

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry for (2) have been deposited with the IUCr (Reference: KA1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,4,4-Trimethyl-2*H*-1,2-benzoselenazin-3(4*H*)-one

L. DUPONT

*Laboratoire de Cristallographie,
Institut de Physique B5,
Université de Liège au Sart Tilman, B-4000 Liège,
Belgium*

P. JACQUEMIN

*Laboratoire de Chimie Organique,
Institut de Chimie B6,
Université de Liège au Sart Tilman, B-4000 Liège,
Belgium*

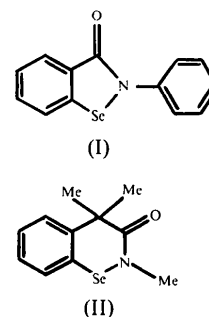
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Abstract

The title compound, $C_{11}H_{13}NOSe$, is a derivative of ebselen, an organoselenium compound which exhibits anti-inflammatory activity. The aim of the modification of the ebselen molecular structure is to increase the solubility of the compound without modifying the pharmacological properties. The Se—N bond [1.859 (5) Å] is included in a six-membered ring, which has a boat conformation. The value of the C—Se—N angle is 95.2 (2)°. The cohesion of the crystal is the result of van der Waals interactions.

Comment

Ebselen (Natterman/RP, 1981), (I), is an organoselenium compound which exhibits anti-inflammatory activity and has the ability to catalyze the reduction of hydroxyperoxidases by glutathion (Parnham & Graf, 1987). The crystal structure of ebselen has been determined previously (Dupont, Dideberg & Jacquemin, 1990). Some modifications of its molecular structure have been attempted in order to increase the solubility of the compound without modifying the pharmacological properties (Jacquemin, Christiaens, Renson, Evers & Dereu, 1992). In particular, a tetrahedral C has been introduced into the heterocycle to form the title compound (II), the crystal structure of which is described here. Fig. 1 shows the molecular structure, with the atomic numbering.



The transformation from the five-membered ring of ebselen to a six-membered heterocycle increases the N1—Se1—O1 angle by about 10°. The Se1—N1 and N1—C10 distances are shortened by about 0.04 Å. The substitution of the phenyl by a methyl group modifies the electronic delocalization around the N atom and this probably contributes to the deviations of the Se—N—(C=O) bond distances from those in ebselen. The benzene ring is planar to

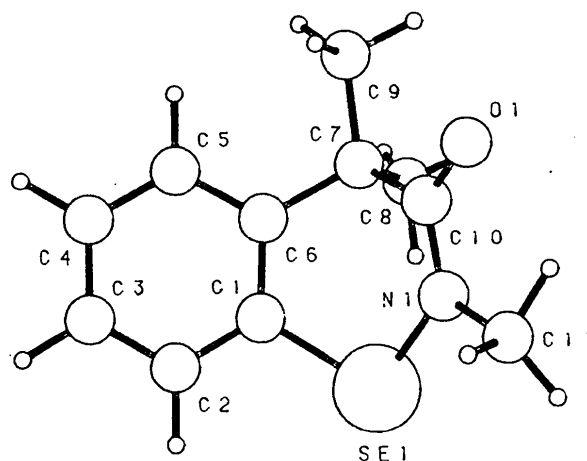


Fig. 1. View of the molecule with atom-labelling scheme.

within the 3 σ limit. The atoms C1–C7 and Se1 are coplanar with a maximum atomic deviation from the mean plane of 0.050 (5) Å (C7). The internal dihedral angles of the heterocycle are characteristic of a boat conformation [puckering parameters $\theta = 91.7$ (4)° and $\varphi = 356.2$ (5)° (Cremer & Pople, 1975)]. The cohesion of the crystal is the result of van der Waals interactions. The short binding contact Se1...O1ⁱ [symmetry code: (i) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; 3.376 (5) Å] is slightly shorter than the sum of the van der Waals radii of Se and O (≈ 3.5 Å).

Experimental

Crystal data

C₁₁H₁₃NOSe $M_r = 254.19$

Monoclinic

 $P2_1/c$ $a = 8.410$ (1) Å $b = 13.762$ (1) Å $c = 9.530$ (1) Å $\beta = 105.57$ (11)° $V = 1062.5$ (1) Å³ $Z = 4$ $D_x = 1.588$ Mg m⁻³Cu $K\alpha$ radiation $\lambda = 1.5418$ Å

Cell parameters from 25

reflections

 $\theta = 37.5$ – 42.1 ° $\mu = 5.07$ mm⁻¹ $T = 290$ K

Prism

 $0.6 \times 0.5 \times 0.3$ mm

Colourless

Crystal source: from cyclohexane solution

Data collection

Siemens diffractometer

 ω scans

Absorption correction:

empirical

 $T_{\min} = 0.14$, $T_{\max} = 0.37$

1504 measured reflections

1334 independent reflections

1162 observed reflections

 $[I \geq 2.5\sigma(I)]$ $R_{\text{int}} = 0.02$ $\theta_{\text{max}} = 55.0$ ° $h = -8 \rightarrow 8$ $k = 0 \rightarrow 14$ $l = 0 \rightarrow 10$

2 standard reflections

frequency: 60 min

intensity variation: 4–8%

Refinement

Refinement on F^2 $R = 0.075$ $wR = 0.070$ $S = 0.63$

1162 reflections

138 parameters

Only H-atom U 's refined $w = 1/[\sigma^2(F_o) + 0.02557F_o^2]$ $(\Delta/\sigma)_{\text{max}} = 0.1$ $\Delta\rho_{\text{max}} = 0.5$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.5$ e Å⁻³

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{eq}
C1	-0.0272 (6)	0.3096 (4)	0.1274 (6)	3.2 (1)
C2	-0.1914 (8)	0.2790 (5)	0.0879 (7)	4.0 (2)
C3	-0.3060 (7)	0.3295 (5)	0.1444 (6)	4.0 (2)
C4	-0.2556 (7)	0.4053 (5)	0.2368 (6)	4.4 (2)
C5	-0.0932 (6)	0.4363 (4)	0.2733 (6)	3.7 (2)
C6	0.0246 (6)	0.3902 (4)	0.2168 (5)	2.8 (1)
C7	0.2050 (6)	0.4268 (4)	0.2503 (5)	2.9 (1)
C8	0.2393 (3)	0.4722 (5)	0.1128 (7)	4.3 (2)

C9	0.2448 (8)	0.5027 (5)	0.3712 (7)	4.6 (2)
C10	0.3214 (7)	0.3386 (4)	0.2995 (6)	3.1 (1)
C11	0.4083 (8)	0.1732 (5)	0.2662 (7)	4.4 (2)
N1	0.3040 (7)	0.2607 (3)	0.2153 (6)	3.6 (2)
O1	0.4333 (5)	0.3414 (3)	0.4176 (4)	4.3 (1)
Se1	0.1261 (1)	0.2398 (1)	0.0537 (1)	4.1 (1)

Table 2. Geometric parameters (Å, °)

C2—C1	1.395 (9)	C8—C7	1.547 (8)
C6—C1	1.396 (8)	C9—C7	1.523 (8)
Se1—C1	1.888 (5)	C10—C7	1.552 (7)
C3—C2	1.408 (9)	N1—C10	1.323 (7)
C4—C3	1.358 (8)	O1—C10	1.259 (6)
C5—C4	1.399 (8)	N1—C11	1.491 (7)
C7—C6	1.548 (7)	Se1—N1	1.859 (5)
C6—C1—C2	121.9 (5)	C9—C7—C8	108.1 (5)
Se1—C1—C2	118.1 (4)	C10—C7—C6	108.2 (4)
Se1—C1—C6	120.0 (4)	C10—C7—C8	109.0 (5)
C3—C2—C1	118.5 (6)	C10—C7—C9	108.4 (4)
C4—C3—C2	120.1 (5)	N1—C10—C7	119.5 (5)
C5—C4—C3	121.0 (6)	O1—C10—C7	120.4 (4)
C6—C5—C4	121.1 (5)	O1—C10—N1	120.1 (5)
C5—C6—C1	117.2 (5)	C11—N1—C10	119.8 (5)
C7—C6—C1	120.8 (5)	Se1—N1—C10	124.2 (4)
C7—C6—C5	121.9 (5)	Se1—N1—C11	114.6 (4)
C8—C7—C6	110.6 (4)	N1—Se1—C1	95.2 (2)
C9—C7—C6	112.5 (5)		
C1—C6—C7—C10	-49.5 (5)	C10—N1—Se1—C1	-30.1 (4)
C6—C7—C10—N1	54.0 (6)	N1—Se1—C1—C6	32.9 (4)
C7—C10—N1—Se1	-10.6 (6)	Se1—C1—C6—C7	3.2 (5)

Data collection and reduction: Siemens software. Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *SHELX76* (Sheldrick, 1976). Program used to calculate Cremer & Pople (1975) parameters: *PUCK2* (Luger & Bülow, 1983). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: PA1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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