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Monoclinic form of 7-nitro-2-phenyl-1,2-benzisoselenazol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.054
wR factor = 0.145
Data-to-parameter ratio = 12.4

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

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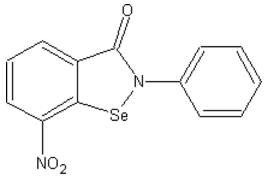
In the course of a study of 1,2-benzoselenazin-4-one heterocyclic homologues of ebselen, a well known anti-oxidizing agent, the crystal structure determination of the title compound, $C_{13}H_8N_2O_3Se$, was carried out and revealed a monoclinic polymorph of the previously reported triclinic form. In both polymorphs, the centroids of the heterocyclic rings are nearly superimposed in projection along their normal. In the title compound, the molecular planes pack along two directions.

Comment

Ebselen (Natterman/RP, 1981; Dupont *et al.*, 1990), a well known anti-inflammatory drug, exhibits some physicochemical failings, mainly a low solubility. In the course of studying new molecules of the benzo[*e*][1,2]selenazin-4-one family which could be better anti-oxidizing agents, crystals of the title compound, (I), were obtained from a chloroform solution. The structure of (I) was determined by X-ray diffraction and shown to be 7-nitro-2-phenyl-1,2-benzisoselenazol-3(2*H)*-one crystallized in a monoclinic form. The triclinic structure, (II), determined from a sample crystallized from a water–methanol solution, has been described previously [Dupont *et al.* (1988); Cambridge Structural Database (Allen, 2002) refcode JABZAN].



(I)

In (I), the maximum and minimum residuals in the final difference map are located at 1.11 and 0.86 Å, respectively, from the Se1 position. The distances and angles are quite similar to those found in (II). The most significant deviation with regard to the bond lengths is for C7—O3, *viz*. 1.213 (6) Å in (I) and 1.234 (5) Å in (II). In (I), the dihedral angle between the phenyl and heterocycle mean planes is 6.0 (3)°, whereas the corresponding value in (II) is 13.2 (1)°. The C1—C2—N1—O1 torsion angles are also slightly different, *viz*. 2.8 (6)° in (I) and 4.9 (5)° in (II). The molecular conformation is consequently more planar in the monoclinic polymorph. The Se1—O1 distances are nevertheless quite similar, *viz*. 2.562 (4) Å in (I) and 2.573 (3) Å in (II). In both polymorphs, the molecules are approximately elongated in the direction of the *c* axis. Each molecule is aligned directly above its equivalent by a $\overline{1}$ symmetry operation. The result is a stacking of almost equidistant molecular planes [alternating distances of 3.333 (5)/3.528 (5) Å in (I) and 3.373 (4)/3.510 (4) Å in (II);

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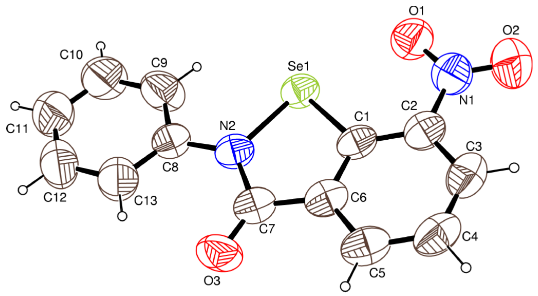

Figure 1
The molecular structure of (I), with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

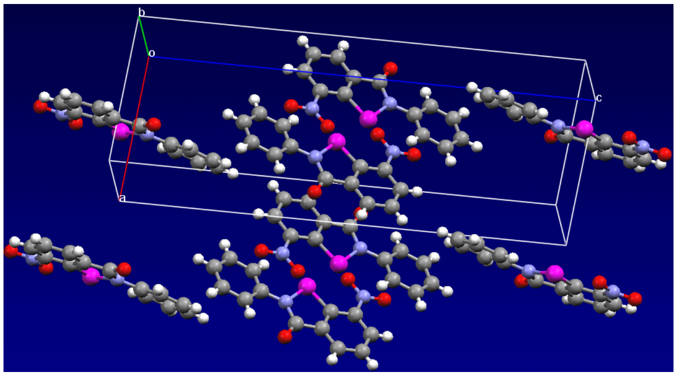

Figure 2
MERCURY (Bruno *et al.*, 2002) view of the packing of (I).

distances are those between the five-membered-ring mean planes (*P*1)], the centroids of the heterocyclic rings being nearly superimposed in projection along the stack. In (II), the normal to *P*1 is almost parallel to the *b* axis, whereas in (I), the molecular planes are packed along two directions. The angle between the corresponding *P*1 planes is 59.4 (4)°. There is no hydrogen bonding in the crystal structure of (I).

Experimental

The title compound was prepared by treatment of 2-methylseleno-3-nitrobenzanilide first with Br_2 and then with Na_2CO_3 (Messali, 2001). Red single crystals were obtained by slow evaporation of a chloroform solution.

Crystal data

$C_{13}H_8N_2O_3Se$
$M_r = 319.17$
Monoclinic, $P2_1/n$
$a = 8.069$ (1) Å
$b = 6.322$ (2) Å
$c = 23.512$ (3) Å
$\beta = 93.38$ (2)°
$V = 1197.3$ (4) Å^3
$Z = 4$
$D_x = 1.771$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 36 reflections
$\theta = 33.0–37.3°$
$\mu = 4.32$ mm^{-1}
$T = 293$ (2) K
Tablet, red
0.58 × 0.57 × 0.08 mm

Data collection

Stoe–Siemens AED four-circle diffractometer
ω scans
Absorption correction: ψ scan (*EMPIR*; Stoe & Cie, 1987)
$T_{\min} = 0.157$, $T_{\max} = 0.724$
2310 measured reflections
2146 independent reflections
1675 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 68.1°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 7$
$l = -28 \rightarrow 28$
2 standard reflections
frequency: 60 min
intensity decay: 5%

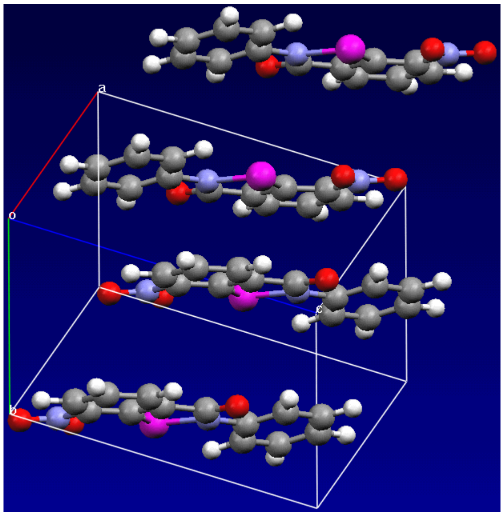

Figure 3
MERCURY (Bruno *et al.*, 2002) view of the packing of (II).

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.145$
$S = 0.99$
2146 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1196P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.05$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0082 (9)

Table 1
Selected geometric parameters (Å, °).

Se1—C1	1.852 (4)	O3—C7	1.213 (6)
Se1—N2	1.897 (4)	N1—C2	1.442 (7)
O1—N1	1.251 (6)	N2—C7	1.399 (6)
O2—N1	1.215 (6)	N2—C8	1.421 (6)
C1—Se1—N2	85.48 (17)	C8—N2—Se1	118.8 (3)
O2—N1—O1	123.5 (5)	C2—C1—Se1	126.2 (4)
O2—N1—C2	120.2 (5)	C6—C1—Se1	113.4 (3)
O1—N1—C2	116.3 (4)	O3—C7—N2	125.5 (4)
C7—N2—C8	125.9 (4)	O3—C7—C6	124.8 (4)
C7—N2—Se1	115.2 (3)	N2—C7—C6	109.7 (4)
C7—N2—C8—C13	6.3 (7)	Se1—N2—C8—C13	−174.1 (4)

All H atoms were included in the refinement in the riding-model approximation, with $U_{\rm iso}$ values fixed at $1.2U_{\rm eq}$ of the parent atom.

Data collection: *DIF*4 (Stoe & Cie, 1987); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL*97.

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