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5-Methylbenzo[*d*][2,1,3]selenadiazole

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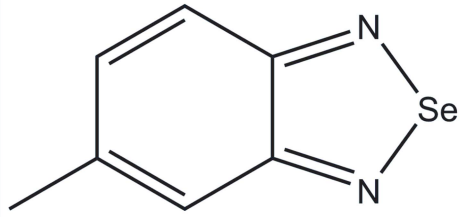
In the crystal of the title compound, $C_7H_6N_2Se$, the molecules are arranged in rods along the *b*-axis direction and form dimeric units due to intermolecular Se···N contacts of 2.982 (2) Å. The molecules are further linked by weak π–π stacking interactions between the 2,1,3-selenadiazole and six-membered aromatic rings [centroid–centroid distance = 3.8509 (11) Å and ring slippage = 1.539 (3) Å].

3D view

Chemical scheme



Structure description

Organoselenium compounds are commonly found to be efficient catalysts in a variety of organic reactions, for example, the allylic chlorination of terpenic olefins (Boualy *et al.*, 2016). Various selenoheterocyclic compounds are widely employed as ligands in asymmetric syntheses (Zhou *et al.*, 2005). They are also used as structure motifs in bioactive molecules, such as antioxidants, anti-inflammatory agents, cytokine inducers, enzyme inhibitors, antitumor and anticancer agents (Mlochowski *et al.*, 2007; Mugesh *et al.*, 2001; Osajda *et al.*, 2001).

The molecule of the title compound (Fig. 1) is almost planar [r.m.s. deviation for the non-H atoms = 0.008 Å; maximum deviation = 0.012 (2) Å for atom C5]. In the crystal, molecules are arranged in rods along the *b* axis. As found for 4,5,6,7-tetramethyl-2,1,3-benzoselenadiazole and their co-crystals, intermolecular Se···N interactions are also observed (Eichstaedt *et al.*, 2016), forming dimeric units. The Se···N distance in the title compound is 2.982 (2) Å. The dimers are further linked by weak π–π stacking interactions between the 2,1,3-selenadiazole and the six-membered aromatic rings [centroid–centroid distance = 3.8509 (11) Å and ring slippage = 1.539 (3) Å] (Fig. 2).

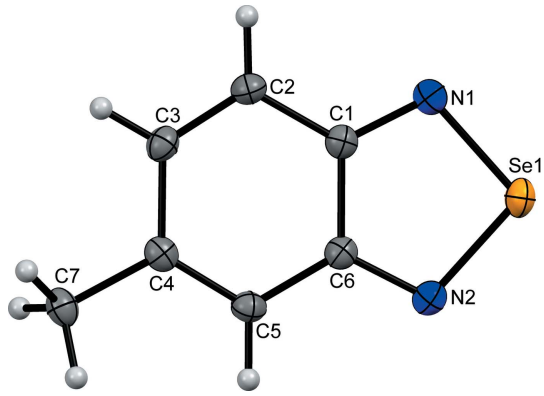


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

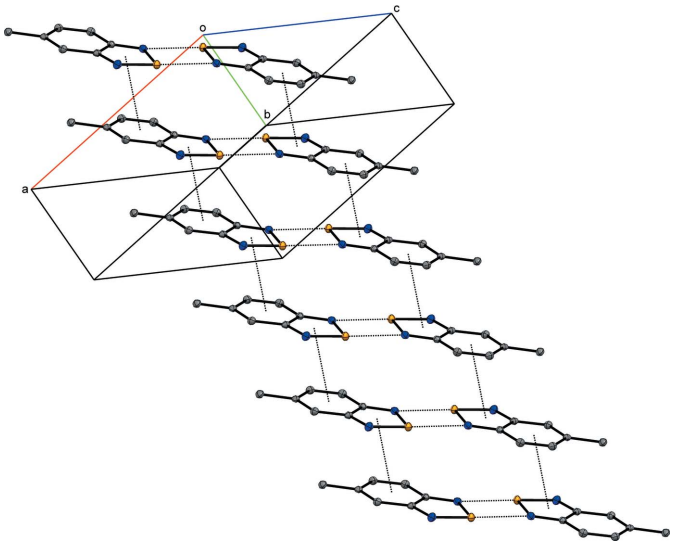


Figure 2
Packing diagram of the title compound, showing Se···N contacts and π–π stacking interactions as dotted lines. Displacement ellipsoids are drawn at the 30% probability level. For clarity, H atoms have been omitted.

Synthesis and crystallization

4-Methyl-*o*-phenylenediamine (0.25 g, 2.04 mmol) and SeO_2 (0.22 g, 1.96 mmol) were dissolved in 5 ml of *N*,*N*-dimethylformamide. After stirring for 24 h at room temperature, the reaction mixture was diluted with 30 ml of water and extracted three times with 20 ml of ethyl acetate. The organic phase was dried over $MgSO_4$ and evaporated under vacuum. The pure product was isolated by column chromatography on silica gel using hexane/ethyl acetate (90:10 *v*/*v*) as eluent (yield 81%). Colourless crystals were obtained by slow evaporation of a chloroform solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were placed geometrically and refined using a riding-atom approximation, with C—H = 0.95–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$ for methyl H atoms. A rotating model was used for the methyl groups.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_7H_6N_2Se$
M_r	197.10
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, b, c (Å)	10.2436 (8), 4.7669 (4), 14.1543 (11)
β (°)	96.6007 (16)
V (Å^3)	686.58 (10)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.38
Crystal size (mm)	0.53 × 0.13 × 0.08
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.41, 0.66
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9766, 1666, 1551
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.660
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.024, 0.066, 1.05
No. of reflections	1666
No. of parameters	92
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.54

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

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full crystallographic data

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Crystal data

$C_7H_6N_2Se$
M_r = 197.10
Monoclinic, $P2_1/c$
a = 10.2436 (8) Å
b = 4.7669 (4) Å
c = 14.1543 (11) Å
β = 96.6007 (16)°
V = 686.58 (10) Å^3
Z = 4
$F(000)$ = 384
D_x = 1.907 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6123 reflections
θ = 2.9–30.5°
μ = 5.38 mm^{-1}
T = 150 K
Needle, colourless
0.53 × 0.13 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.41, T_{max} = 0.66
9766 measured reflections
1666 independent reflections
1551 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.0°, θ_{min} = 2.0°
h = −12→13
k = −6→6
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.066
S = 1.05
1666 reflections
92 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.3807P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.65487 (18)	0.4128 (4)	0.91555 (13)	0.0175 (3)
C2	0.62888 (18)	0.5312 (4)	0.82258 (13)	0.0201 (4)
H2	0.5533	0.4764	0.7815	0.024*
C3	0.71401 (19)	0.7236 (4)	0.79367 (13)	0.0205 (4)
H3	0.6957	0.8033	0.7321	0.025*
C4	0.83091 (18)	0.8116 (4)	0.85274 (14)	0.0181 (4)
C5	0.85896 (18)	0.7016 (4)	0.94160 (14)	0.0196 (4)
H5	0.9363	0.7576	0.9806	0.024*
C6	0.77171 (18)	0.5015 (4)	0.97612 (13)	0.0179 (4)
C7	0.91893 (19)	1.0247 (4)	0.81370 (14)	0.0223 (4)
H7A	0.8820	1.2127	0.8195	0.033*
H7B	0.9256	0.9838	0.7466	0.033*
H7C	1.0065	1.0163	0.8496	0.033*
N1	0.57862 (16)	0.2254 (4)	0.95243 (12)	0.0206 (3)
N2	0.79010 (18)	0.3861 (4)	1.06228 (12)	0.0228 (3)
Se1	0.65506 (2)	0.15213 (4)	1.07010 (2)	0.02169 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0185 (8)	0.0160 (8)	0.0182 (8)	0.0025 (7)	0.0036 (7)	−0.0014 (7)
C2	0.0198 (9)	0.0220 (9)	0.0181 (8)	−0.0001 (7)	0.0000 (7)	−0.0012 (7)
C3	0.0238 (9)	0.0198 (9)	0.0177 (8)	0.0025 (7)	0.0021 (7)	−0.0001 (7)
C4	0.0187 (9)	0.0153 (8)	0.0209 (9)	0.0020 (6)	0.0052 (7)	−0.0028 (7)
C5	0.0195 (9)	0.0187 (9)	0.0204 (9)	−0.0008 (7)	0.0010 (7)	−0.0026 (7)
C6	0.0200 (8)	0.0168 (9)	0.0169 (8)	0.0020 (6)	0.0023 (6)	−0.0015 (6)
C7	0.0256 (9)	0.0171 (9)	0.0251 (9)	−0.0012 (7)	0.0068 (7)	−0.0007 (7)
N1	0.0199 (8)	0.0216 (8)	0.0206 (8)	0.0006 (6)	0.0043 (6)	−0.0006 (6)
N2	0.0249 (9)	0.0236 (8)	0.0197 (8)	−0.0009 (6)	0.0009 (6)	0.0018 (6)
Se1	0.02535 (14)	0.02166 (14)	0.01885 (13)	0.00117 (6)	0.00588 (8)	0.00329 (6)

Geometric parameters (Å, º)

C1—N1	1.332 (3)	C5—C6	1.431 (3)
C1—C2	1.429 (3)	C5—H5	0.9500
C1—C6	1.453 (3)	C6—N2	1.331 (2)
C2—C3	1.361 (3)	C7—H7A	0.9800
C2—H2	0.9500	C7—H7B	0.9800
C3—C4	1.442 (3)	C7—H7C	0.9800
C3—H3	0.9500	N1—Se1	1.7916 (17)
C4—C5	1.363 (3)	N2—Se1	1.7902 (18)
C4—C7	1.505 (3)		
N1—C1—C2	124.78 (18)	C6—C5—H5	120.1
N1—C1—C6	116.39 (17)	N2—C6—C5	124.06 (17)

C2—C1—C6	118.83 (17)	N2—C6—C1	116.14 (17)
C3—C2—C1	119.04 (18)	C5—C6—C1	119.80 (17)
C3—C2—H2	120.5	C4—C7—H7A	109.5
C1—C2—H2	120.5	C4—C7—H7B	109.5
C2—C3—C4	122.69 (18)	H7A—C7—H7B	109.5
C2—C3—H3	118.7	C4—C7—H7C	109.5
C4—C3—H3	118.7	H7A—C7—H7C	109.5
C5—C4—C3	119.75 (18)	H7B—C7—H7C	109.5
C5—C4—C7	121.81 (18)	C1—N1—Se1	106.37 (13)
C3—C4—C7	118.44 (17)	C6—N2—Se1	106.57 (13)
C4—C5—C6	119.87 (18)	N2—Se1—N1	94.54 (8)
C4—C5—H5	120.1		

N1—C1—C2—C3	179.25 (18)	C2—C1—C6—N2	179.36 (17)
C6—C1—C2—C3	−0.1 (3)	N1—C1—C6—C5	179.78 (17)
C1—C2—C3—C4	0.7 (3)	C2—C1—C6—C5	−0.8 (3)
C2—C3—C4—C5	−0.4 (3)	C2—C1—N1—Se1	−179.30 (15)
C2—C3—C4—C7	179.66 (18)	C6—C1—N1—Se1	0.1 (2)
C3—C4—C5—C6	−0.6 (3)	C5—C6—N2—Se1	−179.85 (15)
C7—C4—C5—C6	179.39 (17)	C1—C6—N2—Se1	0.0 (2)
C4—C5—C6—N2	−179.01 (18)	C6—N2—Se1—N1	0.04 (14)
C4—C5—C6—C1	1.1 (3)	C1—N1—Se1—N2	−0.09 (14)
N1—C1—C6—N2	−0.1 (3)		