

N(1)—C(1)	1.476 (4)	C(8)—C(9)	1.518 (5)
N(1)—C(4)	1.333 (5)	C(8)—C(10)	1.528 (5)
N(2)—C(8)	1.483 (5)		
C(4)—O(2)—C(5)	116.6 (3)	O(2)—C(4)—N(1)	109.9 (3)
C(11)—O(4)—C(12)	117.2 (3)	C(1)—C(6)—C(7)	114.9 (3)
C(1)—N(1)—C(4)	124.1 (3)	C(6)—C(7)—C(8)	115.1 (3)
C(8)—N(2)—C(11)	124.3 (3)	N(2)—C(8)—C(7)	111.4 (3)
N(1)—C(1)—C(2)	106.3 (3)	N(2)—C(8)—C(9)	106.1 (3)
N(1)—C(1)—C(3)	109.7 (3)	N(2)—C(8)—C(10)	109.1 (3)
N(1)—C(1)—C(6)	111.7 (3)	C(7)—C(8)—C(9)	111.0 (3)
C(2)—C(1)—C(3)	108.9 (3)	C(7)—C(8)—C(10)	109.5 (3)
C(2)—C(1)—C(6)	110.6 (4)	C(9)—C(8)—C(10)	109.6 (4)
C(3)—C(1)—C(6)	109.6 (3)	O(3)—C(11)—O(4)	123.0 (3)
O(1)—C(4)—O(2)	122.7 (3)	O(3)—C(11)—N(2)	127.8 (3)
O(1)—C(4)—N(1)	127.3 (3)	O(4)—C(11)—N(2)	109.4 (3)
C(5)—O(2)—C(4)—O(1)	-0.2 (5)	C(4)—N(1)—C(1)—C(2)	-174.6 (3)
C(5)—O(2)—C(4)—N(1)	-178.2 (3)	C(4)—N(1)—C(1)—C(3)	67.9 (4)
C(12)—O(4)—C(11)—O(3)	-2.9 (5)	C(4)—N(1)—C(1)—C(6)	-53.8 (4)
C(12)—O(4)—C(11)—N(2)	177.8 (3)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N(1)—H(N1)...O(3 ⁱ)	0.86 (1)	2.171 (5)	3.027 (5)	172.6 (4)
N(2)—H(N2)...O(1 ⁱⁱ)	0.85 (1)	2.198 (5)	3.082 (5)	175.1 (4)

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$.

Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods (*MULTAN11/82*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares with weighting scheme using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAX computer. Anisotropic displacement parameters were used for all the non-H atoms. At an intermediate stage of refinement, difference Fourier maps showed maxima in positions consistent with the locations of all H atoms. The HN atoms were included in the least-squares refinement while all other H atoms were placed in their idealized positions (C—H 0.96 Å) and included as riding atoms in the structure-factor calculations, but were not refined.

The author thanks the University of Hong Kong for support of this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: HL1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1798–1801

Two Octahydroacridines

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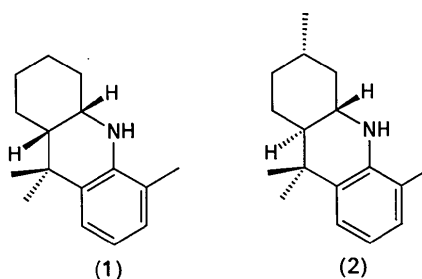
(Received 13 December 1993; accepted 21 April 1994)

Abstract

A *cis*-decalin conformation with congestion at the central heterocyclic ring is found for *cis*-5,9,9-trimethyloctahydroacridine, C₁₆H₂₃N, and a slightly twisted *trans*-decalin conformation is found for (3*RS*,4*aRS*,9*aSR*)-3,5,9,9-tetramethyloctahydroacridine, C₁₇H₂₅N.

Comment

We recently reported the convenient preparation of substituted octahydroacridine derivatives by intramolecular Lewis acid-catalyzed hetero-Diels–Alder reaction of *N*-arylimines, which are tethered to an alkene moiety (Laschat & Lauterwein, 1993). The *cis/trans* ratio was found to be mainly dependent on the substituents at C(3) of the octahydroacridine system. Determination of the stereochemistry of the ring fusion by NMR experiments led to the conclusion that the *cis* isomer prefers a *cis*-decalin conformation, whereas the *trans*-decalin conformation is favoured by the *trans* isomer. This assumption is now confirmed by an X-ray crystallographic analysis of *cis*-5,9,9-trimethyloctahydroacridine, (1) (Fig. 1), and (3*RS*,4*aRS*,9*aSR*)-3,5,9,9-tetramethyloctahydroacridine, (2) (Fig. 2). In addition, the relative *cis* configuration between H(3) and H(4*a*) in (2) can be established from the X-ray data.



Comparison of the important structural features of (1) and (2) with those of tricarbonyl(*trans*-3,3,5,9,9-pentamethyloctahydroacridine)chromium (Laschat, Noe, Riedel & Krüger, 1993) shows the following differences. The aromatic bond lengths of (1) and (2) are very

similar, except for C(8a)—C(10a) and C(5)—C(10a), which are 0.02–0.03 Å longer than the other aromatic bonds. However, all aromatic bonds of (1) and (2) are 0.02–0.03 Å shorter than the corresponding bonds of the *trans* chromium complex, which is probably the result of an increased bond order resulting from the absence of the electron-withdrawing Cr(CO)₃ fragment.

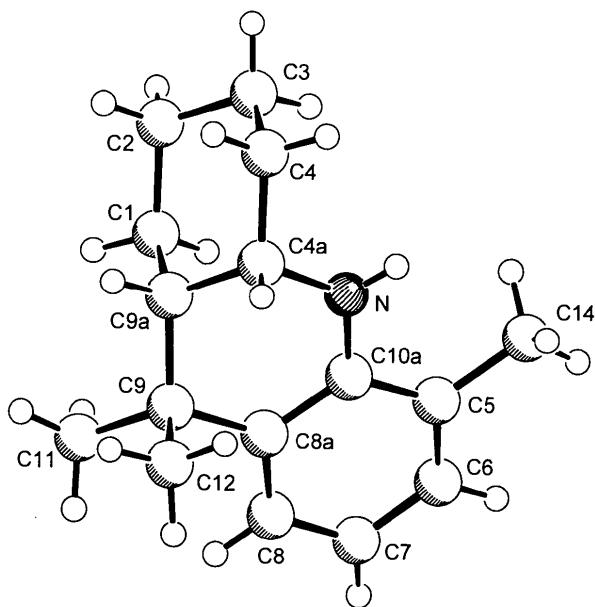


Fig. 1. SCHAKAL (Keller, 1992) plot of (1) with atomic numbering scheme.

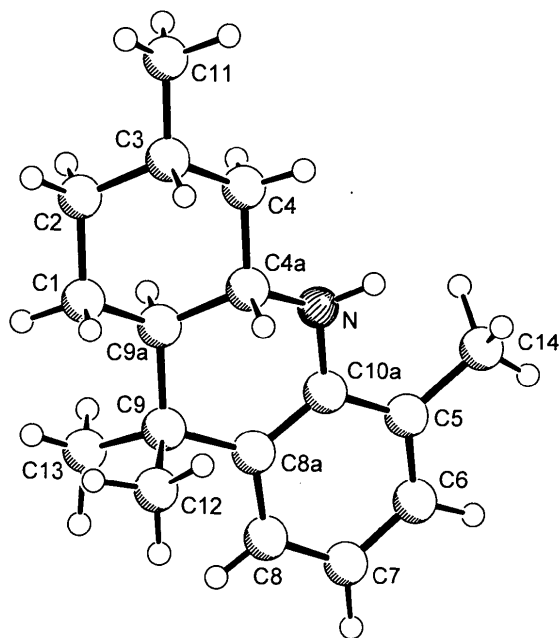


Fig. 2. SCHAKAL (Keller, 1992) plot of (2) with atomic numbering scheme.

Experimental

Compound (1)

Crystal data

C₁₆H₂₃N
M_r = 229.35
 Monoclinic
*P*2₁/*n*
a = 10.058 (1) Å
b = 10.154 (1) Å
c = 13.296 (1) Å
 β = 94.80 (1)°
V = 1353.1 (2) Å³
Z = 4
D_x = 1.126 Mg m⁻³

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 9.84–18.26°
 μ = 0.06 mm⁻¹
T = 293 K
 Irregular
 0.6 × 0.6 × 0.6 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: none
 2397 measured reflections
 2294 independent reflections
 1845 observed reflections [*I* > 2 σ (*I*)]

*R*_{int} = 0.016
 θ_{\max} = 24.8°
h = -11 → 11
k = 0 → 11
l = -15 → 0
 3 standard reflections
 frequency: 120 min
 intensity variation: none

Refinement

Refinement on *F*²
R(*F*) = 0.061
wR(*F*²) = 0.196
S = 1.175
 2290 reflections
 161 parameters
 H-atom parameters calculated, except for H(N) (refined from difference Fourier maps)
 $w = 1/[\sigma^2(F_o)^2 + (0.127P)^2 + 0.2421P]$
 where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001
 $\Delta\rho_{\max}$ = 0.24 e Å⁻³
 $\Delta\rho_{\min}$ = -0.25 e Å⁻³
 Extinction correction: none
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Compound (2)

Crystal data

C₁₇H₂₅N
M_r = 243.38
 Monoclinic
*P*2₁/*n*
a = 9.016 (1) Å
b = 18.475 (2) Å
c = 9.295 (1) Å
 β = 106.85 (1)°
V = 1481.8 (3) Å³
Z = 4
D_x = 1.091 Mg m⁻³

Cu *K*α radiation
 λ = 1.54178 Å
 Cell parameters from 25 reflections
 θ = 22.37–42.10°
 μ = 0.46 mm⁻¹
T = 293 K
 Irregular
 1.4 × 0.4 × 0.1 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

*R*_{int} = 0.047
 θ_{\max} = 70.2°

ω -2 θ scans	$h = -10 \rightarrow 10$
Absorption correction:	$k = -21 \rightarrow 12$
none	$l = -10 \rightarrow 12$
2760 measured reflections	3 standard reflections
2545 independent reflections	frequency: 120 min
1991 observed reflections	intensity variation: none
[$I > 2\sigma(I)$]	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} < 0.001$
$R(F) = 0.072$	$\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.209$	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
$S = 1.035$	Extinction correction: none
2545 reflections	Atomic scattering factors
239 parameters	from <i>International Tables</i>
H-atom parameters refined,	for <i>Crystallography</i> (1992,
positions from difference	Vol. C)
Fourier map	
$w = 1/[\sigma^2(F_o^2) + (0.1312P)^2$	
$+ 0.4555P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (1)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$$

	x	y	z	U_{eq}
N	0.3330 (2)	0.4912 (2)	0.3150 (1)	0.046 (1)
C(1)	0.1373 (2)	0.6904 (2)	0.3826 (2)	0.047 (1)
C(2)	0.1941 (3)	0.7092 (3)	0.4909 (2)	0.060 (1)
C(3)	0.3182 (3)	0.6253 (3)	0.5150 (2)	0.063 (1)
C(4)	0.2892 (2)	0.4823 (2)	0.4914 (2)	0.052 (1)
C(4a)	0.2323 (2)	0.4609 (2)	0.3836 (2)	0.041 (1)
C(5)	0.4101 (2)	0.5566 (2)	0.1561 (2)	0.041 (1)
C(6)	0.3810 (2)	0.6000 (2)	0.0586 (2)	0.047 (1)
C(7)	0.2516 (2)	0.6197 (2)	0.0190 (2)	0.051 (1)
C(8)	0.1484 (2)	0.5968 (2)	0.0791 (2)	0.045 (1)
C(8a)	0.1713 (2)	0.5537 (2)	0.1777 (2)	0.038 (1)
C(9)	0.0580 (2)	0.5243 (2)	0.2443 (2)	0.042 (1)
C(9a)	0.1091 (2)	0.5461 (2)	0.3555 (2)	0.040 (1)
C(10a)	0.3045 (2)	0.5322 (2)	0.2164 (1)	0.036 (1)
C(11)	-0.0639 (2)	0.6121 (3)	0.2169 (2)	0.062 (1)
C(12)	0.0140 (2)	0.3807 (2)	0.2266 (2)	0.059 (1)
C(13)	0.5517 (2)	0.5376 (2)	0.1983 (2)	0.053 (1)

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$$

	x	y	z	U_{eq}
N	0.1021 (2)	0.5377 (1)	0.3194 (2)	0.061 (1)
C(1)	0.0319 (4)	0.3366 (1)	0.2889 (4)	0.080 (1)
C(2)	0.1839 (5)	0.3142 (2)	0.4057 (5)	0.098 (1)
C(3)	0.3181 (4)	0.3586 (2)	0.3978 (4)	0.093 (1)
C(4)	0.2808 (3)	0.4391 (1)	0.4077 (3)	0.074 (1)
C(4a)	0.1320 (3)	0.4614 (1)	0.2926 (3)	0.072 (1)
C(5)	-0.0516 (3)	0.6445 (1)	0.2394 (3)	0.064 (1)
C(6)	-0.1914 (4)	0.6753 (2)	0.1643 (3)	0.083 (1)
C(7)	-0.3201 (4)	0.6339 (2)	0.1000 (4)	0.103 (1)
C(8)	-0.3068 (4)	0.5596 (2)	0.1112 (4)	0.095 (1)
C(8a)	-0.1686 (3)	0.5249 (1)	0.1833 (2)	0.065 (1)
C(9)	-0.1600 (3)	0.4418 (1)	0.1891 (2)	0.066 (1)
C(9a)	-0.0032 (3)	0.4165 (1)	0.3017 (3)	0.070 (1)
C(10a)	-0.0390 (3)	0.5681 (1)	0.2462 (2)	0.057 (1)
C(11)	0.4651 (5)	0.3383 (2)	0.5136 (5)	0.112 (1)
C(12)	-0.1838 (6)	0.4140 (2)	0.0274 (3)	0.097 (1)
C(13)	-0.2910 (4)	0.4127 (2)	0.2472 (4)	0.083 (1)
C(14)	0.0859 (4)	0.6903 (1)	0.3138 (4)	0.080 (1)

Table 3. Selected geometric parameters (\AA , $^\circ$) for (1)

N—C(10a)	1.377 (3)	N—C(4a)	1.470 (3)
C(1)—C(9a)	1.522 (3)	C(1)—C(2)	1.538 (5)
C(2)—C(3)	1.481 (5)	C(3)—C(11)	1.493 (5)
C(3)—C(4)	1.533 (4)	C(4)—C(4a)	1.511 (4)
C(4a)—C(9a)	1.497 (4)	C(5)—C(6)	1.374 (4)
C(5)—C(10a)	1.415 (3)	C(5)—C(14)	1.494 (4)
C(6)—C(7)	1.373 (4)	C(7)—C(8)	1.380 (5)
C(8)—C(8a)	1.389 (4)	C(8a)—C(10a)	1.396 (3)
C(8a)—C(9)	1.537 (3)	C(9)—C(13)	1.531 (4)
C(9)—C(12)	1.543 (3)	C(9)—C(9a)	1.567 (4)
C(10a)—N—C(4a)	119.8 (2)	C(9a)—C(1)—C(2)	111.6 (2)
C(3)—C(2)—C(1)	113.0 (3)	C(2)—C(3)—C(11)	113.2 (3)
C(2)—C(3)—C(4)	109.7 (3)	C(11)—C(3)—C(4)	111.3 (3)
C(4a)—C(4)—C(3)	112.8 (2)	N—C(4a)—C(9a)	109.3 (2)
N—C(4a)—C(4)	108.1 (2)	C(9a)—C(4a)—C(4)	112.2 (2)
C(6)—C(5)—C(10a)	119.0 (2)	C(6)—C(5)—C(14)	121.1 (2)
C(10a)—C(5)—C(14)	119.9 (2)	C(7)—C(6)—C(5)	121.7 (3)
C(6)—C(7)—C(8)	118.5 (3)	C(7)—C(8)—C(8a)	122.8 (3)
C(8)—C(8a)—C(10a)	117.5 (2)	C(8)—C(8a)—C(9)	120.5 (2)
C(10a)—C(8a)—C(9)	122.0 (2)	C(13)—C(9)—C(8a)	109.1 (2)
C(13)—C(9)—C(12)	108.8 (2)	C(8a)—C(9)—C(12)	107.8 (2)
C(13)—C(9)—C(9a)	107.4 (2)	C(8a)—C(9)—C(9a)	110.4 (2)
C(12)—C(9)—C(9a)	113.2 (3)	C(4a)—C(9a)—C(1)	110.0 (2)
C(4a)—C(9a)—C(9)	112.8 (2)	C(1)—C(9a)—C(9)	113.9 (2)
N—C(10a)—C(8a)	121.0 (2)	N—C(10a)—C(5)	118.7 (2)
C(8a)—C(10a)—C(5)	120.4 (2)		

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

N—C(10a)	1.383 (3)	N—C(4a)	1.452 (3)
C(1)—C(2)	1.515 (3)	C(1)—C(9a)	1.530 (3)
C(2)—C(3)	1.522 (4)	C(3)—C(4)	1.509 (4)
C(4)—C(4a)	1.514 (3)	C(4a)—C(9a)	1.531 (3)
C(5)—C(6)	1.377 (3)	C(5)—C(10a)	1.406 (3)
C(5)—C(13)	1.499 (3)	C(6)—C(7)	1.378 (3)
C(7)—C(8)	1.381 (3)	C(8)—C(8a)	1.384 (3)
C(8a)—C(10a)	1.411 (3)	C(8a)—C(9)	1.529 (3)
C(9)—C(11)	1.535 (3)	C(9)—C(12)	1.537 (3)
C(9)—C(9a)	1.540 (3)		
C(10a)—N—C(4a)	124.0 (2)	C(2)—C(1)—C(9a)	113.1 (2)
C(1)—C(2)—C(3)	111.5 (2)	C(4)—C(3)—C(2)	110.7 (2)
C(3)—C(4)—C(4a)	112.7 (2)	N—C(4a)—C(9a)	109.6 (2)
N—C(4a)—C(9a)	108.6 (2)	C(4)—C(4a)—C(9a)	112.6 (2)
C(6)—C(5)—C(10a)	118.9 (2)	C(6)—C(5)—C(13)	121.0 (2)
C(10a)—C(5)—C(13)	120.2 (2)	C(5)—C(6)—C(7)	121.7 (2)
C(6)—C(7)—C(8)	119.1 (2)	C(7)—C(8)—C(8a)	121.9 (2)
C(8)—C(8a)—C(10a)	118.1 (2)	C(8)—C(8a)—C(9)	122.5 (2)
C(10a)—C(8a)—C(9)	119.3 (2)	C(8a)—C(9)—C(11)	111.6 (2)
C(8a)—C(9)—C(12)	108.4 (2)	C(11)—C(9)—C(12)	107.5 (2)
C(8a)—C(9)—C(9a)	108.8 (2)	C(11)—C(9)—C(9a)	109.9 (2)
C(12)—C(9)—C(9a)	110.6 (2)	C(1)—C(9a)—C(4a)	110.7 (2)
C(1)—C(9a)—C(9)	113.8 (2)	C(4a)—C(9a)—C(9)	110.6 (2)
N—C(10a)—C(5)	119.2 (2)	N—C(10a)—C(8a)	120.5 (2)
C(5)—C(10a)—C(8a)	120.3 (2)		

The positions of the H atoms of (1) could be found from difference Fourier maps. Refinement resulted in slightly deformed arrangements. Thus, for final refinement, H atoms were set at calculated positions. All calculations were performed on a PC486 with *SHELXS86* (Sheldrick, 1985) and *SHELXL93* (Sheldrick, 1993). Molecular graphics were obtained with *SCHAKAL92* (Keller, 1992).

Generous financial support by the Alfried von Bohlen und Halbach-Stiftung is gratefully acknowledged. SL thanks the Wissenschaftsministerium Nordrhein-Westfalen for a Lise-Meitner fellowship.

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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Acta Cryst. (1994). **C50**, 1801–1802

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

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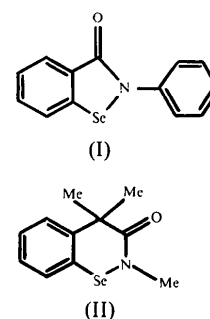
(Received 15 October 1993; accepted 8 February 1994)

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

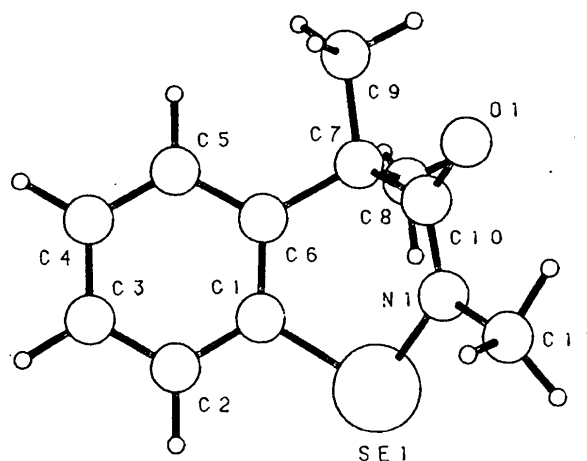


Fig. 1. View of the molecule with atom-labelling scheme.