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Tris(4-methoxyphenyl)phosphine selenide

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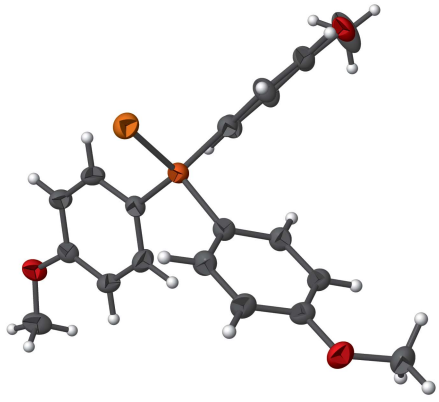
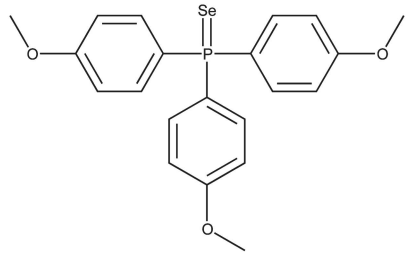
The title compound, $C_{21}H_{21}O_3PSe$, is comprised of a P atom in a distorted tetrahedral environment, attached to the Se atom and three C atoms of the phenyl rings. The P—Se bond length is 2.1214 (12) Å. All three methoxy groups are near coplanar with their respective phenyl rings, with the angles between the phenyl ring and the C—O bond of the methoxy groups being 5.7 (2), 1.5 (4), and 5.7 (3)°. The torsion angles of the phenyl rings relative to the P═Se bond are 35.62 (10), 35.07 (13), and 44.50 (11)°. No strong intermolecular interactions were observed, but that in addition to van der Waals forces, there are C—H···π and C—H···Se close contacts.

3D view



Chemical scheme



Structure description

The title compound $C_{21}H_{21}O_3PSe$ or $SeP(C_7H_7O)_3$ (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 2.1214 (15) Å, 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). This implies minimal effect of the substituent group on the bond distance between the phosphorus and selenium atoms. The torsion angles relative to the P═Se bond in the *para* methoxy derivative are 35.62 (10), 35.07 (13) and 44.50 (11)° for the aryl rings containing C1, C8, and C15, respectively. The methoxy carbon–oxygen bond alignments can be described as a propeller in three dimensions. The cone angle is 128.2 (7)° for the cone swept out by the phenyl rings during a rotation around the Se═P bond (averaged value for the three phenyl rings). The compound presents extremely weak C—H···Se and C—H···π intermolecular interactions (Table 1). The crystal packing is shown in Fig. 2.

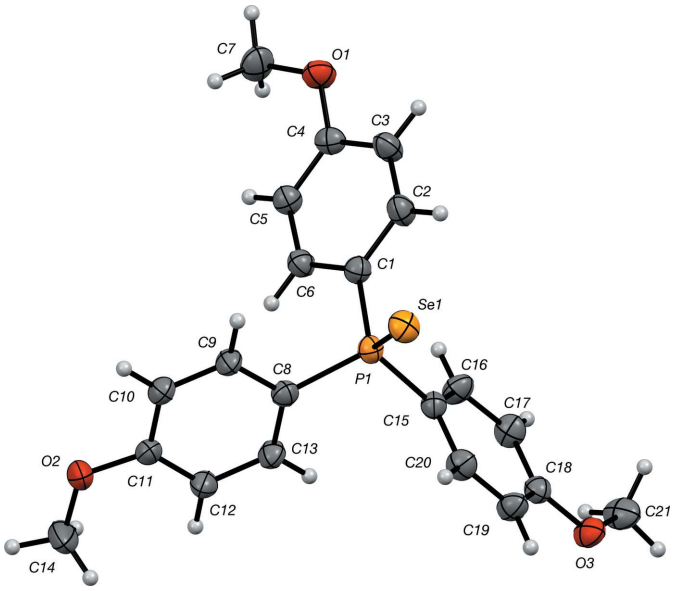


Figure 1
A view of the molecular structure of the title compound showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

The title compound was synthesized by dissolving 0.25 g (0.71 mmol) of tris-4-methoxyphenylphosphine in 20 ml of methanol. This solution was brought to a boil and an equimolar amount of selenium (0.056 g, 0.71 mmol) was added in one portion. The solution was heated at reflux for 15 minutes and then filtered hot to remove any unreacted selenium metal. Colorless crystals were grown by slow evaporation of the solvent at room temperature. The yield was 70% based on the phosphine starting material. This is an adaptation of a literature preparation by Dakternieks *et al.* (1994).

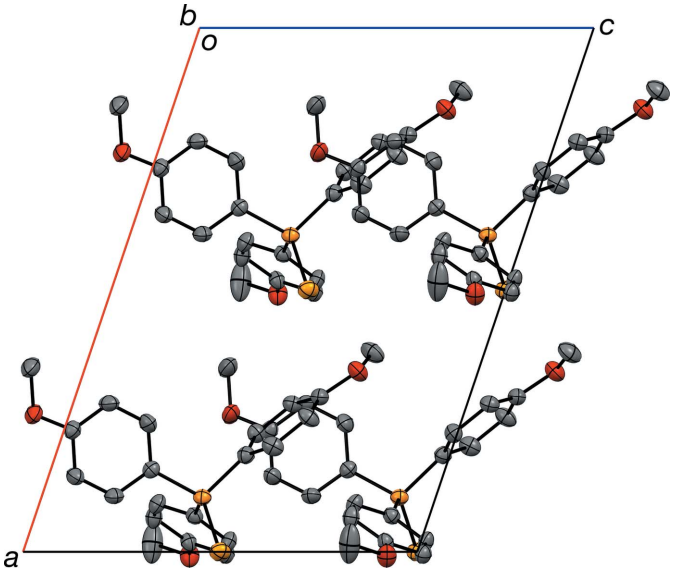


Figure 2
Crystal packing of the title compound viewed along the *b* axis. All H atoms have been omitted for clarity.

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*C*···Se1^{i}	0.98	2.96	3.798 (6)	145
C7—H7*A*···Se1^{ii}	0.98	2.99	3.809 (5)	142
C12—H12···*Cg*1^{iii}	0.95	2.82	3.515 (6)	130
C21—H21*B*···*Cg*2^{iv}	0.98	2.95	3.606 (6)	126

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{21}O_3PSe$
M_r	431.31
Crystal system, space group	Monoclinic, *Cc*
Temperature (K)	173
a, *b*, *c* (Å)	16.442 (11), 10.991 (7), 11.722 (8)
β (°)	108.611 (7)
V (Å^3)	2008 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.97
Crystal size (mm)	0.4 × 0.2 × 0.2
Data collection	
Diffractometer	Rigaku XtaLAB mini
Absorption correction	Multi-scan (*REQAB*; Rigaku, 1998)
T_{min}, T_{max}	0.532, 0.675
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4535, 4535, 4111
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.065, 0.92
No. of reflections	4535
No. of parameters	238
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.47
Absolute structure	Flack *x* determined using 1735 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.002 (7)

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161271 [doi:10.1107/S2414314616012712]

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Tris(4-methoxyphenyl)phosphine selenide

Crystal data

$C_{21}H_{21}O_3PSe$
M_r = 431.31
Monoclinic, *Cc*
a = 16.442 (11) Å
b = 10.991 (7) Å
c = 11.722 (8) Å
β = 108.611 (7)°
V = 2008 (2) Å^3
Z = 4
F(000) = 880
D_x = 1.427 Mg m^{-3}
Mo *K*α radiation, λ = 0.71075 Å
Cell parameters from 2952 reflections
θ = 2.3–27.5°
μ = 1.97 mm^{-1}
T = 173 K
Prism, colorless
0.4 × 0.2 × 0.2 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} = 0.532, T_{max} = 0.675
4535 measured reflections
4535 independent reflections
4111 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 2.3°
h = −21→21
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.065
S = 0.92
4535 reflections
238 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: Flack *x* determined using 1735 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.002 (7)

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	0.99996 (3)	0.83595 (4)	0.99958 (4)	0.03525 (12)
P1	0.89609 (7)	0.72080 (9)	0.90804 (9)	0.0264 (2)
O1	0.6556 (2)	0.6964 (3)	1.1948 (3)	0.0375 (8)
O2	1.0079 (2)	0.2046 (3)	0.9260 (3)	0.0368 (8)
O3	0.7375 (2)	0.9091 (3)	0.4097 (3)	0.0374 (8)
C1	0.8154 (3)	0.7122 (4)	0.9826 (4)	0.0285 (9)
C2	0.7986 (3)	0.8144 (4)	1.0434 (4)	0.0346 (10)
H2	0.8256	0.8898	1.0388	0.042*
C3	0.7432 (3)	0.8061 (4)	1.1096 (4)	0.0359 (11)
H3	0.7313	0.8764	1.1487	0.043*
C4	0.7043 (3)	0.6962 (4)	1.1201 (4)	0.0301 (9)
C5	0.7181 (3)	0.5943 (4)	1.0573 (4)	0.0305 (10)
H5	0.6896	0.5198	1.0604	0.037*
C6	0.7738 (3)	0.6030 (4)	0.9905 (4)	0.0303 (10)
H6	0.7839	0.5333	0.9491	0.036*
C7	0.6197 (3)	0.5831 (5)	1.2162 (5)	0.0455 (13)
H7A	0.5753	0.5569	1.1424	0.068*
H7B	0.5943	0.5936	1.2806	0.068*
H7C	0.6650	0.5214	1.2401	0.068*
C8	0.9314 (3)	0.5653 (4)	0.9040 (4)	0.0274 (9)
C9	0.9805 (3)	0.5112 (4)	1.0133 (4)	0.0304 (10)
H9	0.9971	0.5581	1.0850	0.036*
C10	1.0045 (4)	0.3915 (4)	1.0170 (4)	0.0304 (10)
H10	1.0367	0.3555	1.0914	0.036*
C11	0.9818 (3)	0.3226 (4)	0.9118 (4)	0.0285 (9)
C12	0.9342 (3)	0.3753 (4)	0.8025 (4)	0.0339 (10)
H12	0.9192	0.3288	0.7304	0.041*
C13	0.9090 (3)	0.4966 (4)	0.7998 (4)	0.0310 (9)
H13	0.8760	0.5323	0.7256	0.037*
C14	0.9843 (5)	0.1284 (5)	0.8213 (5)	0.074 (2)
H14A	1.0099	0.1604	0.7625	0.111*
H14B	0.9217	0.1273	0.7856	0.111*
H14C	1.0052	0.0456	0.8441	0.111*
C15	0.8431 (3)	0.7702 (4)	0.7547 (4)	0.0267 (9)
C16	0.7547 (3)	0.7820 (4)	0.7069 (4)	0.0351 (11)
H16	0.7196	0.7584	0.7536	0.042*
C17	0.7166 (3)	0.8280 (4)	0.5913 (4)	0.0351 (11)
H17	0.6560	0.8370	0.5602	0.042*
C18	0.7675 (3)	0.8607 (4)	0.5215 (4)	0.0295 (9)
C19	0.8560 (3)	0.8467 (4)	0.5681 (4)	0.0344 (10)
H19	0.8910	0.8677	0.5203	0.041*
C20	0.8934 (3)	0.8026 (4)	0.6832 (4)	0.0336 (10)
H20	0.9540	0.7941	0.7141	0.040*
C21	0.6481 (3)	0.9361 (5)	0.3620 (4)	0.0448 (12)
H21A	0.6353	0.9693	0.2806	0.067*

H21B	0.6327	0.9959	0.4136	0.067*
H21C	0.6148	0.8614	0.3590	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0326 (2)	0.0340 (2)	0.0374 (2)	−0.0059 (2)	0.00885 (16)	−0.0040 (2)
P1	0.0250 (5)	0.0250 (5)	0.0288 (6)	0.0011 (4)	0.0081 (4)	0.0008 (4)
O1	0.0371 (18)	0.0396 (18)	0.043 (2)	−0.0021 (16)	0.0221 (16)	−0.0015 (15)
O2	0.049 (2)	0.0280 (16)	0.0346 (18)	0.0103 (15)	0.0153 (15)	0.0035 (13)
O3	0.0367 (19)	0.0473 (19)	0.0257 (17)	0.0091 (17)	0.0061 (14)	0.0057 (14)
C1	0.029 (2)	0.028 (2)	0.028 (2)	0.0009 (19)	0.0087 (18)	0.0006 (17)
C2	0.035 (3)	0.026 (2)	0.045 (3)	0.000 (2)	0.016 (2)	−0.0005 (19)
C3	0.036 (3)	0.029 (2)	0.049 (3)	0.001 (2)	0.023 (2)	−0.004 (2)
C4	0.025 (2)	0.036 (2)	0.029 (2)	0.0003 (19)	0.0083 (18)	−0.0012 (19)
C5	0.027 (2)	0.030 (2)	0.035 (3)	−0.0011 (19)	0.0106 (19)	−0.0003 (19)
C6	0.033 (3)	0.026 (2)	0.032 (2)	−0.0009 (19)	0.012 (2)	−0.0028 (18)
C7	0.042 (3)	0.044 (3)	0.058 (3)	−0.001 (3)	0.028 (3)	0.010 (2)
C8	0.028 (2)	0.028 (2)	0.028 (2)	−0.0002 (18)	0.0111 (17)	0.0017 (17)
C9	0.036 (3)	0.030 (2)	0.021 (2)	0.004 (2)	0.0046 (18)	0.0020 (17)
C10	0.033 (2)	0.031 (2)	0.026 (3)	0.002 (2)	0.008 (2)	0.0032 (18)
C11	0.033 (2)	0.028 (2)	0.027 (2)	0.0025 (19)	0.0136 (19)	0.0034 (17)
C12	0.047 (3)	0.027 (2)	0.027 (2)	−0.001 (2)	0.012 (2)	−0.0016 (18)
C13	0.036 (2)	0.030 (2)	0.024 (2)	0.001 (2)	0.0051 (18)	0.0023 (17)
C14	0.147 (7)	0.035 (3)	0.047 (4)	0.027 (4)	0.040 (4)	0.003 (3)
C15	0.028 (2)	0.024 (2)	0.027 (2)	0.0006 (18)	0.0072 (17)	0.0012 (16)
C16	0.032 (2)	0.043 (3)	0.034 (3)	−0.003 (2)	0.015 (2)	0.006 (2)
C17	0.023 (2)	0.041 (3)	0.038 (3)	0.001 (2)	0.0065 (19)	0.0039 (19)
C18	0.032 (2)	0.028 (2)	0.027 (2)	0.0027 (19)	0.0077 (19)	−0.0012 (17)
C19	0.029 (2)	0.043 (3)	0.033 (3)	0.006 (2)	0.013 (2)	0.0061 (19)
C20	0.023 (2)	0.042 (3)	0.035 (3)	0.004 (2)	0.0078 (19)	0.004 (2)
C21	0.039 (3)	0.061 (3)	0.030 (3)	0.012 (3)	0.004 (2)	0.000 (2)

Geometric parameters (Å, º)

Se1—P1	2.1214 (15)	C9—H9	0.9500
P1—C1	1.809 (4)	C9—C10	1.370 (5)
P1—C8	1.810 (4)	C10—H10	0.9500
P1—C15	1.812 (4)	C10—C11	1.393 (6)
O1—C4	1.362 (5)	C11—C12	1.397 (6)
O1—C7	1.433 (5)	C12—H12	0.9500
O2—C11	1.360 (5)	C12—C13	1.393 (6)
O2—C14	1.433 (6)	C13—H13	0.9500
O3—C18	1.353 (5)	C14—H14A	0.9800
O3—C21	1.428 (5)	C14—H14B	0.9800
C1—C2	1.405 (6)	C14—H14C	0.9800
C1—C6	1.399 (6)	C15—C16	1.387 (6)
C2—H2	0.9500	C15—C20	1.399 (6)

C2—C3	1.376 (6)	C16—H16	0.9500
C3—H3	0.9500	C16—C17	1.394 (6)
C3—C4	1.390 (6)	C17—H17	0.9500
C4—C5	1.398 (6)	C17—C18	1.392 (6)
C5—H5	0.9500	C18—C19	1.390 (6)
C5—C6	1.385 (6)	C19—H19	0.9500
C6—H6	0.9500	C19—C20	1.380 (6)
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	1.409 (6)	C21—H21C	0.9800
C8—C13	1.383 (6)		
H14C···Se1^{i}	2.956 (2)	H12···Cg1^{iii}	2.824 (2)
H7A···Se1^{ii}	2.985 (2)	H21B···Cg2^{iv}	2.946 (3)
C1—P1—Se1	112.53 (15)	C11—C10—H10	120.0
C1—P1—C8	104.71 (19)	O2—C11—C10	115.2 (4)
C1—P1—C15	107.5 (2)	O2—C11—C12	124.8 (4)
C8—P1—Se1	111.31 (15)	C10—C11—C12	120.0 (4)
C8—P1—C15	108.28 (19)	C11—C12—H12	120.2
C15—P1—Se1	112.15 (14)	C13—C12—C11	119.6 (4)
C4—O1—C7	118.0 (4)	C13—C12—H12	120.2
C11—O2—C14	117.6 (4)	C8—C13—C12	120.6 (4)
C18—O3—C21	118.1 (4)	C8—C13—H13	119.7
C2—C1—P1	119.8 (3)	C12—C13—H13	119.7
C6—C1—P1	121.8 (3)	O2—C14—H14A	109.5
C6—C1—C2	118.2 (4)	O2—C14—H14B	109.5
C1—C2—H2	119.8	O2—C14—H14C	109.5
C3—C2—C1	120.4 (4)	H14A—C14—H14B	109.5
C3—C2—H2	119.8	H14A—C14—H14C	109.5
C2—C3—H3	119.5	H14B—C14—H14C	109.5
C2—C3—C4	120.9 (4)	C16—C15—P1	122.6 (3)
C4—C3—H3	119.5	C16—C15—C20	118.5 (4)
O1—C4—C3	115.9 (4)	C20—C15—P1	118.8 (3)
O1—C4—C5	124.6 (4)	C15—C16—H16	119.5
C3—C4—C5	119.5 (4)	C15—C16—C17	121.0 (4)
C4—C5—H5	120.3	C17—C16—H16	119.5
C6—C5—C4	119.3 (4)	C16—C17—H17	120.1
C6—C5—H5	120.3	C18—C17—C16	119.8 (4)
C1—C6—H6	119.2	C18—C17—H17	120.1
C5—C6—C1	121.5 (4)	O3—C18—C17	124.8 (4)
C5—C6—H6	119.2	O3—C18—C19	115.8 (4)
O1—C7—H7A	109.5	C19—C18—C17	119.4 (4)
O1—C7—H7B	109.5	C18—C19—H19	119.7
O1—C7—H7C	109.5	C20—C19—C18	120.5 (4)
H7A—C7—H7B	109.5	C20—C19—H19	119.7
H7A—C7—H7C	109.5	C15—C20—H20	119.6

H7B—C7—H7C	109.5	C19—C20—C15	120.7 (4)
C9—C8—P1	118.0 (3)	C19—C20—H20	119.6
C13—C8—P1	122.8 (3)	O3—C21—H21A	109.5
C13—C8—C9	119.1 (4)	O3—C21—H21B	109.5
C8—C9—H9	119.7	O3—C21—H21C	109.5
C10—C9—C8	120.7 (4)	H21A—C21—H21B	109.5
C10—C9—H9	119.7	H21A—C21—H21C	109.5
C9—C10—H10	120.0	H21B—C21—H21C	109.5
C9—C10—C11	120.0 (4)		

Se1—P1—C1—C2	33.7 (4)	C7—O1—C4—C5	3.7 (7)
Se1—P1—C1—C6	−140.8 (3)	C8—P1—C1—C2	154.7 (4)
Se1—P1—C8—C9	51.3 (4)	C8—P1—C1—C6	−19.7 (4)
Se1—P1—C8—C13	−131.7 (3)	C8—P1—C15—C16	105.4 (4)
Se1—P1—C15—C16	−131.4 (3)	C8—P1—C15—C20	−77.3 (4)
Se1—P1—C15—C20	45.9 (4)	C8—C9—C10—C11	1.2 (7)
P1—C1—C2—C3	−174.0 (4)	C9—C8—C13—C12	0.2 (7)
P1—C1—C6—C5	173.8 (3)	C9—C10—C11—O2	−179.2 (4)
P1—C8—C9—C10	176.0 (4)	C9—C10—C11—C12	−0.2 (8)
P1—C8—C13—C12	−176.8 (3)	C10—C11—C12—C13	−0.8 (7)
P1—C15—C16—C17	175.7 (4)	C11—C12—C13—C8	0.7 (7)
P1—C15—C20—C19	−176.6 (4)	C13—C8—C9—C10	−1.2 (7)
O1—C4—C5—C6	−175.5 (4)	C14—O2—C11—C10	178.4 (5)
O2—C11—C12—C13	178.1 (4)	C14—O2—C11—C12	−0.5 (7)
O3—C18—C19—C20	177.5 (4)	C15—P1—C1—C2	−90.3 (4)
C1—P1—C8—C9	−70.5 (4)	C15—P1—C1—C6	95.3 (4)
C1—P1—C8—C13	106.5 (4)	C15—P1—C8—C9	175.0 (3)
C1—P1—C15—C16	−7.2 (4)	C15—P1—C8—C13	−7.9 (4)
C1—P1—C15—C20	170.1 (3)	C15—C16—C17—C18	1.2 (7)
C1—C2—C3—C4	1.5 (7)	C16—C15—C20—C19	0.8 (7)
C2—C1—C6—C5	−0.7 (7)	C16—C17—C18—O3	−178.2 (4)
C2—C3—C4—O1	175.4 (4)	C16—C17—C18—C19	0.2 (7)
C2—C3—C4—C5	−3.5 (7)	C17—C18—C19—C20	−1.0 (7)
C3—C4—C5—C6	3.3 (7)	C18—C19—C20—C15	0.5 (7)
C4—C5—C6—C1	−1.2 (7)	C20—C15—C16—C17	−1.6 (7)
C6—C1—C2—C3	0.6 (7)	C21—O3—C18—C17	4.2 (7)
C7—O1—C4—C3	−175.2 (4)	C21—O3—C18—C19	−174.2 (4)

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

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*C*···Se1^{i}	0.98	2.96	3.798 (6)	145
C7—H7*A*···Se1^{ii}	0.98	2.99	3.809 (5)	142

C12—H12···*Cg*1^{iii}	0.95	2.82	3.515 (6)	130
C21—H21*B*···*Cg*2^{iv}	0.98	2.95	3.606 (6)	126

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