

2-Amino-4-phenylselenazolium bromide  
monohydrateXiu-Rong Hu,<sup>a\*</sup> You-Chu Zhang<sup>b</sup>  
and Jian-Ming Gu<sup>a</sup><sup>a</sup>Center of Analysis and Measurement, Zhejiang University, Hangzhou, Zhejiang 310028, People's Republic of China, and <sup>b</sup>Department of Chemistry, Shaoxing College of Arts and Sciences, Shaoxing, Zhejiang 312000, People's Republic of ChinaCorrespondence e-mail:  
huxiurong@yahoo.com.cnThe components of the title compound, C<sub>9</sub>H<sub>9</sub>N<sub>2</sub>Se<sup>+</sup>·Br<sup>-</sup>·H<sub>2</sub>O, are linked to each other *via* N—H···O, N—H···Br and O—H···Br hydrogen bonds.

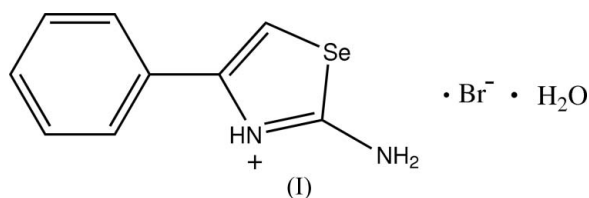
Received 11 October 2005

Accepted 20 October 2005

Online 27 October 2005

## Comment

Selenazoles have attracted considerable interest because they are small building blocks of complex natural products and biologically active compounds (Back, 1999).



## Key indicators

Single-crystal X-ray study  
T = 296 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
R factor = 0.030  
wR factor = 0.077  
Data-to-parameter ratio = 20.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the structure of (I), the selenazole ring is planar, forming a dihedral angle of 23.93 (13)<sup>o</sup> with the benzene ring. All components are involved in a hydrogen-bond network (Table 2), the Br<sup>-</sup> ions and the water molecules bridging the 2-amino-4-phenylselenazolium cations into a centrosymmetric dimer (Fig. 2). The dimers are further linked into a layer by an O1—H102···Br<sup>ii</sup> hydrogen bond (see Table 2 for symmetry code).

## Experimental

To a solution of 2-bromo-1-phenylethanone (1.99 g, 10 mmol) in EtOH (30 ml) was added selenourea (1.23 g, 10 mmol). The mixture was refluxed for 3 h. After cooling, the mixture was extracted with Et<sub>2</sub>O and water, dried with anhydrous MgSO<sub>4</sub>, acidified with dry HBr and recrystallized from ethanol to give the salt 4-phenyl-2-amino-1,3-selenazole bromide (yield: 2.4 g, 79%) (Geisler *et al.*, 2004). This was recrystallized from ethanol (95%), giving colourless crystals of (I) suitable for X-ray diffraction.

## Crystal data

C<sub>9</sub>H<sub>9</sub>N<sub>2</sub>Se<sup>+</sup>·Br<sup>-</sup>·H<sub>2</sub>O  
M<sub>r</sub> = 322.06  
Monoclinic, P2<sub>1</sub>/c  
a = 11.493 (7) Å  
b = 9.591 (5) Å  
c = 10.456 (4) Å  
β = 94.211 (19)<sup>o</sup>  
V = 1149.6 (10) Å<sup>3</sup>  
Z = 4D<sub>x</sub> = 1.861 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 8209 reflections  
θ = 3.3–27.5<sup>o</sup>  
μ = 6.72 mm<sup>-1</sup>  
T = 296 (1) K  
Platelet, colourless  
0.24 × 0.18 × 0.08 mm

Data collection

Rigaku R-AXIS RAPID  
diffractometer  
 $\omega$  scans  
Absorption correction: multi-scan  
(*ABSCOR*; Higashi, 1995)  
 $T_{\min} = 0.263$ ,  $T_{\max} = 0.584$   
10800 measured reflections

2622 independent reflections  
1834 reflections with  $F^2 > 2\sigma(F^2)$   
 $R_{\text{int}} = 0.054$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -14 \rightarrow 14$   
 $k = -12 \rightarrow 12$   
 $l = -12 \rightarrow 13$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.077$   
 $S = 1.00$   
2622 reflections  
128 parameters  
H-atom parameters constrained

$w = 1/[0.77\sigma(F_o^2)]/(4F_o^2)$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.51 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.60 \text{ e } \text{Å}^{-3}$   
Extinction correction: Larson  
(1970)  
Extinction coefficient: 42 (7)

Table 1

Selected geometric parameters (Å, °).

Se1—C1	1.874 (2)	N12—C1	1.338 (3)
Se1—C2	1.875 (3)	N12—C3	1.403 (3)
N11—C1	1.309 (3)	C2—C3	1.339 (4)
C1—Se1—C2	85.34 (13)	C1—N12—C3	117.0 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N12—H121...O1	0.86	1.97	2.805 (3)	165
N11—H111...Br1	0.86	2.50	3.330 (3)	162
N11—H112...Br1 <sup>i</sup>	0.86	2.76	3.521 (3)	148
O1—H101...Br1 <sup>i</sup>	0.85	2.52	3.342 (2)	164
O1—H102...Br1 <sup>ii</sup>	0.84	2.56	3.388 (3)	167

Symmetry codes: (i)  $-x + 1, -y + 1, -z$ ; (ii)  $-x + 1, +y + \frac{1}{2}, -z + \frac{1}{2}$

H atoms attached to O and N atoms were located in a difference Fourier map and included in the refinement based on the as-found O—H and N—H bond lengths; their isotropic displacement parameters were initially refined, then fixed in the final stage. All other H atoms were placed in calculated positions, with C—H = 0.93 Å, and included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

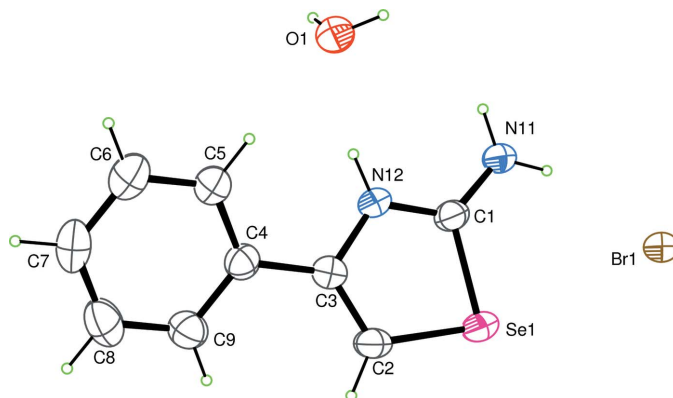


Figure 1

A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

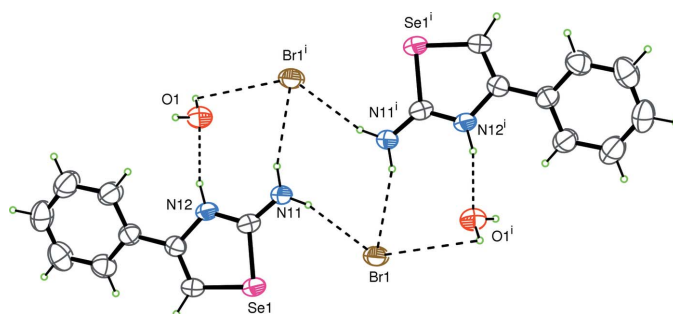


Figure 2

A hydrogen-bonded dimer of (I) [symmetry code: (i)  $1 - x, 1 - y, -z$ ].

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Back, T. G. (1999). *Organoselenium Chemistry*. Oxford University Press.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Geisler, K., Pfeiffer, W. D., Kuenzler, A., Below, H., Bulka, E. & Langer, P. (2004). *Synthesis*, **6**, 875–884.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan.
- Rigaku/MS (2004). *CrystalStructure*. Version 3.60. Rigaku/MS, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.