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1-Methyl-2,3-dihydro-1*H*-benzimidazole-2-selone

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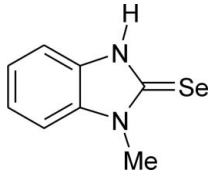
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 22.8.

The title compound $C_8H_8N_2Se$, is the product of the reaction of 2-chloro-1-methylbenzimidazole with sodium hydroselenide. The molecule is almost planar (r.m.s. deviation = 0.041 Å) owing to the presence of the long chain of conjugated bonds (Se=C–NMe–C=C–C=C–C=C–NH). The C=Se bond length [1.838 (2) Å] corresponds well to those found in the close analogs and indicates its pronounced double-bond character. In the crystal, molecules form helicoidal chains along the b axis by means of N—H···Se hydrogen bonds.

Related literature

For selones as potential antithyroid drugs, see: Taurog *et al.* (1994); Roy & Mugesh (2005, 2006); Roy *et al.* (2007, 2011). For related compounds, see: Guziec & Guziec (1994); Husebye *et al.* (1997); Aydin *et al.* (1999); Akkurt *et al.* (2004, 2011); Landry *et al.* (2006); Nakanishi *et al.* (2008); Mammadova *et al.* (2011). For hypervalent adducts of selones with dihalogens and interhalogens, see: Aragoni *et al.* (2001); Boyle & Godfrey (2001); Roy *et al.* (2011).



Experimental

Crystal data

$C_8H_8N_2Se$
M_r = 211.12
Monoclinic, $P2_1/n$
a = 9.9434 (13) Å
b = 5.8472 (8) Å
c = 13.6387 (18) Å
β = 95.360 (2)°
V = 789.50 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.69 mm^{-1}
T = 100 K
0.24 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.399, T_{max} = 0.454
9470 measured reflections
2304 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.00
2304 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*N*···Se1^{i}	0.91	2.58	3.471 (2)	168

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank Professor Abel M. Maharramov for fruitful discussions and help in this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2347).

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supplementary materials

Acta Cryst. (2012). E**68**, o1381 [doi:10.1107/S1600536812013700]

1-Methyl-2,3-dihydro-1*H*-benzimidazole-2-selone

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Comment

In the last years, the selone derivatives have attracted considerable attention owing to their antithyroid properties (Taurog *et al.*, 1994; Roy & Mugesh, 2005, 2006; Roy *et al.*, 2007, 2011) as well as selone-selenol tautomerism (Guziec & Guziec, 1994; Husebye *et al.*, 1997; Landry *et al.*, 2006; Mammadova *et al.*, 2011). Moreover, they are used as substrates in the preparation of hypervalent adducts in reactions with dihalogens and interhalogens (Aydin *et al.*, 1999; Aragoni *et al.*, 2001; Boyle & Godfrey, 2001; Akkurt *et al.*, 2004, 2011; Roy *et al.*, 2011).

The title compound - 1-methyl-2,3-dihydro-1*H*-benzimidazole-2-selone, **I** was obtained by a reaction of 2-chloro-1-methylbenzimidazole with sodium hydroselenide (Fig. 1). The molecule of **I** is almost planar (r.m.s. deviation = 0.041Å) owing to the presence of the long chain of conjugated bonds (Se═C—NMe—C═C—C═C—C═C—NH) (Fig. 2). The length of the C═Se bond (1.838 (2)Å) corresponds well to those found in the closer analogs of **I** - 1,3-dimethyl-benzimidazole-2-selone (1.825 (7)Å) (Aydin *et al.*, 1999), 1-ethyl-3-(2-phenylethyl)benzimidazole-2-selone (1.829 (3)Å) (Akkurt *et al.*, 2004) and 1,3-bis(3-phenylpropyl)-1*H*-1,3-benzimidazole-2(3*H*)-selone (1.828 (2)Å) (Akkurt *et al.*, 2011) indicating its pronounced double character.

In the crystal, molecules form helicoidal chains along the *b* axis by means of intermolecular N—H···Se^{i} hydrogen bonds (Fig. 3, Table 1). Symmetry code: (i) -*x*+1/2, *y*+1/2, -*z*+3/2.

Experimental

A solution of $NaBH_4$ (3.83 g, 100.0 mmol) in water (25 ml) was added to a suspension of selenium (3.77 g, 47.7 mmol) in water (30 ml) with stirring at room temperature under argon. After 15 min, a solution of 2-chloro-1-methylbenzimidazole (6.60 g, 39.6 mmol) in C_2H_5OH (25 ml) was added. The resulting mixture was refluxed for 5 h. At the end of the reaction the solvents were evaporated *in vacuo*, and formed precipitate was extracted with CH_2Cl_2. Then the extract was dried over $MgSO_4$. Further crystallization from CH_2Cl_2 gives the selone **I** as colourless crystals. Yield is 7.81 g (93%). *M*.p. = 470-471 K. IR(KBr), ν/cm^{-1}: 3095, 1618, 1438, 1382, 11330, 1223, 1091, 746, 709. ^{1}H NMR (*DMSO*-d_6, 600 MHz, 303 K): δ = 3.63 (s, 3H, *Me*), 7.15 (t, 1H, H6, *J* = 7.1), 7.19 (t, 1H, H5, *J* = 7.1), 7.37 (d, 1H, H4, *J* = 7.1), 7.43 (d, 1H, H7, *J* = 7.1), 13.25 (s, 1H, H3). Anal. Calc. for $C_8H_8N_2Se$: C, 45.53; H, 3.82; N, 13.27. Found: C, 45.43; H, 2.78; N, 13.19.

Refinement

The hydrogen atom of the amino group was localized in the difference-Fourier map and included in the refinement with fixed positional and isotropic displacement parameters [U_{iso}(H) = 1.2U_{eq}(N)]. The other hydrogen atoms were placed in calculated positions with C—H = 0.95–0.98Å and refined in the riding model with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C) for the methyl group and 1.2U_{eq}(C) for the other groups].

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

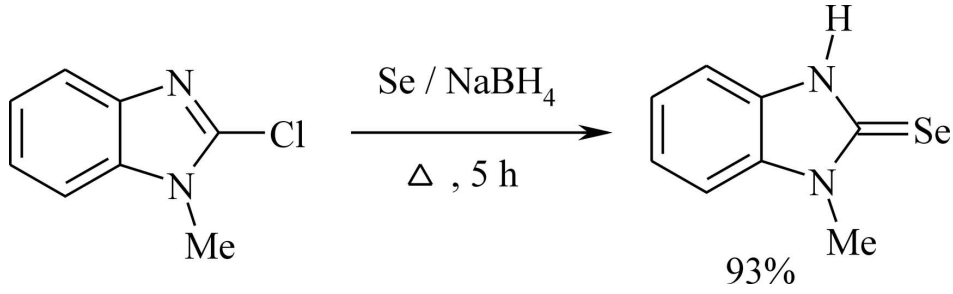

Figure 1

Reaction of 2-chloro-1-methylbenzimidazole with sodium hydroselenide.

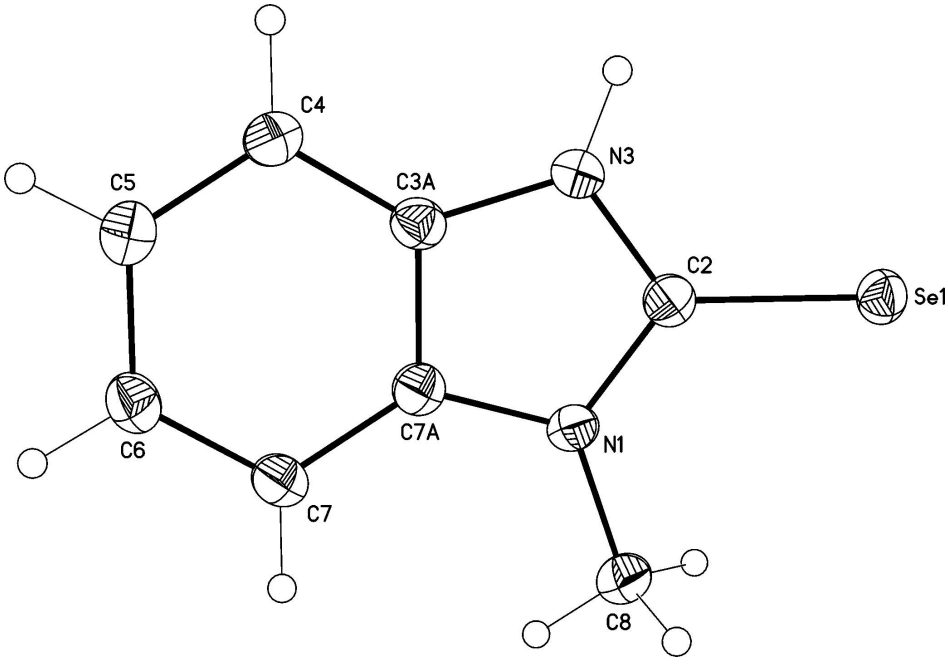

Figure 2

Molecular structure of **I** with the atom numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

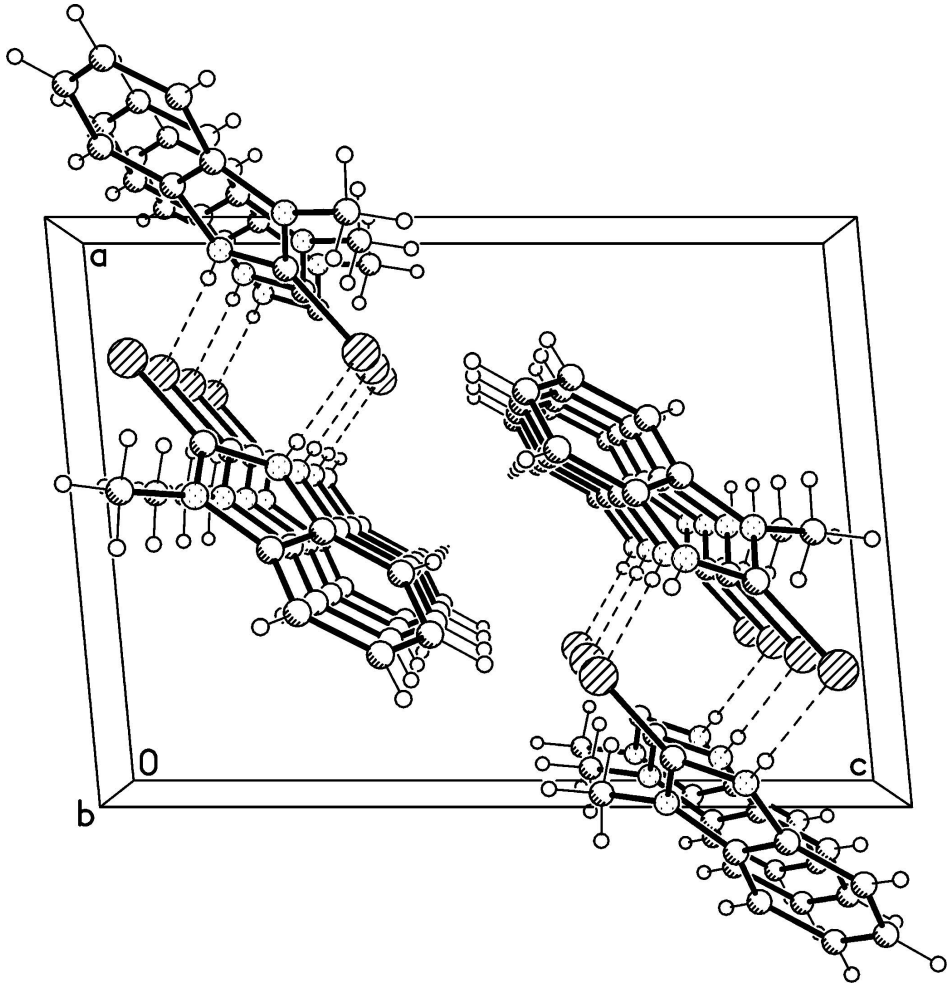

Figure 3

The helicoidal chains of **I** along the *b* axis. Dashed lines indicate the intermolecular N–H···Se hydrogen bonds.

1-Methyl-2,3-dihydro-1*H*-benzimidazole-2-selone

Crystal data

$C_8H_8N_2Se$
$M_r = 211.12$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.9434$ (13) Å
$b = 5.8472$ (8) Å
$c = 13.6387$ (18) Å
$\beta = 95.360$ (2)°
$V = 789.50$ (18) Å^3
$Z = 4$
$F(000) = 416$
$D_x = 1.776$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3916 reflections
$\theta = 2.4$–32.5°
$\mu = 4.69$ mm^{-1}
$T = 100$ K
Prism, yellow
0.24 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ– and ω–scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.399$, $T_{max} = 0.454$

9470 measured reflections
2304 independent reflections
1941 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.4°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.068$
$S = 1.00$
2304 reflections
101 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.10$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.26128 (2)	1.12016 (4)	0.878637 (16)	0.02213 (7)
N1	0.47551 (17)	0.8571 (3)	0.80856 (13)	0.0200 (3)
C2	0.3951 (2)	1.0457 (4)	0.79997 (16)	0.0203 (4)
N3	0.43002 (18)	1.1660 (3)	0.72130 (13)	0.0209 (3)
H3N	0.3892	1.2987	0.7005	0.025*
C3A	0.5294 (2)	1.0505 (4)	0.67611 (16)	0.0206 (4)
C4	0.5950 (2)	1.0978 (4)	0.59324 (16)	0.0224 (4)
H4	0.5785	1.2350	0.5568	0.027*
C5	0.6860 (2)	0.9352 (4)	0.56600 (17)	0.0242 (4)
H5	0.7320	0.9611	0.5090	0.029*
C6	0.7122 (2)	0.7339 (4)	0.62006 (16)	0.0240 (4)
H6	0.7745	0.6260	0.5986	0.029*
C7	0.6488 (2)	0.6890 (4)	0.70443 (16)	0.0220 (4)
H7	0.6674	0.5539	0.7419	0.026*
C7A	0.5571 (2)	0.8506 (3)	0.73148 (15)	0.0200 (4)
C8	0.4688 (2)	0.6817 (4)	0.88271 (17)	0.0255 (4)
H8A	0.4578	0.7533	0.9464	0.038*
H8B	0.5524	0.5919	0.8876	0.038*
H8C	0.3918	0.5810	0.8644	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.02158 (11)	0.02058 (11)	0.02486 (11)	−0.00119 (8)	0.00548 (7)	−0.00250 (8)
N1	0.0202 (8)	0.0177 (8)	0.0225 (8)	−0.0010 (6)	0.0030 (6)	0.0026 (6)
C2	0.0191 (9)	0.0196 (9)	0.0219 (9)	−0.0018 (7)	0.0011 (7)	−0.0011 (7)
N3	0.0219 (8)	0.0174 (8)	0.0236 (8)	0.0020 (6)	0.0037 (6)	0.0018 (6)
C3A	0.0195 (9)	0.0184 (9)	0.0236 (10)	−0.0006 (7)	0.0007 (7)	0.0000 (7)
C4	0.0227 (9)	0.0221 (10)	0.0222 (9)	−0.0013 (8)	0.0017 (7)	0.0027 (8)
C5	0.0231 (10)	0.0272 (11)	0.0227 (10)	−0.0019 (8)	0.0037 (8)	−0.0019 (8)
C6	0.0204 (10)	0.0242 (10)	0.0274 (11)	0.0021 (8)	0.0029 (8)	−0.0023 (8)
C7	0.0200 (9)	0.0192 (9)	0.0263 (10)	0.0007 (7)	0.0005 (8)	−0.0008 (8)
C7A	0.0184 (9)	0.0199 (9)	0.0216 (9)	−0.0015 (7)	0.0009 (7)	−0.0005 (7)
C8	0.0258 (10)	0.0237 (10)	0.0275 (11)	0.0005 (8)	0.0058 (8)	0.0063 (8)

Geometric parameters (Å, º)

Se1—C2	1.838 (2)	C4—H4	0.9500
N1—C2	1.361 (3)	C5—C6	1.400 (3)
N1—C7A	1.387 (3)	C5—H5	0.9500
N1—C8	1.446 (3)	C6—C7	1.388 (3)
C2—N3	1.355 (3)	C6—H6	0.9500
N3—C3A	1.388 (3)	C7—C7A	1.387 (3)
N3—H3N	0.9090	C7—H7	0.9500
C3A—C4	1.385 (3)	C8—H8A	0.9800
C3A—C7A	1.405 (3)	C8—H8B	0.9800
C4—C5	1.387 (3)	C8—H8C	0.9800
C2—N1—C7A	109.74 (17)	C6—C5—H5	119.0
C2—N1—C8	124.74 (18)	C7—C6—C5	121.3 (2)
C7A—N1—C8	125.35 (18)	C7—C6—H6	119.4
N3—C2—N1	107.24 (18)	C5—C6—H6	119.4
N3—C2—Se1	126.35 (16)	C7A—C7—C6	116.9 (2)
N1—C2—Se1	126.40 (16)	C7A—C7—H7	121.5
C2—N3—C3A	110.20 (18)	C6—C7—H7	121.5
C2—N3—H3N	123.4	C7—C7A—N1	131.8 (2)
C3A—N3—H3N	126.3	C7—C7A—C3A	121.6 (2)
C4—C3A—N3	132.4 (2)	N1—C7A—C3A	106.60 (18)
C4—C3A—C7A	121.4 (2)	N1—C8—H8A	109.5
N3—C3A—C7A	106.12 (18)	N1—C8—H8B	109.5
C3A—C4—C5	116.8 (2)	H8A—C8—H8B	109.5
C3A—C4—H4	121.6	N1—C8—H8C	109.5
C5—C4—H4	121.6	H8A—C8—H8C	109.5
C4—C5—C6	121.9 (2)	H8B—C8—H8C	109.5
C4—C5—H5	119.0		
C7A—N1—C2—N3	−3.4 (2)	C5—C6—C7—C7A	1.1 (3)
C8—N1—C2—N3	−178.94 (19)	C6—C7—C7A—N1	178.4 (2)
C7A—N1—C2—Se1	175.35 (15)	C6—C7—C7A—C3A	0.0 (3)
C8—N1—C2—Se1	−0.2 (3)	C2—N1—C7A—C7	−175.6 (2)

N1—C2—N3—C3A	2.5 (2)	C8—N1—C7A—C7	−0.1 (4)
Se1—C2—N3—C3A	−176.20 (15)	C2—N1—C7A—C3A	2.9 (2)
C2—N3—C3A—C4	178.0 (2)	C8—N1—C7A—C3A	178.45 (19)
C2—N3—C3A—C7A	−0.7 (2)	C4—C3A—C7A—C7	−1.5 (3)
N3—C3A—C4—C5	−176.7 (2)	N3—C3A—C7A—C7	177.40 (19)
C7A—C3A—C4—C5	1.9 (3)	C4—C3A—C7A—N1	179.74 (19)
C3A—C4—C5—C6	−0.8 (3)	N3—C3A—C7A—N1	−1.3 (2)
C4—C5—C6—C7	−0.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···Se1^{i}	0.91	2.58	3.471 (2)	168

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+3/2.