

O-4-Chlorobenzoyl diphenylselenophosphate

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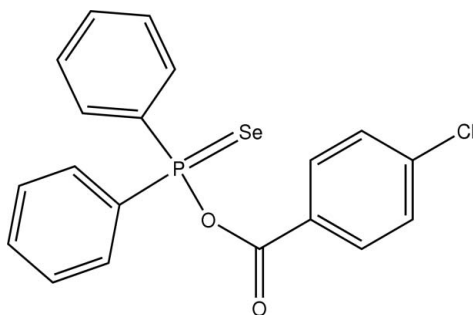
Received 23 January 2009; accepted 13 March 2009

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)°. Packing of the molecules in the crystal is reinforced by π - π 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)°
 $V = 1722.64$ (19) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.44$ mm⁻¹
 $T = 120$ 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⁻³
 $\Delta\rho_{\min} = -0.56$ e Å⁻³

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 ⁱ	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).

JC and JP thank the Gdansk University of Technology for financial support (internal grant No. 014668 t. 005).

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

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supplementary materials

Acta Cryst. (2009). E65, o855 [doi:10.1107/S1600536809009313]

***O*-4-Chlorobenzoyl diphenylselenophosphinate**

G. Cholewinski, J. Chojnacki, J. Pikies and J. Rachon

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, C₁₉H₁₄ClO₂PSe, 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 π - π 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.

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 ¹H, ¹³C, ³¹P NMR, MS and IR spectra were recorded and are consistent with the formula anticipated - see the supporting information for the article cited.

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.

Figures

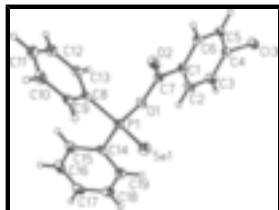


Fig. 1. View of the title compound showing the atom-numbering scheme (50% probability displacement ellipsoids).

O-4-Chlorobenzoyl diphenylselenophosphinate

Crystal data

$C_{19}H_{14}ClO_2PSe$	$F_{000} = 840$
$M_r = 419.68$	$D_x = 1.618 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Melting point: 379(2) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation
$a = 9.3390 (5) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 9.7132 (5) \text{ \AA}$	Cell parameters from 9084 reflections
$c = 19.1353 (15) \text{ \AA}$	$\theta = 2.1\text{--}32.4^\circ$
$\beta = 97.059 (6)^\circ$	$\mu = 2.44 \text{ mm}^{-1}$
$V = 1722.64 (19) \text{ \AA}^3$	$T = 120 \text{ K}$
$Z = 4$	Prism, colourless
	$0.46 \times 0.33 \times 0.26 \text{ mm}$

Data collection

Oxford Diffraction KM-4-CCD diffractometer	3376 reflections with $I > 2\sigma(I)$
Radiation source: enhance	$R_{\text{int}} = 0.045$
Monochromator: graphite	$\theta_{\text{max}} = 28^\circ$
ω scans (0.75° width)	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.325$, $T_{\text{max}} = 0.53$	$k = -12 \rightarrow 12$
14128 measured reflections	$l = -21 \rightarrow 25$
4158 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H-atom parameters constrained
$wR(F^2) = 0.162$	$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 5.5587P]$
$S = 1.16$	where $P = (F_o^2 + 2F_c^2)/3$
	$(\Delta/\sigma)_{\text{max}} = 0.001$

4158 reflections $\Delta\rho_{\max} = 1.82 \text{ e } \text{\AA}^{-3}$
 217 parameters $\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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)

supplementary materials

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 (\AA^2)

	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 (\AA , $^\circ$)

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—C13	119.3 (3)	C16—C15—C14	119.4 (4)
C5—C4—C13	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)

supplementary materials

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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C16—H16 \cdots O2 ⁱ	0.95	2.59	3.359 (6)	138

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

Fig. 1

