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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.039
 wR factor = 0.110
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis(2-methylbenzyl) diselenide

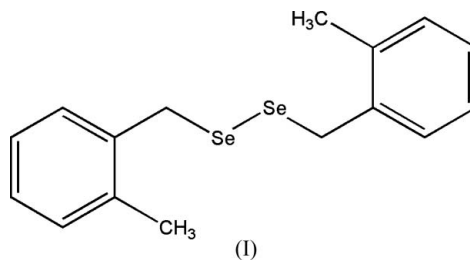
In the title molecule, $\text{C}_{16}\text{H}_{18}\text{Se}_2$, the dihedral angle between the benzene rings is $93.1(3)^\circ$. In the crystal structure, there are no significant hydrogen bonding interactions or π - π stacking interactions.

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Comment

Diorganoselenides are recognized as important antioxidants (Zhao & Holmgren, 2002) and are essential components of the active sites of a number of enzymes. Therefore, the pharmacology, biology and biochemistry of these materials have become subjects of considerable interest and are spurring efforts to develop synthetic selenium-containing compounds as possible therapeutic agents (Favero *et al.*, 2005). We are currently studying the syntheses of a new series of organoselenium compounds, such as diselenides and macrocyclic Schiff bases containing selenium. We report here the synthesis of the title compound, (I), by the reaction of 2-methylbenzyl chloride with sodium diselenide, and its X-ray crystal structure determination.



In (I) (Fig. 1), the Se—Se bond as well as the Se—C bonds (Table 1) are slightly longer than those in bisdiphenylmethyl diselenide [2.285 (3) and 1.97 (1) Å, respectively; Palmer & Palmer, 1969]. The dihedral angle between the two benzene rings is $93.1(3)^\circ$ and the dihedral angles between the planes defined by each Se—Se—C unit and the adjoining benzene ring are $68.6(1)$ and $76.7(1)^\circ$ for rings C1—C6 and C10—C15, respectively. The central C8—Se1—Se1—C9 torsion angle is within the range expected for this type of compound and indicates that steric strain is being relieved through bond lengthening rather than by an increase in this torsion angle (Bergson, 1962; Back & Codding, 1983). In the crystal structure, there are no significant hydrogen bonds or π - π stacking interactions.

Experimental

To a vigorously stirred suspension of selenium powder (2.0 g, 25 mmol) and hexadecyltrimethylammonium bromide (0.05 g, 0.14 mmol) in aqueous sodium hydroxide (25 ml, 2 mol l⁻¹), a cold

solution of sodium hydroxide (0.1 g, 2.5 mmol) and sodium borohydride (0.13 g, 3.3 mmol) in water (5 ml) was added dropwise under a nitrogen atmosphere. The reaction was maintained for 30 min at room temperature and for a further 30 min at 363 K. 2-Methylbenzyl chloride (5.1 ml, 39 mmol) was then added to the resulting brown solution with stirring, and the reaction was allowed to proceed for 3 h. The reaction mixture was extracted with ethyl acetate (3 × 50 ml), and the extract was washed with water (3 × 50 ml) and dried with sodium sulfate. The solvent was removed *in vacuo* to give a residue. The solid residue was recrystallized from ethyl acetate under refrigeration to give colorless crystals of the title compound (I) in 87% yield (m.p. 357 K), which were suitable for X-ray analysis.

Crystal data

$C_{16}H_{18}Se_2$ $V = 755.29 (17) \text{ \AA}^3$
 $M_r = 368.22$ $Z = 2$
 Triclinic, $P\bar{1}$ $D_x = 1.619 \text{ Mg m}^{-3}$
 $a = 8.2965 (10) \text{ \AA}$ Mo $K\alpha$ radiation
 $b = 9.0307 (11) \text{ \AA}$ $\mu = 4.88 \text{ mm}^{-1}$
 $c = 10.9729 (16) \text{ \AA}$ $T = 292 (2) \text{ K}$
 $\alpha = 83.598 (3)^\circ$ Block, yellow
 $\beta = 67.935 (2)^\circ$ $0.20 \times 0.10 \times 0.10 \text{ mm}$
 $\gamma = 83.878 (2)^\circ$

Data collection

Bruker SMART-APEX CCD 4310 measured reflections
 diffractometer 2918 independent reflections
 φ and ω scans 2193 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.080$
 (SADABS; Sheldrick, 2001) $\theta_{max} = 26.0^\circ$
 $T_{min} = 0.424$, $T_{max} = 0.615$

Refinement

Refinement on F^2 H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.039$ $w = 1/[\sigma^2(F_o^2) + (0.0588P)^2]$
 $wR(F^2) = 0.110$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 0.97$ $(\Delta/\sigma)_{max} = 0.001$
 2918 reflections $\Delta\rho_{max} = 0.80 \text{ e \AA}^{-3}$
 165 parameters $\Delta\rho_{min} = -0.63 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C8—Se1	1.989 (4)	Se1—Se2	2.3031 (7)
C9—Se2	1.995 (4)		
C6—C8—Se1	114.0 (3)	C8—Se1—Se2	100.73 (13)
C10—C9—Se2	113.0 (3)	C9—Se2—Se1	99.17 (13)
C7—C1—C6—C8	−3.5 (6)	C9—C10—C11—C16	−0.9 (6)
C1—C6—C8—Se1	84.4 (4)	C6—C8—Se1—Se2	68.3 (3)
C5—C6—C8—Se1	−97.0 (4)	C10—C9—Se2—Se1	−62.7 (3)
Se2—C9—C10—C15	101.5 (4)	C8—Se1—Se2—C9	86.97 (18)
Se2—C9—C10—C11	−77.4 (4)		

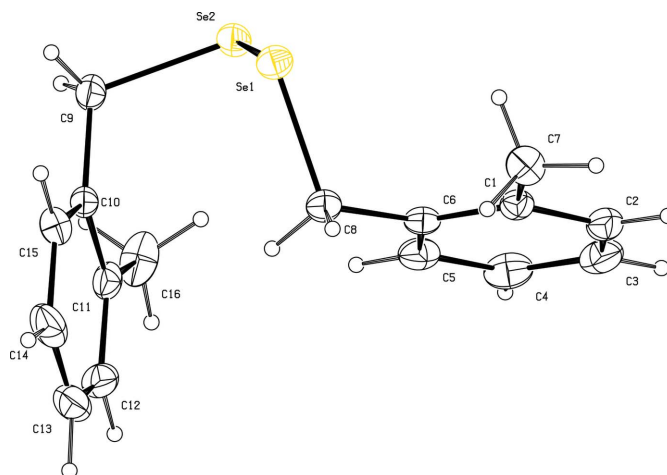


Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level and H atoms as small spheres.

All H atoms were placed in calculated positions (C—H = 0.93–0.97 \AA) and included in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl C})$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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