

Crystal structure of 1,1'-[selanediyl-bis(4,1-phenylene)]bis(2-chloroethan-1-one)

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In the title molecule, C₁₆H₁₂Cl₂O₂Se, the C–Se–C angle is 100.05 (14)°, with the dihedral angle between the planes of the benzene rings being 69.92 (17)°. The average endocyclic angles (Se–C_{ar}–C_{ar}; ar = aromatic) facing the Se atom are 120.0 (3) and 119.4 (3)°. The Se atom is essentially coplanar with the benzene rings, with Se–C_{ar}–C_{ar}–C_{ar} torsion angles of –179.2 (3) and –179.7 (3)°. In the crystal, molecules are linked *via* C–H···O hydrogen bonds forming chains propagating along the *a*-axis direction. The chains are linked *via* C–H···π interactions, forming a three-dimensional network.

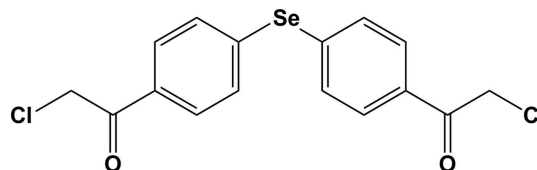
Keywords: crystal structure; organoselenium; selenium; C–H···O hydrogen bonds.

CCDC reference: 1432588

1. Related literature

For a review of organoselenium chemistry, see: Procter (2001). For their uses as reagents and intermediates in organic synthesis, see: Zade *et al.* (2005). For their use as intermediates in the synthesis of pharmaceuticals, see: Woods *et al.* (1993), and fine chemicals and polymers, see: Hellberg *et al.* (1997). For their biological properties, see: Zhu & Jiang (2008); Anderson *et al.* (1996); Abdel-Hafez (2008). For details of how selenium compounds play important roles in protecting the heart, preventing cancer and cardiovascular diseases, see: Yang *et al.* (2005). For details of how selenium functions as an antioxidant in conjunction with vitamin E, see: Ellis *et al.*

(1984). For the synthesis, see: Mechehoud *et al.* (2010). For related structures, see: Zuo (2013); Bouraoui *et al.* (2011).



2. Experimental

2.1. Crystal data

C ₁₆ H ₁₂ Cl ₂ O ₂ Se	$\gamma = 91.102 (8)^\circ$
$M_r = 386.12$	$V = 775.14 (14) \text{ \AA}^3$
Triclinic $P\bar{1}$	$Z = 2$
$a = 7.9664 (7) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.2804 (10) \text{ \AA}$	$\mu = 2.77 \text{ mm}^{-1}$
$c = 10.9045 (12) \text{ \AA}$	$T = 293 \text{ K}$
$\alpha = 104.612 (9)^\circ$	$0.13 \times 0.08 \times 0.04 \text{ mm}$
$\beta = 95.886 (8)^\circ$	

2.2. Data collection

Oxford Diffraction Xcalibur diffractometer	8193 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2013)	4862 independent reflections
$T_{\min} = 0.519$, $T_{\max} = 1.000$	2878 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.033$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$	190 parameters
$wR(F^2) = 0.134$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.59 \text{ e \AA}^{-3}$
4862 reflections	$\Delta\rho_{\text{min}} = -0.49 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

*Cg*1 and *Cg*2 are the centroids of benzene rings C1–C6 and C7–C12, respectively.

<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>
C6–H61···O1 ⁱ	0.93	2.41	3.288 (4)	158
C16–H161···Cg2 ⁱⁱ	0.97	2.82	3.611 (4)	140
C16–H162···Cg1 ⁱⁱⁱ	0.97	2.92	3.749 (4)	144

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $x, y + 1, z$.

Data collection: *Xcalibur* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* and *PLATON*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5221).

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supporting information

Acta Cryst. (2015). E71, o935–o936 [https://doi.org/10.1107/S2056989015019969]

Crystal structure of 1,1'-[selanediylbis(4,1-phenylene)]bis(2-chloroethan-1-one)**Hazem Bouraoui, Ali Boudjada, Noudjoud Hamdouni, Youcef Mechehoud and Jean Meinel****S1. Comment**

During the last few years, organoselenium chemistry (Procter, 2001) has been the subject of constant scientific interest and organoselenium compounds have been used intensively as important reagents and intermediates in organic synthesis (Zade *et al.*, 2005). Moreover, organoselenium compounds are of considerable interest as anti-cancer (Zhu & Jiang, 2008), anti-oxydant (Anderson *et al.*, 1996), anti-inflammatory, and antiallergic agents (Abdel-Hafez, 2008) and they are key intermediates for the synthesis of pharmaceuticals (Woods *et al.*, 1993), fine chemicals and polymers (Hellberg *et al.*, 1997). Selenium compounds have been found to play important roles in protecting the heart, preventing cancer and cardiovascular diseases (Yang *et al.*, 2005). Because selenium functions as an antioxidant it works in conjunction with vitamin E (Ellis *et al.*, 1984).

In the title compound, Fig. 1, the C1—Se1—C7 angle is 100.05 (14) °, similar to the value observed in two very similar compounds, viz. ca 99.47 ° in bis(4-nitrophenyl)selenide (Zuo, 2013), and 99.59 (14)° in bis(4-acetylphenyl) selenide (Bouraoui *et al.*, 2011) where the Se atom lies on a two-fold rotation axis. In the title compound the two benzene rings are inclined to one another by 69.92 (17) °. This is similar to the same angle observed for the 4-nitrophenyl derivative, ca 63.77 °, but considerably different to that observed for the acetylphenyl derivative, viz. 87.08 (15) °.

In the crystal of the title compound, molecules are linked by C—H···O hydrogen bonds forming chains along the *a* axis direction (Table 1 and Fig. 2). The chains are linked by C—H··· π interactions (Table 1) forming a three-dimensional structure.

S2. Synthesis and crystallization

The title compound was prepared according to a method proposed by (Mechehoud *et al.*, 2010). Methylene chloride acyl chloride, ClCH₂COCl, (36.5 mmol) and anhydrous aluminium chloride 5 g (37.5 mmol, 3 eq) were taken in dry CH₂Cl₂ (100 ml). The reaction mixture was cooled to 273-278 K, protected from atmospheric moisture, and stirred continuously for 15 min. A solution of diphenyl selenide (1) 3 g (1.87 mmol) in CH₂Cl₂ was added drop wise over a period of 5 min. The reaction mixture was allowed to reach room temperature gradually and then stirred at this temperature overnight. The solution was then washed with ice water-HCl (80 ml) and extracted with CH₂Cl₂. The organic layer was separated and dried (Na₂SO₄). Removal of the solvent under reduced pressure afforded the crude product which was recrystallized from ether-petrol to yield 4.2 g of the title compound. Yellow single crystals suitable for X-ray diffraction were obtained by recrystallization from CH₂Cl₂.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All the H atoms were localized in difference Fourier maps but introduced in calculated positions and treated as riding atoms: C—H = 0.93-0.97 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

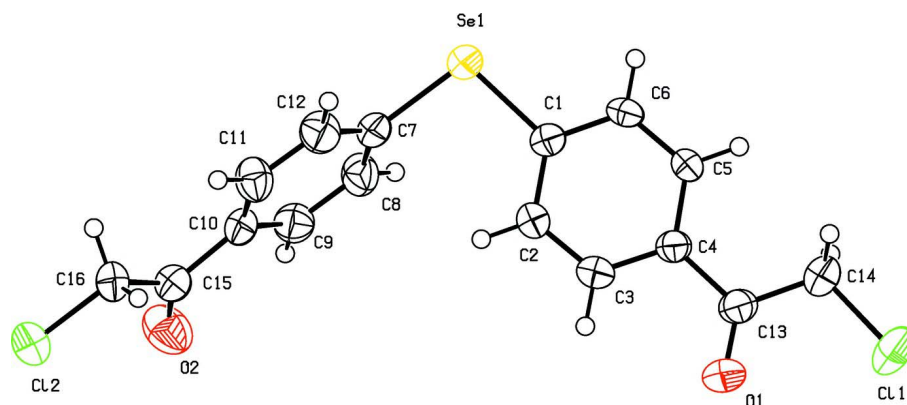


Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

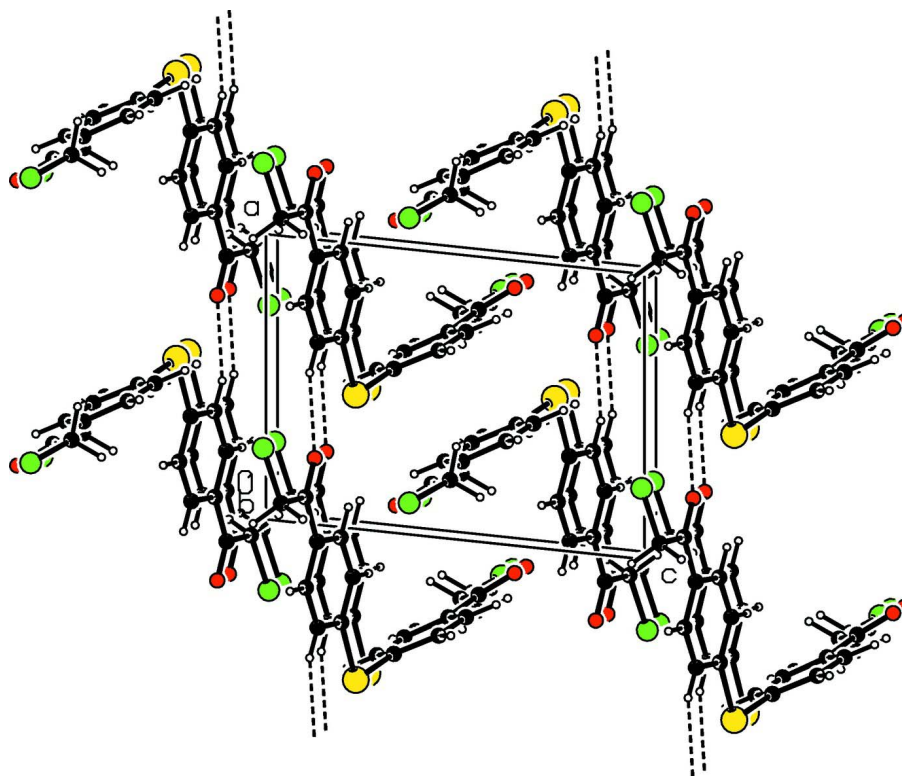


Figure 2

Crystal packing of the title compound, viewed along the *b* axis. The hydrogen bonds are shown as dashed lines (see Table 1).

1,1'-[Selanediylbis(4,1-phenylene)]bis(2-chloroethan-1-one)

Crystal data

$C_{16}H_{12}Cl_2O_2Se$

$M_r = 386.12$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.9664 (7) \text{ \AA}$

$b = 9.2804 (10) \text{ \AA}$

$c = 10.9045 (12) \text{ \AA}$
 $\alpha = 104.612 (9)^\circ$
 $\beta = 95.886 (8)^\circ$
 $\gamma = 91.102 (8)^\circ$
 $V = 775.14 (14) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 384$
 $D_x = 1.654 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$
 Cell parameters from 1842 reflections
 $\theta = 3.9\text{--}27.7^\circ$
 $\mu = 2.77 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Needle, colourless
 $0.13 \times 0.08 \times 0.04 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer
 Graphite monochromator
 $\omega/2\theta$ scans
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Agilent, 2013)
 $T_{\min} = 0.519$, $T_{\max} = 1.000$
 8193 measured reflections

4862 independent reflections
 2878 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\max} = 32.0^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -11 \rightarrow 11$
 $k = -13 \rightarrow 13$
 $l = -16 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.134$
 $S = 1.04$
 4862 reflections
 190 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
 $w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.1701P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.59 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.49 \text{ e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.53262 (4)	0.10191 (4)	0.76583 (4)	0.05281 (16)
Cl1	-0.25879 (14)	-0.40473 (13)	0.99518 (12)	0.0730 (3)
Cl2	0.13155 (14)	0.78058 (13)	0.40236 (15)	0.0818 (4)
O1	-0.2370 (3)	-0.1702 (3)	0.8662 (3)	0.0610 (7)
O2	0.1088 (4)	0.4605 (3)	0.3572 (3)	0.0714 (8)
C1	0.3337 (4)	0.0067 (4)	0.8013 (3)	0.0394 (8)
C2	0.1712 (4)	0.0498 (4)	0.7722 (4)	0.0440 (8)
H21	0.1548	0.1271	0.7328	0.053*
C3	0.0353 (4)	-0.0224 (4)	0.8020 (3)	0.0423 (8)
H31	-0.0729	0.0071	0.7825	0.051*
C4	0.0557 (4)	-0.1385 (3)	0.8605 (3)	0.0362 (7)
C5	0.2200 (4)	-0.1800 (4)	0.8892 (3)	0.0392 (8)
H51	0.2365	-0.2574	0.9285	0.047*

C6	0.3576 (4)	-0.1082 (4)	0.8603 (3)	0.0406 (8)
H61	0.4661	-0.1368	0.8803	0.049*
C7	0.4308 (4)	0.2302 (4)	0.6692 (4)	0.0415 (8)
C8	0.3485 (5)	0.1721 (4)	0.5489 (4)	0.0488 (9)
H81	0.3415	0.0695	0.5139	0.059*
C9	0.2762 (5)	0.2656 (4)	0.4798 (4)	0.0470 (8)
H91	0.2200	0.2256	0.3989	0.056*
C10	0.2872 (4)	0.4188 (4)	0.5306 (3)	0.0402 (8)
C11	0.3757 (5)	0.4760 (4)	0.6494 (4)	0.0490 (9)
H111	0.3879	0.5787	0.6829	0.059*
C12	0.4456 (5)	0.3831 (4)	0.7184 (4)	0.0500 (9)
H121	0.5033	0.4232	0.7989	0.060*
C13	-0.0979 (4)	-0.2116 (4)	0.8894 (3)	0.0425 (8)
C14	-0.0706 (5)	-0.3401 (4)	0.9497 (4)	0.0514 (9)
H141	0.0117	-0.3087	1.0244	0.062*
H142	-0.0248	-0.4214	0.8898	0.062*
C15	0.1994 (4)	0.5136 (4)	0.4539 (4)	0.0465 (9)
C16	0.2244 (5)	0.6811 (4)	0.5070 (4)	0.0540 (10)
H161	0.3444	0.7076	0.5243	0.065*
H162	0.1758	0.7101	0.5872	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.03407 (19)	0.0680 (3)	0.0687 (3)	-0.00036 (16)	0.00438 (18)	0.0409 (2)
Cl1	0.0585 (6)	0.0786 (7)	0.0896 (9)	-0.0193 (5)	0.0163 (6)	0.0345 (6)
Cl2	0.0582 (6)	0.0802 (7)	0.1284 (12)	0.0049 (5)	0.0010 (7)	0.0700 (8)
O1	0.0318 (12)	0.0782 (19)	0.083 (2)	0.0059 (12)	0.0080 (13)	0.0385 (16)
O2	0.081 (2)	0.0686 (19)	0.060 (2)	0.0050 (16)	-0.0141 (17)	0.0170 (16)
C1	0.0312 (15)	0.0460 (18)	0.044 (2)	0.0008 (13)	0.0040 (15)	0.0170 (16)
C2	0.0387 (17)	0.0477 (19)	0.053 (2)	0.0035 (15)	0.0039 (16)	0.0268 (17)
C3	0.0326 (15)	0.0475 (19)	0.050 (2)	0.0065 (14)	0.0018 (15)	0.0183 (17)
C4	0.0303 (15)	0.0424 (17)	0.0371 (19)	0.0027 (13)	0.0037 (14)	0.0124 (15)
C5	0.0334 (15)	0.0426 (18)	0.048 (2)	0.0066 (13)	0.0045 (15)	0.0242 (16)
C6	0.0277 (14)	0.0510 (19)	0.048 (2)	0.0090 (14)	0.0006 (14)	0.0218 (16)
C7	0.0338 (16)	0.0500 (19)	0.046 (2)	-0.0004 (14)	0.0077 (16)	0.0214 (17)
C8	0.055 (2)	0.0373 (18)	0.054 (2)	-0.0036 (16)	0.0018 (19)	0.0130 (17)
C9	0.0465 (19)	0.050 (2)	0.043 (2)	-0.0039 (16)	-0.0016 (17)	0.0108 (17)
C10	0.0383 (17)	0.0430 (18)	0.044 (2)	-0.0007 (14)	0.0103 (16)	0.0178 (16)
C11	0.061 (2)	0.0396 (19)	0.045 (2)	-0.0020 (17)	0.0022 (19)	0.0107 (17)
C12	0.054 (2)	0.055 (2)	0.040 (2)	-0.0037 (18)	-0.0008 (18)	0.0127 (18)
C13	0.0337 (16)	0.053 (2)	0.042 (2)	-0.0001 (14)	0.0036 (15)	0.0146 (17)
C14	0.0443 (19)	0.057 (2)	0.059 (3)	-0.0034 (17)	0.0112 (19)	0.0253 (19)
C15	0.0394 (17)	0.054 (2)	0.053 (2)	0.0040 (16)	0.0098 (18)	0.0240 (19)
C16	0.047 (2)	0.049 (2)	0.077 (3)	0.0029 (16)	0.009 (2)	0.036 (2)

Geometric parameters (Å, °)

Se1—C7	1.919 (3)	C7—C8	1.377 (5)
Se1—C1	1.920 (3)	C7—C12	1.383 (5)
C11—C14	1.766 (4)	C8—C9	1.383 (5)
C12—C16	1.757 (4)	C8—H81	0.9300
O1—C13	1.203 (4)	C9—C10	1.387 (5)
O2—C15	1.202 (5)	C9—H91	0.9300
C1—C6	1.384 (5)	C10—C11	1.382 (5)
C1—C2	1.392 (4)	C10—C15	1.495 (5)
C2—C3	1.371 (5)	C11—C12	1.372 (5)
C2—H21	0.9300	C11—H111	0.9300
C3—C4	1.388 (5)	C12—H121	0.9300
C3—H31	0.9300	C13—C14	1.510 (5)
C4—C5	1.398 (4)	C14—H141	0.9700
C4—C13	1.487 (4)	C14—H142	0.9700
C5—C6	1.376 (4)	C15—C16	1.517 (5)
C5—H51	0.9300	C16—H161	0.9700
C6—H61	0.9300	C16—H162	0.9700
C7—Se1—C1	100.05 (14)	C11—C10—C9	118.8 (3)
C6—C1—C2	120.0 (3)	C11—C10—C15	123.3 (3)
C6—C1—Se1	117.0 (2)	C9—C10—C15	117.9 (3)
C2—C1—Se1	123.0 (3)	C12—C11—C10	120.8 (3)
C3—C2—C1	119.7 (3)	C12—C11—H111	119.6
C3—C2—H21	120.2	C10—C11—H111	119.6
C1—C2—H21	120.2	C11—C12—C7	120.4 (4)
C2—C3—C4	121.5 (3)	C11—C12—H121	119.8
C2—C3—H31	119.2	C7—C12—H121	119.8
C4—C3—H31	119.2	O1—C13—C4	121.8 (3)
C3—C4—C5	118.0 (3)	O1—C13—C14	121.6 (3)
C3—C4—C13	118.3 (3)	C4—C13—C14	116.7 (3)
C5—C4—C13	123.7 (3)	C13—C14—C11	112.6 (3)
C6—C5—C4	121.1 (3)	C13—C14—H141	109.1
C6—C5—H51	119.4	C11—C14—H141	109.1
C4—C5—H51	119.4	C13—C14—H142	109.1
C5—C6—C1	119.7 (3)	C11—C14—H142	109.1
C5—C6—H61	120.1	H141—C14—H142	107.8
C1—C6—H61	120.1	O2—C15—C10	121.9 (3)
C8—C7—C12	119.4 (3)	O2—C15—C16	121.3 (4)
C8—C7—Se1	120.7 (3)	C10—C15—C16	116.7 (3)
C12—C7—Se1	119.9 (3)	C15—C16—C12	112.6 (3)
C7—C8—C9	120.2 (3)	C15—C16—H161	109.1
C7—C8—H81	119.9	C12—C16—H161	109.1
C9—C8—H81	119.9	C15—C16—H162	109.1
C8—C9—C10	120.4 (4)	C12—C16—H162	109.1
C8—C9—H91	119.8	H161—C16—H162	107.8
C10—C9—H91	119.8		

C6—C1—C2—C3	-0.2 (5)	C15—C10—C11—C12	176.3 (3)
Se1—C1—C2—C3	-179.2 (3)	C10—C11—C12—C7	1.0 (6)
C1—C2—C3—C4	-0.1 (6)	C8—C7—C12—C11	1.5 (5)
C2—C3—C4—C5	0.3 (5)	Se1—C7—C12—C11	179.0 (3)
C2—C3—C4—C13	-179.8 (3)	C3—C4—C13—O1	-1.9 (5)
C3—C4—C5—C6	0.0 (5)	C5—C4—C13—O1	178.0 (4)
C13—C4—C5—C6	-180.0 (3)	C3—C4—C13—C14	178.4 (3)
C4—C5—C6—C1	-0.3 (5)	C5—C4—C13—C14	-1.7 (5)
C2—C1—C6—C5	0.4 (5)	O1—C13—C14—C11	-6.1 (5)
Se1—C1—C6—C5	179.4 (3)	C4—C13—C14—C11	173.6 (3)
C12—C7—C8—C9	-2.2 (5)	C11—C10—C15—O2	-172.1 (4)
Se1—C7—C8—C9	-179.7 (3)	C9—C10—C15—O2	6.9 (5)
C7—C8—C9—C10	0.5 (5)	C11—C10—C15—C16	5.5 (5)
C8—C9—C10—C11	1.9 (5)	C9—C10—C15—C16	-175.5 (3)
C8—C9—C10—C15	-177.1 (3)	O2—C15—C16—C12	-7.9 (5)
C9—C10—C11—C12	-2.7 (5)	C10—C15—C16—C12	174.6 (2)

Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of benzene rings C1-C6 and C7-C12, respectively.

<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>
C6—H61...O1 ⁱ	0.93	2.41	3.288 (4)	158
C16—H161...Cg2 ⁱⁱ	0.97	2.82	3.611 (4)	140
C16—H162...Cg1 ⁱⁱⁱ	0.97	2.92	3.749 (4)	144

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, y+1, z$.