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5-Benzylidene-2,3-diphenyl-1,2-selenaphosphole-2-selenide

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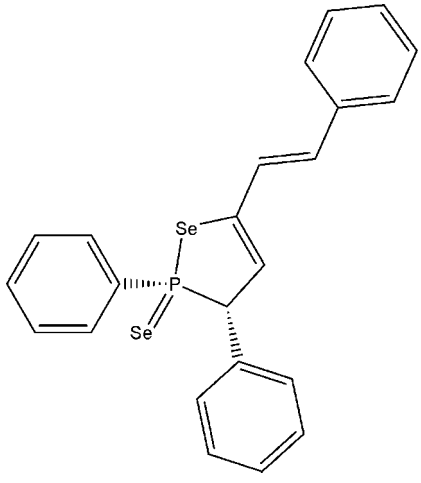
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.005 Å; R factor = 0.035; wR factor = 0.077; data-to-parameter ratio = 15.6.

The title compound, $C_{23}H_{19}PSe_2$, has a central five-membered twist C_3PSe ring conformation. One phenyl ring substituent, attached to an sp^2 carbon, is approximately coplanar with the C_3PSe ring whilst the other organic substituents, attached to an sp^3-carbon and a P^V atom, lie on the same side of the ring.

Related literature

For related literature, see: Yoshifuji *et al.* (1998); Fitzmaurice *et al.* (1988); Gray, Bhattacharyya *et al.* (2005); Gray, Slawin *et al.* (2005); Hua & Woollins (2007) and literature cited therein; Hua *et al.* (2006); Mugesh *et al.* (2001); Shi *et al.* (2006, 2007); Sommen *et al.* (2005).



Experimental

Crystal data

$C_{23}H_{19}PSe_2$
M_r = 484.27
Monoclinic, $C2/c$
a = 22.385 (2) Å
b = 14.4348 (14) Å
c = 12.4433 (12) Å
β = 94.847 (2)°
V = 4006.4 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.78 mm^{-1}
T = 93 (2) K
0.30 × 0.15 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku,2004)
T_{min} = 0.515, T_{max} = 0.692
11508 measured reflections
3667 independent reflections
3125 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.077
S = 1.04
3667 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Se2—C9	1.941 (3)	P1—C7	1.862 (3)
Se2—P1	2.2523 (9)	C8—C9	1.334 (5)
Se1—P1	2.1044 (9)	C8—C7	1.500 (5)
P1—C1	1.817 (3)		
C9—Se2—P1	87.97 (10)	C7—P1—Se2	96.47 (10)
C1—P1—C7	109.16 (15)	Se1—P1—Se2	115.54 (4)
C1—P1—Se1	112.86 (11)	C9—C8—C7	123.3 (3)
C7—P1—Se1	115.65 (11)	C8—C7—P1	107.5 (2)
C1—P1—Se2	105.64 (11)	C8—C9—Se2	117.1 (2)

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: Bruker *SHELXTL* (Sheldrick, 2003); software used to prepare material for publication: Bruker *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2054).

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S1. Comment

Organoselenium chemistry is attracting increasing attention because of chemo-, regio-, and stereoselective reactions and useful biological activity(Mugesh *et al.*, 2001). However, the synthesis of selenium-containing organic heterocycles can be problematic involving use of toxic selenium reagents which are often difficult to handle. 2,4-bis(phenyl)-1,3-diselenadiphosphetane-2,4-diselenide [PhP(Se)(μ-Se)]$_2$, known as Woollins reagent (WR) excels in efficiency and broad utility, capable of preparing a wide range selenium-containing heterocycles and the related compounds (Gray, Bhattacharyya *et al.* (2005); Gray, Slawin *et al.* (2005); Shi *et al.*, 2006, 2007). In our new five membered P—Se heterocycle the P = Se bond length (2.1044 (9) Å) and the P—Se distance (2.2523 (9) Å, Table 1) are consistent with the related selenides-containing P^V= Se bonds (2.081 (2) - 2.123 (3) Å) and P^V—Se single bonds (Fitzmaurice *et al.* 1988, Yoshifuji *et al.* 1998).

S2. Experimental

A mixture of dibenzoylideneacetone (0.47 g, 2 mmol) and Woollins′ reagent (0.54 g, 1 mmol) in 10 ml of dry toluene was refluxed for 20 hr. The red suspension disappeared and a red solution was formed along with a small amount of elemental selenium in the bottom of flask. Upon cooling to room temperature the mixture was purified by silica gel column chromatograhy (toluene as eluent) to give the title compound in 83% yield. Colorless crystal were grown from dichloro-methane with slow diffusion of n-hexane. Anal. Calcd for $C_{23}H_{19}PSe_2$: C, 57.04; H, 3.95. Found: C, 57.01; H, 3.99. ^{1}H NMR ($CDCl_3$): 7.63–7.47 (m, 2H, ArH), 7.37–7.30 (m, 3H, ArH), 7.21–7.12 (m, 4H, ArH), 7.11–7.04 (m, 4H, AeH), 6.96–6.93 (m, 2H, ArH), 7.05 (d, 1H, CH=CH), 6.95 (d, 1H, CH=CH), 6.66 (dd, 1H, CH=CH), 6.36 (dd, 1H, CH=CH). ^{31}P NMR ($CDCl_3$): 69.85 (s, J(P,Se$_{endo}$) = 350 Hz, J(P,Se$_{exo}$) = 782 Hz). ^{77}Se NMR ($CDCl_3$): 354.85 (J(P,Se$_{endo}$) = 350 Hz), -169.99 (J(P,Se$_{exo}$) = 780 Hz).

S3. Refinement

All H atoms were included in calculated positions (C—H distances are 0.98 Å for methyl H atoms, 0.99 Å for methylene H atoms and 0.95 Å for aryl H atoms) and were refined as riding atoms with U_{iso}(H) = 1.2 U_{eq} (parent atom, methylene and aryl H atoms) or U_{iso}(H) = 1.5 U_{eq} (parent atom,methyl H atoms).

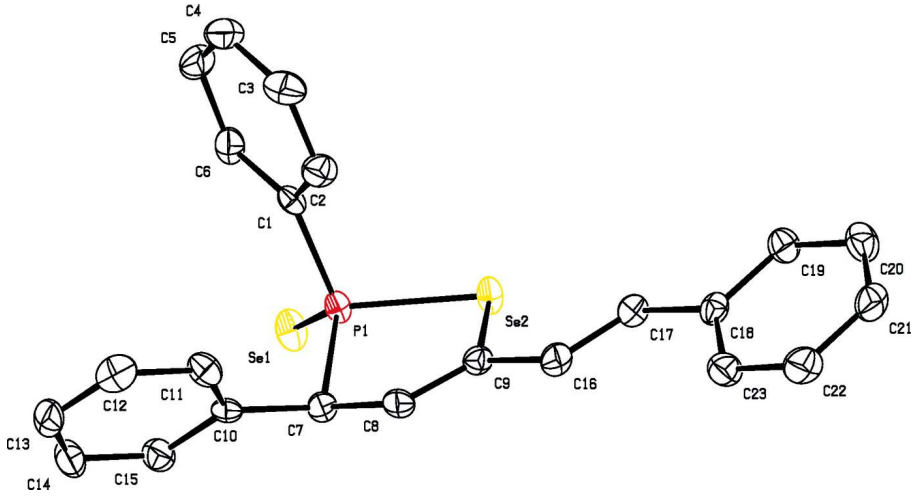

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

5-Benzylidene-2,3-diphenyl-1,2-selenaphosphole-2-selenide

Crystal data

$C_{23}H_{19}PSe_2$
$M_r = 484.27$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 22.385\ (2)$ Å
$b = 14.4348\ (14)$ Å
$c = 12.4433\ (12)$ Å
$\beta = 94.847\ (2)°$
$V = 4006.4\ (7)$ Å^3
$Z = 8$
$F(000) = 1920$
$D_x = 1.606$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8628 reflections
$\theta = 1.6–25.3°$
$\mu = 3.78$ mm^{-1}
$T = 93$ K
Prism, colorless
0.30 × 0.15 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: rotating anode
Confocal monochromator
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku,2004)
$T_{min} = 0.515$, $T_{max} = 0.692$
11508 measured reflections
3667 independent reflections
3125 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.3°$
$h = -26\rightarrow17$
$k = -18\rightarrow18$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.077$
$S = 1.04$
3667 reflections
235 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
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 14.4806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.95$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Se2	0.946290 (15)	−0.03631 (2)	0.57968 (3)	0.02008 (10)
Se1	0.960236 (16)	0.21801 (2)	0.57205 (3)	0.02484 (11)
P1	0.90715 (4)	0.10413 (6)	0.60975 (7)	0.01702 (19)
C19	0.93533 (15)	−0.4014 (2)	0.5818 (3)	0.0236 (8)
H19	0.9426	−0.3743	0.5145	0.028*
C6	0.81730 (16)	0.1775 (2)	0.4664 (3)	0.0237 (8)
H6	0.8439	0.2281	0.4600	0.028*
C10	0.85050 (14)	0.1485 (2)	0.7960 (2)	0.0182 (7)
C2	0.79343 (16)	0.0315 (2)	0.5460 (3)	0.0254 (8)
H2	0.8042	−0.0188	0.5931	0.030*
C11	0.79206 (15)	0.1186 (3)	0.8016 (3)	0.0240 (8)
H11	0.7813	0.0573	0.7800	0.029*
C5	0.76207 (17)	0.1763 (3)	0.4063 (3)	0.0326 (9)
H5	0.7515	0.2253	0.3573	0.039*
C13	0.76399 (17)	0.2669 (3)	0.8696 (3)	0.0286 (9)
H13	0.7346	0.3072	0.8945	0.034*
C1	0.83355 (14)	0.1048 (2)	0.5360 (3)	0.0188 (7)
C15	0.86516 (16)	0.2385 (2)	0.8277 (3)	0.0245 (8)
H15	0.9050	0.2602	0.8245	0.029*
C22	0.91438 (18)	−0.4819 (3)	0.7761 (3)	0.0326 (9)
H22	0.9070	−0.5096	0.8430	0.039*
C18	0.92438 (14)	−0.3447 (2)	0.6681 (3)	0.0201 (7)
C3	0.73807 (17)	0.0323 (3)	0.4872 (3)	0.0330 (9)
H3	0.7106	−0.0169	0.4949	0.040*
C12	0.74889 (16)	0.1772 (3)	0.8384 (3)	0.0294 (8)
H12	0.7090	0.1556	0.8422	0.035*
C4	0.72257 (18)	0.1045 (3)	0.4173 (3)	0.0349 (9)
H4	0.6846	0.1046	0.3769	0.042*
C8	0.89178 (15)	−0.0157 (2)	0.7738 (2)	0.0202 (7)
H8	0.8729	−0.0357	0.8354	0.024*
C14	0.82195 (17)	0.2971 (3)	0.8641 (3)	0.0299 (9)
H14	0.8325	0.3586	0.8854	0.036*
C21	0.92538 (16)	−0.5374 (2)	0.6896 (3)	0.0294 (8)
H21	0.9258	−0.6029	0.6971	0.035*
C16	0.90956 (15)	−0.1790 (2)	0.7240 (3)	0.0216 (7)

H16	0.8966	−0.2003	0.7904	0.026*
C7	0.89842 (15)	0.0864 (2)	0.7559 (3)	0.0186 (7)
H7	0.9372	0.1052	0.7955	0.022*
C23	0.91395 (17)	−0.3864 (3)	0.7666 (3)	0.0274 (8)
H23	0.9066	−0.3490	0.8269	0.033*
C17	0.92483 (14)	−0.2435 (2)	0.6540 (3)	0.0196 (7)
H17	0.9374	−0.2214	0.5876	0.024*
C9	0.91106 (14)	−0.0799 (2)	0.7077 (3)	0.0185 (7)
C20	0.93580 (16)	−0.4975 (3)	0.5923 (3)	0.0292 (8)
H20	0.9433	−0.5354	0.5325	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se2	0.02552 (19)	0.01480 (18)	0.02139 (18)	0.00227 (13)	0.01059 (13)	0.00242 (14)
Se1	0.0290 (2)	0.01641 (18)	0.0306 (2)	−0.00455 (14)	0.01166 (15)	0.00100 (15)
P1	0.0214 (4)	0.0136 (4)	0.0169 (4)	−0.0008 (3)	0.0065 (3)	0.0003 (3)
C19	0.0256 (19)	0.0207 (18)	0.0248 (19)	−0.0015 (14)	0.0034 (14)	−0.0006 (15)
C6	0.0312 (19)	0.0211 (18)	0.0200 (18)	0.0048 (15)	0.0083 (14)	0.0030 (15)
C10	0.0246 (18)	0.0223 (18)	0.0078 (15)	0.0015 (14)	0.0013 (12)	0.0015 (14)
C2	0.032 (2)	0.0220 (18)	0.0214 (18)	−0.0023 (15)	−0.0003 (14)	0.0039 (16)
C11	0.0277 (19)	0.0263 (19)	0.0190 (18)	−0.0022 (15)	0.0076 (14)	−0.0020 (15)
C5	0.041 (2)	0.036 (2)	0.0207 (19)	0.0131 (18)	−0.0013 (16)	0.0044 (17)
C13	0.036 (2)	0.032 (2)	0.0183 (18)	0.0138 (17)	0.0096 (15)	0.0010 (16)
C1	0.0226 (17)	0.0199 (17)	0.0142 (16)	0.0005 (14)	0.0039 (13)	−0.0046 (14)
C15	0.0289 (19)	0.0238 (19)	0.0208 (18)	0.0001 (15)	0.0018 (14)	−0.0012 (15)
C22	0.048 (2)	0.023 (2)	0.026 (2)	−0.0017 (17)	−0.0025 (17)	0.0092 (17)
C18	0.0152 (16)	0.0176 (17)	0.0272 (19)	−0.0003 (13)	−0.0010 (13)	0.0009 (15)
C3	0.032 (2)	0.039 (2)	0.028 (2)	−0.0071 (18)	−0.0020 (16)	−0.0057 (19)
C12	0.0220 (18)	0.042 (2)	0.0252 (19)	−0.0006 (16)	0.0071 (14)	0.0067 (17)
C4	0.034 (2)	0.046 (3)	0.023 (2)	0.0075 (19)	−0.0058 (16)	−0.0061 (19)
C8	0.0263 (18)	0.0233 (19)	0.0114 (16)	0.0011 (14)	0.0036 (13)	0.0018 (14)
C14	0.042 (2)	0.022 (2)	0.026 (2)	0.0046 (16)	0.0036 (16)	−0.0053 (17)
C21	0.031 (2)	0.0151 (18)	0.040 (2)	−0.0019 (15)	−0.0068 (16)	0.0023 (17)
C16	0.0279 (19)	0.0191 (17)	0.0183 (17)	0.0011 (14)	0.0035 (14)	0.0067 (14)
C7	0.0214 (17)	0.0181 (17)	0.0164 (17)	−0.0001 (13)	0.0029 (13)	0.0022 (14)
C23	0.038 (2)	0.0219 (19)	0.0219 (19)	−0.0003 (16)	−0.0006 (15)	0.0008 (16)
C17	0.0195 (17)	0.0192 (17)	0.0204 (17)	−0.0013 (13)	0.0031 (13)	0.0034 (14)
C9	0.0205 (17)	0.0190 (17)	0.0159 (17)	0.0007 (13)	0.0013 (13)	0.0029 (14)
C20	0.031 (2)	0.0215 (19)	0.036 (2)	−0.0027 (15)	0.0029 (16)	−0.0058 (17)

Geometric parameters (Å, º)

Se2—C9	1.941 (3)	C15—C14	1.389 (5)
Se2—P1	2.2523 (9)	C15—H15	0.9500
Se1—P1	2.1044 (9)	C22—C21	1.381 (5)
P1—C1	1.817 (3)	C22—C23	1.383 (5)
P1—C7	1.862 (3)	C22—H22	0.9500

C19—C18	1.389 (5)	C18—C23	1.402 (5)
C19—C20	1.393 (5)	C18—C17	1.472 (5)
C19—H19	0.9500	C3—C4	1.383 (6)
C6—C5	1.390 (5)	C3—H3	0.9500
C6—C1	1.390 (5)	C12—H12	0.9500
C6—H6	0.9500	C4—H4	0.9500
C10—C11	1.385 (5)	C8—C9	1.334 (5)
C10—C15	1.389 (5)	C8—C7	1.500 (5)
C10—C7	1.515 (4)	C8—H8	0.9500
C2—C3	1.385 (5)	C14—H14	0.9500
C2—C1	1.400 (5)	C21—C20	1.378 (5)
C2—H2	0.9500	C21—H21	0.9500
C11—C12	1.390 (5)	C16—C17	1.339 (5)
C11—H11	0.9500	C16—C9	1.446 (5)
C5—C4	1.377 (6)	C16—H16	0.9500
C5—H5	0.9500	C7—H7	1.0000
C13—C14	1.376 (5)	C23—H23	0.9500
C13—C12	1.385 (5)	C17—H17	0.9500
C13—H13	0.9500	C20—H20	0.9500

C9—Se2—P1	87.97 (10)	C4—C3—C2	120.3 (4)
C1—P1—C7	109.16 (15)	C4—C3—H3	119.8
C1—P1—Se1	112.86 (11)	C2—C3—H3	119.8
C7—P1—Se1	115.65 (11)	C13—C12—C11	120.0 (3)
C1—P1—Se2	105.64 (11)	C13—C12—H12	120.0
C7—P1—Se2	96.47 (10)	C11—C12—H12	120.0
Se1—P1—Se2	115.54 (4)	C5—C4—C3	120.1 (4)
C18—C19—C20	120.9 (3)	C5—C4—H4	120.0
C18—C19—H19	119.5	C3—C4—H4	120.0
C20—C19—H19	119.5	C9—C8—C7	123.3 (3)
C5—C6—C1	120.0 (3)	C9—C8—H8	118.4
C5—C6—H6	120.0	C7—C8—H8	118.4
C1—C6—H6	120.0	C13—C14—C15	120.6 (3)
C11—C10—C15	118.6 (3)	C13—C14—H14	119.7
C11—C10—C7	122.0 (3)	C15—C14—H14	119.7
C15—C10—C7	119.4 (3)	C20—C21—C22	119.7 (3)
C3—C2—C1	119.9 (3)	C20—C21—H21	120.1
C3—C2—H2	120.1	C22—C21—H21	120.1
C1—C2—H2	120.1	C17—C16—C9	125.9 (3)
C10—C11—C12	120.9 (3)	C17—C16—H16	117.1
C10—C11—H11	119.6	C9—C16—H16	117.1
C12—C11—H11	119.6	C8—C7—C10	116.8 (3)
C4—C5—C6	120.3 (4)	C8—C7—P1	107.5 (2)
C4—C5—H5	119.8	C10—C7—P1	112.1 (2)
C6—C5—H5	119.8	C8—C7—H7	106.6
C14—C13—C12	119.4 (3)	C10—C7—H7	106.6
C14—C13—H13	120.3	P1—C7—H7	106.6
C12—C13—H13	120.3	C22—C23—C18	120.2 (3)

C6—C1—C2	119.4 (3)	C22—C23—H23	119.9
C6—C1—P1	119.8 (3)	C18—C23—H23	119.9
C2—C1—P1	120.8 (3)	C16—C17—C18	127.4 (3)
C14—C15—C10	120.5 (3)	C16—C17—H17	116.3
C14—C15—H15	119.7	C18—C17—H17	116.3
C10—C15—H15	119.7	C8—C9—C16	126.0 (3)
C21—C22—C23	120.8 (3)	C8—C9—Se2	117.1 (2)
C21—C22—H22	119.6	C16—C9—Se2	116.8 (2)
C23—C22—H22	119.6	C21—C20—C19	119.9 (3)
C19—C18—C23	118.4 (3)	C21—C20—H20	120.0
C19—C18—C17	119.3 (3)	C19—C20—H20	120.0
C23—C18—C17	122.3 (3)		

C9—Se2—P1—C1	−90.70 (14)	C23—C22—C21—C20	−0.3 (6)
C9—Se2—P1—C7	21.31 (14)	C9—C8—C7—C10	149.3 (3)
C9—Se2—P1—Se1	143.79 (10)	C9—C8—C7—P1	22.4 (4)
C15—C10—C11—C12	−0.2 (5)	C11—C10—C7—C8	−29.5 (4)
C7—C10—C11—C12	−179.8 (3)	C15—C10—C7—C8	150.9 (3)
C1—C6—C5—C4	1.7 (5)	C11—C10—C7—P1	95.1 (3)
C5—C6—C1—C2	−1.0 (5)	C15—C10—C7—P1	−84.5 (3)
C5—C6—C1—P1	177.2 (3)	C1—P1—C7—C8	82.6 (2)
C3—C2—C1—C6	−0.3 (5)	Se1—P1—C7—C8	−148.82 (19)
C3—C2—C1—P1	−178.5 (3)	Se2—P1—C7—C8	−26.4 (2)
C7—P1—C1—C6	128.1 (3)	C1—P1—C7—C10	−47.0 (3)
Se1—P1—C1—C6	−2.0 (3)	Se1—P1—C7—C10	81.5 (2)
Se2—P1—C1—C6	−129.2 (2)	Se2—P1—C7—C10	−156.1 (2)
C7—P1—C1—C2	−53.7 (3)	C21—C22—C23—C18	0.4 (6)
Se1—P1—C1—C2	176.2 (2)	C19—C18—C23—C22	−0.3 (5)
Se2—P1—C1—C2	49.1 (3)	C17—C18—C23—C22	−179.6 (3)
C11—C10—C15—C14	−0.1 (5)	C9—C16—C17—C18	179.4 (3)
C7—C10—C15—C14	179.6 (3)	C19—C18—C17—C16	172.9 (3)
C20—C19—C18—C23	0.2 (5)	C23—C18—C17—C16	−7.9 (5)
C20—C19—C18—C17	179.4 (3)	C7—C8—C9—C16	175.7 (3)
C1—C2—C3—C4	1.0 (5)	C7—C8—C9—Se2	−2.5 (4)
C14—C13—C12—C11	−0.3 (5)	C17—C16—C9—C8	174.8 (3)
C10—C11—C12—C13	0.3 (5)	C17—C16—C9—Se2	−6.9 (5)
C6—C5—C4—C3	−1.0 (6)	P1—Se2—C9—C8	−14.4 (3)
C2—C3—C4—C5	−0.4 (6)	P1—Se2—C9—C16	167.2 (3)
C12—C13—C14—C15	0.0 (5)	C22—C21—C20—C19	0.2 (5)
C10—C15—C14—C13	0.2 (5)	C18—C19—C20—C21	−0.1 (5)