IUCrData

ISSN 2414-3146

rac-4*H*,5*H*,6*H*,7*H*,8*H*,9*H*,10*H*,11*H*-Cyclodeca[*d*]-[1,2,3]selenadiazol-4-ol

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Received 27 January 2026
Accepted 29 January 2026

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

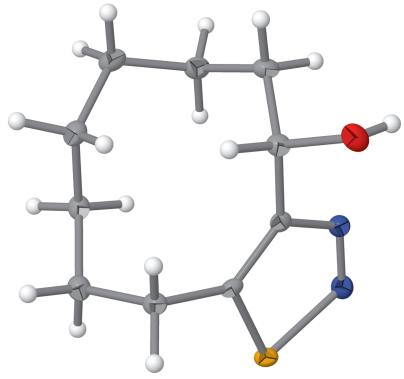
Keywords: crystal structure; heterocycle; selenium; medium-sized ring; hydrogen bond.

CCDC reference: 2526821

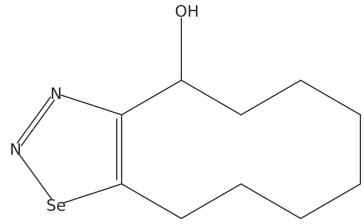
Structural data: full structural data are available from iucrdata.iucr.org

Two molecules of the title compound, $C_{10}H_{16}N_2OSe$, with a chair conformation are connected *via* hydrogen bonds into centrosymmetric dimers. C—H···O hydrogen bonds interconnect the dimers.

3D view



Chemical scheme



Structure description

The title compound, $C_{10}H_{16}N_2OSe$ (Fig. 1), was prepared in a project focusing on transannular cyclizations (Detert *et al.*, 1992; Krämer *et al.*, 2009; Meier *et al.*,) in medium-sized cycloalkynes (Detert & Schollmeyer, 2021; Herges *et al.*, 2005). The molecule adopts a chair conformation. There are two planes, one is the heterocycle and adjacent C atoms (C5, C12), the other is composed of C5, C6, C7 and C10, C11, C12. The former is planar within 0.0357 (16) Å at C5, the latter within 0.1002 (17) Å at C7. Both planes are close to orthogonal, making a dihedral angle of 85.02 (5)°. The methylene groups CH_2-8, CH_2-9 are staggered. In the crystal, pairs of molecules form centrosymmetric dimers, connected *via* O14—H14···N3^{i} hydrogen bonds (Table 1, Fig. 2). The molecules of the dimers are connected to neighbouring molecules, one *via* a *c*-glide plane, the other one *via* translation along the *c*-axis. For details of the C—H···O hydrogen bonds connecting the dimers, see Table 1.

Synthesis and crystallization

The sample was prepared from sebacoin (Prelog *et al.*, 1947; Rühlmann, 1971) *via* acetylation (Carlson & Bateman 1967), formation and oxidation of its semicarbazone and deacetylation with 2-aminoethanol, m.p. 418 K. Crystallization was by slow evaporation of a solution in methanol/dichloromethane. ^{1}H-NMR (250 MHz, $CDCl_3$): 5.18 (*dd*, 1 H, 4-H), 3.24 (*m*, 2H), 2.18-2.43 (*m*, 3 H), 1.58-1.90 (*m*, 2H), 1.15-1.55 (*m*, 8 H). ^{13}C-NMR (100 MHz, $CDCl_3$): 161.96 (C-3a, $^2J_{C-Se}$ = 27 Hz), 161.34 (C-11*a*, $^1J_{C-Se}$ = 135 Hz), 67.53

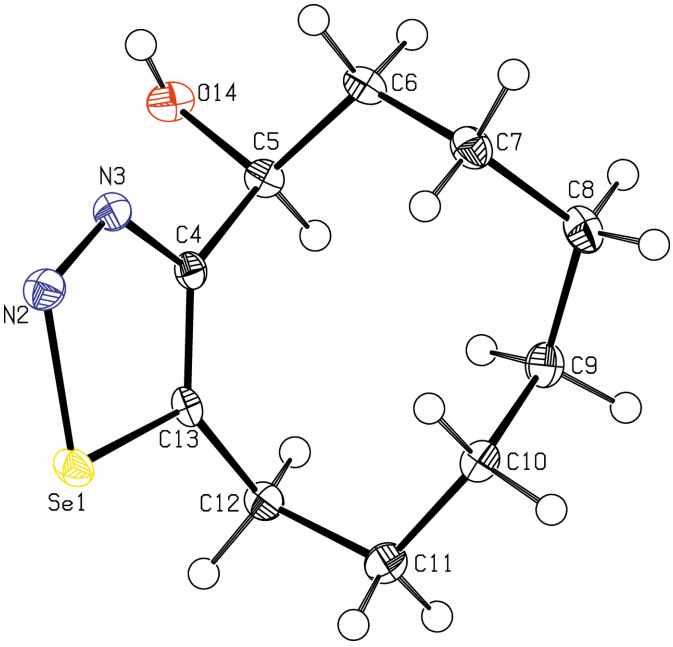


Figure 1
View of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

(C-4), 38.78 (C-11), 30.32, 26.94, 25.04, 24.21, 21.88, 19.70. ^{77}Se-NMR (76.3 MHz, $CDCl_3$): 1525.8 p.p.m. (Me_2Se = 0).

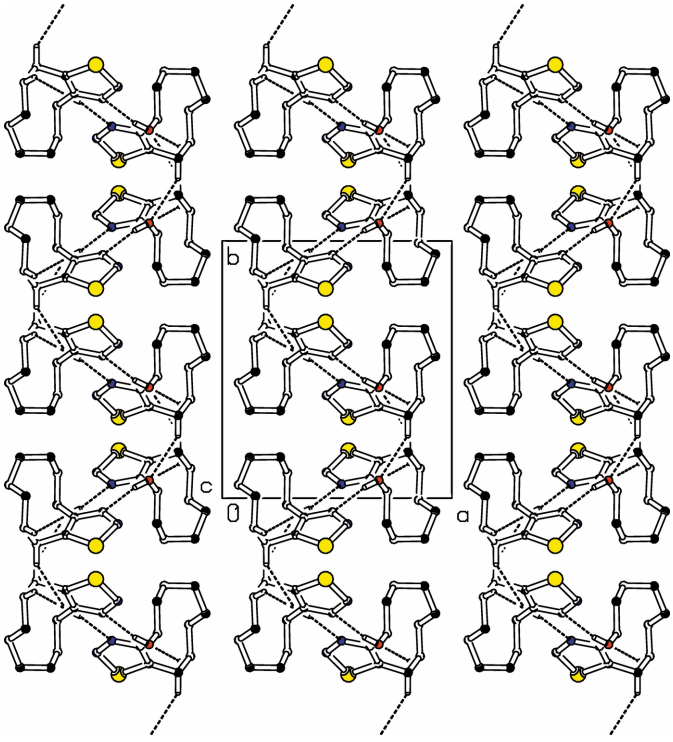


Figure 2
Part of the packing diagram. Hydrogen bonds are drawn with dashed lines. View along *c*-axis direction. Only hydrogen atoms involved in hydrogen bonds are shown for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12B···O14^{i}	0.99	2.58	3.430 (2)	144
O14—H14···N3^{ii}	0.78 (3)	2.22 (3)	2.9801 (18)	165 (2)
C11—H11A···O14^{iii}	0.99	2.56	3.419 (2)	145

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{16}N_2OSe$
M_r	259.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	11.3765 (5), 12.3700 (4), 7.6033 (3)
β (°)	103.747 (3)
V (Å^3)	1039.34 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.58
Crystal size (mm)	0.35 × 0.32 × 0.08
Data collection	
Diffractometer	Stoe Stadivari
Absorption correction	Integration [*X-RED32* (Stoe & Cie, 2020), absorption correction by Gaussian integration, analogous to Coppens (1970)]
T_{min}, T_{max}	0.672, 0.911
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9011, 2636, 2338
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.671
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.025, 0.068, 1.08
No. of reflections	2636
No. of parameters	131
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.63, −0.43

Computer programs: *X-AREA WinXpose*, *Recipe* and *Integrate* (Stoe & Cie, 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**, x260095 [https://doi.org/10.1107/S2414314626000957]

rac-4*H*,5*H*,6*H*,7*H*,8*H*,9*H*,10*H*,11*H*-Cyclodeca[*d*][1,2,3]selenadiazol-4-ol

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Crystal data

$C_{10}H_{16}N_2OSe$
M_r = 259.21
Monoclinic, $P2_1/c$
a = 11.3765 (5) Å
b = 12.3700 (4) Å
c = 7.6033 (3) Å
β = 103.747 (3)°
V = 1039.34 (7) Å^3
Z = 4
$F(000)$ = 528
D_x = 1.657 Mg m^{-3}
Melting point: 418 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 15860 reflections
θ = 2.5–33.7°
μ = 3.58 mm^{-1}
T = 120 K
Plate, brown
0.35 × 0.32 × 0.08 mm

Data collection

Stoe Stadivari diffractometer
Radiation source: Axo Mo
Detector resolution: 13.33 pixels mm^{-1}
rotation method, ω scans
Absorption correction: integration [X-Red32 (Stoe & Cie, 2020), absorption correction by Gaussian integration, analogous to Coppens (1970)]
T_{min} = 0.672, T_{max} = 0.911
9011 measured reflections
2636 independent reflections
2338 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 28.5°, θ_{min} = 2.5°
h = −15→15
k = −16→16
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.068
S = 1.08
2636 reflections
131 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.0208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 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 $C_{methylene}$–H = 0.99 Å, $C_{tertiary}$–H = 1.00 Å, and with U_{iso}(H) = 1.2 U_{eq}(C). Hydrogen atom H14 attached to O14 was refined isotropically.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	0.45006 (2)	0.68523 (2)	0.48407 (2)	0.01632 (7)
N2	0.54559 (12)	0.60029 (12)	0.37125 (19)	0.0175 (3)
N3	0.48019 (12)	0.55443 (11)	0.23022 (18)	0.0148 (3)
C4	0.35750 (14)	0.57504 (12)	0.1901 (2)	0.0128 (3)
C5	0.28163 (15)	0.52610 (13)	0.0178 (2)	0.0143 (3)
H5	0.195907	0.548864	0.007480	0.017*
C6	0.28386 (16)	0.40234 (13)	0.0134 (2)	0.0179 (3)
H6A	0.365938	0.379757	0.005564	0.021*
H6B	0.227068	0.378654	−0.099816	0.021*
C7	0.25231 (16)	0.34018 (14)	0.1707 (2)	0.0185 (3)
H7A	0.303042	0.369285	0.285035	0.022*
H7B	0.276149	0.263773	0.161795	0.022*
C8	0.12010 (16)	0.34178 (15)	0.1858 (3)	0.0206 (4)
H8A	0.068247	0.320765	0.066855	0.025*
H8B	0.110468	0.285582	0.274041	0.025*
C9	0.07213 (15)	0.44839 (14)	0.2427 (2)	0.0182 (3)
H9A	−0.012947	0.437897	0.249508	0.022*
H9B	0.072662	0.503203	0.148010	0.022*
C10	0.14468 (14)	0.49220 (13)	0.4253 (2)	0.0157 (3)
H10A	0.113867	0.458365	0.523539	0.019*
H10B	0.230420	0.470593	0.442049	0.019*
C11	0.13838 (15)	0.61496 (14)	0.4428 (2)	0.0182 (3)
H11A	0.182414	0.635982	0.566559	0.022*
H11B	0.052684	0.636301	0.427624	0.022*
C12	0.19156 (15)	0.67821 (12)	0.3053 (2)	0.0154 (3)
H12A	0.139014	0.667721	0.182503	0.018*
H12B	0.191917	0.756274	0.334065	0.018*
C13	0.31837 (14)	0.64318 (13)	0.3054 (2)	0.0132 (3)
O14	0.31845 (12)	0.56888 (10)	−0.13529 (16)	0.0180 (3)
H14	0.379 (2)	0.542 (2)	−0.143 (3)	0.037 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.01354 (10)	0.01686 (11)	0.01669 (11)	−0.00094 (6)	−0.00010 (7)	−0.00468 (6)
N2	0.0136 (6)	0.0191 (7)	0.0192 (7)	0.0020 (6)	0.0030 (5)	0.0000 (6)
N3	0.0138 (6)	0.0145 (7)	0.0156 (7)	0.0008 (5)	0.0028 (5)	0.0003 (6)
C4	0.0141 (7)	0.0106 (7)	0.0134 (7)	−0.0012 (6)	0.0029 (6)	0.0017 (6)
C5	0.0146 (7)	0.0148 (8)	0.0125 (7)	0.0012 (6)	0.0010 (6)	0.0007 (6)
C6	0.0209 (8)	0.0148 (8)	0.0174 (8)	−0.0005 (7)	0.0033 (7)	−0.0038 (7)
C7	0.0201 (8)	0.0134 (8)	0.0216 (8)	0.0011 (7)	0.0040 (7)	0.0002 (7)

C8	0.0194 (8)	0.0178 (8)	0.0232 (9)	−0.0039 (7)	0.0019 (7)	−0.0023 (7)
C9	0.0148 (7)	0.0197 (8)	0.0196 (8)	−0.0017 (7)	0.0028 (6)	0.0006 (7)
C10	0.0136 (8)	0.0185 (8)	0.0144 (7)	0.0009 (6)	0.0024 (6)	0.0025 (6)
C11	0.0166 (8)	0.0196 (8)	0.0189 (8)	0.0013 (6)	0.0051 (7)	0.0001 (7)
C12	0.0140 (8)	0.0150 (8)	0.0162 (8)	0.0010 (6)	0.0019 (6)	−0.0007 (6)
C13	0.0136 (7)	0.0103 (7)	0.0146 (7)	−0.0027 (6)	0.0012 (6)	0.0003 (6)
O14	0.0214 (6)	0.0183 (6)	0.0147 (6)	0.0046 (5)	0.0048 (5)	0.0027 (5)

Geometric parameters (Å, º)

Se1—C13	1.8418 (16)	C8—H8A	0.9900
Se1—N2	1.8612 (14)	C8—H8B	0.9900
N2—N3	1.2830 (19)	C9—C10	1.536 (2)
N3—C4	1.380 (2)	C9—H9A	0.9900
C4—C13	1.365 (2)	C9—H9B	0.9900
C4—C5	1.513 (2)	C10—C11	1.527 (2)
C5—O14	1.4294 (19)	C10—H10A	0.9900
C5—C6	1.532 (2)	C10—H10B	0.9900
C5—H5	1.0000	C11—C12	1.540 (2)
C6—C7	1.534 (2)	C11—H11A	0.9900
C6—H6A	0.9900	C11—H11B	0.9900
C6—H6B	0.9900	C12—C13	1.506 (2)
C7—C8	1.536 (2)	C12—H12A	0.9900
C7—H7A	0.9900	C12—H12B	0.9900
C7—H7B	0.9900	O14—H14	0.78 (3)
C8—C9	1.528 (2)		
C13—Se1—N2	87.85 (7)	H8A—C8—H8B	107.3
N3—N2—Se1	110.41 (11)	C8—C9—C10	114.14 (14)
N2—N3—C4	117.28 (13)	C8—C9—H9A	108.7
C13—C4—N3	116.05 (14)	C10—C9—H9A	108.7
C13—C4—C5	126.66 (14)	C8—C9—H9B	108.7
N3—C4—C5	117.17 (14)	C10—C9—H9B	108.7
O14—C5—C4	109.85 (13)	H9A—C9—H9B	107.6
O14—C5—C6	110.03 (13)	C11—C10—C9	113.78 (14)
C4—C5—C6	114.20 (13)	C11—C10—H10A	108.8
O14—C5—H5	107.5	C9—C10—H10A	108.8
C4—C5—H5	107.5	C11—C10—H10B	108.8
C6—C5—H5	107.5	C9—C10—H10B	108.8
C5—C6—C7	118.40 (14)	H10A—C10—H10B	107.7
C5—C6—H6A	107.7	C10—C11—C12	114.32 (14)
C7—C6—H6A	107.7	C10—C11—H11A	108.7
C5—C6—H6B	107.7	C12—C11—H11A	108.7
C7—C6—H6B	107.7	C10—C11—H11B	108.7
H6A—C6—H6B	107.1	C12—C11—H11B	108.7
C6—C7—C8	117.81 (15)	H11A—C11—H11B	107.6
C6—C7—H7A	107.9	C13—C12—C11	112.58 (14)
C8—C7—H7A	107.9	C13—C12—H12A	109.1

C6—C7—H7B	107.9	C11—C12—H12A	109.1
C8—C7—H7B	107.9	C13—C12—H12B	109.1
H7A—C7—H7B	107.2	C11—C12—H12B	109.1
C9—C8—C7	117.02 (15)	H12A—C12—H12B	107.8
C9—C8—H8A	108.0	C4—C13—C12	129.46 (14)
C7—C8—H8A	108.0	C4—C13—Se1	108.42 (11)
C9—C8—H8B	108.0	C12—C13—Se1	122.04 (12)
C7—C8—H8B	108.0	C5—O14—H14	109.8 (18)
C13—Se1—N2—N3	−0.03 (12)	C7—C8—C9—C10	−57.3 (2)
Se1—N2—N3—C4	−0.13 (17)	C8—C9—C10—C11	153.10 (15)
N2—N3—C4—C13	0.3 (2)	C9—C10—C11—C12	−62.33 (19)
N2—N3—C4—C5	176.55 (14)	C10—C11—C12—C13	−52.79 (19)
C13—C4—C5—O14	113.06 (17)	N3—C4—C13—C12	−177.09 (15)
N3—C4—C5—O14	−62.74 (18)	C5—C4—C13—C12	7.1 (3)
C13—C4—C5—C6	−122.78 (18)	N3—C4—C13—Se1	−0.29 (17)
N3—C4—C5—C6	61.42 (18)	C5—C4—C13—Se1	−176.14 (13)
O14—C5—C6—C7	177.50 (14)	C11—C12—C13—C4	99.6 (2)
C4—C5—C6—C7	53.4 (2)	C11—C12—C13—Se1	−76.78 (16)
C5—C6—C7—C8	70.6 (2)	N2—Se1—C13—C4	0.18 (12)
C6—C7—C8—C9	−70.8 (2)	N2—Se1—C13—C12	177.26 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*B*···O14^{i}	0.99	2.58	3.430 (2)	144
O14—H14···N3^{ii}	0.78 (3)	2.22 (3)	2.9801 (18)	165 (2)
C11—H11*A*···O14^{iii}	0.99	2.56	3.419 (2)	145

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