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3-Methyl-*N*-(4-phenyl-1,3-diselenol-2-ylidene)-benzamide

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In the crystal structure of the title compound, $C_{17}H_{13}NOSe_2$, the exocyclic C═N double bond has a cis configuration.

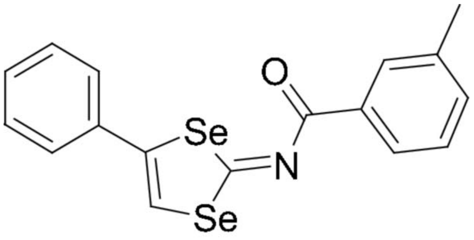
Key indicators

Single-crystal X-ray study
T = 118 K
Mean σ(C–C) = 0.009 Å
R factor = 0.071
wR factor = 0.125
Data-to-parameter ratio = 17.7

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

Comment

The title compound, (I), belongs to the class of *N*-(4-aryl-1,3-diselenol-2-ylidene)amides (Shafiee & Fanaii, 1984). The structures of the *N*-(4-aryl-1,3-diselenol-2-ylidene)amides were proved by comparison with *N*-(4-aryl-1,3-dithiol-2-ylidene)carbamic acid esters (Chauhan & McKinnon, 1976), *N*-(4-aryl-1,3-thiaselenol-2-ylidene)carbamic acid esters (Shafiee *et al.*, 1980) and their chemical transformations. 3-Methyl-*N*-(4-phenyl-1,3-diselenol-2-ylidene)benzamide, (I), was prepared by the reaction of lithium phenylethyneselenoate with 3-methylbenzoyl isoselenocyanate.



(I)

The molecular structure of (I) is shown in Fig. 1. The compound possesses an exocyclic C═N double bond with a *cis* configuration. Atoms Se1, C1, Se2, C2, C3, N1, C4, C5 and O1 are almost coplanar, with a dihedral angle between the plane of atoms Se1/C1/Se2/C2/C3 and that of N1/C4/C5/O1 of 5.4 (4)°. The Se1—C1—N1 angle is larger than the Se2—C1—N1 angle.

Experimental

To a tetrahydrofuran solution (15 ml) of phenylacetylene (110 µl, 1.0 mmol) was added *n*-BuLi (1.56 mol%, 1.2 mmol) at 273 K, and the mixture was stirred for 15 min. Elemental selenium powder (79 mg, 1 mmol) was then added to the mixture. The reaction mixture was warmed to room temperature and stirred for 1 h under an argon atmosphere. Lithium phenylethyneselenoate formed *in situ* and was then ready for further reaction. The acyl isoselenocyanate generated from potassium selenocyanate (432 mg, 3 mmol) and *m*-toluoyl chloride (190 µl, 1.5 mmol) was added to the above lithium phenylethyneselenoate at 248 K and the reaction mixture was stirred at 248 K for 2 h. The mixture was then extracted with diethyl ether and washed with water. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash chromatography on silica gel with diethyl ether–*n*-hexane (20:1) as eluent to give (I) (yield 334 mg, 82%) as a yellow solid. Crystals of the *cis*-isomer suitable for X-ray analysis were prepared by recrystallization from a solution in chloroform–hexane (10:1) (m.p. 394.1–398.3 K).

Crystal data

$C_{17}H_{13}NOSe_2$
$M_r = 405.20$
Monoclinic, $P2_1/c$
$a = 4.847$ (4) Å
$b = 24.786$ (19) Å
$c = 12.664$ (11) Å
$\beta = 103.504$ (10)°
$V = 1479$ (2) Å^3
$Z = 4$
$D_x = 1.819$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 5.00$ mm^{-1}
$T = 118$ (2) K
Prism, yellow
0.30 × 0.10 × 0.05 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: integration (*ABSCOR*; Higashi, 1999)
$T_{\min} = 0.407$, $T_{\max} = 0.845$
11879 measured reflections
3374 independent reflections
2961 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.125$
$S = 1.29$
3374 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 7.6207P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.03$ e Å^{-3}
$\Delta\rho_{\min} = -1.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Se1—C1	1.893 (6)	C2—C3	1.353 (8)
Se1—C3	1.904 (6)	C3—C12	1.472 (8)
C1—N1	1.299 (7)	N1—C4	1.386 (7)
C1—Se2	1.877 (6)	O1—C4	1.223 (7)
Se2—C2	1.866 (6)	C4—C5	1.492 (8)
C1—Se1—C3	93.9 (3)	C2—C3—Se1	117.4 (4)
N1—C1—Se2	118.4 (4)	C12—C3—Se1	117.4 (4)
N1—C1—Se1	127.4 (4)	C1—N1—C4	116.4 (5)
Se2—C1—Se1	114.2 (3)	O1—C4—N1	123.5 (5)
C2—Se2—C1	94.3 (3)	O1—C4—C5	121.4 (5)
C3—C2—Se2	120.1 (4)	N1—C4—C5	115.1 (5)
C2—C3—C12	125.2 (5)		

C-bound H atoms were placed in idealized positions and treated as riding, with C—H distances in the range 0.95–0.98 Å, and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms or $1.2U_{\rm eq}({\rm C})$ for the remaining H atoms. The highest peak is located 1.73 Å from atom Se1 and the deepest hole 1.51 Å from atom O1.

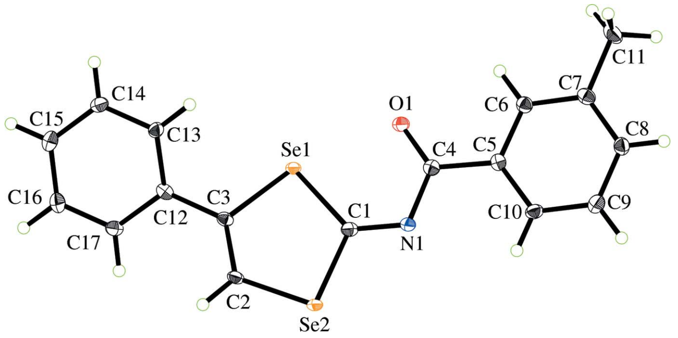


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *TEXSAN*.

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