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4-Hydroxy-4-methyl-6-phenyl-2-*p*-tolyl-5,6-dihydro-4*H*-1,3-selenazine

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.068
Data-to-parameter ratio = 19.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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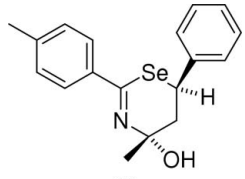
In the title crystal structure, $C_{18}H_{19}NOSe$, there are two molecules in the asymmetric unit. Pairs of molecules are linked into a centrosymmetric dimer *via* intermolecular O—H···N hydrogen bonds [O···N = 2.872 (2) and 2.893 (2) Å].

Comment

4-Hydroxy-5,6-dihydro-4*H*-1,3-selenazine derivatives have been reported to show antibacterial activity and antitumor effects (Cho *et al.*, 2000; Gutzkow *et al.*, 2003; Koketsu & Ishihara, 2003; Koketsu *et al.*, 1998; Koketsu, Ishihara *et al.*, 1999; Wu *et al.*, 1999). In order to study structure–biological activity relationships of selenazine derivatives, it is essential to determine the configuration of the selenazine ring. 4,6-Disubsitututed 4-hydroxy-5,6-dihydro-4*H*-1,3-selenazines have been obtained by reaction of primary selenoamides with α,β-unsaturated ketones in the presence of $BF_3{\cdot}Et_2O$ (Koketsu, Senda *et al.*, 1999; Koketsu *et al.*, 2001, 2003). They were obtained as diastereomers resulting from the asymmetric centers at the 4- and 6-positions of the selenazine ring. From the structure determination of the title compound, (I), the diastereomer was confirmed to have a *trans* relationship between the OH group bonded to atom C2 (or C20) and the phenyl group bonded to atom C4 (or C22).



(I)

In the crystal structure of (I) there are two independent molecules in the asymmetric unit. The molecular structures are shown in Figs. 1 and 2. There are no significant geometric differences between the molecules, except the orientations of the phenyl rings (Table 1). The selenazine ring is essentially planar except for one C atom (C3 and C21), forming a sofa conformation. Each of the independent molecules forms a centrosymmetric hydrogen-bonded dimer (Fig. 3). Hydrogen-bond distances are similar in both independent dimers (Table 2).

Experimental

4-Phenyl-3-buten-2-one (0.15 g, 1.0 mmol) was added to a stirred solution of 4-methylselenobenzamide (0.20 g, 1.0 mmol) in dry chloroform (10 ml) under an argon atmosphere. Then $BF_3{\cdot}Et_2O$ (1 equivalent) was added to the mixture. The reaction mixture was

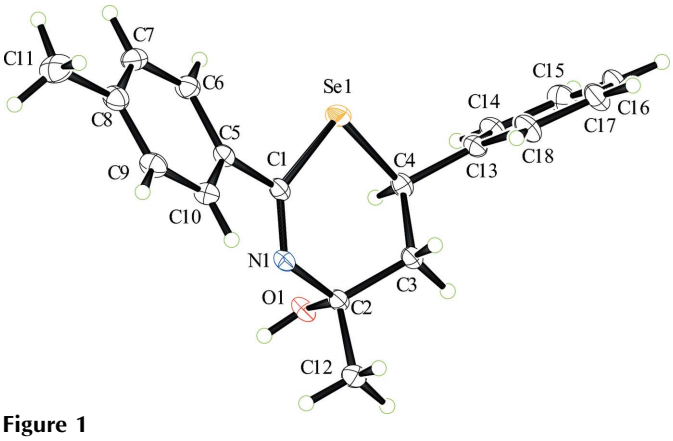


Figure 1
The molecular structure of the first of the two independent molecules in (I) which includes atom Se1. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

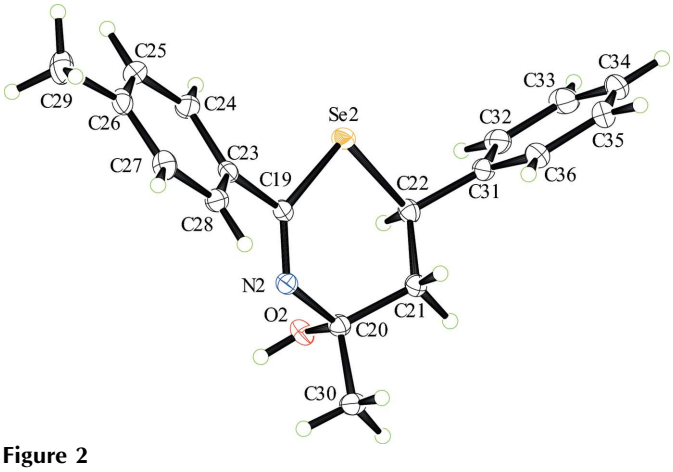


Figure 2
The molecular structure of the second independent molecule in (I) which includes atom Se2. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

stirred at 273 K for 3 h. The mixture was extracted with dichloromethane and washed with saturated sodium carbonate solution. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash chromatography on silica gel with dichloromethane to give (I) in quantitative yield (0.35 g). Crystals were prepared by recrystallization of (I) from diethyl ether/hexane (4:6) (m.p. 378.3–379.3 K). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.49 (3H, *s*, CH_3), 1.96 (1H, *t*, J = 13.6 Hz), 2.19 (1H, *dd*, J = 4.4, 13.6 Hz), 2.36 (3H, *s*, CH_3), 3.13 (1H, *br*, OH), 4.69 (1H, *dd*, J = 4.4, 13.6 Hz, C6), 7.18 (2H, *d*, J = 7.8 Hz, Ar), 7.24–7.29 (1H, *m*, Ar), 7.33–7.38 (4H, *m*, Ar), 7.66 (2H, *d*, J = 7.8 Hz, Ar).

Crystal data

$C_{18}H_{19}NOSe$
M_r = 344.30
Monoclinic, $P2_1/c$
a = 18.0846 (10) Å
b = 16.9821 (10) Å
c = 10.8354 (10) Å
β = 103.190 (10)°
V = 3239.9 (4) Å^3
Z = 8
D_x = 1.412 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13476 reflections
θ = 2.9–27.5°
μ = 2.32 mm^{-1}
T = 190 (2) K
Prism, colorless
0.32 × 0.20 × 0.10 mm

Data collection

Bruker-Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.524, T_{max} = 0.801
26451 measured reflections
7417 independent reflections
6160 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°
$h = -23 \rightarrow 23$
$k = -22 \rightarrow 20$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.068
S = 1.03
7417 reflections
387 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2 + 1.8946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Se1—C1	1.9197 (18)	Se2—C19	1.9206 (18)
Se1—C4	1.968 (2)	Se2—C22	1.9731 (19)
C1—N1	1.266 (2)	C19—N2	1.267 (2)
C1—C5	1.485 (3)	C19—C23	1.486 (3)
N1—C2	1.478 (2)	N2—C20	1.483 (2)
C2—O1	1.418 (2)	C20—O2	1.417 (2)
C2—C12	1.521 (3)	C20—C30	1.520 (3)
C2—C3	1.525 (2)	C20—C21	1.528 (2)
C3—C4	1.522 (3)	C21—C22	1.521 (3)
C4—C13	1.509 (3)	C22—C31	1.514 (3)
C1—Se1—C4	98.50 (8)	C19—Se2—C22	99.28 (8)
N1—C1—C5	119.01 (16)	N2—C19—C23	120.36 (16)
N1—C1—Se1	127.61 (15)	N2—C19—Se2	127.74 (14)
C5—C1—Se1	113.36 (13)	C23—C19—Se2	111.87 (13)
C1—N1—C2	123.50 (15)	C19—N2—C20	122.60 (16)
O1—C2—N1	108.14 (15)	O2—C20—N2	107.84 (15)
O1—C2—C12	111.18 (16)	O2—C20—C30	111.12 (16)
N1—C2—C12	106.23 (15)	N2—C20—C30	106.71 (16)
O1—C2—C3	107.03 (15)	O2—C20—C21	106.79 (15)
N1—C2—C3	114.14 (15)	N2—C20—C21	114.48 (15)
C12—C2—C3	110.16 (16)	C30—C20—C21	109.94 (16)
C4—C3—C2	113.34 (16)	C22—C21—C20	114.04 (16)
C13—C4—C3	113.66 (16)	C31—C22—C21	115.87 (16)
C13—C4—Se1	109.10 (13)	C31—C22—Se2	106.47 (12)
C3—C4—Se1	108.48 (13)	C21—C22—Se2	108.80 (13)
N1—C1—C5—C10	31.1 (3)	N2—C19—C23—C28	39.3 (3)
Se1—C1—C5—C10	−147.47 (15)	Se2—C19—C23—C28	−139.10 (15)
N1—C1—C5—C6	−147.67 (19)	N2—C19—C23—C24	−140.36 (19)
Se1—C1—C5—C6	33.8 (2)	Se2—C19—C23—C24	41.2 (2)
C3—C4—C13—C18	−53.1 (3)	C21—C22—C31—C36	−48.0 (3)
Se1—C4—C13—C18	68.1 (2)	Se2—C22—C31—C36	73.1 (2)
C3—C4—C13—C14	124.3 (2)	C21—C22—C31—C32	134.69 (19)
Se1—C4—C13—C14	−114.51 (18)	Se2—C22—C31—C32	−104.19 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···N1^{i}	0.84	2.04	2.872 (2)	173
O2—H30···N2^{ii}	0.84	2.06	2.893 (2)	175

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x, -y+1, -z+1$.

All H atoms were placed in idealized positions and treated as riding atoms, with C—H = 0.95–0.99 Å and O—H = 0.84 Å, and with U_{iso}(H) = 1.2U_{eq}(C,O) or 1.5U_{eq}(methyl C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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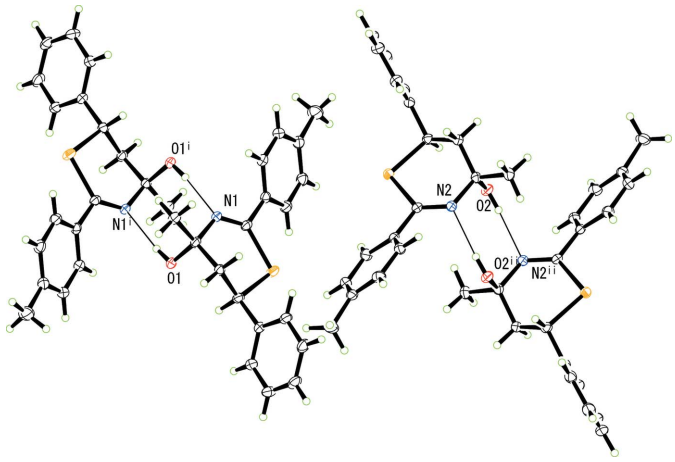

Figure 3
The hydrogen-bonded (thin lines) dimeric structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Symmetry codes are as given in Table 2.

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