Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

A T-shaped selenenyl halide

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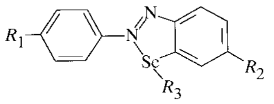
Received 20 December 1999
Accepted 2 February 2000

Data validation number: IUC0000033

The title selenenyl halide complex, 3-iodo-2-phenyl-3*H*-3-selenaindazole, $C_{12}H_9IN_2Se$, has an almost planar conformation and a nearly ideal T-shape for the Se(INC) moiety [Se—I 2.8122 (12), Se—C 1.881 (7) and Se—N2 2.051 (6) Å; C—Se—N 79.6 (3), C—Se—I 96.8 (2) and N—Se—I 176.17 (17)°]. This arrangement, together with the two selenium lone pairs, leads to a distorted trigonal-bipyrimidal geometry about the Se atom. Intermolecular interactions are largely limited to stacking forces.

Comment

X-ray structure determination of the title compound, (I), revealed the complex to be a T-shaped selenenyl iodide with the I and N atoms occupying the *trans* positions. The main nine-atom moeity (Se1, N1, N2, C1–C6) is essentially planar (r.m.s. atomic deviations of 0.030 Å), as is the phenyl ring (r.m.s. deviations of 0.013 Å), and the two are rotated relative to each other by 6.4 (3)° about the N2—C7 bond.



(I) R_1=H, R_2=H, R_3=I
(II) R_1=H, R_2=H, R_3=Cl/I
(III) R_1=H, R_2=H, R_3=Cl
(IV) R_1=Me, R_2=Me, R_3=Cl

As with similar compounds investigated, (II) and (III) (Majeed, 1999), and (IV) (Jones & Ramirez de Arellano, 1995), the Se atom is formally bonded to the halogen atom and to the aromatic C1 atom, with a secondary link to N2 which is *trans* to the halogen. The Se1—I1 bond length in (I) is 2.8122 (12) Å, which compares very favourably with the equivalent bond in the T-shaped selenium chloroiodide complex (II) of 2.837 (2) Å. The Se1—N2 distance of 2.051 (6) Å is significantly longer than the mean value of 1.83 Å for Se—N single bonds in the Cambridge Structural Database (Allen *et al.*, 1987), but again is close to the values determined for the selenenyl halides (II), (III) and (IV) which are 2.040 (6), 2.011 (5) and 2.025 (3) Å, respectively. The Se1—C1 bond length of 1.881 (7) Å is as expected for an Se—Csp^2 single covalent bond and compares favourably with the value quoted by Allen *et al.* (1987) of 1.893 Å.

In the Se1—N2 interaction, N2 donates an electron pair to Se1 to complete a trigonal-bipyrimidal type of coordination about the central Se atom. The two lone pairs and C1 are equatorial and the more electronegative N and I atoms are axial, this being in accord with the valence-shell electron-pair repulsion (VSEPR) theory (Gillespie, 1963). The angle N2—Se1—I1 is close to linear [176.17 (17)°], and the slight bending in the direction of C1 is probably due to lone-pair repulsion as suggested by Jones & Ramirez de Arellano (1995). The main deviation from an ideal T-shape is seen in the N2—Se1—C1 angle of 79.6 (3)°. This is characteristic of this group of compounds with (II), (III) and (IV) having angles of 80.3 (3), 80.8 (2) and 79.8 (2)°, respectively.

The main intermolecular forces are largely associated with stacking along the short *a* axis.

Experimental

Azobenzene (55 mmol), selenium(IV) tetrachloride (5 mmol) and anhydrous aluminium chloride (5 mmol) were heated to 433 K under argon for 3 h in 1,2-dichlorobenzene (50 ml). The reaction mixture was cooled to 353 K and methanol added until the vigorous reaction ceased. The brown precipitate of (III) which formed after cooling was recrystallized from methanol (yield 10%). (III) (1.67 mmol) was added to a solution of diiodine (1.67 mmol) in AR grade propanone (200 ml). The mixture was heated for 10 min and the solvent removed leaving a deep-red crystalline solid (yield 33%). Crystals suitable for diffraction work were obtained by slow evaporation from propanone.

Crystal data

$C_{12}H_9IN_2Se$
$M_r = 387.07$
Monoclinic, $P2_1/n$
$a = 4.845$ (2) Å
$b = 18.369$ (6) Å
$c = 13.7745$ (12) Å
$\beta = 91.77$ (2)°
$V = 1225.3$ (7) Å^3
$Z = 4$

$D_x = 2.098$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.79$–17.02°
$\mu = 5.557$ mm^{-1}
$T = 293$ (2) K
Plate, red
0.40 × 0.32 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.186$, $T_{\max} = 0.250$
2956 measured reflections
2140 independent reflections
1728 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.037$
$\theta_{\max} = 24.95$°
$h = -1 \rightarrow 5$
$k = 0 \rightarrow 21$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity decay: 1.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.122$
$S = 1.168$
2140 reflections
145 parameters
H atoms constrained

$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 5.5805P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.89$ e Å^{-3}
$\Delta\rho_{\min} = -0.82$ e Å^{-3}

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CADABS* (Gould & Smith, 1986); program(s) used to solve structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

We thank Dr Carl H. Schwalbe for his helpful discussions in preparing this document.

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