

# 3,14-Diselena-4,5,12,13-tetraazatricyclo[9.3.0.0<sup>2,6</sup>]-tetradeca-1(11),2(6),4,12-tetraene

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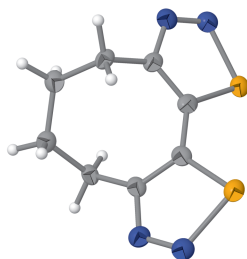
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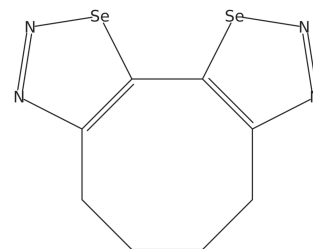
**Keywords:** crystal structure; heterocycle; selenium; medium-sized ring.**CCDC reference:** 2524974**Structural data:** full structural data are available from iucrdata.iucr.org

The crystal structure of the third isomer of cyclooctenobis-1,2,3-selenadiazoles,  $C_8H_8N_4Se_2$ , is reported. The molecule is located on a twofold rotation axis. The eight-membered ring adopts a twist-chair conformation with planar heterocycles.  $C-H \cdots N$  hydrogen bonds connect the molecules into layers parallel to  $(\bar{1}01)$ .

3D view



Chemical scheme



## Structure description

The title compound,  $C_8H_8N_4Se_2$  (Fig. 1), is the third isomer in the series of cyclooctenobis-1,2,3-selenadiazoles (Detert & Schollmeyer, 2020; Schollmeyer & Detert, 2025). These were prepared as part of a project on medium-sized cycloalkynes with functional and sterically demanding groups (Bissinger *et al.*, 1988; Detert *et al.*, 1994; Detert & Meier, 1997). Bis-1,2,3-selenadiazoles are antimicrobial agents (Al-Smadi *et al.*, 2008) and important sources for medium-sized cycloalkadiynes (Gleiter *et al.*, 1988; Morales & Fronczek, 1994). The molecule is  $C_2$ -symmetric, having the eight-membered ring in a nearly twist-chair conformation with staggered  $C-H$  bonds. Both selenadiazole rings are planar within 0.02 (6) Å at atom C5 and the torsion angle between the heterocycles is 55.5 (3)°. This is significantly larger than the torsion angle [ $-43.5$  (11)°] between the selenadiazole rings in the isomeric molecule with an inverted orientation of the annulated heterocycles (Detert & Schollmeyer, 2020), probably due to the large atomic radii of the vicinal Se atoms in the title compound. The unit cell contains four molecules connected *via* four hydrogen bonds [ $C7-H7b \cdots N2^i$ ; symmetry code: (i)  $-x, y + \frac{1}{2}, -z + \frac{1}{2}$ ]. The hydrogen bonds (Table 1, Fig. 2) connect the molecules to form layers parallel to  $(\bar{1}01)$ .

## Synthesis and crystallization

The title compound appeared in the synthesis of its homo-conjugated isomer (Schollmeyer & Detert, 2025) from 1,4-cyclooctanedione bissemicarbazone and selenium

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C7-H7B\cdots N2^i$	0.99	2.61	3.466 (9)	144

Symmetry code: (i)  $-x, y - \frac{1}{2}, -z + \frac{1}{2}$ .

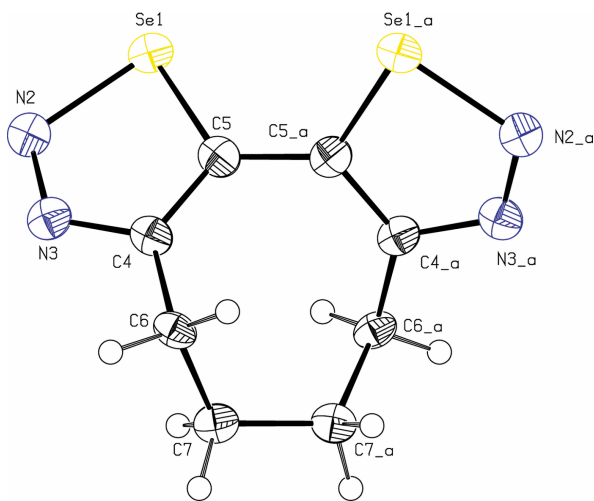
dioxide in 1,4-dioxane in 12% yield. Recrystallization from a solution in chloroform/ligroin gave brownish crystals (m.p. 393 K). IR (KBr): 2900, 2840, 1515, 1460, 1440, 1430, 1340, 1295, 1275, 1250, 1215, 945, 880, 850  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.20 (bs, 4H), 1.90 (bs, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  158.8, 144.9 (C-1,2,7,10), 27.4, 25.1 (C-6,7,8,9);  $^{77}\text{Se}$  NMR (73 MHz,  $\text{CDCl}_3$ ,  $\text{SeO}_2/\text{D}_2\text{O}$  as reference):  $\delta$  295.9;  $^{15}\text{N}$  NMR (40,5 MHz,  $\text{CDCl}_3$ ,  $\text{HC}_3\text{NO}_2$  as reference = 0):  $\delta$  93.9, 93.1; UV (EtOH,  $\lambda$ ,  $\log \epsilon$ ): 207 (3.85), 220 (3.86), 261 (3.57), 302 (sh, 3.33), 339 nm (sh, 3.12); MS (FD): 290 ( $M - \text{N}_2^+$ ).

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were placed at calculated positions and refined in the riding-model approximation, with  $C-H = 0.99$  Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

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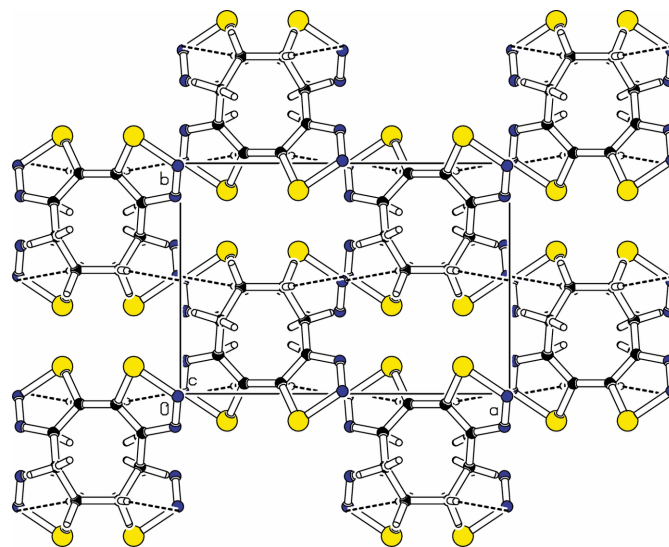
**Figure 1**  
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$\text{C}_8\text{H}_8\text{N}_4\text{Se}_2$
$M_r$	318.10
Crystal system, space group	Monoclinic, $I2/a$
Temperature (K)	120
$a, b, c$ (Å)	11.746 (2), 8.1617 (10), 10.0922 (15)
$\beta$ (°)	97.121 (14)
$V$ (Å <sup>3</sup> )	960.1 (3)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	7.66
Crystal size (mm)	0.48 × 0.09 × 0.04
Data collection	
Diffractometer	STOE IPDS 2T
Absorption correction	Integration
$T_{\text{min}}, T_{\text{max}}$	0.468, 0.893
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	2481, 1144, 930
$R_{\text{int}}$	0.037
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.663
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.056, 0.148, 1.10
No. of reflections	1144
No. of parameters	64
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	1.04, -1.75

Computer programs: *X-AREA WinXpose* (Stoe & Cie, 2020), *X-AREA Recipe* (Stoe & Cie, 2020), *X-AREA Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015a), *SHELXL2019* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

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**Figure 2**  
Part of the packing diagram, viewed along the  $c$ -axis direction.

## full crystallographic data

*IUCrData* (2026). **11**, x260063 [<https://doi.org/10.1107/S2414314626000635>]

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#### Crystal data

C<sub>8</sub>H<sub>8</sub>N<sub>4</sub>Se<sub>2</sub>

$M_r = 318.10$

Monoclinic,  $I2/a$

$a = 11.746$  (2) Å

$b = 8.1617$  (10) Å

$c = 10.0922$  (15) Å

$\beta = 97.121$  (14)°

$V = 960.1$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 608$

$D_x = 2.201$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5444 reflections

$\theta = 3.1$ – $28.2$ °

$\mu = 7.66$  mm<sup>-1</sup>

$T = 120$  K

Plate, colorless

$0.48 \times 0.09 \times 0.04$  mm

#### Data collection

STOE IPDS 2T

diffractometer

Detector resolution: 6.67 pixels mm<sup>-1</sup>

rotation method,  $\omega$  scans

Absorption correction: integration

$T_{\min} = 0.468$ ,  $T_{\max} = 0.893$

2481 measured reflections

1144 independent reflections

930 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 28.1$ °,  $\theta_{\min} = 3.1$ °

$h = -15 \rightarrow 13$

$k = -10 \rightarrow 10$

$l = -13 \rightarrow 13$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.148$

$S = 1.10$

1144 reflections

64 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0748P)^2 + 11.7676P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.04$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.75$  e Å<sup>-3</sup>

#### Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.14172 (6)	0.61729 (7)	0.33965 (6)	0.0274 (3)
N2	0.0080 (5)	0.4890 (7)	0.3007 (5)	0.0291 (12)
N3	0.0157 (5)	0.3578 (7)	0.3698 (5)	0.0289 (12)
C4	0.1149 (6)	0.3307 (8)	0.4573 (6)	0.0259 (13)
C5	0.1943 (5)	0.4546 (8)	0.4581 (6)	0.0269 (13)
C6	0.1228 (6)	0.1830 (8)	0.5420 (6)	0.0254 (12)
H6A	0.044169	0.148577	0.555241	0.031*
H6B	0.163678	0.211823	0.630606	0.031*
C7	0.1843 (6)	0.0368 (8)	0.4861 (6)	0.0284 (13)
H7A	0.155565	-0.065021	0.523630	0.034*
H7B	0.162450	0.033262	0.388198	0.034*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.0327 (4)	0.0269 (4)	0.0241 (4)	0.0027 (3)	0.0096 (3)	0.0013 (2)
N2	0.031 (3)	0.033 (3)	0.024 (2)	0.001 (2)	0.007 (2)	-0.001 (2)
N3	0.030 (3)	0.031 (3)	0.028 (3)	0.003 (2)	0.011 (2)	0.000 (2)
C4	0.027 (3)	0.027 (3)	0.026 (3)	-0.003 (3)	0.012 (2)	-0.003 (2)
C5	0.027 (3)	0.029 (3)	0.027 (3)	-0.001 (3)	0.011 (2)	-0.006 (2)
C6	0.029 (3)	0.024 (3)	0.025 (3)	-0.006 (3)	0.010 (2)	-0.002 (2)
C7	0.034 (3)	0.027 (3)	0.026 (3)	0.001 (3)	0.008 (3)	0.002 (3)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Se1—C5	1.841 (7)	C6—C7	1.538 (9)
Se1—N2	1.888 (6)	C6—H6A	0.9900
N2—N3	1.275 (8)	C6—H6B	0.9900
N3—C4	1.390 (9)	C7—C7 <sup>i</sup>	1.534 (13)
C4—C5	1.375 (9)	C7—H7A	0.9900
C4—C6	1.474 (9)	C7—H7B	0.9900
C5—C5 <sup>i</sup>	1.467 (13)		
C5—Se1—N2	86.5 (3)	C7—C6—H6A	108.5
N3—N2—Se1	110.6 (5)	C4—C6—H6B	108.5
N2—N3—C4	118.6 (6)	C7—C6—H6B	108.5
C5—C4—N3	113.7 (6)	H6A—C6—H6B	107.5
C5—C4—C6	127.2 (6)	C7 <sup>i</sup> —C7—C6	116.2 (5)
N3—C4—C6	119.1 (6)	C7 <sup>i</sup> —C7—H7A	108.2
C4—C5—C5 <sup>i</sup>	124.0 (5)	C6—C7—H7A	108.2
C4—C5—Se1	110.7 (5)	C7 <sup>i</sup> —C7—H7B	108.2
C5 <sup>i</sup> —C5—Se1	125.3 (3)	C6—C7—H7B	108.2
C4—C6—C7	114.9 (5)	H7A—C7—H7B	107.4
C4—C6—H6A	108.5		

C5—Se1—N2—N3	-0.1 (4)	C6—C4—C5—Se1	-177.1 (5)
Se1—N2—N3—C4	-0.1 (7)	N2—Se1—C5—C4	0.2 (4)
N2—N3—C4—C5	0.3 (8)	N2—Se1—C5—C5 <sup>i</sup>	179.9 (6)
N2—N3—C4—C6	177.4 (5)	C5—C4—C6—C7	-87.5 (8)
N3—C4—C5—C5 <sup>i</sup>	180.0 (6)	N3—C4—C6—C7	95.9 (7)
C6—C4—C5—C5 <sup>i</sup>	3.2 (11)	C4—C6—C7—C7 <sup>i</sup>	83.7 (8)
N3—C4—C5—Se1	-0.4 (7)		

Symmetry code: (i)  $-x+1/2, y, -z+1$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C7—H7B $\cdots$ N2 <sup>ii</sup>	0.99	2.61	3.466 (9)	144

Symmetry code: (ii)  $-x, y-1/2, -z+1/2$ .