (6)	0.0050 (4)	0.0049 (4)	0.0057 (4)
P1	0.0138 (5)	0.0119 (5)	0.0183 (5)	-0.0001 (4)	0.0032 (4)	0.0002 (4)
O1	0.0148 (13)	0.0164 (15)	0.0231 (14)	0.0027 (11)	0.0044 (11)	0.0033 (11)
O2	0.0193 (15)	0.0260 (17)	0.0286 (16)	0.0038 (13)	0.0079 (12)	0.0092 (13)
C1	0.0147 (18)	0.015 (2)	0.0198 (19)	-0.0007 (15)	0.0009 (15)	-0.0001 (15)
C2	0.0164 (19)	0.020 (2)	0.027 (2)	0.0034 (16)	0.0084 (16)	0.0015 (17)
C3	0.023 (2)	0.022 (2)	0.027 (2)	-0.0003 (18)	0.0119 (17)	0.0052 (18)
C4	0.0179 (19)	0.0130 (19)	0.024 (2)	-0.0018 (16)	-0.0004 (15)	0.0008 (16)
C5	0.0167 (19)	0.019 (2)	0.024 (2)	0.0048 (16)	0.0042 (15)	-0.0031 (16)
C6	0.019 (2)	0.022 (2)	0.0178 (19)	0.0028 (17)	0.0034 (15)	-0.0014 (16)
C7	0.0133 (18)	0.020 (2)	0.0196 (19)	0.0015 (16)	0.0013 (14)	-0.0012 (16)
C8	0.0096 (16)	0.0099 (18)	0.0217 (19)	0.0005 (14)	0.0036 (14)	0.0013 (15)
C9	0.020 (2)	0.0115 (19)	0.026 (2)	0.0035 (16)	0.0041 (16)	0.0015 (15)
C10	0.026 (2)	0.020 (2)	0.025 (2)	0.0035 (18)	0.0029 (17)	0.0077 (17)
C11	0.023 (2)	0.027 (2)	0.020 (2)	0.0013 (19)	0.0012 (16)	0.0019 (18)
C12	0.021 (2)	0.017 (2)	0.026 (2)	-0.0016 (17)	0.0017 (16)	-0.0032 (17)
C13	0.0181 (19)	0.0122 (19)	0.026 (2)	-0.0018 (16)	0.0039 (15)	-0.0005 (16)
C14	0.0144 (18)	0.0081 (17)	0.025 (2)	0.0001 (14)	0.0047 (15)	0.0018 (15)
C15	0.018 (2)	0.021 (2)	0.023 (2)	0.0001 (17)	0.0035 (16)	0.0026 (17)
C16	0.0127 (19)	0.030 (3)	0.036 (2)	0.0012 (18)	0.0030 (17)	0.007 (2)
C17	0.020 (2)	0.021 (2)	0.039 (3)	0.0026 (18)	0.0132 (18)	0.0050 (19)
C18	0.029 (2)	0.020 (2)	0.024 (2)	-0.0018 (18)	0.0112 (17)	-0.0012 (17)
C19	0.020 (2)	0.017 (2)	0.024 (2)	-0.0010 (17)	0.0035 (16)	-0.0006 (16)

*Geometric parameters (Å, °)*

Se1—P1	2.0774 (11)	C9—C10	1.392 (6)
Cl3—C4	1.740 (4)	C9—H9	0.95
P1—O1	1.653 (3)	C10—C11	1.384 (7)
P1—C8	1.802 (4)	C10—H10	0.95
P1—C14	1.809 (4)	C11—C12	1.388 (6)
O1—C7	1.380 (5)	C11—H11	0.95
O2—C7	1.197 (5)	C12—C13	1.382 (6)

C1—C6	1.396 (6)	C12—H12	0.95
C1—C2	1.397 (6)	C13—H13	0.95
C1—C7	1.479 (6)	C14—C15	1.394 (6)
C2—C3	1.392 (6)	C14—C19	1.394 (6)
C2—H2	0.95	C15—C16	1.393 (6)
C3—C4	1.380 (6)	C15—H15	0.95
C3—H3	0.95	C16—C17	1.383 (7)
C4—C5	1.385 (6)	C16—H16	0.95
C5—C6	1.389 (6)	C17—C18	1.387 (7)
C5—H5	0.95	C17—H17	0.95
C6—H6	0.95	C18—C19	1.387 (6)
C8—C9	1.395 (6)	C18—H18	0.95
C8—C13	1.398 (6)	C19—H19	0.95
O1—P1—C8	105.05 (17)	C10—C9—H9	120
O1—P1—C14	98.28 (17)	C8—C9—H9	120
C8—P1—C14	106.94 (18)	C11—C10—C9	120.0 (4)
O1—P1—Se1	114.93 (11)	C11—C10—H10	120
C8—P1—Se1	115.52 (13)	C9—C10—H10	120
C14—P1—Se1	114.32 (14)	C10—C11—C12	120.1 (4)
C7—O1—P1	119.8 (3)	C10—C11—H11	119.9
C6—C1—C2	120.0 (4)	C12—C11—H11	119.9
C6—C1—C7	117.4 (4)	C13—C12—C11	120.4 (4)
C2—C1—C7	122.6 (4)	C13—C12—H12	119.8
C3—C2—C1	119.8 (4)	C11—C12—H12	119.8
C3—C2—H2	120.1	C12—C13—C8	119.9 (4)
C1—C2—H2	120.1	C12—C13—H13	120.1
C4—C3—C2	119.2 (4)	C8—C13—H13	120.1
C4—C3—H3	120.4	C15—C14—C19	120.2 (4)
C2—C3—H3	120.4	C15—C14—P1	120.9 (3)
C3—C4—C5	122.0 (4)	C19—C14—P1	118.8 (3)
C3—C4—Cl3	119.3 (3)	C16—C15—C14	119.4 (4)
C5—C4—Cl3	118.7 (3)	C16—C15—H15	120.3
C4—C5—C6	118.8 (4)	C14—C15—H15	120.3
C4—C5—H5	120.6	C17—C16—C15	120.1 (4)
C6—C5—H5	120.6	C17—C16—H16	119.9
C5—C6—C1	120.2 (4)	C15—C16—H16	119.9
C5—C6—H6	119.9	C16—C17—C18	120.6 (4)
C1—C6—H6	119.9	C16—C17—H17	119.7
O2—C7—O1	122.2 (4)	C18—C17—H17	119.7
O2—C7—C1	125.4 (4)	C19—C18—C17	119.8 (4)
O1—C7—C1	112.4 (3)	C19—C18—H18	120.1
C9—C8—C13	119.7 (4)	C17—C18—H18	120.1
C9—C8—P1	119.1 (3)	C18—C19—C14	119.9 (4)
C13—C8—P1	121.1 (3)	C18—C19—H19	120
C10—C9—C8	119.9 (4)	C14—C19—H19	120
C8—P1—O1—C7	65.5 (3)	Se1—P1—C8—C13	156.6 (3)

C14—P1—O1—C7	175.7 (3)	C13—C8—C9—C10	-0.7 (6)
Se1—P1—O1—C7	-62.6 (3)	P1—C8—C9—C10	-178.4 (3)
C6—C1—C2—C3	2.5 (7)	C8—C9—C10—C11	-0.5 (7)
C7—C1—C2—C3	-175.0 (4)	C9—C10—C11—C12	1.2 (7)
C1—C2—C3—C4	-1.5 (7)	C10—C11—C12—C13	-0.8 (7)
C2—C3—C4—C5	-1.3 (7)	C11—C12—C13—C8	-0.4 (6)
C2—C3—C4—C13	176.7 (4)	C9—C8—C13—C12	1.1 (6)
C3—C4—C5—C6	2.9 (7)	P1—C8—C13—C12	178.8 (3)
C13—C4—C5—C6	-175.0 (3)	O1—P1—C14—C15	-108.5 (4)
C4—C5—C6—C1	-1.9 (7)	C8—P1—C14—C15	0.1 (4)
C2—C1—C6—C5	-0.8 (7)	Se1—P1—C14—C15	129.3 (3)
C7—C1—C6—C5	176.8 (4)	O1—P1—C14—C19	73.5 (4)
P1—O1—C7—O2	-15.8 (6)	C8—P1—C14—C19	-177.9 (3)
P1—O1—C7—C1	164.5 (3)	Se1—P1—C14—C19	-48.7 (4)
C6—C1—C7—O2	-9.5 (7)	C19—C14—C15—C16	0.0 (7)
C2—C1—C7—O2	168.1 (5)	P1—C14—C15—C16	-178.0 (4)
C6—C1—C7—O1	170.3 (4)	C14—C15—C16—C17	0.6 (7)
C2—C1—C7—O1	-12.2 (6)	C15—C16—C17—C18	-0.9 (8)
O1—P1—C8—C9	-153.4 (3)	C16—C17—C18—C19	0.5 (7)
C14—P1—C8—C9	102.8 (3)	C17—C18—C19—C14	0.1 (7)
Se1—P1—C8—C9	-25.7 (4)	C15—C14—C19—C18	-0.3 (7)
O1—P1—C8—C13	28.9 (4)	P1—C14—C19—C18	177.6 (4)
C14—P1—C8—C13	-74.9 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C16—H16...O2 <sup>i</sup>	0.95	2.59	3.359 (6)	138

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

