IUCrData

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4*H*,5*H*,6*H*,7*H*,8*H*,9*H*-Cycloocta[*d*][1,2,3]selenadiazol-7-one

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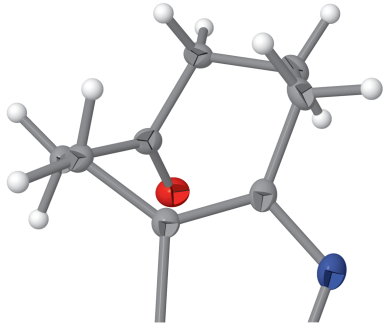
Keywords: crystal structure; selenium; Se-NMR; heterocycle; medium sized ring.

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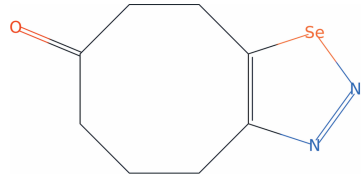
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_8H_{10}N_2OSe$, an almost planar 1,2,3-selenadiazole ring is annulated to a cyclooctenone ring with a twist-boat conformation. The heterocycle and C—C(=O)—C carbonyl moieties subtend a dihedral angle of 54.25 (11)°. In the extended structure, weak C—H···O and C—H···N interactions link the molecules into double layers lying parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

The title compound, $C_8H_{10}N_2OSe$ (**I**) (Fig. 1), was prepared in a project on medium sized rings with geometrical strain (Bissinger *et al.* 1988; Detert & Meier, 1997*a*,*b*). 1,2,3-Selenadiazoles are important precursors for the synthesis of strained cycloalkynes (Yalpani *et al.* 1971; Meier & Voigt,1972). The title compound had been prepared for the first synthesis of cyclooctynone (Meier & Petersen, 1978). The crystal structures of related cyclooctено-1,2,3-selanadiazoles have been reported recently (Schollmeyer & Detert, 2020, 2025). The C4/C8/N7/N6/Se5 selenadiazole ring in (**I**) is planar to within 0.0044 (2) Å and the C2—C1(═O12)—C11 carbonyl moiety to within 0.004 (2) Å at C1; these planes subtend a dihedral angle of 54.25 (11)°. Further characteristics are a dihedral angle of 3.51 (19)° for the selenadiazole C4/C8 fragment and its adjacent methylene C atoms (C3—C4—C8—C9) and a twist-boat conformation of the cyclooctene ring.

In the extended structure of (**I**), the molecules are connected *via* weak C—H···O and C—H···N hydrogen bonds with H···*X* (*X* = O, N) lengths of 2.58 to 2.68 Å (Table 1, Fig. 2). These bridges connect the molecules into a double-layer system with the layers lying parallel to the *bc* plane. Within one layer, the molecules are related by translation symmetry along the *b* axis and a *c* glide plane; a centre of inversion connects molecules of both layers, as centrosymmetric dimers are formed. While the C—H···O and C—H···N hydrogen bonds are within a layer and connect the double layer, the selenadiazole rings are on both surfaces of this subsystem.

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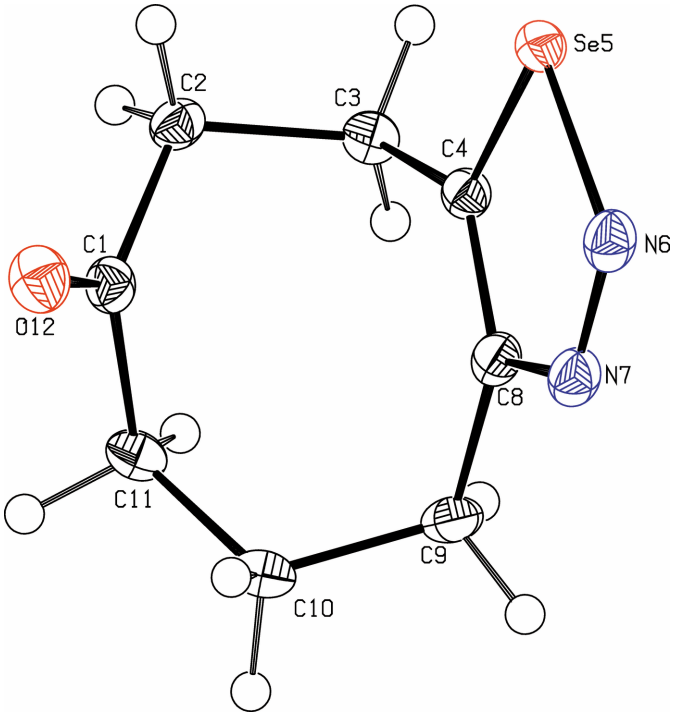


Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The synthesis of the title compound was performed according to the literature (Meier & Petersen, 1978) from cycloocta-diene-1,5 *via* hydroboration/oxidation, partial oxidation of the diol, transformation of the ketone to a 1,2,3-selenadiazole and oxidation of the remaining alcohol group. After recrystallization from the mixed solvents of chloroform and propanol-2-ol, colourless blocks with m.p. 371 K were formed. Analytical data are given by Meier and Petersen (1978), ^{1}H-NMR (400 MHz, $CDCl_2$): δ = 3.45 (*m*, 2 H), 3.26 (*m*, 2 H), 2.73 (*m*, 2 H), 2.47 (*m*, 2 H), 1.98 (*qui*, 2 H). ^{77}Se-NMR (73 MHz, $CDCl_3$, SeO_2/D_2O as reference): δ = 223.7.

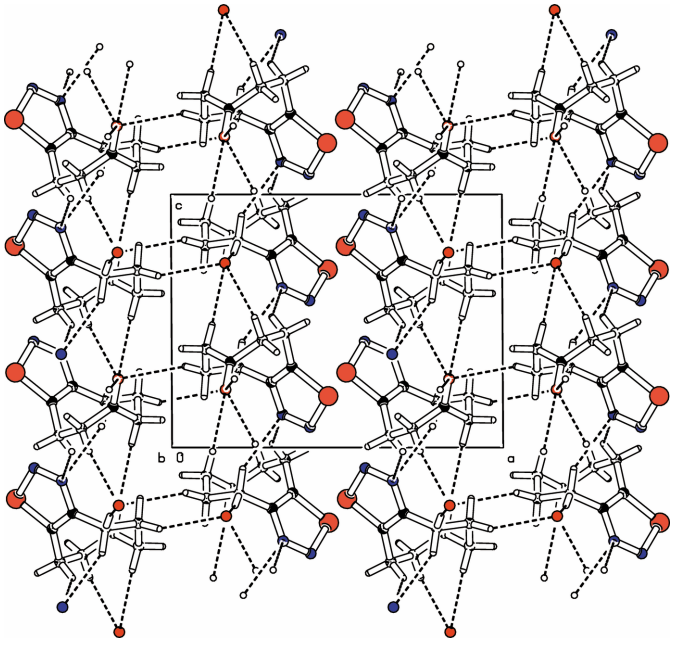


Figure 2
Partial packing diagram of (**I**) viewed along the *b*-axis direction. Hydrogen bonds are shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O12^{i}	0.99	2.58	3.339 (3)	133
C3—H3A···N7^{ii}	0.99	2.68	3.473 (3)	138
C9—H9A···O12^{iii}	0.99	2.65	3.608 (3)	162
C9—H9B···N7^{ii}	0.99	2.64	3.547 (3)	152
C10—H10B···O12^{iv}	0.99	2.63	3.461 (3)	142
C11—H11A···O12^{i}	0.99	2.63	3.467 (3)	143

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{10}N_2OSe$
M_r	229.14
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	12.7015 (6), 7.2450 (2), 9.7070 (5)
β (°)	90.288 (4)
V (Å^3)	893.25 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.16
Crystal size (mm)	0.95 × 0.53 × 0.21
Data collection	
Diffractometer	Stoe IPDS 2T
Absorption correction	Integration
T_{min}, T_{max}	0.071, 0.405
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4432, 2134, 2070
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.084, 1.14
No. of reflections	2134
No. of parameters	109
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.89

Computer programs: *X-AREA WinXpose*, *X-AREA Recipe* and *X-AREA Integrate* (Stoe, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2026). **11**, x261138 [https://doi.org/10.1107/S2414314626011387]

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Crystal data

$C_8H_{10}N_2OSe$
M_r = 229.14
Monoclinic, $P2_1/c$
a = 12.7015 (6) Å
b = 7.2450 (2) Å
c = 9.7070 (5) Å
β = 90.288 (4)°
V = 893.25 (7) Å^3
Z = 4
$F(000)$ = 456
D_x = 1.704 Mg m^{-3}
Melting point: 371 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8908 reflections
θ = 2.6–28.6°
μ = 4.16 mm^{-1}
T = 120 K
Block, colorless
0.95 × 0.53 × 0.21 mm

Data collection

Stoe IPDS 2T diffractometer
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
Absorption correction: integration
T_{min} = 0.071, T_{max} = 0.405
4432 measured reflections
2134 independent reflections
2070 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.0°, θ_{min} = 3.2°
h = −16→16
k = −9→9
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.084
S = 1.14
2134 reflections
109 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.6455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}

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.

Refinement. Hydrogen atoms attached to carbons were placed at calculated positions and were refined in the riding-model approximation with C—H = 0.99 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.17825 (15)	0.2685 (3)	0.3378 (2)	0.0186 (4)
C2	0.27398 (16)	0.2237 (3)	0.4249 (2)	0.0185 (4)
H2A	0.317187	0.129893	0.377209	0.022*
H2B	0.251056	0.170810	0.513922	0.022*
C3	0.34108 (16)	0.3969 (3)	0.4520 (2)	0.0179 (4)
H3A	0.303370	0.480159	0.515785	0.022*
H3B	0.408426	0.360662	0.496112	0.022*
C4	0.36298 (15)	0.4966 (3)	0.3194 (2)	0.0162 (4)
Se5	0.47241 (2)	0.43962 (3)	0.20325 (2)	0.01750 (10)
N6	0.41692 (15)	0.6158 (3)	0.08121 (18)	0.0211 (4)
N7	0.33379 (14)	0.6881 (2)	0.13071 (18)	0.0204 (3)
C8	0.30090 (16)	0.6286 (3)	0.2587 (2)	0.0177 (4)
C9	0.20190 (17)	0.7117 (3)	0.3166 (2)	0.0208 (4)
H9A	0.189133	0.831963	0.271053	0.025*
H9B	0.212906	0.735749	0.416032	0.025*
C10	0.10337 (17)	0.5907 (3)	0.2989 (2)	0.0222 (4)
H10A	0.097547	0.553333	0.201022	0.027*
H10B	0.040500	0.665579	0.321089	0.027*
C11	0.10277 (17)	0.4157 (3)	0.3892 (2)	0.0204 (4)
H11A	0.122305	0.449694	0.484777	0.024*
H11B	0.030593	0.364205	0.390915	0.024*
O12	0.16234 (13)	0.1908 (2)	0.22902 (17)	0.0251 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0176 (9)	0.0184 (9)	0.0198 (9)	−0.0023 (7)	0.0034 (7)	0.0011 (7)
C2	0.0202 (9)	0.0165 (9)	0.0188 (9)	0.0017 (7)	0.0022 (7)	0.0028 (7)
C3	0.0184 (9)	0.0207 (9)	0.0147 (8)	0.0011 (7)	−0.0005 (7)	0.0003 (7)
C4	0.0163 (9)	0.0163 (9)	0.0159 (8)	−0.0007 (7)	−0.0016 (7)	−0.0001 (7)
Se5	0.01565 (14)	0.01792 (15)	0.01893 (14)	−0.00037 (6)	0.00089 (9)	0.00129 (6)
N6	0.0239 (9)	0.0198 (8)	0.0195 (8)	−0.0040 (7)	−0.0010 (7)	0.0037 (7)
N7	0.0231 (8)	0.0181 (8)	0.0200 (8)	−0.0025 (6)	−0.0021 (7)	0.0037 (6)
C8	0.0203 (9)	0.0152 (9)	0.0176 (9)	−0.0014 (7)	−0.0028 (7)	0.0010 (7)
C9	0.0215 (10)	0.0173 (9)	0.0237 (9)	0.0059 (7)	−0.0026 (8)	−0.0003 (8)
C10	0.0170 (10)	0.0250 (9)	0.0245 (10)	0.0058 (8)	−0.0007 (8)	0.0002 (8)
C11	0.0152 (9)	0.0247 (10)	0.0212 (10)	0.0030 (7)	0.0019 (7)	−0.0004 (7)
O12	0.0256 (8)	0.0265 (8)	0.0232 (7)	−0.0025 (6)	0.0001 (6)	−0.0048 (6)

Geometric parameters (Å, º)

C1—O12	1.213 (3)	N6—N7	1.275 (3)
C1—C2	1.513 (3)	N7—C8	1.381 (3)
C1—C11	1.520 (3)	C8—C9	1.506 (3)
C2—C3	1.539 (3)	C9—C10	1.537 (3)

C2—H2A	0.9900	C9—H9A	0.9900
C2—H2B	0.9900	C9—H9B	0.9900
C3—C4	1.503 (3)	C10—C11	1.542 (3)
C3—H3A	0.9900	C10—H10A	0.9900
C3—H3B	0.9900	C10—H10B	0.9900
C4—C8	1.371 (3)	C11—H11A	0.9900
C4—Se5	1.8410 (19)	C11—H11B	0.9900
Se5—N6	1.8766 (19)		

O12—C1—C2	121.13 (19)	C4—C8—N7	115.38 (18)
O12—C1—C11	120.60 (19)	C4—C8—C9	126.73 (18)
C2—C1—C11	118.27 (17)	N7—C8—C9	117.89 (18)
C1—C2—C3	111.31 (16)	C8—C9—C10	114.28 (17)
C1—C2—H2A	109.4	C8—C9—H9A	108.7
C3—C2—H2A	109.4	C10—C9—H9A	108.7
C1—C2—H2B	109.4	C8—C9—H9B	108.7
C3—C2—H2B	109.4	C10—C9—H9B	108.7
H2A—C2—H2B	108.0	H9A—C9—H9B	107.6
C4—C3—C2	110.52 (17)	C9—C10—C11	114.31 (18)
C4—C3—H3A	109.5	C9—C10—H10A	108.7
C2—C3—H3A	109.5	C11—C10—H10A	108.7
C4—C3—H3B	109.5	C9—C10—H10B	108.7
C2—C3—H3B	109.5	C11—C10—H10B	108.7
H3A—C3—H3B	108.1	H10A—C10—H10B	107.6
C8—C4—C3	126.52 (18)	C1—C11—C10	112.67 (17)
C8—C4—Se5	109.12 (14)	C1—C11—H11A	109.1
C3—C4—Se5	124.05 (14)	C10—C11—H11A	109.1
C4—Se5—N6	87.23 (8)	C1—C11—H11B	109.1
N7—N6—Se5	110.53 (14)	C10—C11—H11B	109.1
N6—N7—C8	117.74 (18)	H11A—C11—H11B	107.8

O12—C1—C2—C3	122.5 (2)	C3—C4—C8—C9	4.8 (3)
C11—C1—C2—C3	−56.7 (2)	Se5—C4—C8—C9	178.63 (17)
C1—C2—C3—C4	−50.3 (2)	N6—N7—C8—C4	0.3 (3)
C2—C3—C4—C8	87.7 (2)	N6—N7—C8—C9	−179.16 (19)
C2—C3—C4—Se5	−85.2 (2)	C4—C8—C9—C10	−79.8 (3)
C8—C4—Se5—N6	0.78 (15)	N7—C8—C9—C10	99.6 (2)
C3—C4—Se5—N6	174.77 (17)	C8—C9—C10—C11	70.4 (2)
C4—Se5—N6—N7	−0.63 (15)	O12—C1—C11—C10	−67.1 (3)
Se5—N6—N7—C8	0.3 (2)	C2—C1—C11—C10	112.1 (2)
C3—C4—C8—N7	−174.62 (19)	C9—C10—C11—C1	−73.7 (2)
Se5—C4—C8—N7	−0.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···Se5^{i}	0.99	3.11	4.025 (2)	154
C2—H2*B*···O12^{ii}	0.99	2.58	3.339 (3)	133

C3—H3*A*···N7^{iii}	0.99	2.68	3.473 (3)	138
C3—H3*B*···Se5^{ii}	0.99	3.07	3.825 (2)	134
C9—H9*A*···O12^{iv}	0.99	2.65	3.608 (3)	162
C9—H9*B*···N7^{iii}	0.99	2.64	3.547 (3)	152
C10—H10*B*···O12^{v}	0.99	2.63	3.461 (3)	142
C11—H11*A*···O12^{ii}	0.99	2.63	3.467 (3)	143

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) *x*, −*y*+3/2, *z*+1/2; (iv) *x*, *y*+1, *z*; (v) −*x*, *y*+1/2, −*z*+1/2.