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Tris(2-methoxyphenyl)phosphine selenide

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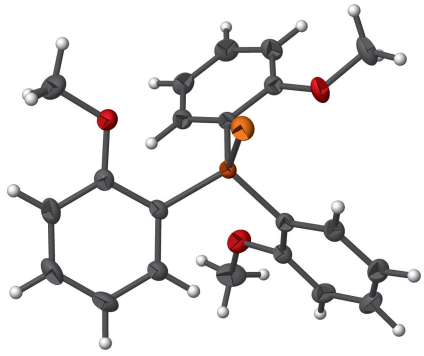
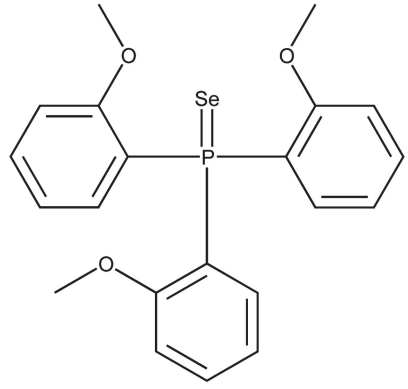
The title compound $C_{21}H_{21}O_3PSe$, is comprised of a P atom in a distorted tetrahedral environment, attached to the selenium atom and three carbons from the phenyl rings. The phosphorus–selenium bond length is 2.1194 (11) Å. All three methoxy groups are nearly co-planar with their respective phenyl rings, with the angles between the phenyl ring and the C—O bond of the methoxy groups being 6.2 (2), 3.1 (2), and 5.7 (2)°. The torsion angles of the phenyl rings relative to the P═Se bond are 55.84 (19), 176.18 (16), and 63.9 (2)°. No strong interactions were observed, but in addition to van der Waals forces, there are close contacts between C—H···π and C—H···Se.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is composed of a distorted tetrahedral phosphorus atom attached to the selenium atom and three carbons from three different phenyl rings. The P═Se bond distance is 2.1194 (11) Å. This distance is similar to those reported previously for the phenyl (Codding & Kerr, 1979), *p*-fluorophenyl (Muller & Meijboom, 2007), *p*-tolyl (Muller, 2011), and *o*-tolyl (Cameron & Dahlèn, 1975) derivatives (all 2.10–2.12 Å). The average P—C bond distance is 1.820 (3) Å with an average C—P—C bond angle of 106.29 (11)°. The average Se—P—C bond angle is determined to be 112.48 (9)°. The torsion angles of the phenyl rings relative to the P═Se bond are 55.84 (19)° for Se1—P1—C1—C2, 176.18 (16)° for Se1—P1—C8—C9, and 63.9 (2)° for Se1—P1—C15—C16. The compound presents extremely weak C—H···Se and C—H···π intermolecular interactions and displays an intramolecular C13—H13···Se1 close contact (Table 1). The crystal packing is illustrated in Fig. 2.

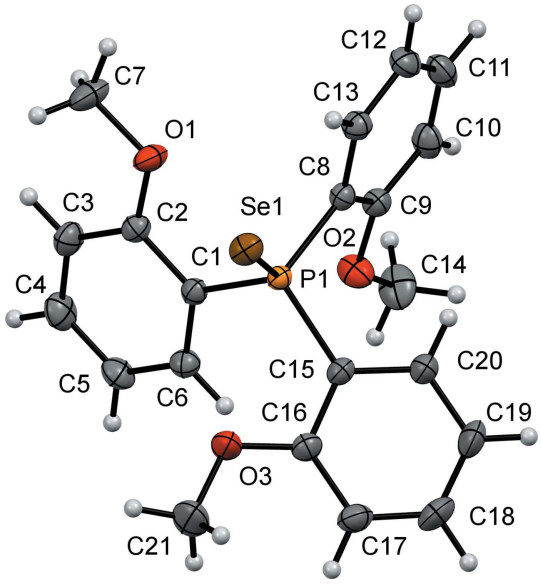


Figure 1
A view of the molecular structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

The title compound was synthesized by dissolving 0.25 g (0.71 mmol) of tris-2-methoxyphenylphosphine in 20 mL of methanol and this solution was brought to a boil. To this solution was added an equimolar amount of selenium (0.056 g, 0.71 mmol) in one portion. The solution was heated at reflux for 15 minutes. The solution was filtered hot to remove any unreacted selenium metal. Colorless crystals were then grown by slow evaporation of the solvent at room temperature. Yields were between 70–75% based on the phosphine starting material. This is an adaptation of a literature preparation by Dakternieks *et al.* (1994) and is similar to that described by Raymundo, *et al.* (2016).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

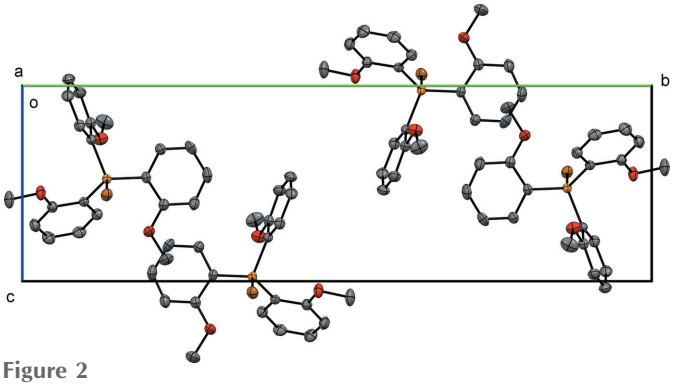


Figure 2
Crystal packing diagram of the title compound, viewed along the *a* axis. All H atoms have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···Se1	0.95	2.77	3.356 (3)	120
C19—H19···Se1^{i}	0.95	2.94	3.761 (3)	145
C7—H7*B*···*Cg*1^{ii}	0.98	2.76	3.602 (4)	144
C10—H10···*Cg*1^{iii}	0.95	2.83	3.658 (3)	146

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x, -y+1, -z$; (iii) $x, y, z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{21}O_3PSe$
M_r	431.31
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, *b*, *c* (Å)	8.351 (4), 27.156 (14), 8.545 (4)
β (°)	99.414 (7)
V (Å^3)	1911.7 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.07
Crystal size (mm)	0.4 × 0.2 × 0.15
Data collection	
Diffractometer	Rigaku XtaLAB mini diffractometer
Absorption correction	Multi-scan (*REQAB*; Rigaku, 1998)
T_{min}, T_{max}	0.556, 0.734
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4389, 4389, 3819
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.084, 1.09
No. of reflections	4389
No. of parameters	238
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.36

Computer programs: *CrystalClear SM Expert* (Rigaku, 2011), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170009 [https://doi.org/10.1107/S2414314617000098]

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Crystal data

$C_{21}H_{21}O_3PSe$
M_r = 431.31
Monoclinic, $P2_1/n$
a = 8.351 (4) Å
b = 27.156 (14) Å
c = 8.545 (4) Å
β = 99.414 (7)°
V = 1911.7 (16) Å^3
Z = 4
$F(000)$ = 880
D_x = 1.499 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 5181 reflections
θ = 2.4–27.5°
μ = 2.07 mm^{-1}
T = 173 K
Prism, colorless
0.4 × 0.2 × 0.15 mm

Data collection

Rigaku XtaLAB mini diffractometer
Detector resolution: 6.827 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*REQAB*; Rigaku, 1998)
T_{min} = 0.556, T_{max} = 0.734
4389 measured reflections
4389 independent reflections
3819 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −10→10
k = −35→35
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.084
S = 1.09
4389 reflections
238 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 1.2505P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.46784 (3)	0.63422 (2)	−0.05815 (3)	0.02628 (8)
P1	0.24136 (7)	0.63436 (2)	0.02326 (7)	0.01802 (12)
O1	0.2527 (2)	0.52932 (6)	−0.0484 (2)	0.0295 (4)
O2	−0.0163 (2)	0.62419 (7)	0.2283 (2)	0.0313 (4)
O3	0.1419 (2)	0.70152 (6)	−0.2456 (2)	0.0328 (4)
C1	0.0819 (3)	0.59807 (8)	−0.0936 (2)	0.0202 (4)
C2	0.1084 (3)	0.54796 (8)	−0.1201 (3)	0.0226 (5)
C3	−0.0102 (3)	0.52009 (9)	−0.2140 (3)	0.0302 (5)
H3	0.0089	0.4864	−0.2339	0.036*
C4	−0.1558 (3)	0.54181 (10)	−0.2780 (3)	0.0337 (6)
H4	−0.2365	0.5228	−0.3424	0.040*
C5	−0.1860 (3)	0.59059 (10)	−0.2498 (3)	0.0297 (5)
H5	−0.2872	0.6050	−0.2933	0.036*
C6	−0.0674 (3)	0.61862 (9)	−0.1575 (3)	0.0247 (5)
H6	−0.0883	0.6522	−0.1377	0.030*
C7	0.2889 (3)	0.47934 (9)	−0.0828 (4)	0.0385 (6)
H7A	0.2945	0.4761	−0.1960	0.058*
H7B	0.2036	0.4577	−0.0557	0.058*
H7C	0.3935	0.4700	−0.0203	0.058*
C8	0.2598 (3)	0.60968 (8)	0.2241 (3)	0.0208 (4)
C9	0.1291 (3)	0.60840 (8)	0.3091 (3)	0.0230 (5)
C10	0.1516 (3)	0.59136 (9)	0.4641 (3)	0.0308 (5)
H10	0.0640	0.5918	0.5225	0.037*
C11	0.3022 (3)	0.57369 (9)	0.5338 (3)	0.0334 (6)
H11	0.3179	0.5624	0.6404	0.040*
C12	0.4297 (3)	0.57243 (9)	0.4491 (3)	0.0300 (5)
H12	0.5313	0.5589	0.4955	0.036*
C13	0.4087 (3)	0.59105 (8)	0.2959 (3)	0.0252 (5)
H13	0.4976	0.5911	0.2393	0.030*
C14	−0.1478 (3)	0.63054 (12)	0.3142 (4)	0.0419 (7)
H14A	−0.2449	0.6408	0.2412	0.063*
H14B	−0.1193	0.6558	0.3960	0.063*
H14C	−0.1694	0.5993	0.3645	0.063*
C15	0.1653 (3)	0.69686 (8)	0.0308 (3)	0.0226 (5)
C16	0.1256 (3)	0.72478 (8)	−0.1088 (3)	0.0252 (5)
C17	0.0718 (3)	0.77322 (9)	−0.1014 (3)	0.0318 (6)
H17	0.0434	0.7919	−0.1959	0.038*
C18	0.0603 (4)	0.79385 (10)	0.0433 (3)	0.0404 (7)
H18	0.0207	0.8265	0.0477	0.048*
C19	0.1054 (4)	0.76776 (10)	0.1826 (3)	0.0409 (7)
H19	0.1008	0.7828	0.2822	0.049*
C20	0.1571 (3)	0.71963 (9)	0.1754 (3)	0.0297 (5)
H20	0.1878	0.7017	0.2710	0.036*
C21	0.1161 (5)	0.72893 (11)	−0.3881 (3)	0.0523 (8)
H21A	0.1326	0.7076	−0.4767	0.078*

H21B	0.1930	0.7564	−0.3798	0.078*
H21C	0.0049	0.7417	−0.4067	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.02445 (14)	0.02560 (14)	0.03081 (14)	−0.00104 (9)	0.01052 (10)	0.00186 (9)
P1	0.0201 (3)	0.0154 (3)	0.0187 (3)	0.0008 (2)	0.0037 (2)	0.0000 (2)
O1	0.0259 (9)	0.0186 (8)	0.0417 (10)	0.0034 (7)	−0.0018 (7)	−0.0005 (7)
O2	0.0210 (9)	0.0437 (10)	0.0299 (9)	0.0038 (7)	0.0067 (7)	0.0032 (8)
O3	0.0511 (11)	0.0251 (9)	0.0225 (8)	0.0054 (8)	0.0063 (8)	0.0028 (7)
C1	0.0236 (11)	0.0189 (10)	0.0188 (10)	−0.0005 (8)	0.0050 (8)	0.0000 (8)
C2	0.0227 (11)	0.0215 (11)	0.0241 (11)	−0.0003 (9)	0.0051 (9)	0.0003 (9)
C3	0.0335 (13)	0.0221 (12)	0.0341 (13)	−0.0045 (10)	0.0032 (10)	−0.0043 (10)
C4	0.0298 (13)	0.0352 (14)	0.0338 (14)	−0.0098 (11)	−0.0020 (10)	−0.0037 (11)
C5	0.0226 (12)	0.0361 (14)	0.0281 (12)	−0.0016 (10)	−0.0026 (9)	0.0013 (10)
C6	0.0229 (11)	0.0250 (12)	0.0258 (12)	0.0017 (9)	0.0031 (9)	−0.0004 (9)
C7	0.0373 (15)	0.0199 (12)	0.0580 (18)	0.0070 (11)	0.0075 (13)	−0.0003 (12)
C8	0.0221 (11)	0.0183 (10)	0.0217 (11)	−0.0005 (8)	0.0028 (8)	−0.0009 (8)
C9	0.0247 (11)	0.0205 (11)	0.0239 (11)	0.0007 (9)	0.0040 (9)	−0.0006 (9)
C10	0.0385 (14)	0.0320 (13)	0.0233 (12)	−0.0049 (11)	0.0094 (10)	−0.0004 (10)
C11	0.0447 (16)	0.0321 (13)	0.0211 (12)	−0.0071 (11)	−0.0016 (11)	0.0033 (10)
C12	0.0292 (13)	0.0292 (13)	0.0281 (12)	0.0003 (10)	−0.0058 (10)	0.0005 (10)
C13	0.0240 (12)	0.0232 (11)	0.0279 (12)	−0.0001 (9)	0.0028 (9)	−0.0005 (9)
C14	0.0235 (13)	0.061 (2)	0.0434 (16)	0.0027 (12)	0.0127 (11)	−0.0108 (14)
C15	0.0261 (12)	0.0172 (10)	0.0245 (11)	0.0018 (9)	0.0044 (9)	−0.0018 (9)
C16	0.0288 (12)	0.0202 (11)	0.0265 (12)	−0.0008 (9)	0.0041 (9)	0.0012 (9)
C17	0.0350 (14)	0.0210 (12)	0.0379 (14)	0.0029 (10)	0.0011 (11)	0.0032 (10)
C18	0.0530 (18)	0.0187 (12)	0.0492 (17)	0.0071 (11)	0.0078 (14)	−0.0043 (11)
C19	0.0614 (19)	0.0271 (13)	0.0352 (15)	0.0051 (13)	0.0110 (13)	−0.0116 (11)
C20	0.0409 (14)	0.0230 (12)	0.0246 (12)	0.0056 (10)	0.0041 (10)	−0.0022 (9)
C21	0.092 (3)	0.0396 (16)	0.0259 (14)	0.0070 (17)	0.0108 (15)	0.0078 (12)

Geometric parameters (Å, º)

Se1—P1	2.1194 (11)	C9—C10	1.387 (3)
P1—C1	1.818 (2)	C10—H10	0.9500
P1—C8	1.825 (2)	C10—C11	1.386 (4)
P1—C15	1.817 (2)	C11—H11	0.9500
O1—C2	1.358 (3)	C11—C12	1.382 (4)
O1—C7	1.432 (3)	C12—H12	0.9500
O2—C9	1.364 (3)	C12—C13	1.387 (3)
O2—C14	1.428 (3)	C13—H13	0.9500
O3—C16	1.354 (3)	C14—H14A	0.9800
O3—C21	1.414 (3)	C14—H14B	0.9800
C1—C2	1.403 (3)	C14—H14C	0.9800
C1—C6	1.393 (3)	C15—C16	1.407 (3)
C2—C3	1.391 (3)	C15—C20	1.393 (3)

C3—H3	0.9500	C16—C17	1.394 (3)
C3—C4	1.382 (4)	C17—H17	0.9500
C4—H4	0.9500	C17—C18	1.376 (4)
C4—C5	1.377 (4)	C18—H18	0.9500
C5—H5	0.9500	C18—C19	1.384 (4)
C5—C6	1.388 (3)	C19—H19	0.9500
C6—H6	0.9500	C19—C20	1.381 (3)
C7—H7A	0.9800	C20—H20	0.9500
C7—H7B	0.9800	C21—H21A	0.9800
C7—H7C	0.9800	C21—H21B	0.9800
C8—C9	1.407 (3)	C21—H21C	0.9800
C8—C13	1.389 (3)		

C1—P1—Se1	115.57 (8)	C11—C10—H10	120.1
C1—P1—C8	105.00 (10)	C10—C11—H11	119.8
C8—P1—Se1	111.43 (8)	C12—C11—C10	120.4 (2)
C15—P1—Se1	110.44 (8)	C12—C11—H11	119.8
C15—P1—C1	107.27 (11)	C11—C12—H12	120.1
C15—P1—C8	106.60 (10)	C11—C12—C13	119.7 (2)
C2—O1—C7	117.57 (19)	C13—C12—H12	120.1
C9—O2—C14	118.3 (2)	C8—C13—H13	119.5
C16—O3—C21	118.4 (2)	C12—C13—C8	121.0 (2)
C2—C1—P1	119.58 (17)	C12—C13—H13	119.5
C6—C1—P1	121.75 (17)	O2—C14—H14A	109.5
C6—C1—C2	118.7 (2)	O2—C14—H14B	109.5
O1—C2—C1	116.14 (19)	O2—C14—H14C	109.5
O1—C2—C3	123.6 (2)	H14A—C14—H14B	109.5
C3—C2—C1	120.3 (2)	H14A—C14—H14C	109.5
C2—C3—H3	120.2	H14B—C14—H14C	109.5
C4—C3—C2	119.6 (2)	C16—C15—P1	120.56 (17)
C4—C3—H3	120.2	C20—C15—P1	120.96 (18)
C3—C4—H4	119.5	C20—C15—C16	118.3 (2)
C5—C4—C3	121.0 (2)	O3—C16—C15	115.9 (2)
C5—C4—H4	119.5	O3—C16—C17	123.9 (2)
C4—C5—H5	120.2	C17—C16—C15	120.2 (2)
C4—C5—C6	119.5 (2)	C16—C17—H17	120.2
C6—C5—H5	120.2	C18—C17—C16	119.7 (2)
C1—C6—H6	119.6	C18—C17—H17	120.2
C5—C6—C1	120.8 (2)	C17—C18—H18	119.5
C5—C6—H6	119.6	C17—C18—C19	121.1 (2)
O1—C7—H7A	109.5	C19—C18—H18	119.5
O1—C7—H7B	109.5	C18—C19—H19	120.4
O1—C7—H7C	109.5	C20—C19—C18	119.3 (2)
H7A—C7—H7B	109.5	C20—C19—H19	120.4
H7A—C7—H7C	109.5	C15—C20—H20	119.3
H7B—C7—H7C	109.5	C19—C20—C15	121.4 (2)
C9—C8—P1	122.77 (17)	C19—C20—H20	119.3
C13—C8—P1	118.76 (17)	O3—C21—H21A	109.5

C13—C8—C9	118.5 (2)	O3—C21—H21B	109.5
O2—C9—C8	115.6 (2)	O3—C21—H21C	109.5
O2—C9—C10	124.0 (2)	H21A—C21—H21B	109.5
C10—C9—C8	120.4 (2)	H21A—C21—H21C	109.5
C9—C10—H10	120.1	H21B—C21—H21C	109.5
C11—C10—C9	119.8 (2)		

Se1—P1—C1—C2	55.84 (19)	C7—O1—C2—C1	−175.3 (2)
Se1—P1—C1—C6	−124.48 (17)	C7—O1—C2—C3	5.3 (3)
Se1—P1—C8—C9	176.18 (16)	C8—P1—C1—C2	−67.4 (2)
Se1—P1—C8—C13	−4.3 (2)	C8—P1—C1—C6	112.3 (2)
Se1—P1—C15—C16	63.9 (2)	C8—P1—C15—C16	−174.95 (18)
Se1—P1—C15—C20	−111.01 (19)	C8—P1—C15—C20	10.2 (2)
P1—C1—C2—O1	2.9 (3)	C8—C9—C10—C11	−2.5 (4)
P1—C1—C2—C3	−177.69 (18)	C9—C8—C13—C12	−1.2 (3)
P1—C1—C6—C5	178.31 (18)	C9—C10—C11—C12	−0.7 (4)
P1—C8—C9—O2	3.7 (3)	C10—C11—C12—C13	3.0 (4)
P1—C8—C9—C10	−176.97 (18)	C11—C12—C13—C8	−2.0 (4)
P1—C8—C13—C12	179.20 (18)	C13—C8—C9—O2	−175.8 (2)
P1—C15—C16—O3	2.4 (3)	C13—C8—C9—C10	3.5 (3)
P1—C15—C16—C17	−178.10 (19)	C14—O2—C9—C8	−171.1 (2)
P1—C15—C20—C19	177.5 (2)	C14—O2—C9—C10	9.6 (3)
O1—C2—C3—C4	177.9 (2)	C15—P1—C1—C2	179.50 (17)
O2—C9—C10—C11	176.7 (2)	C15—P1—C1—C6	−0.8 (2)
O3—C16—C17—C18	−179.5 (2)	C15—P1—C8—C9	55.6 (2)
C1—P1—C8—C9	−58.0 (2)	C15—P1—C8—C13	−124.83 (19)
C1—P1—C8—C13	121.56 (19)	C15—C16—C17—C18	1.0 (4)
C1—P1—C15—C16	−62.9 (2)	C16—C15—C20—C19	2.5 (4)
C1—P1—C15—C20	122.2 (2)	C16—C17—C18—C19	1.8 (4)
C1—C2—C3—C4	−1.5 (4)	C17—C18—C19—C20	−2.4 (5)
C2—C1—C6—C5	−2.0 (3)	C18—C19—C20—C15	0.2 (4)
C2—C3—C4—C5	−0.3 (4)	C20—C15—C16—O3	177.4 (2)
C3—C4—C5—C6	0.9 (4)	C20—C15—C16—C17	−3.1 (4)
C4—C5—C6—C1	0.3 (4)	C21—O3—C16—C15	−175.0 (2)
C6—C1—C2—O1	−176.8 (2)	C21—O3—C16—C17	5.5 (4)
C6—C1—C2—C3	2.6 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···Se1	0.95	2.77	3.356 (3)	120
C19—H19···Se1^{i}	0.95	2.94	3.761 (3)	145
C7—H7*B*···*Cg*1^{ii}	0.98	2.76	3.602 (4)	144
C10—H10···*Cg*1^{iii}	0.95	2.83	3.658 (3)	146

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*+1/2; (ii) −*x*, −*y*+1, −*z*; (iii) *x*, *y*, *z*+1.