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N-(2-Bromophenyl)-1,3-selenazolo-[5,4-*b*]pyridin-2-amine

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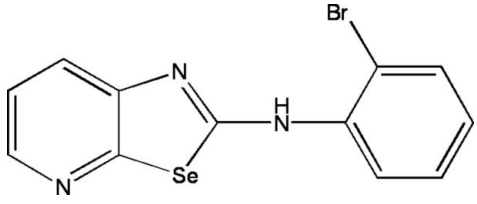
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.007 Å; R factor = 0.052; wR factor = 0.132; data-to-parameter ratio = 12.4.

The molecular structure of the title molecule, $C_{12}H_8BrN_3Se$, is built up from fused selenazolo and pyridine rings, linked to a 2-bromoaniline group. In the crystal, pairs of molecules are linked by N—H···N hydrogen bonds into dimers, forming eight-membered ring motifs.

Related literature

For the bioactivity of organoselenium compounds, see: Garud *et al.* (2007); Ling *et al.* (2010); Plamen *et al.* (2010). For crystallographic studies of selenazolo derivatives, see: Plamen *et al.* (2004).



Experimental

Crystal data

$C_{12}H_8BrN_3Se$ M_r = 353.08
Monoclinic, $P2_1/n$
a = 12.5312 (5) Å
b = 7.4562 (3) Å
c = 13.8913 (5) Å
β = 112.331 (4)°
V = 1200.60 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 7.96 mm^{-1}
T = 295 K
0.30 × 0.30 × 0.10 mm

Data collection

Agilent Xcalibur (Sapphire3, Gemini ultra) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.199, T_{max} = 0.299
4420 measured reflections
1921 independent reflections
1779 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.132
S = 1.11
1921 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −1.88 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2^{i}	1.06	1.88	2.933 (4)	174

Symmetry code: (i) $-x+1, -y, -z$.

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2639).

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supplementary materials

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N-(2-Bromophenyl)-1,3-selenazolo[5,4-*b*]pyridin-2-amine

Zhou Bo, Huang Du Shu, Liu Wei and Zhou Mei Yun

1. Comment

Since the discovery of the importance of Se as a microelement in bacteria and animals, and the function of the seleno-enzyme glutathione peroxidase (GPx) as an antioxidant, the interest in organoselenium compounds has increased significantly (Garud, *et al.* 2007; Ling, *et al.* 2010; Plamen, *et al.* 2010,2004). The design and synthesis of organoselenium compounds, especially Se-containing heterocycles, are of our current interest. The title molecule (Fig.1) is built up from two fused rings, *viz*. selenazolo and pyridine, linked to 2-bromoaniline group. In the crystal, pairs of molecules are linked by N—H—N hydrogen bonds (H—N=2.933 Å) into dimers, forming eight- membered rings motifs.

2. Experimental

To a stirred solution of *N*-phenyllformamide (10 mmol) in toluene (100 ml) in an ice bath, Et_3N (4.0 g, 40 mmol) and Se black powder were added. Then, phosgene (8 g of a 20% solution in toluene,) was added slowly over 30 min. An exothermic reaction took place. After complete addition, the suspension was heated under reflux for 10 h (TLC control). The mixture was filtered and washed with several portions of toluene, and then the filtrate was concentrated and afforded the raw isoselenocyanatobenzene. Isoselenocyanatobenzene was added to a stirred solution of 2-chloropyridin -3-amine (1.28 g, 10 mmol) in 2-propanol at room temperature, and the mixture was heated to reflux for 3 h. After filtration, the precipitate was collected as a yellow solid. The impure product was dissolved in CCl_2H_2 at room temperature. Colorless crystals suitable for X-ray analysis (90.4% yield) grew over a period of one week when the solution was exposed to the air.

3. Refinement

The structure was solved by direct methods and refined by least squares method on F2 using the *SHELXTL* program package. All atoms were refined anisotropically. Hydrogen atoms were placed at the calculated positions using a riding model with C(aromatic)-H = 0.95 Å and U_{iso}(H) = 1.2Ueq(C), and with N—H = 0.95 Å and U_{iso}(H) =1.5Ueq(N).

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO* (Agilent, 2010); data reduction: *CrysAlis PRO* (Agilent, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

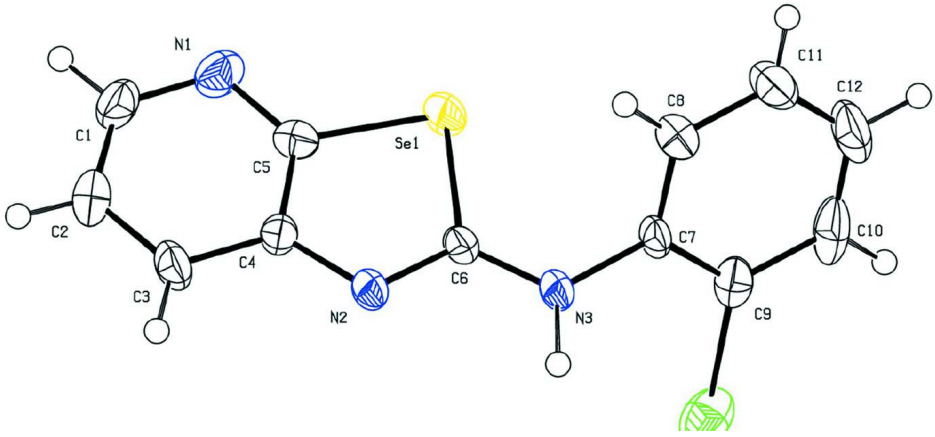

Figure 1

The molecular structure of the title compound in (I) showing the atom numbering Scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

$C_{12}H_8BrN_3Se$
$M_r = 353.08$
Monoclinic, $P2_1/n$
$a = 12.5312$ (5) Å
$b = 7.4562$ (3) Å
$c = 13.8913$ (5) Å
$\beta = 112.331$ (4)°
$V = 1200.60$ (8) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.953$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 1921 reflections
$\theta = 63.3$–$4.1°$
$\mu = 7.96$ mm^{-1}
$T = 295$ K
Prism, colorless
0.30 × 0.30 × 0.10 mm

Data collection

Agilent Xcalibur (Sapphire3, Gemini ultra) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0288 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.199$, $T_{max} = 0.299$
4420 measured reflections
1921 independent reflections
1779 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 62.8°$, $\theta_{min} = 4.1°$
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 8$
$l = -16 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.132$
$S = 1.11$
1921 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.66$ e Å^{-3}
$\Delta\rho_{min} = -1.88$ e Å^{-3}

Special details

Experimental. CrysAlisPro, Agilent Technologies, Version 1.171.34.49 (release 20-01-2011 CrysAlis171 .NET) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.54412 (4)	0.09933 (7)	0.29605 (3)	0.0347 (2)
Br1	0.25269 (5)	−0.30042 (7)	0.03920 (4)	0.0499 (2)
C1	0.8930 (4)	0.1246 (6)	0.3969 (4)	0.0391 (10)
H1	0.9579	0.1449	0.4594	0.047*
N1	0.7879 (3)	0.1260 (5)	0.4012 (3)	0.0390 (9)
C6	0.5142 (3)	0.0633 (5)	0.1513 (3)	0.0244 (8)
N2	0.6067 (3)	0.0572 (5)	0.1289 (2)	0.0283 (7)
C5	0.7000 (4)	0.1010 (5)	0.3106 (3)	0.0294 (9)
C10	0.1263 (4)	−0.0412 (8)	0.1036 (3)	0.0467 (13)
H10	0.0714	−0.1346	0.0929	0.056*
N3	0.4086 (3)	0.0347 (5)	0.0812 (2)	0.0307 (8)
H3	0.4002	0.0097	0.0038	0.11 (3)*
C4	0.7091 (3)	0.0741 (5)	0.2149 (3)	0.0255 (8)
C9	0.2266 (4)	−0.0716 (6)	0.0859 (3)	0.0320 (9)
C3	0.8190 (4)	0.0674 (6)	0.2134 (3)	0.0354 (10)
H3A	0.8303	0.0447	0.1507	0.042*
C7	0.3078 (3)	0.0625 (6)	0.1015 (3)	0.0264 (8)
C8	0.2863 (4)	0.2307 (6)	0.1358 (3)	0.0359 (10)
H8	0.3407	0.3248	0.1466	0.043*
C2	0.9120 (4)	0.0950 (6)	0.3064 (4)	0.0409 (11)
H2	0.9885	0.0937	0.3079	0.049*
C11	0.1872 (4)	0.2608 (8)	0.1538 (4)	0.0471 (12)
H11	0.1743	0.3748	0.1779	0.056*
C12	0.1068 (4)	0.1272 (9)	0.1372 (4)	0.0534 (15)
H12	0.0379	0.1496	0.1486	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0303 (3)	0.0541 (4)	0.0266 (3)	−0.00181 (18)	0.0184 (2)	−0.00480 (18)
Br1	0.0459 (4)	0.0368 (4)	0.0550 (4)	−0.0067 (2)	0.0055 (3)	0.0005 (2)
C1	0.028 (2)	0.044 (2)	0.034 (2)	0.000 (2)	−0.0008 (18)	−0.0034 (19)
N1	0.038 (2)	0.045 (2)	0.0263 (18)	−0.0011 (17)	0.0045 (16)	−0.0022 (16)
C6	0.0217 (19)	0.0271 (19)	0.0281 (19)	−0.0004 (15)	0.0135 (16)	−0.0024 (15)
N2	0.0222 (17)	0.0396 (18)	0.0269 (17)	−0.0023 (14)	0.0137 (14)	−0.0037 (14)

C5	0.034 (2)	0.028 (2)	0.028 (2)	0.0000 (16)	0.0148 (18)	−0.0021 (15)
C10	0.022 (2)	0.075 (4)	0.041 (3)	−0.009 (2)	0.010 (2)	0.016 (2)
N3	0.0199 (17)	0.047 (2)	0.0287 (17)	−0.0016 (15)	0.0134 (14)	−0.0077 (15)
C4	0.022 (2)	0.0283 (19)	0.0254 (19)	−0.0012 (15)	0.0082 (16)	−0.0003 (15)
C9	0.024 (2)	0.044 (2)	0.027 (2)	−0.0020 (18)	0.0082 (17)	0.0063 (17)
C3	0.024 (2)	0.048 (3)	0.039 (2)	0.0023 (18)	0.0179 (18)	0.0001 (19)
C7	0.0174 (18)	0.042 (2)	0.0230 (18)	−0.0001 (16)	0.0111 (15)	−0.0002 (16)
C8	0.031 (2)	0.042 (2)	0.039 (2)	0.0025 (19)	0.0177 (19)	−0.0041 (19)
C2	0.023 (2)	0.049 (3)	0.046 (3)	−0.0020 (18)	0.008 (2)	0.001 (2)
C11	0.038 (3)	0.067 (3)	0.040 (3)	0.015 (2)	0.018 (2)	−0.007 (2)
C12	0.028 (3)	0.098 (4)	0.041 (3)	0.010 (3)	0.021 (2)	0.005 (3)

Geometric parameters (Å, º)

Se1—C5	1.886 (4)	N3—C7	1.410 (5)
Se1—C6	1.920 (4)	N3—H3	1.0555
Br1—C9	1.897 (5)	C4—C3	1.387 (6)
C1—N1	1.340 (7)	C9—C7	1.384 (6)
C1—C2	1.384 (7)	C3—C2	1.387 (6)
C1—H1	0.9500	C3—H3A	0.9500
N1—C5	1.334 (6)	C7—C8	1.404 (6)
C6—N2	1.309 (5)	C8—C11	1.375 (6)
C6—N3	1.328 (5)	C8—H8	0.9500
N2—C4	1.388 (5)	C2—H2	0.9500
C5—C4	1.390 (6)	C11—C12	1.372 (8)
C10—C9	1.388 (7)	C11—H11	0.9500
C10—C12	1.393 (8)	C12—H12	0.9500
C10—H10	0.9500		
C5—Se1—C6	83.98 (17)	C7—C9—C10	121.1 (4)
N1—C1—C2	123.6 (4)	C7—C9—Br1	119.4 (3)
N1—C1—H1	118.2	C10—C9—Br1	119.5 (4)
C2—C1—H1	118.2	C4—C3—C2	117.9 (4)
C5—N1—C1	115.4 (4)	C4—C3—H3A	121.0
N2—C6—N3	123.0 (3)	C2—C3—H3A	121.0
N2—C6—Se1	114.5 (3)	C9—C7—C8	118.3 (4)
N3—C6—Se1	122.3 (3)	C9—C7—N3	121.6 (4)
C6—N2—C4	113.9 (3)	C8—C7—N3	120.1 (4)
N1—C5—C4	125.7 (4)	C11—C8—C7	120.7 (5)
N1—C5—Se1	123.5 (3)	C11—C8—H8	119.6
C4—C5—Se1	110.7 (3)	C7—C8—H8	119.6
C9—C10—C12	119.5 (5)	C1—C2—C3	119.7 (4)
C9—C10—H10	120.3	C1—C2—H2	120.1
C12—C10—H10	120.3	C3—C2—H2	120.1
C6—N3—C7	123.2 (3)	C12—C11—C8	120.5 (5)
C6—N3—H3	117.4	C12—C11—H11	119.8
C7—N3—H3	118.6	C8—C11—H11	119.8
C3—C4—N2	125.6 (4)	C11—C12—C10	120.0 (4)
C3—C4—C5	117.5 (4)	C11—C12—H12	120.0
N2—C4—C5	116.8 (4)	C10—C12—H12	120.0

C2—C1—N1—C5	−1.6 (7)	C12—C10—C9—C7	0.3 (7)
C5—Se1—C6—N2	−0.2 (3)	C12—C10—C9—Br1	−179.6 (4)
C5—Se1—C6—N3	175.3 (4)	N2—C4—C3—C2	177.1 (4)
N3—C6—N2—C4	−174.2 (4)	C5—C4—C3—C2	−2.5 (6)
Se1—C6—N2—C4	1.3 (4)	C10—C9—C7—C8	0.0 (6)
C1—N1—C5—C4	0.1 (6)	Br1—C9—C7—C8	179.8 (3)
C1—N1—C5—Se1	−179.5 (3)	C10—C9—C7—N3	−178.3 (4)
C6—Se1—C5—N1	178.7 (4)	Br1—C9—C7—N3	1.5 (5)
C6—Se1—C5—C4	−0.9 (3)	C6—N3—C7—C9	−125.4 (4)
N2—C6—N3—C7	−172.0 (4)	C6—N3—C7—C8	56.4 (6)
Se1—C6—N3—C7	12.8 (6)	C9—C7—C8—C11	0.3 (6)
C6—N2—C4—C3	178.2 (4)	N3—C7—C8—C11	178.7 (4)
C6—N2—C4—C5	−2.1 (5)	N1—C1—C2—C3	0.9 (8)
N1—C5—C4—C3	2.0 (6)	C4—C3—C2—C1	1.2 (7)
Se1—C5—C4—C3	−178.4 (3)	C7—C8—C11—C12	−0.9 (7)
N1—C5—C4—N2	−177.7 (4)	C8—C11—C12—C10	1.1 (8)
Se1—C5—C4—N2	1.9 (4)	C9—C10—C12—C11	−0.8 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N2^{i}	1.06	1.88	2.933 (4)	174

Symmetry code: (i) −*x*+1, −*y*, −*z*.