Jian-Ge Wang*

Crystal structure of 3-nitrophenol-2,1,3-benzoselenadiazole (1/1), $C_{12}H_9N_3O_3Se$

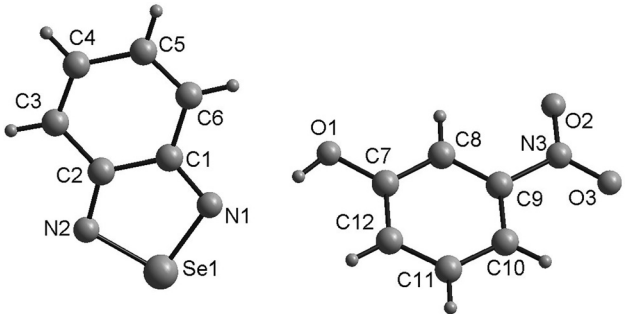

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.24 × 0.21 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	3.04 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	28.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9024, 2709, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2124
$N(param)_{refined}$:	173
Programs:	CrysAlis^{Pro} [1], SHELX [2, 3], Olex2 [4]

https://doi.org/10.1515/ncrs-2023-0098
Received March 2, 2023; accepted March 24, 2023;
published online October 2, 2024

Abstract

$C_{12}H_9N_3O_3Se$, monoclinic, $P2_1/c$ (no. 14), a = 3.9106(3) Å, b = 23.5453(14) Å, c = 13.4611(8) Å, β = 92.697(6)°, V = 1238.07(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0432, $\omega R_{ref}(F^2)$ = 0.0786, T = 290 K.

CCDC no.: 2251363

The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

All reagents and solvents were used as obtained without further purification. The methanol solution (10 mL) of 3-nitrophenol (0.10 mmol, 0.0139 g) was slowly added to another methanol solution (10 mL) of 2,1,3-benzoselenadiazole (0.1 mmol, 0.0183 g). The mixture was stirred for 2 h at room temperature. The solution was filtered, and the filtrate was kept at the room temperature. After 1 week colorless crystals of the title compound were obtained.

2 Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors U_{iso}(H) = 1.2 times U_{eq} (C, aromatic ring), U_{iso}(H) = 1.5 times U_{eq}(OH). The final refinement by using geometrical restraints, with C–H = 0.93 Å (aromatic ring), O–H = 0.82 Å (OH).

3 Comment

With their unique structures and interesting physicochemical properties, supramolecular compounds attract much attention [5–7]. In general, the different components are linked together by one or more intermolecular interactions such as hydrogen bonds, forming high ordered supramolecular cocrystal structures [8–10]. As known, the nitrogen atoms of polyaza heteroaromatic compounds can serve as weak base to accept protons from carboxylic acid to form acid-base conjugate pair incorporating charge transfer interactions, which enable the potential characteristic of the construction of cocrystals. In this paper, we report the reaction product of the nitrogen heterocyclic compound 2,1,3-benzoselenadiazole together with 3-nitrophenol.

The asymmetric unit of the title structure contains one 2,1,3-benzoselenadiazole and one 3-nitrophenol. The dimers of two 2,1,3-benzoselenadiazole molecules are formed by a pair of N–Se···N chalcogen bonds. And the adjacent dimers are connected by O–H···N hydrogen bonds and N–Se···O chalcogen bonds to form a two dimensional

***Corresponding author: Jian-Ge Wang**, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471022, P.R. China, E-mail: wang_jiange2@126.com

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.44256 (9)	0.55392 (2)	0.89226 (2)	0.04105 (13)
N1	0.2846 (7)	0.55800 (11)	0.7653 (2)	0.0421 (7)
N2	0.3121 (7)	0.48169(11)	0.90576 (19)	0.0395 (7)
C1	0.1481 (8)	0.50795 (13)	0.7420 (2)	0.0345 (7)
C2	0.1642 (8)	0.46575 (13)	0.8197 (2)	0.0326 (7)
C3	0.0239 (9)	0.41080 (14)	0.7998 (3)	0.0444 (8)
H3	0.0307	0.3831	0.8491	0.053*
C4	−0.1188 (9)	0.39948 (15)	0.7089 (3)	0.0477 (9)
H4	−0.2091	0.3636	0.6959	0.057*
C5	−0.1344 (9)	0.44102 (15)	0.6326 (3)	0.0479 (9)
H5	−0.2365	0.4318	0.5709	0.057*
C6	−0.0055 (9)	0.49346 (15)	0.6471 (3)	0.0453 (9)
H6	−0.0162	0.5200	0.5958	0.054*
O1	0.5223 (7)	0.61149 (10)	0.59512 (18)	0.0574 (7)
H1	0.4605	0.6031	0.6506	0.086*
O2	0.8942 (8)	0.73963 (12)	0.3359 (2)	0.0691 (8)
O3	0.7645 (8)	0.82264 (11)	0.3821 (2)	0.0724 (9)
N3	0.7669 (8)	0.77224 (14)	0.3933 (2)	0.0489 (8)
C7	0.4997 (8)	0.66826 (13)	0.5824 (2)	0.0353 (7)
C8	0.6323(8)	0.69083 (13)	0.4970 (2)	0.0362 (8)
H8	0.7305	0.6674	0.4505	0.043*
C9	0.6148 (8)	0.74837 (13)	0.4830 (2)	0.0338 (7)
C10	0.4733 (9)	0.78504 (15)	0.5490 (3)	0.0455 (9)
H10	0.4659	0.8240	0.5376	0.055*
C11	0.3425 (9)	0.76131 (16)	0.6331 (3)	0.0490 (9)
H11	0.2439	0.7848	0.6793	0.059*
C12	0.3546 (8)	0.70396 (15)	0.6501 (2)	0.0416 (8)
H12	0.2651	0.6890	0.7073	0.050*

network. Bond lengths and angles are all in the expected ranges [11].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: None declared.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Agilent Technologies. CrysAlis^{Pro}; Agilent Technologies: Santa Clara, CA, USA, 2017.
2. Sheldrick G. M. A short history of *SHELX*. *Acta Crystallogr.* 2008, *A64*, 112–122.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
5. Liu R., Gao Y. J., Jin W. J. Color-tunable phosphorescence of 1,10-phenanthrolines by 4,7-methyl/-diphenyl/-dichloro substituents in cocrystals assembled via bifurcated C–I···N halogen bonds using 1,4-diiodotetrafluorobenzene as a bonding donor. *Acta Crystallogr.* 2017, *B73*, 247–254.
6. Kapadia P. P., Ditzler L. R., Baltrusaitis J., Swenson D. C., Tivanski A. V., Pigge C. Semiconducting organic complexes prepared from tetraphenylethylene tetracarboxylic acid and bis(pyridine)s via charge-assisted hydrogen bonding. *J. Am. Chem. Soc.* 2011, *133*, 8490–8493.
7. Thomas S., Satheeshkumar K., Mugesh G., Guru Row T. N. Unusually short chalcogen bonds involving organoselenium: insights into the Se–N bond cleavage mechanism of the antioxidant ebselen and analogues. *Chem. Eur J.* 2015, *21*, 1–9.
8. Wang H., Jin W. J. Cocrystals assembled by 1,4-diiodotetrafluorobenzene and phenothiazine based on C–I···Π/N/S halogen bond and other assisting interactions. *Acta Crystallogr.* 2017, *B73*, 210–216.
9. Colquhoun H. M., Williams D. J., Zhu Z. Macrocyclic aromatic ether-imide-sulfones: versatile supramolecular receptors with extreme thermochemical and oxidative stability. *J. Am. Chem. Soc.* 2002, *124*, 13346–13347.
10. Eichstaedt K., Wasilewska A., Wicher B., Gdaniec M., Polonski T. Supramolecular synthesis based on a combination of Se···N secondary bonding interactions with hydrogen and halogen bonds. *Cryst. Growth Des.* 2016, *16*, 1282–1293.
11. Hubner T., Gieren A. The crystal structures of two polyiodide salts of organic cations. *Acta Crystallogr.* 1984, *A40*, C266.