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2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)-ethaneselenol

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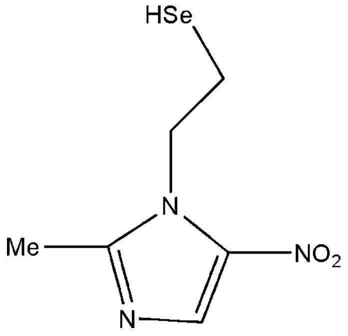
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.065; wR factor = 0.177; data-to-parameter ratio = 14.7.

In the title compound, $C_6H_9N_3O_2Se$, which is a selenol substituent derivative of metronidazole, all bond lengths and angles are normal. The imidazole ring and nitro group make a dihedral angle of 6.6 (1)°, while the N—C—C—Se torsion angle is 59.5 (8)°.

Related literature

The crystal structures of chloro- and iodometronidazole were reported by Pi *et al.* (2005) and Yang *et al.* (2005), respectively.



Experimental

Crystal data

$C_6H_9N_3O_2Se$
M_r = 234.12
Monoclinic, $P2_1/c$
a = 12.079 (2) Å
b = 11.089 (2) Å
c = 6.3800 (13) Å
β = 97.57 (3)°
V = 847.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.39 mm^{-1}
T = 293 (2) K
0.36 × 0.32 × 0.25 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.272, T_{max} = 0.352 (expected range = 0.258–0.333)
1804 measured reflections
1647 independent reflections
960 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.178
S = 1.05
1647 reflections
112 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2303).

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supplementary materials

Acta Cryst. (2007). E**63**, o4096 [**doi:10.1107/S1600536807045588**]

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethaneselenol

H. Zhao and **H.-B. Gong**

Comment

The title compound, (I) (Fig. 1), is a selenol substituent derivative of metronidazole, which is extensively used in the treatment of anaerobic infections and is under continuing investigation. Earlier, the crystal structures of chloro- and iodometronidazole were reported by Pi *et al.* (2005) and Yang *et al.* (2005), respectively.

In (I), the imidazole ring and nitro group make a dihedral angle of 6.6 (1)°, while the torsion angle N2—C2—C1—Se is 59.5 (8)°.

Experimental

Metronidazole (96%) and HSe aqua-solution (50%) wee purchased from Nanjing Chemical Company, Nanjing, P. *R*. China. TLC was run on the silica gel coated aluminium sheets (silica gel 60 GF254, E. Merk, Germany) and visualized in UV light (254 nm).

Metronidazole was dissolved in anhydrous DMF and the solution was stirred at 100 °C for 15 min. Then HSe in anhydrous DMF (20 ml) was carefully added, the mixed solution was stirred at 100 °C for 4 h. The mixture was cooled to the room temperature, and the solvent was removed under reduced pressure. The residue was suspended in EtOH and filtered, the solvent was removed under reduced pressure, and the residue was chromatographed. Elution with EtOAc gave an oil, which was recrystallized from EtOAc/petroleum ether (3/1) to give the title compound. Mp: 88.5–89.5 °C; Yield: 73%; 1H NMR (DMSO-d6): 2.50 (s, 3H, –CH3-); 3.51 (t, J = 8.68 Hz, 2H, N—CH2-); 4.61 (t, J = 8.51 Hz, 2H, –CH2-); 8.05 (s, 1H).

Refinement

H atoms attached to C atoms were refined in the riding model approximation, with C–H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The H atom attached to Se atom was located from a difference Fourier map and refined with bond restraint Se–H = 0.97 (1) Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm iso}$(Se).

Figures

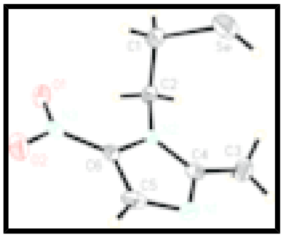

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethaneselenol

Crystal data

$C_6H_9N_3O_2Se$

$M_r = 234.12$

Monoclinic, $P2_1/c$

$a = 12.079$ (2) Å

$b = 11.089$ (2) Å

$c = 6.3800$ (13) Å

$\beta = 97.57$ (3)°

$V = 847.1$ (3) Å^3

$Z = 4$

$F_{000} = 464$

$D_x = 1.836$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 1542 reflections

$\theta = 3.9$–26.4°

$\mu = 4.40$ mm^{-1}

$T = 293$ (2) K

Prism, yellow

0.36 × 0.32 × 0.25 mm

Data collection

Bruker APEX area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.272$, $T_{max} = 0.352$

1804 measured reflections

1647 independent reflections

960 reflections with $I > 2\sigma(I)$

$R_{int} = 0.046$

$\theta_{max} = 26.0$°

$\theta_{min} = 1.7$°

$h = -14 \rightarrow 14$

$k = -13 \rightarrow 0$

$l = 0 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.065$

$wR(F^2) = 0.178$

$S = 1.05$

1647 reflections

112 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 4.5156P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.48$ e Å^{-3}

$\Delta\rho_{min} = -0.63$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Se	0.27158 (9)	0.08541 (10)	1.19615 (16)	0.0625 (4)
O1	0.0341 (5)	−0.1194 (5)	0.6752 (10)	0.0483 (15)
O2	0.1029 (5)	−0.2980 (6)	0.6766 (10)	0.0546 (17)
N1	0.4065 (6)	−0.1286 (7)	0.7038 (10)	0.0402 (17)
N2	0.2522 (5)	−0.0209 (5)	0.7252 (8)	0.0263 (14)
N3	0.1135 (6)	−0.1880 (7)	0.6843 (9)	0.0364 (16)
C1	0.1470 (6)	0.0813 (8)	0.9825 (13)	0.0408 (19)
H1B	0.1035	0.0093	0.9999	0.049*
H1C	0.0999	0.1507	0.9979	0.049*
C2	0.1822 (6)	0.0817 (7)	0.7604 (12)	0.0331 (17)
H2A	0.1159	0.0805	0.6564	0.040*
H2B	0.2225	0.1556	0.7407	0.040*
C3	0.4326 (7)	0.0927 (8)	0.7434 (14)	0.051 (2)
H3A	0.5097	0.0723	0.7408	0.076*
H3B	0.4246	0.1328	0.8740	0.076*
H3C	0.4079	0.1452	0.6270	0.076*
C4	0.3641 (6)	−0.0192 (8)	0.7257 (11)	0.0343 (19)
C5	0.3193 (7)	−0.2051 (8)	0.6905 (12)	0.040 (2)
H5A	0.3237	−0.2883	0.6758	0.048*
C6	0.2233 (6)	−0.1415 (7)	0.7020 (11)	0.0275 (16)
H1	0.3517 (14)	0.079 (7)	1.193 (12)	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se	0.0718 (8)	0.0693 (7)	0.0437 (6)	0.0085 (6)	−0.0024 (5)	−0.0086 (5)
O1	0.029 (3)	0.064 (4)	0.050 (4)	−0.005 (3)	−0.002 (3)	−0.009 (3)
O2	0.052 (4)	0.043 (4)	0.067 (4)	−0.014 (3)	0.005 (3)	−0.012 (3)
N1	0.031 (4)	0.059 (5)	0.032 (4)	0.005 (3)	0.007 (3)	−0.002 (3)
N2	0.027 (4)	0.037 (4)	0.015 (3)	0.001 (3)	0.003 (2)	0.002 (3)
N3	0.040 (4)	0.050 (5)	0.020 (3)	−0.007 (4)	0.004 (3)	−0.010 (3)
C1	0.028 (4)	0.046 (5)	0.048 (5)	0.010 (4)	0.003 (4)	−0.004 (4)

C2	0.025 (4)	0.037 (4)	0.036 (4)	0.002 (4)	−0.002 (3)	0.004 (4)
C3	0.039 (5)	0.062 (6)	0.051 (5)	−0.014 (5)	0.007 (4)	0.004 (5)
C4	0.036 (5)	0.051 (5)	0.016 (4)	−0.005 (4)	0.007 (3)	0.001 (3)
C5	0.050 (5)	0.041 (5)	0.029 (4)	0.013 (4)	0.008 (4)	−0.006 (4)
C6	0.030 (4)	0.035 (4)	0.016 (4)	−0.002 (4)	0.001 (3)	−0.002 (3)

Geometric parameters (Å, °)

Se—C1	1.893 (8)	C1—H1B	0.9700
Se—H1	0.974 (10)	C1—H1C	0.9700
O1—N3	1.220 (8)	C2—H2A	0.9700
O2—N3	1.226 (8)	C2—H2B	0.9700
N1—C4	1.331 (10)	C3—C4	1.489 (11)
N1—C5	1.346 (10)	C3—H3A	0.9600
N2—C4	1.350 (9)	C3—H3B	0.9600
N2—C6	1.385 (9)	C3—H3C	0.9600
N2—C2	1.454 (9)	C5—C6	1.367 (10)
N3—C6	1.413 (10)	C5—H5A	0.9300
C1—C2	1.532 (11)		

C1—Se—H1	133 (5)	C1—C2—H2B	109.2
C4—N1—C5	105.8 (7)	H2A—C2—H2B	107.9
C4—N2—C6	104.6 (6)	C4—C3—H3A	109.5
C4—N2—C2	126.3 (6)	C4—C3—H3B	109.5
C6—N2—C2	128.9 (6)	H3A—C3—H3B	109.5
O1—N3—O2	122.7 (7)	C4—C3—H3C	109.5
O1—N3—C6	120.0 (7)	H3A—C3—H3C	109.5
O2—N3—C6	117.3 (7)	H3B—C3—H3C	109.5
C2—C1—Se	112.0 (5)	N1—C4—N2	112.7 (7)
C2—C1—H1B	109.2	N1—C4—C3	123.4 (7)
Se—C1—H1B	109.2	N2—C4—C3	123.9 (8)
C2—C1—H1C	109.2	N1—C5—C6	109.5 (7)
Se—C1—H1C	109.2	N1—C5—H5A	125.3
H1B—C1—H1C	107.9	C6—C5—H5A	125.3
N2—C2—C1	112.2 (6)	C5—C6—N2	107.4 (7)
N2—C2—H2A	109.2	C5—C6—N3	126.9 (7)
C1—C2—H2A	109.2	N2—C6—N3	125.6 (7)
N2—C2—H2B	109.2		

C4—N2—C2—C1	−103.4 (8)	N1—C5—C6—N2	−0.5 (8)
C6—N2—C2—C1	70.6 (9)	N1—C5—C6—N3	176.9 (7)
Se—C1—C2—N2	59.5 (8)	C4—N2—C6—C5	0.1 (8)
C5—N1—C4—N2	−0.6 (8)	C2—N2—C6—C5	−174.9 (6)
C5—N1—C4—C3	−179.2 (7)	C4—N2—C6—N3	−177.3 (6)
C6—N2—C4—N1	0.3 (8)	C2—N2—C6—N3	7.7 (11)
C2—N2—C4—N1	175.5 (6)	O1—N3—C6—C5	−172.2 (7)
C6—N2—C4—C3	178.9 (7)	O2—N3—C6—C5	7.1 (11)
C2—N2—C4—C3	−5.9 (11)	O1—N3—C6—N2	4.7 (11)
C4—N1—C5—C6	0.7 (8)	O2—N3—C6—N2	−176.0 (7)

Fig. 1