N(1)—O(1)—N(2)	113.3 (1)	C(3)—C(4)—C(5)	119.5 (2)
O(1)—N(1)—C(2)	104.1 (1)	C(4)—C(5)—C(6)	120.3 (2)
O(1)—N(2)—C(3)	104.0 (2)	N(4)—C(6)—C(1)	118.9 (1)
O(2)—N(3)—O(3)	125.6 (2)	N(4)—C(6)—C(5)	116.7 (2)
O(2)—N(3)—C(4)	117.4 (2)	C(1)—C(6)—C(5)	124.4 (1)
O(3)—N(3)—C(4)	117.0 (2)	O(6)—C(7)—C(1)	119.1 (2)
O(4)—N(4)—O(5)	124.9 (1)	O(6)—C(7)—C(8)	124.2 (2)
O(4)—N(4)—C(6)	117.8 (1)	C(1)—C(7)—C(8)	116.0 (1)
O(5)—N(4)—C(6)	117.3 (2)	O(7)—C(8)—C(7)	114.2 (1)
C(2)—C(1)—C(6)	114.6 (1)	O(7)—C(8)—C(9)	125.2 (2)
C(2)—C(1)—C(7)	119.3 (1)	C(7)—C(8)—C(9)	120.6 (1)
C(6)—C(1)—C(7)	125.9 (1)	C(8)—C(9)—C(10)	118.8 (2)
N(1)—C(2)—C(1)	127.6 (2)	C(8)—C(9)—C(14)	121.1 (2)
N(1)—C(2)—C(3)	109.6 (1)	C(10)—C(9)—C(14)	120.0 (1)
C(1)—C(2)—C(3)	122.8 (2)	C(9)—C(10)—C(11)	119.5 (2)
N(2)—C(3)—C(2)	109.0 (2)	C(10)—C(11)—C(12)	120.4 (2)
N(2)—C(3)—C(4)	132.9 (2)	C(11)—C(12)—C(13)	120.6 (2)
C(2)—C(3)—C(4)	118.1 (1)	C(12)—C(13)—C(14)	119.8 (2)
N(3)—C(4)—C(3)	121.0 (1)	C(9)—C(14)—C(13)	119.7 (2)
N(3)—C(4)—C(5)	119.5 (2)		

Crystals were obtained by recrystallization from acetonitrile. Data were corrected for Lorentz and polarization factors. The structure was solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and by Fourier methods. Full-matrix least-squares refinement minimized $\Sigma w|F_o - F_c|^2$. H atoms were located by difference Fourier synthesis and refined isotropically. The program system used was Enraf-Nonius *MolEN* (Fair, 1990).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71181 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1037]

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Structure of 5,10-Dihydro-3-phenylimino-1,2,4-thiaselenazolo[4,5-*b*][2,4]benzodiazepine

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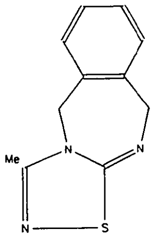
Abstract

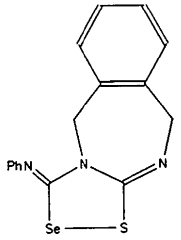
The Se—S bond length is 2.199 (2) Å. The C1—Se and C10—S bonds are essentially single bonds of lengths 1.926 (6) and 1.771 (6) Å, respectively. The C1—Se—S and C10—S—Se bond angles are 90.4 (2) and 97.4 (2)°, respectively. The N1=C1 and N3=C10 bonds are localized N=C double bonds. The diazepine ring is folded about the C2–C9 axis into two planar moieties inclined at 119.3 (1)° to one another.

Comment

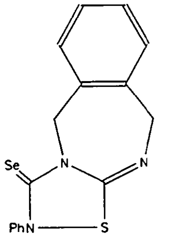
The determination of the structure of the title compound was carried out to establish whether the product from the reaction of the 1,2,4-thiadiazolo[4,5-*a*][1,3]diazepine (1) with phenyl isoselenocyanate in boiling toluene possesses structure (2) or structure (3), the reaction having taken place with elimination of acetonitrile from (1) and comcomitant addition of one molecule of phenyl isoselenocyanate (Lai, 1990, 1992; Lai & Reid, 1989).



(1)



(2)



(3)

The reaction product possesses structure (2) (title compound, Fig. 1). Compound (2) is the first reported member of a new heterocyclic system. The length of the Se—S bond is virtually the same as the accepted covalent Se—S single-bond length [2.195 (15) Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987]. The C1—Se and C10—S bonds are essentially C(sp^2)—Se and C(sp^2)—S single bonds, respectively, and C1=N1 and C10=N3 are localized C(sp^2)=N double bonds. The diazepine ring is folded about the C2–C9 axis, with the five-membered ring and atoms N1, N3, C2 and C9 lying virtually in one plane which is inclined at 119.3 (1)° to the plane of the *o*-xylyl moiety.

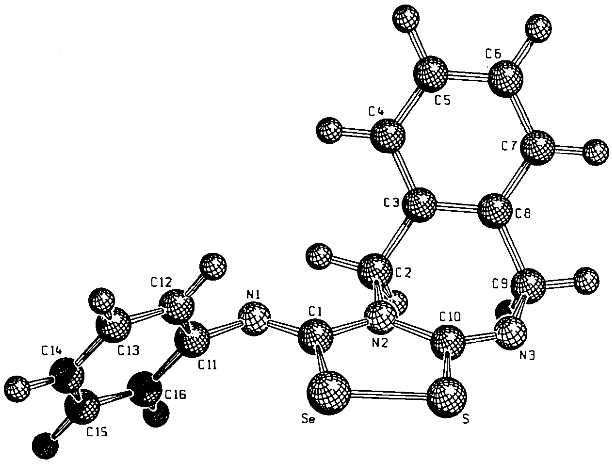


Fig. 1. Molecular structure and atomic labelling of the molecule.

Experimental

Crystal data

$C_{16}H_{13}N_3SSe$
$M_r = 358.325$
Monoclinic
$P2_1/c$
$a = 10.1029$ (7) Å
$b = 14.5274$ (9) Å
$c = 10.8606$ (6) Å
$\beta = 109.922$ (5)°
$V = 1498.6$ Å^3
$Z = 4$

$D_x = 1.588$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\mu = 2.541$ mm^{-1}
$T = 298$ K
Needle
1.3 × 0.3 × 0.2 mm
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
2709 measured reflections
2104 independent reflections
1727 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int} = 0.0327$
$\theta_{\max} = 40.0°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 12$
2 standard reflections
frequency: 60 min
intensity variation: 26.9% (linear correction)

Refinement

Refinement on F
Final $R = 0.0431$
$wR = 0.0349$
1727 reflections
191 parameters

$w = 1.5183/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.162$
$\Delta\rho_{\max} = 0.3139$ e Å^{-3}
$\Delta\rho_{\min} = -0.6066$ e Å^{-3}

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Se	0.2393 (1)	0.0216	0.1312 (1)	0.045 (0)
S	0.1142 (2)	0.1435 (1)	0.0420 (2)	0.048 (0)
N1	0.2841 (5)	−0.0968 (3)	−0.0584 (5)	0.040 (1)
N2	0.1353 (4)	0.0287 (3)	−0.1434 (4)	0.036 (1)
N3	0.0016 (5)	0.1689 (3)	−0.2042 (5)	0.042 (1)
C1	0.2226 (6)	−0.0247 (4)	−0.0396 (5)	0.036 (1)
C2	0.1119 (6)	−0.0045 (4)	−0.2794 (5)	0.045 (2)
C3	−0.0402 (6)	−0.0132 (5)	−0.3551 (5)	0.040 (2)
C4	−0.1045 (9)	−0.0993 (5)	−0.3904 (6)	0.055 (2)
C5	−0.2464 (9)	−0.1047 (6)	−0.4643 (7)	0.072 (3)
C6	−0.3285 (8)	−0.0268 (7)	−0.5038 (7)	0.068 (2)
C7	−0.2659 (7)	0.0590 (5)	−0.4665 (6)	0.055 (2)
C8	−0.1220 (7)	0.0658 (4)	−0.3927 (6)	0.041 (2)
C9	−0.0529 (7)	0.1579 (4)	−0.3480 (6)	0.049 (2)
C10	0.0771 (6)	0.1117 (4)	−0.1238 (6)	0.034 (2)
C11	0.3665 (6)	−0.1466 (4)	0.0539 (6)	0.037 (2)
C12	0.3038 (6)	−0.1951 (4)	0.1297 (6)	0.043 (2)
C13	0.3856 (7)	−0.2478 (4)	0.2357 (6)	0.049 (2)
C14	0.5299 (7)	−0.2519 (4)	0.2611 (6)	0.056 (2)
C15	0.5902 (7)	−0.2046 (5)	0.1840 (7)	0.059 (2)
C16	0.5102 (6)	−0.1520 (4)	0.0803 (6)	0.049 (2)

Table 2. *Geometric parameters* (Å, °)

S—Se	2.199 (2)	C1—Se	1.926 (6)
C10—S	1.771 (6)	C1—N1	1.269 (7)
C11—N1	1.419 (7)	C1—N2	1.404 (7)
C2—N2	1.493 (6)	C10—N2	1.390 (7)
C9—N3	1.476 (7)	C10—N3	1.257 (7)
C3—C2	1.481 (7)	C4—C3	1.400 (8)
C8—C3	1.392 (8)	C5—C4	1.387 (9)
C6—C5	1.382 (10)	C7—C6	1.394 (9)
C8—C7	1.405 (8)	C9—C8	1.511 (8)
C12—C11	1.389 (7)	C16—C11	1.383 (7)
C13—C12	1.395 (8)	C14—C13	1.390 (8)
C15—C14	1.375 (9)	C16—C15	1.373 (8)
C1—Se—S	90.4 (2)	C10—S—Se	97.4 (2)
C11—N1—C1	117.4 (5)	C2—N2—C1	117.4 (5)
C10—N2—C1	122.6 (5)	C10—N2—C2	119.9 (4)
C10—N3—C9	125.7 (5)	N1—C1—Se	123.8 (4)
N2—C1—Se	113.9 (4)	N2—C1—N1	122.3 (5)
C3—C2—N2	111.2 (4)	C4—C3—C2	121.7 (6)
C8—C3—C2	119.5 (6)	C8—C3—C4	118.8 (6)
C5—C4—C3	120.1 (7)	C6—C5—C4	121.8 (7)
C7—C6—C5	118.5 (7)	C8—C7—C6	120.5 (6)
C7—C8—C3	120.3 (6)	C9—C8—C3	118.2 (6)
C9—C8—C7	121.4 (6)	C8—C9—N3	113.7 (5)
N2—C10—S	115.3 (4)	N3—C10—S	113.8 (4)
N3—C10—N2	130.9 (5)	C12—C11—N1	121.0 (5)
C16—C11—N1	118.4 (5)	C16—C11—C12	120.5 (5)
C13—C12—C11	120.3 (6)	C14—C13—C12	118.3 (6)
C15—C14—C13	120.6 (6)	C16—C15—C14	121.3 (6)
C15—C16—C11	119.0 (6)		

The title compound (2) crystallized from dichloromethane-acetonitrile (1:3) as pale-orange crystals, m.p. 493–501 K (decomposition). Data collection and reduction was carried out using the standard routines supplied by Enraf-Nonius. As the crystal is a weak diffractor, useful data could only be collected over a limited θ range. A trial data collection at higher angles ($\theta > 40°$)

found no measurable reflections. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refined by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). H atoms were placed in geometrically calculated positions and allowed to refine, riding on the atoms to which they are attached, with a common isotropic displacement parameter. Fig. 1 was produced using *SCHAKAL* (Keller, 1988).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71086 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1038]

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6,6a,7,11b-Tetrahydro-6a-methyl-5-phenyl-5,7-methano-5*H*-benzo[*c*]fluorene and 9-Methyl-1,3,9-triphenylfluorene

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Abstract

The two crystalline products of the non-steam volatile fraction from the reaction of acetophenone and methanesulfonic acid have been identified, by single-crystal strucure analysis, as 6,6a,7,11b-tetrahydro-6a-methyl-5-phenyl-5,7-methano-5*H*-benzo[*c*]fluorene and 9-methyl-1,3,9-triphenylfluorene. The mechanism leading to their formation is not obvious.

Comment

Treatment of acetophenone with hot neat methanesulfonic acid at 383 K (3 h) yields a complex product mixture. Steam distillation of the product mixture followed by chromatography on silica yielded 1,1,3-trimethyl-3-phenylindan, 3-methyl-3-phenyl-1-indanone, 1-methyl-3-phenylnaphthalene and 3,3,3′,3′-tetramethyl-1,1′-spirobiindan. Chromatography on silica of the steam distillation residue gave 1,3,5-triphenylbenzene, 9-methyl-1,3,9-triphenylfluorene, 4a-methyl-1,3,9-triphenyl-4a*H*-fluorene and 6,6a,7,11b-tetrahydro-6a-methyl-5-phenyl-5,7-methano-5*H*-benzo[*c*]fluorene. 9-Methyl-1,3,9-triphenylfluorene was isolated previously among

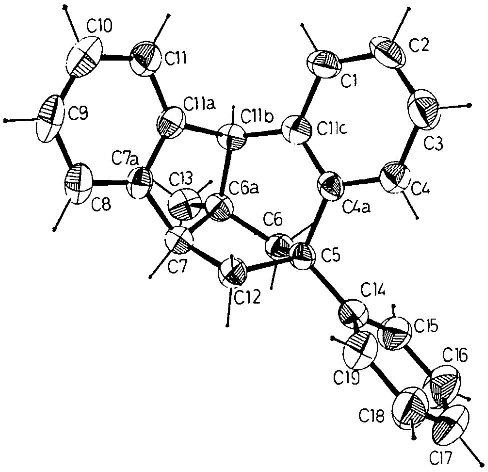

Fig. 1. Projection view of 6,6a,7,11b-tetrahydro-6a-methyl-5-phenyl-5,7-methano-5*H*-benzo[*c*]fluorene.

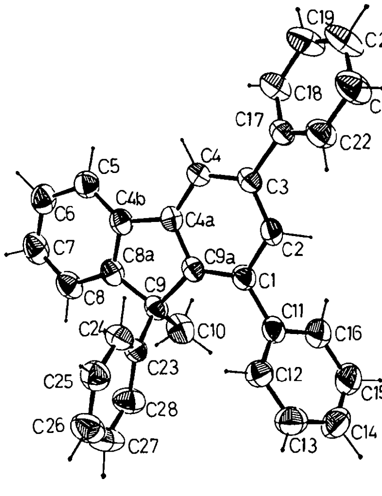

Fig. 2. Projection view of 9-methyl-1,3,9-triphenylfluorene.

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