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5,14-Diselena-3,4,15,16-tetraazatricyclo-[11.3.0.0^{2,6}]hexadeca-1(13),2(6),3,15-tetraene

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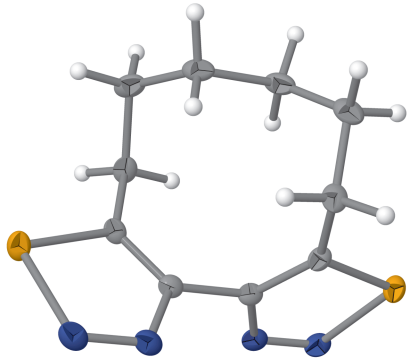
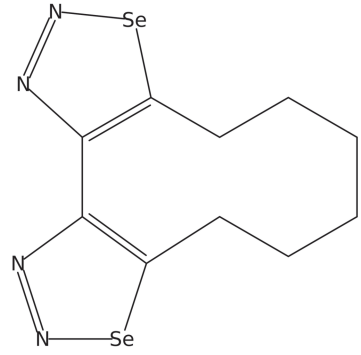
The complete molecule of the title compound, $C_8H_8N_4Se_2$, is generated by a crystallographic twofold axis: the almost planar 1,2,3-selenadiazole ring and its symmetry-generated partner are annulated to a cyclodeca-1,3-diene ring. Their dihedral angle amounts to 88.4 (2)° and the conformation of the hexamethylene chain is mostly staggered (*gauche*) except for a near-*anti* arrangement in the middle.

3D view



Chemical scheme



Structure description

The title compound,$C_8H_8N_4Se_2$ (**I**), was prepared in a project focusing on medium-sized cycloalkynes with additional sterically demanding groups (Bissinger *et al.* 1988; Detert *et al.* 1994; Detert & Meier, 1997). Bis-1,2,3-selanadiazoles are important sources for medium-sized cycloalkadiynes (Gleiter *et al.* 1988) and the structures of three bis-selenadiazolo-cyclooctadienes have recently been reported (Detert & Schollmeyer, 2020, 2026, Schollmeyer & Detert, 2025). Compound (**I**) is thermally unstable and in the presence of tetraphenyl cyclopentadienone, the formal mono-trapping product of cyclodeca-1,3-diyne is formed (42%, m.p. 529 K).

The complete molecule of (**I**) (Fig. 1) is generated by crystallographic twofold symmetry with the rotation axis passing through the mid-points of the C1—C1^{i} and C8—C8^{i} bonds [symmetry code: (i) $1-x, y, \frac{1}{2}-z$] with a dihedral angle between the 1,2,3-selenadiazole ring and its symmetry-generated partner of 88.4 (2)°. This angle is more than twice as large as in the smaller eight-ring homologue (43.6°, Detert & Schollmeyer, 2020). The C1/N2/N3/Se4/C5 selenadiazole ring is planar to within 0.05 (4) Å and the N2—C1—C1^{i}—N2^{i} torsion angle is 91.8 (4)°. The hexamethylene tether shows a nearly perfect staggered conformation with torsion angles of *ca* 60°; only the central C7—C8—C8^{i}—C7^{i} torsion angle of −149.8 (4)° indicates some Pitzer strain. Within the orthorhombic crystal, the twofold symmetry axis parallel to the *b*-axis and a twofold screw axis along the *c*-axis pack the molecules effectively (Fig. 2).

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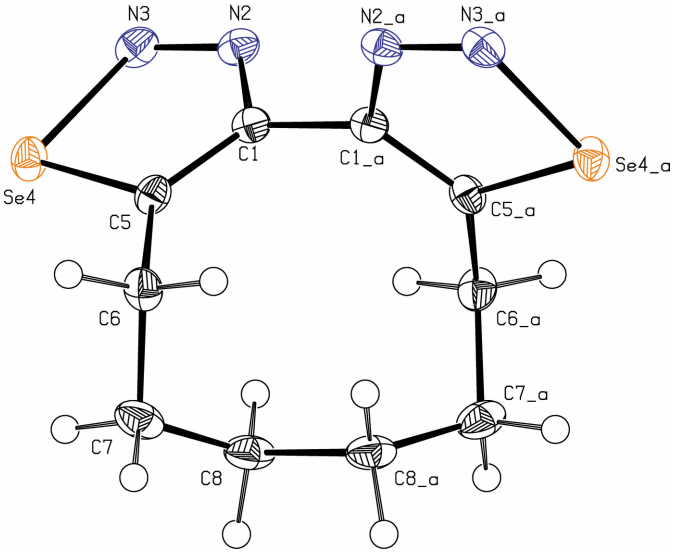

Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level. Atoms with suffix _a are generated by the symmetry operation $1 - x, y, \frac{1}{2} - z$.

Synthesis and crystallization

The title compound was prepared from sebacoic acid *via* esterification with ethanol, an acyloin condensation according to a combination of the Prelog and Rühlmann procedures (Prelog *et al.* 1947; Rühlmann 1971), oxidation with copper acetate (Blomquist & Goldstein 1956), formation of the semicarbazone and reaction with selenium dioxide. The semicarbazone (8.5 g, m.p. 556 K) was suspended in dioxane (70 ml), and selenium dioxide (26.4 g) and water (15 ml) were added and the mixture was stirred at 318 K for 4 days. Filtration, removal of 50 ml dioxane, addition of 50 ml of ethyl acetate, washing of the organic layer with brine, sodium bicarbonate solution, and brine was followed by drying over sodium sulfate and chromatography on silica gel with toluene/ethyl acetate 7/3 yielded 3.5 g (34%) of hexagonal, pinkish crystals with m.p. 398–400 K (dec.). Single crystals of (**I**) were grown by slow evaporation of a solution in toluene. Spectroscopic data: (assignment of signals follows IUPAC nomenclature): ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 3.15, 2.90 (2 × *dt*, 2 × 2H, *J* = 15 Hz; *J*′ = 5 Hz, fast exchange of H—CH'-7,12 and HC—H′-7,12), 1.65 (*m*, 4 H, H_2C-8, 12), 1.35, 0.75 (2 × *bs*, 2 × 2 H, fast exchange of H—CH'-9,10 and HC—H′-9,10); ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 168.5, 152.3 (C-1,2,6,13), 30.0 (C-7, 12,), 24.8, 20.0 (C-8,9,10,11); ^{77}Se-NMR (73 MHz, $CDCl_3$, SeO_2/D_2O as reference): δ = 213.4; UV(EtOH): λ (log ε) = 210 (4.10), 228 (4.16), 286 nm (3.42), MS (FD): 348 ($M^{+\cdot}$, Se_2-isotope pattern), 320 ($M^{+\cdot}$—N_2, Se_2-isotope pattern), 240 ($M^{+\cdot}$—N_2—Se, Se-isotope pattern); MS (EI): 292 (8%, $M^{+\cdot} - 2$ N_2, Se_2-isotope pattern), 212 (5%, $M^{+\cdot}$-2 N_2– Se) 183 (50%), 129 (77%), 91 (100%); IR (KBr): ν = 2920, 2840, 1502, 1448, 1101, 1055, 860, 842 cm^{-1}

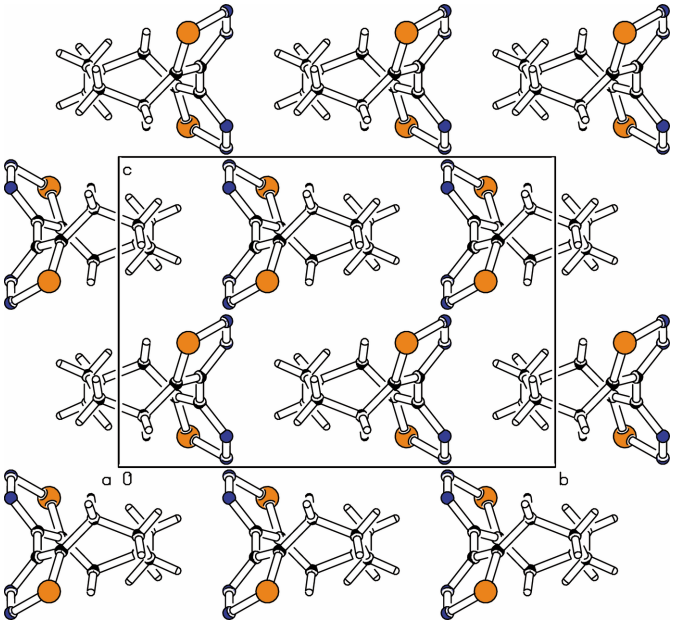

Figure 2
Part of the packing diagram of (**I**) viewed along the *a*-axis direction.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{12}N_4Se_2$
M_r	346.16
Crystal system, space group	Orthorhombic, $C222_1$
Temperature (K)	120
a, *b*, *c* (Å)	8.4957 (7), 14.0651 (10), 9.9897 (6)
V (Å^3)	1193.70 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	6.17
Crystal size (mm)	0.37 × 0.26 × 0.23
Data collection	
Diffractometer	Stoe IPDS 2T
Absorption correction	Integration
T_{min}, T_{max}	0.230, 0.329
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	1663, 1254, 1224
R_{int}	0.014
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.024, 0.060, 1.15
No. of reflections	1254
No. of parameters	73
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.31
Absolute structure	Classical Flack method preferred over Parsons because s.u. lower
Absolute structure parameter	0.08 (3)

Computer programs: *X-AREA WinXpose*, *X-AREA Recipe* and *X-AREA 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 1.

References

Bissinger, H. J., Detert, H. & Meier, H. (1988). *Liebigs Ann. Chem.* pp. 221–224.

Blomquist, A. T. & Goldstein, A. (1956). *Org. Synth.* **36**, 77–79.

Detert, H. & Meier, H. (1997). *Liebigs Ann. Recl* pp. 1557–1563.

Detert, H., Rose, B., Mayer, W. & Meier, H. (1994). *Chem. Ber.* **127**, 1529–1532.

Detert, H. & Schollmeyer, D. (2020). *IUCr Data* **5** x201585.

Detert, H. & Schollmeyer, D. (2026). *IUCr Data* **11**, x260063.

Gleiter, R., Karcher, M., Jahn, R. & Irngartinger, H. (1988). *Chem. Ber.* **121**, 735–740.

Prelog, V., Frenkiel, L., Kobelt, M. & Barman, P. (1947). *Helv. Chim. Acta* **30**, 1741–1749.

Rühlmann, K. (1971). *Synthesis* pp. 236–253.

Schollmeyer, D. & Detert, H. (2025). *IUCr Data* **10**, x250324.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Stoe & Cie (2020). *X-AREA WinXpose*, *X-AREA Recipe* and *X-AREA Integrate*. Stoe & Cie, Darmstadt, Germany.

full crystallographic data

IUCrData (2026). **11**, x261095 [https://doi.org/10.1107/S2414314626010953]

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5,14-Diselena-3,4,15,16-tetraazatricyclo[11.3.0.0^{2,6}]hexadeca-1(13),2(6),3,15-tetraene

Crystal data

$C_{10}H_{12}N_4Se_2$
M_r = 346.16
Orthorhombic, $C222_1$
a = 8.4957 (7) Å
b = 14.0651 (10) Å
c = 9.9897 (6) Å
V = 1193.70 (15) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.926 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3676 reflections
θ = 2.8–28.3°
μ = 6.17 mm^{-1}
T = 120 K
Block, colorless
0.37 × 0.26 × 0.23 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed tube
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
Absorption correction: integration
T_{min} = 0.230, T_{max} = 0.329
1663 measured reflections
1254 independent reflections
1224 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.9°, θ_{min} = 2.8°
h = −11→10
k = −12→18
l = −13→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
S = 1.15
1254 reflections
73 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 2.4527P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Classical Flack method preferred over Parsons because s.u. lower
Absolute structure parameter: 0.08 (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. The H atoms were placed at calculated positions with C—H = 0.99 Å and were refined in the riding-model approximation 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	0.4277 (4)	0.1859 (3)	0.2089 (4)	0.0163 (8)
N2	0.4228 (4)	0.2468 (2)	0.0997 (4)	0.0186 (6)
N3	0.2985 (5)	0.2466 (3)	0.0296 (4)	0.0206 (7)
Se4	0.15092 (4)	0.15946 (3)	0.09906 (4)	0.02012 (13)
C5	0.2978 (5)	0.1314 (3)	0.2288 (4)	0.0174 (8)
C6	0.2739 (5)	0.0574 (3)	0.3352 (4)	0.0202 (8)
H6A	0.171981	0.069125	0.380357	0.024*
H6B	0.358169	0.063595	0.403035	0.024*
C7	0.2750 (6)	−0.0446 (3)	0.2791 (5)	0.0250 (9)
H7A	0.259725	−0.089763	0.353998	0.030*
H7B	0.185083	−0.052100	0.216997	0.030*
C8	0.4257 (6)	−0.0702 (3)	0.2058 (5)	0.0244 (9)
H8A	0.441258	−0.024463	0.131619	0.029*
H8B	0.413158	−0.134127	0.165772	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0180 (18)	0.0161 (18)	0.0147 (16)	0.0009 (14)	0.0040 (15)	0.0011 (14)
N2	0.0211 (16)	0.0173 (15)	0.0173 (14)	0.0024 (12)	0.0037 (15)	0.0004 (16)
N3	0.0230 (16)	0.0207 (17)	0.0182 (17)	0.0031 (14)	0.0039 (14)	0.0054 (15)
Se4	0.01690 (17)	0.02506 (19)	0.01841 (18)	0.00027 (17)	−0.00221 (16)	0.00498 (17)
C5	0.0170 (16)	0.0195 (18)	0.0158 (16)	0.0028 (15)	−0.0010 (14)	0.0023 (15)
C6	0.0187 (19)	0.024 (2)	0.0176 (19)	−0.0023 (16)	0.0015 (16)	0.0071 (16)
C7	0.032 (2)	0.019 (2)	0.024 (2)	−0.0069 (18)	−0.006 (2)	0.0067 (18)
C8	0.035 (3)	0.0148 (18)	0.023 (2)	−0.0014 (17)	−0.010 (2)	−0.0013 (17)

Geometric parameters (Å, º)

C1—C5	1.358 (6)	C6—H6A	0.9900
C1—N2	1.388 (5)	C6—H6B	0.9900
C1—C1^{i}	1.477 (8)	C7—C8	1.518 (7)
N2—N3	1.267 (5)	C7—H7A	0.9900
N3—Se4	1.885 (4)	C7—H7B	0.9900
Se4—C5	1.842 (4)	C8—C8^{i}	1.541 (8)
C5—C6	1.501 (5)	C8—H8A	0.9900
C6—C7	1.540 (6)	C8—H8B	0.9900
C5—C1—N2	116.0 (4)	H6A—C6—H6B	107.8
C5—C1—C1^{i}	126.5 (4)	C8—C7—C6	113.7 (4)
N2—C1—C1^{i}	117.5 (3)	C8—C7—H7A	108.8
N3—N2—C1	117.3 (3)	C6—C7—H7A	108.8

N2—N3—Se4	110.7 (3)	C8—C7—H7B	108.8
C5—Se4—N3	86.99 (17)	C6—C7—H7B	108.8
C1—C5—C6	127.2 (4)	H7A—C7—H7B	107.7
C1—C5—Se4	109.1 (3)	C7—C8—C8^{i}	114.5 (5)
C6—C5—Se4	123.7 (3)	C7—C8—H8A	108.6
C5—C6—C7	112.8 (4)	C8^{i}—C8—H8A	108.6
C5—C6—H6A	109.0	C7—C8—H8B	108.6
C7—C6—H6A	109.0	C8^{i}—C8—H8B	108.6
C5—C6—H6B	109.0	H8A—C8—H8B	107.6
C7—C6—H6B	109.0		

C5—C1—N2—N3	0.5 (5)	C1^{i}—C1—C5—Se4	179.6 (3)
C1^{i}—C1—N2—N3	−179.9 (3)	N3—Se4—C5—C1	0.7 (3)
C1—N2—N3—Se4	0.1 (4)	N3—Se4—C5—C6	−177.7 (4)
N2—N3—Se4—C5	−0.5 (3)	C1—C5—C6—C7	−108.5 (5)
N2—C1—C5—C6	177.5 (4)	Se4—C5—C6—C7	69.6 (5)
C1^{i}—C1—C5—C6	−2.0 (7)	C5—C6—C7—C8	58.0 (5)
N2—C1—C5—Se4	−0.9 (5)	C6—C7—C8—C8^{i}	63.7 (4)

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.