Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Mesitylselenenyl iodide

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Key indicators

Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.010 Å
R factor = 0.038
wR factor = 0.109
Data-to-parameter ratio = 17.6

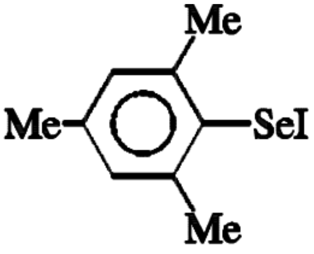
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 February 2002
Accepted 26 February 2002
Online 8 March 2002

The title compound, $C_9H_{11}ISe$, displays the following dimensions involving selenium: Se—I 2.5360 (11) Å, Se—C1 1.923 (6) Å and C—Se—I 100.2 (2)°. Short Se···I and I···I contacts link the molecules into ribbons parallel to the *b* axis.

Comment

We are interested in the structural chemistry of organoselenium derivatives. The first selenenyl iodide, 2,4,6-tri-*tert*-butylphenylselenenyl iodide, was synthesized and characterized by X-ray structure analysis by du Mont *et al.* (1987). It displayed crystallographic mirror symmetry, with Se—I = 2.529 (1) Å, Se—C = 1.941 (3) Å and C—Se—I = 97.5 (1)°. A search of the Cambridge Structural Database (version of October 2001; Allen & Kennard, 1993) revealed no other neutral selenenyl iodides, although two structures described as such contain very short intramolecular Se···N contacts of 2.242 (5) and 2.074 (6) Å that we would prefer to describe as genuine bonds, thus making the selenium three-coordinate (Panda *et al.*, 1993; Mugesh *et al.*, 1999).



(I)

We present here the structure of mesitylselenenyl iodide, (I), which we have previously described briefly in a review of selenium–iodine contacts (du Mont *et al.*, 2001). The molecule, which has no crystallographically imposed symmetry, is shown in Fig. 1. The geometry at selenium is essentially the same as in the structure of du Mont *et al.* (1987), with Se—I = 2.5360 (11) Å, Se—C1 = 1.923 (6) Å and C—Se—I 100.2 (2)°. The Se—I bond is not symmetrically disposed with respect to the ring; the relevant torsion angles are −99.3 (5) and 84.4 (5)°.

The molecular packing (Fig. 2) involves the following short contacts: I···I^{ii} and I···I^{iii} 3.840 (1) Å, and Se···I^{iv} 3.839 (2) Å (symmetry codes: see Table 1). The overall effect is to form a ribbon parallel to the *b* axis. Appreciably shorter Se···I contacts were observed in the 2:1 adduct between di(2,4,6-

DOI: 10.1107/S1600536802003677

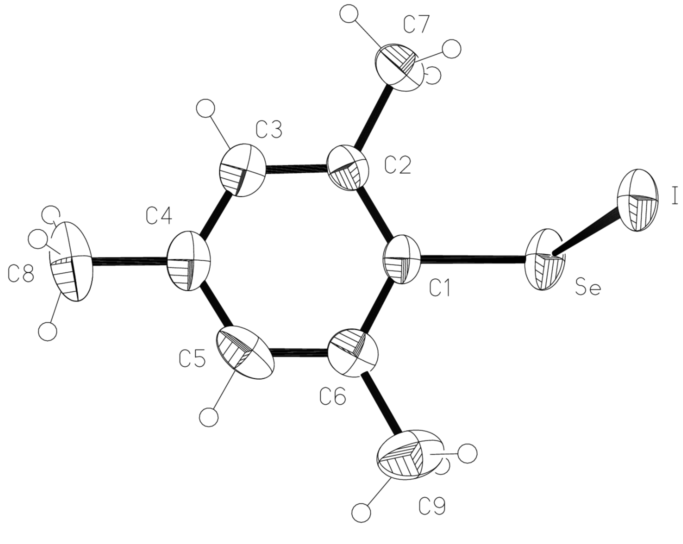


Figure 1
The molecule of the title compound in the crystal. Ellipsoids are drawn at 50% probability levels. H-atom radii are arbitrary. Only one orientation of the disordered methyl group at C8 is shown.

triisopropylphenyl) diselenide and molecular iodine [3.483 (1) Å; du Mont *et al.*, 1990]. The selenenyl iodide structure of du Mont *et al.* (1987) involves no such contacts (no Se···I < 5.7 Å).

Experimental

Dimesityl diselenide was allowed to react with the equivalent amount of elemental iodine in dichloromethane to form the title compound. Single crystals were obtained by slow evaporation of the solution.

Crystal data

$C_9H_{11}ISe$
$M_r = 325.04$
Monoclinic, $P2_1/c$
$a = 13.964\ (5)$ Å
$b = 4.606\ (2)$ Å
$c = 16.214\ (6)$ Å
$\beta = 99.87\ (3)^\circ$
$V = 1027.5\ (7)$ Å^3
$Z = 4$

$D_x = 2.101$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–11.5°
$\mu = 6.60$ mm^{-1}
$T = 178\ (2)$ K
Needle, dark red
0.60 × 0.20 × 0.05 mm

Data collection

Nicolet *P*3 diffractometer
ω scans
Absorption correction: ψ scan (*XEMP*; Siemens 1994)
$T_{\min} = 0.590$, $T_{\max} = 0.932$
5385 measured reflections
1811 independent reflections
1442 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.069$
$\theta_{\max} = 25.1^\circ$
$h = -16 \rightarrow 16$
$k = -5 \rightarrow 3$
$l = -19 \rightarrow 19$
3 standard reflections every 147 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.109$
$S = 1.19$
1811 reflections
103 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.8646P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.09$ e Å^{-3}
$\Delta\rho_{\min} = -1.32$ e Å^{-3}

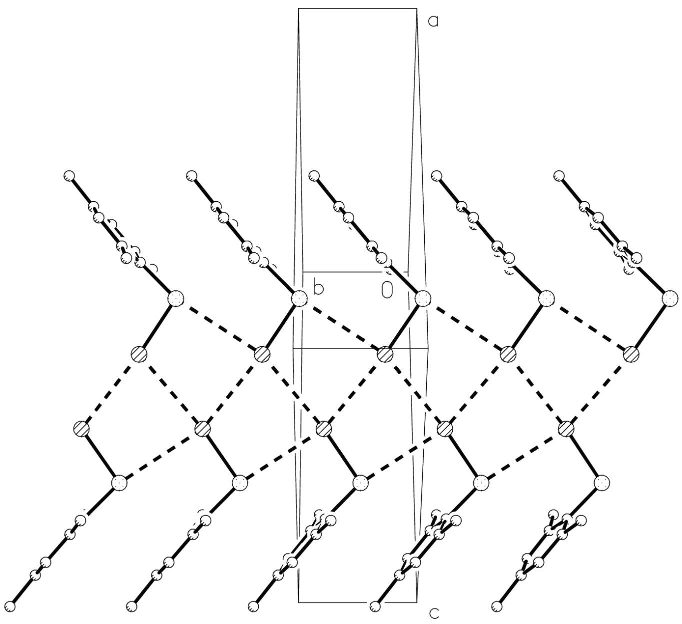


Figure 2
Packing diagram of the title compound. I···I and Se···I contacts are indicated by broken lines. H atoms have been omitted for clarity.

Table 1
Selected geometric parameters (Å, °).

I—Se	2.5360 (11)	I···I$^{\rm iii}$	3.8398 (13)
I···Se$^{\rm i}$	3.8393 (15)	Se—C1	1.923 (6)
I···I$^{\rm ii}$	3.8398 (13)	Se···I$^{\rm iv}$	3.8393 (15)
Se—I···Se$^{\rm i}$	90.13 (4)	I$^{\rm ii}$···I···I$^{\rm iii}$	73.71 (3)
Se—I···I$^{\rm ii}$	169.63 (3)	C1—Se—I	100.21 (18)
Se$^{\rm i}$···I···I$^{\rm ii}$	86.00 (3)	C1—Se···I$^{\rm iv}$	163.54 (19)
Se—I···I$^{\rm iii}$	108.72 (4)	I—Se···I$^{\rm iv}$	90.13 (4)
Se$^{\rm i}$···I···I$^{\rm iii}$	158.68 (2)		

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

H atoms at C7 and C9 were included using rigid methyl groups, and at C8 a rigid, ideally disordered methyl group with components mutually rotated by 60°; starting positions were taken from difference syntheses and then allowed to rotate but not tip. Other H atoms were included, using a riding model, starting from calculated positions. The maximum features of residual electron density are *ca* 1 Å from the Se and I atoms and can probably be ascribed to residual absorption errors.

Data collection: *P*3 *Software* (Nicolet, 1987); cell refinement: *P*3 *Software*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Mont, W.-W. du, Kubiniok, S., Peters, K. & von Schnering, H. G. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 780–781.

Mont, W.-W. du, Martens, A., Pohl, S. & Saak, W. (1990). *Inorg. Chem.* **29**, 4847–4848.

Mont, W.-W. du, Martens-von Salzen, A., Ruthe, F., Seppälä, E., Mugesh, G., Devillanova, F. A., Lippolis, V. & Kuhn, N. (2001). *J. Organomet. Chem.* **623**, 14–28.

Mugesh, G., Singh, H. B. & Butcher, R. J. (1999). *Tetrahedron Asymmetry*, **10**, 237–242.

Nicolet (1987). *P*3 *Software*. Nicolet Analytical X-ray Instruments, Madison, Wisconsin, USA.

Panda, A., Mugesh, G., Singh, H. B. & Butcher, R. J. (1993). *Organometallics*, **18**, 1986–1993.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Siemens (1994). *XEMP* and *XP* (Version 5.03). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.