data reports

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3-(2-Ethoxy-2-oxoethyl)-4,5,6,7,8,9-hexahydrocycloocta[*d*][1,2,3]selenadiazol-3-ium bromide

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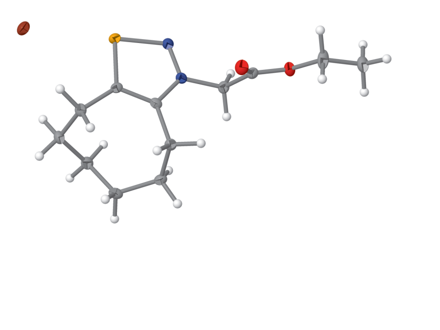
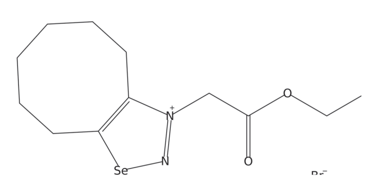
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{12}H_{19}N_2O_2Se^+\cdot Br^-$, features a selenadiazole five-membered ring attached to a cyclooctene ring. A bromine anion is located in the vicinity of the selenium atom [3.0197 (5) Å]

3D view



Chemical scheme



Structure description

1,2,3-Selenadiazoles are known as precursors for alkynes, especially strained cycloalkynes (Bissinger *et al.*, 1988; Detert & Meier, 1997). Jaffari *et al.* (1970) reported benzo-annulated selenadiazolium salts, and the first 1,2,3-selenadiazolium salt was described by Butler & Fox (2001). Recently, *N*-methylated selenadiazoles were characterized by us (Schollmeyer & Detert, 2016, 2017).

The molecular structure of the title compound (Fig. 1) is composed of a cyclooctene ring with a boat-twist conformation, a 1,2,3-selenadiazole ring, an ethylacetate unit, and a bromide anion in the vicinity of the selenium atom. The selenadiazole ring is planar with a maximum deviation of 0.018 (3) Å from the mean plane at N2 whereas N3 is slightly below the ring. In spite of the conformational freedom, the ester unit, C12–C17, is almost planar; here the maximum deviation from the mean plane is 0.117 (2) Å at O15. These planes subtend a dihedral angle of 77.24 (14)°. The cyclooctene ring adopts a distorted boat-chair conformation (Evans & Boeyens, 1988). The bromide ion is located in the vicinity of the selenium atom [3.0197 (5) Å], opposite to the carbonyl group and slightly below the selenadiazole plane [0.5364 (3) Å]. The packing is shown in Fig. 2.

Synthesis and crystallization

The title compound was prepared by adding ethyl bromoacetate (2.5 ml) to a solution of cycloocteno-1,2,3-selenadiazole (0.9 g, 4 mmol) (Meier & Voigt, 1972) in nitromethane (12 ml). The mixture was kept for one month at room temperature under exclusion of light. Two isomeric selenadiazolium salts in a 2.5:1 ratio were formed (^{1}H-NMR), the

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Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{19}N_2O_2Se^+\cdot Br^-$
M_r	382.16
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	120
a, b, c (Å)	8.4924 (7), 9.3927 (7), 9.7799 (8)
α, β, γ (°)	71.379 (6), 86.439 (7), 74.119 (6)
V (Å^3)	710.78 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.45
Crystal size (mm)	0.37 × 0.35 × 0.18
Data collection	
Diffractometer	Stoe *IPDS* 2T
Absorption correction	Integration [*X-RED32* (Stoe, & Cie, 2020), absorption correction by Gaussian integration, analogous to Coppens (1970)]
T_{min}, T_{max}	0.163, 0.405
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6548, 3370, 3085
R_{int}	0.030
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.659
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.035, 0.096, 1.10
No. of reflections	3370
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	1.39, −0.84

Computer programs: *X-AREA WinXpose*, *Recipe*, *Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *Mercury* (Macrae *et al.*, 2020).

main isomer was isolated by evaporation of the solvent and chromatography on silica gel using chloroform/propanol-2 as eluent. Yield: 0.65 g of the pure title compound (43%), m.p.: 435 K. IR (KBr): 2975, 2912, 2855, 1733, 1711, 1522, 1472, 1447, 1368, 1339, 1240, 1220, 1022 cm^{-1}. ^{1}H-NMR (CDCl$_3$, 400 MHz): 5.63 (*s*, 2 H, N—CH$_2$; ^{13}C-satellites, *J* = 148 Hz), 4.26, (*q*, *J* = 7.5 Hz, OCH$_2$), 3.75 (pseudo-*t*, 2 H, 10-CH$_2$), 3.17 (pseudo-*t*, 2 H, 5-CH$_2$), 1.90 (*qui*, 2 H, 9-CH$_2$), 1.78 (*qui*, 2 H, 6-CH$_2$), 1.40 (*m*, 4 H, CH$_2$), 1.26 ppm (*t*, 3 H, CH$_3$). NOE: Irradiation into 5.63: positive NOE at 3.17, 1.78 ppm. 175.6 (C11, Se-satellites, $^1J_{C\text{–}Se}$ = 160 Hz), 164.0 (C=O), 154.4 (C-4) 64.4 (OCH$_2$), 61.0 (NCH$_2$), 31.2 (C-9), 30.5 (C-10), 28.2 (C-6), 26.8 (C-5), 25.6 (C-7), 24.8 (C-7), 13.9 (CH$_3$) ppm. Numbering of atoms according to scheme 1. MS: (EI): 685 (19%, Se_2Br-isotope pattern), $(C_{12}H_{19}O_2N_2Se)_2Br^+$); 381 (4%, *M*+).

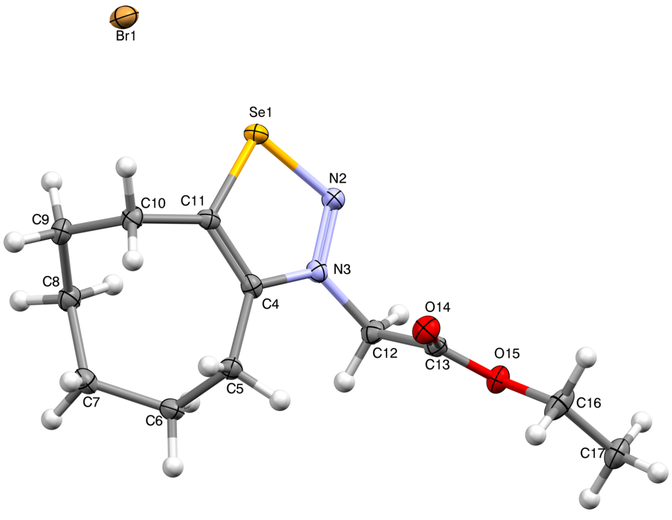

Figure 1
View (Macrae *et al.*, 2020) of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

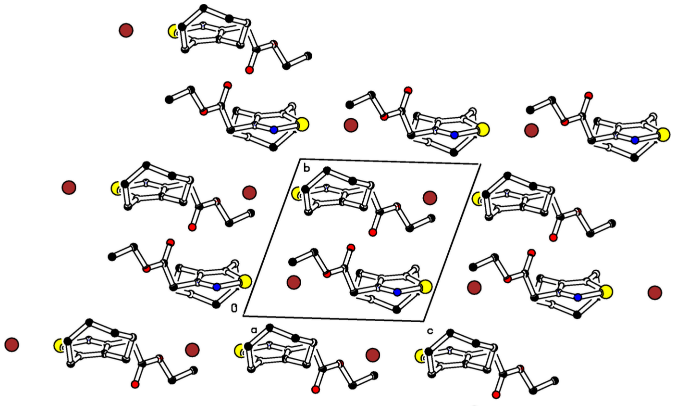

Figure 2
Part of the packing diagram. View along *a*-axis direction (Spek, 2020).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

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full crystallographic data

IUCrData (2025). **10**, x250143 [https://doi.org/10.1107/S2414314625001439]

3-(2-Ethoxy-2-oxoethyl)-4,5,6,7,8,9-hexahydrocycloocta[*d*][1,2,3]selenadiazol-3-ium bromide

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3-(2-Ethoxy-2-oxoethyl)-4,5,6,7,8,9-hexahydrocycloocta[*d*][1,2,3]selenadiazol-3-ium bromide

Crystal data

$C_{12}H_{19}N_2O_2Se^+\cdot Br^-$	$F(000) = 380$
$M_r = 382.16$	$D_x = 1.786$ Mg m^{-3}
Triclinic, $P\overline{1}$	Melting point: 435 K
$a = 8.4924$ (7) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.3927$ (7) Å	Cell parameters from 10409 reflections
$c = 9.7799$ (8) Å	$\theta = 2.5$–$28.4°$
$\alpha = 71.379$ (6)°	$\mu = 5.45$ mm^{-1}
$\beta = 86.439$ (7)°	$T = 120$ K
$\gamma = 74.119$ (6)°	Block, colorless
$V = 710.78$ (10) Å^3	0.37 × 0.35 × 0.18 mm
$Z = 2$	

Data collection

Stoe IPDS 2T diffractometer	$T_{\min} = 0.163$, $T_{\max} = 0.405$
Radiation source: sealed X-ray tube, 12x0.4mm long-fine focus	6548 measured reflections
Detector resolution: 6.67 pixels mm^{-1}	3370 independent reflections
rotation method, ω scans	3085 reflections with $I > 2\sigma(I)$
Absorption correction: integration [X-Red32 (Stoe, & Cie, 2020), absorption correction by Gaussian integration, analogous to Coppens (1970)]	$R_{\text{int}} = 0.030$
	$\theta_{\max} = 27.9°$, $\theta_{\min} = 2.5°$
	$h = -9\rightarrow 11$
	$k = -12\rightarrow 12$
	$l = -12\rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 1.110P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.10$	$\Delta\rho_{\max} = 1.39$ e Å^{-3}
3370 reflections	$\Delta\rho_{\min} = -0.84$ e Å^{-3}
164 parameters	
0 restraints	
Primary atom site location: dual	

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 were refined as riding on their parent atoms with C—H = 0.99 Å for methylene groups and with C—H = 0.98 Å for methyl groups. Isotropic displacement parameters of the H atoms were set to $1.2U_{eq}$(C) or $1.5U_{eq}$(C_{methyl}).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.49934 (4)	0.22173 (3)	1.21107 (3)	0.02302 (10)
Se1	0.34890 (3)	0.21865 (3)	0.94061 (3)	0.01662 (10)
O14	0.1838 (3)	0.4488 (2)	0.4557 (2)	0.0206 (4)
O15	0.0967 (3)	0.2826 (2)	0.3758 (2)	0.0178 (4)
N2	0.2602 (3)	0.1785 (3)	0.7980 (3)	0.0173 (5)
N3	0.3497 (3)	0.2043 (3)	0.6843 (3)	0.0144 (4)
C4	0.4827 (3)	0.2615 (3)	0.6848 (3)	0.0143 (5)
C5	0.5890 (3)	0.2910 (3)	0.5563 (3)	0.0164 (5)
H5A	0.626019	0.384490	0.548184	0.020*
H5B	0.522922	0.313715	0.468181	0.020*
C6	0.7403 (4)	0.1532 (3)	0.5634 (3)	0.0187 (5)
H6A	0.704205	0.056317	0.590697	0.022*
H6B	0.785283	0.168197	0.465499	0.022*
C7	0.8790 (3)	0.1298 (3)	0.6690 (3)	0.0197 (6)
H7A	0.977802	0.055683	0.648121	0.024*
H7B	0.905037	0.230735	0.650026	0.024*
C8	0.8431 (4)	0.0691 (3)	0.8295 (3)	0.0191 (5)
H8A	0.940889	−0.013424	0.878496	0.023*
H8B	0.752247	0.019621	0.838569	0.023*
C9	0.7976 (3)	0.1894 (3)	0.9106 (3)	0.0187 (5)
H9A	0.893461	0.229792	0.911700	0.022*
H9B	0.776542	0.135081	1.012018	0.022*
C10	0.6482 (3)	0.3294 (3)	0.8502 (3)	0.0163 (5)
H10A	0.614406	0.385169	0.922162	0.020*
H10B	0.679218	0.402734	0.761909	0.020*
C11	0.5076 (3)	0.2798 (3)	0.8151 (3)	0.0139 (5)
C12	0.2965 (3)	0.1714 (3)	0.5601 (3)	0.0167 (5)
H12A	0.237092	0.089818	0.594280	0.020*
H12B	0.393413	0.131917	0.507588	0.020*
C13	0.1851 (3)	0.3189 (3)	0.4589 (3)	0.0161 (5)
C16	−0.0031 (4)	0.4150 (4)	0.2620 (4)	0.0263 (7)
H16A	0.064370	0.484369	0.207894	0.032*
H16B	−0.094754	0.476150	0.304881	0.032*
C17	−0.0676 (4)	0.3498 (4)	0.1638 (4)	0.0245 (6)
H17A	−0.131725	0.435342	0.084350	0.037*
H17B	−0.137472	0.284484	0.217717	0.037*

H17C	0.024140	0.286559	0.124624	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02640 (17)	0.02727 (17)	0.01503 (16)	−0.00549 (12)	0.00133 (12)	−0.00775 (12)
Se1	0.01565 (15)	0.02022 (16)	0.01349 (16)	−0.00509 (11)	0.00309 (10)	−0.00490 (11)
O14	0.0246 (10)	0.0171 (9)	0.0214 (10)	−0.0071 (8)	−0.0009 (8)	−0.0062 (8)
O15	0.0195 (10)	0.0160 (9)	0.0174 (10)	−0.0055 (8)	−0.0041 (8)	−0.0030 (8)
N2	0.0151 (11)	0.0218 (11)	0.0152 (11)	−0.0053 (9)	0.0007 (9)	−0.0056 (9)
N3	0.0135 (10)	0.0147 (10)	0.0158 (11)	−0.0046 (8)	0.0007 (8)	−0.0050 (8)
C4	0.0124 (11)	0.0122 (11)	0.0166 (12)	−0.0033 (9)	0.0003 (9)	−0.0021 (9)
C5	0.0161 (12)	0.0181 (12)	0.0147 (13)	−0.0068 (10)	0.0014 (10)	−0.0032 (10)
C6	0.0173 (12)	0.0230 (13)	0.0172 (13)	−0.0063 (11)	0.0046 (10)	−0.0081 (11)
C7	0.0139 (12)	0.0205 (13)	0.0232 (15)	−0.0033 (10)	0.0026 (11)	−0.0062 (11)
C8	0.0179 (13)	0.0187 (13)	0.0173 (13)	−0.0028 (10)	−0.0016 (10)	−0.0027 (10)
C9	0.0163 (12)	0.0225 (13)	0.0173 (13)	−0.0046 (10)	−0.0031 (10)	−0.0060 (11)
C10	0.0178 (12)	0.0155 (12)	0.0169 (13)	−0.0060 (10)	−0.0007 (10)	−0.0053 (10)
C11	0.0149 (12)	0.0117 (11)	0.0138 (12)	−0.0025 (9)	0.0022 (9)	−0.0037 (9)
C12	0.0184 (13)	0.0168 (12)	0.0173 (13)	−0.0061 (10)	0.0002 (10)	−0.0074 (10)
C13	0.0170 (12)	0.0194 (12)	0.0145 (12)	−0.0088 (10)	0.0033 (10)	−0.0059 (10)
C16	0.0371 (17)	0.0187 (13)	0.0204 (15)	−0.0025 (12)	−0.0127 (13)	−0.0043 (11)
C17	0.0245 (15)	0.0270 (15)	0.0213 (15)	−0.0055 (12)	−0.0067 (12)	−0.0064 (12)

Geometric parameters (Å, º)

Se1—N2	1.811 (3)	C8—C9	1.533 (4)
Se1—C11	1.850 (3)	C8—H8A	0.9900
O14—C13	1.208 (3)	C8—H8B	0.9900
O15—C13	1.316 (3)	C9—C10	1.541 (4)
O15—C16	1.469 (4)	C9—H9A	0.9900
N2—N3	1.303 (3)	C9—H9B	0.9900
N3—C4	1.378 (3)	C10—C11	1.490 (4)
N3—C12	1.470 (4)	C10—H10A	0.9900
C4—C11	1.375 (4)	C10—H10B	0.9900
C4—C5	1.498 (4)	C12—C13	1.523 (4)
C5—C6	1.541 (4)	C12—H12A	0.9900
C5—H5A	0.9900	C12—H12B	0.9900
C5—H5B	0.9900	C16—C17	1.491 (4)
C6—C7	1.538 (4)	C16—H16A	0.9900
C6—H6A	0.9900	C16—H16B	0.9900
C6—H6B	0.9900	C17—H17A	0.9800
C7—C8	1.532 (4)	C17—H17B	0.9800
C7—H7A	0.9900	C17—H17C	0.9800
C7—H7B	0.9900		

N2—Se1—C11	89.04 (12)	C10—C9—H9A	108.2
C13—O15—C16	115.5 (2)	C8—C9—H9B	108.2

N3—N2—Se1	109.05 (18)	C10—C9—H9B	108.2
N2—N3—C4	120.2 (2)	H9A—C9—H9B	107.4
N2—N3—C12	115.6 (2)	C11—C10—C9	111.8 (2)
C4—N3—C12	124.2 (2)	C11—C10—H10A	109.3
C11—C4—N3	112.7 (2)	C9—C10—H10A	109.3
C11—C4—C5	125.8 (2)	C11—C10—H10B	109.3
N3—C4—C5	121.5 (2)	C9—C10—H10B	109.3
C4—C5—C6	113.5 (2)	H10A—C10—H10B	107.9
C4—C5—H5A	108.9	C4—C11—C10	124.5 (2)
C6—C5—H5A	108.9	C4—C11—Se1	109.0 (2)
C4—C5—H5B	108.9	C10—C11—Se1	126.3 (2)
C6—C5—H5B	108.9	N3—C12—C13	110.1 (2)
H5A—C5—H5B	107.7	N3—C12—H12A	109.6
C7—C6—C5	115.8 (2)	C13—C12—H12A	109.6
C7—C6—H6A	108.3	N3—C12—H12B	109.6
C5—C6—H6A	108.3	C13—C12—H12B	109.6
C7—C6—H6B	108.3	H12A—C12—H12B	108.2
C5—C6—H6B	108.3	O14—C13—O15	126.4 (3)
H6A—C6—H6B	107.4	O14—C13—C12	123.5 (3)
C8—C7—C6	115.6 (2)	O15—C13—C12	110.1 (2)
C8—C7—H7A	108.4	O15—C16—C17	107.3 (2)
C6—C7—H7A	108.4	O15—C16—H16A	110.3
C8—C7—H7B	108.4	C17—C16—H16A	110.3
C6—C7—H7B	108.4	O15—C16—H16B	110.3
H7A—C7—H7B	107.4	C17—C16—H16B	110.3
C7—C8—C9	116.7 (2)	H16A—C16—H16B	108.5
C7—C8—H8A	108.1	C16—C17—H17A	109.5
C9—C8—H8A	108.1	C16—C17—H17B	109.5
C7—C8—H8B	108.1	H17A—C17—H17B	109.5
C9—C8—H8B	108.1	C16—C17—H17C	109.5
H8A—C8—H8B	107.3	H17A—C17—H17C	109.5
C8—C9—C10	116.3 (2)	H17B—C17—H17C	109.5
C8—C9—H9A	108.2		

C11—Se1—N2—N3	2.47 (19)	C5—C4—C11—C10	−1.3 (4)
Se1—N2—N3—C4	−3.2 (3)	N3—C4—C11—Se1	0.0 (3)
Se1—N2—N3—C12	177.90 (18)	C5—C4—C11—Se1	−177.0 (2)
N2—N3—C4—C11	2.2 (4)	C9—C10—C11—C4	−87.9 (3)
C12—N3—C4—C11	−179.0 (2)	C9—C10—C11—Se1	87.2 (3)
N2—N3—C4—C5	179.4 (2)	N2—Se1—C11—C4	−1.4 (2)
C12—N3—C4—C5	−1.8 (4)	N2—Se1—C11—C10	−177.1 (2)
C11—C4—C5—C6	82.8 (3)	N2—N3—C12—C13	93.4 (3)
N3—C4—C5—C6	−94.1 (3)	C4—N3—C12—C13	−85.5 (3)
C4—C5—C6—C7	−74.3 (3)	C16—O15—C13—O14	4.8 (4)
C5—C6—C7—C8	71.2 (3)	C16—O15—C13—C12	−173.6 (2)
C6—C7—C8—C9	−102.0 (3)	N3—C12—C13—O14	21.8 (4)
C7—C8—C9—C10	57.4 (3)	N3—C12—C13—O15	−159.8 (2)
C8—C9—C10—C11	45.7 (3)	C13—O15—C16—C17	169.4 (3)

N3—C4—C11—C10	175.8 (2)