

# *rac*-11-Selena-12,13-diazabicyclo[10.3.0]penta-deca-10a(13a),12-dien-1-ol

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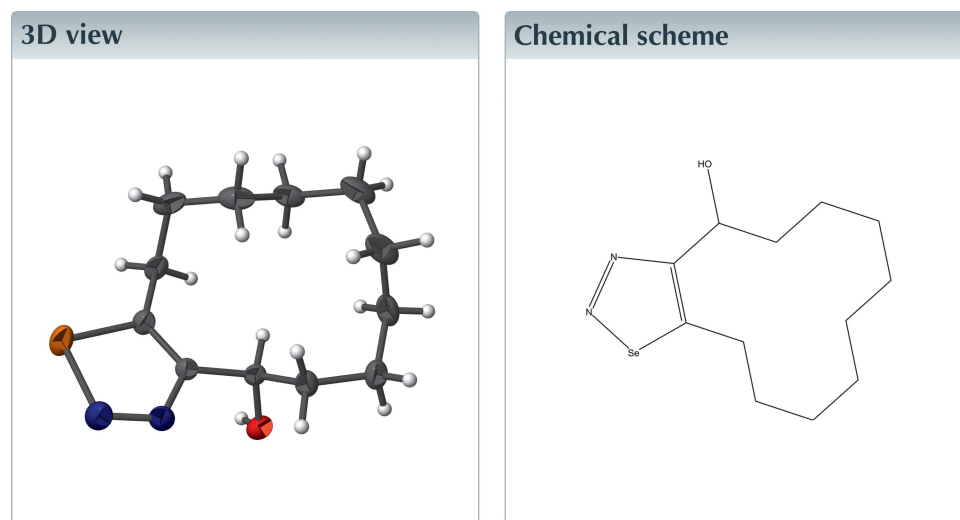
Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

Keywords: crystal structure; heterocycles; medium-sized ring; selenium.

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Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

The title compound, C<sub>12</sub>H<sub>20</sub>N<sub>2</sub>OSe, crystallizes in strands of enantiomeric molecules connected *via* O—H···N hydrogen bonds. There are only slight deviations from an ideal *gauche* conformation in the decamethylene chain, indicating just a little strain.



## Structure description

1,2,3-Selenadiazoles are intermediates in the synthesis of other heterocycles (Detert, 2011), strained (Bissinger *et al.*, 1988) and functionalized cycloalkynes. In addition, they are important for strain-accelerated 3 + 2-cycloadditions (Ziegler & Wilms, 1950; Agard *et al.*, 2004).

There is one molecule of the title compound in the asymmetric unit (Fig. 1), resulting in four molecules filling the unit cell. The crystal is formed from two strands of molecules without directional bonding between the strands (Fig. 2). Within the strands, the enantiomeric molecules are connected *via* *c*-glide symmetry. Additionally, the hydrogen bond O16—H16···N14 (Table 1) consolidates the structure. The geometry of the heterocycle matches nearly perfectly that of a recently reported congener (Detert & Schollmeyer, 2020). Within the decamethylene tether, strain is only visible at C7—C8—C9—C10: the torsion angle of  $-149.0(2)^\circ$  differs by more than  $30^\circ$  from the ideal *trans* conformation. C—C—C bond angles in the tether are  $112\text{--}115^\circ$ , giving further proof of a nearly strain-free ring system.

## Synthesis and crystallization

Acyloin condensation of diethyl dodecanedioate (Stoll & Rouvé, 1947; Rühlmann, 1971), acetylation with acetic anhydride in pyridine, reaction with semicarbazide and oxidation with SeO<sub>2</sub> (Lalezari *et al.*, 1972) to acetoxydodeceno-1,2,3-selenadiazole followed by

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O16—H16 $\cdots$ N14 <sup>i</sup>	0.77 (3)	2.22 (3)	2.976 (2)	170 (3)

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

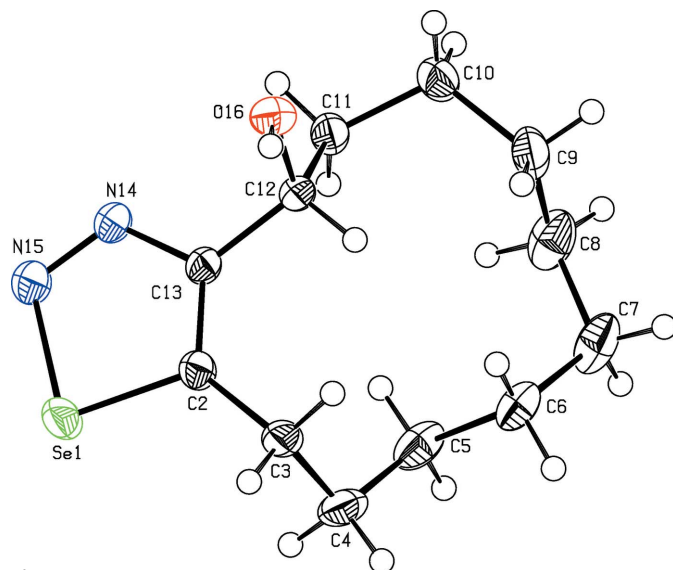
aminolysis of the ester led to the title compound as a viscous oil. Crystallization *via* slow evaporation of a solution in CDCl<sub>3</sub> gave colorless crystals with m.p. = 380 K. Characterization: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): 4.98 (*dd*, 1 H), 3.14 (*ddd*, 1 H), 3.01 (*ddd*, 1 H), 2.30–2.00 (*m*, 3 H), 1.88 (*m*, 1 H), 1.62 (*m*, 1 H). 1.55–0.90 (*m*, 13 H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 75 MHz): 163.3 (Se-satellites,  $J = 134$  Hz), 162.1 (Se-satellites,  $J = 27$  Hz), 37.1, 32.2, 25.0, 24.9, 24.9, 24.3, 24.3, 22.8, 22.8, 22.1. <sup>77</sup>Se NMR: (CDCl<sub>3</sub>, 76.4 MHz, Me<sub>2</sub>Se = 0 p.p.m.)  $\delta = 1528.23$  p.p.m. MS (EI)  $m/z = 174$  (9%, M—N<sub>2</sub>—Se), 146 (14%, M—N<sub>2</sub>—Se—C<sub>2</sub>H<sub>4</sub>), 94 (100%).

## Refinement

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

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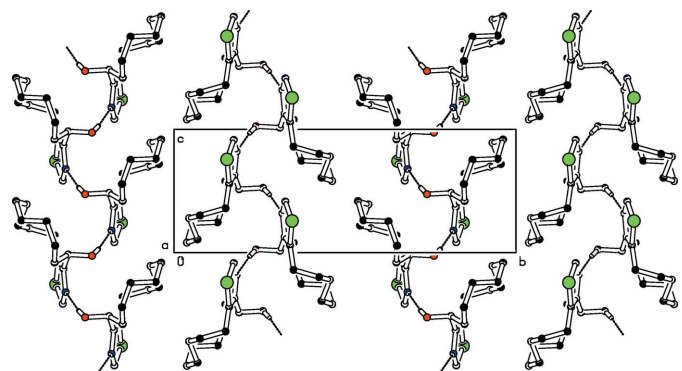
**Figure 1**  
Perspective view of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>12</sub> H <sub>20</sub> N <sub>2</sub> OSe
$M_r$	287.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	193
$a, b, c$ (Å)	8.9261 (5), 19.9277 (9), 7.3829 (4)
$\beta$ (°)	103.321 (4)
$V$ (Å <sup>3</sup> )	1277.91 (12)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	2.92
Crystal size (mm)	0.71 × 0.26 × 0.20
Data collection	
Diffractometer	Stoe IPDS 2T
Absorption correction	Integration [X-RED32 (Stoe & Cie, 2019), absorption correction by Gaussian integration, analogous to Coppens (1970)]
$T_{\min}, T_{\max}$	0.294, 0.601
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	6412, 3020, 2721
$R_{\text{int}}$	0.017
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.029, 0.070, 1.14
No. of reflections	3020
No. of parameters	216
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.41, -0.68

Computer programs: X-AREA WinXpose, Recipe and Integrate (Stoe & Cie, 2019), SIR2004 (Burla *et al.*, 2005), SHELXL2018/3 (Sheldrick, 2015) and PLATON (Spek, 2020).

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**Figure 2**  
Partial packing diagram of the title compound. View along the  $a$ -axis. The dotted line indicates the O—H $\cdots$ N hydrogen bond.

## full crystallographic data

*IUCrData* (2021). 6, x210069 [https://doi.org/10.1107/S2414314621000699]

***rac*-11-Selena-12,13-diazabicyclo[10.3.0]pentadeca-10a(13a),12-dien-1-ol**

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***rac*-11-Selena-12,13-diazabicyclo[10.3.0]pentadeca-10a(13a),12-dien-1-ol***Crystal data*

$C_{12}H_{20}N_2OSe$

$M_r = 287.26$

Monoclinic,  $P2_1/c$

$a = 8.9261$  (5) Å

$b = 19.9277$  (9) Å

$c = 7.3829$  (4) Å

$\beta = 103.321$  (4)°

$V = 1277.91$  (12) Å<sup>3</sup>

$Z = 4$

$F(000) = 592$

$D_x = 1.493$  Mg m<sup>-3</sup>

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

Cell parameters from 9207 reflections

$\theta = 2.6$ – $28.4$ °

$\mu = 2.92$  mm<sup>-1</sup>

$T = 193$  K

Block, colourless

$0.71 \times 0.26 \times 0.20$  mm

*Data collection*

Stoe IPDS 2T

diffractometer

Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus

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

rotation method,  $\omega$  scans

Absorption correction: integration

[X-Red32 (Stoe & Cie, 2019), absorption

correction by Gaussian integration, analogous to Coppens (1970)]

$T_{\min} = 0.294$ ,  $T_{\max} = 0.601$

6412 measured reflections

3020 independent reflections

2721 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 27.9$ °,  $\theta_{\min} = 2.6$ °

$h = -11 \rightarrow 10$

$k = -24 \rightarrow 26$

$l = -9 \rightarrow 9$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.070$

$S = 1.14$

3020 reflections

216 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 0.7552P]$

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

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

$\Delta\rho_{\max} = 0.41$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.67$  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.

**Refinement.** Hydrogen atoms were refined with isotropic displacement parameters constraining the  $U_s$  of two H atoms bonded to the same C atom to the same value.

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	0.82417 (2)	0.15493 (2)	0.75710 (3)	0.03612 (8)
C2	0.6550 (2)	0.16090 (9)	0.5593 (2)	0.0263 (3)
C3	0.6625 (2)	0.15035 (10)	0.3601 (3)	0.0296 (4)
H3A	0.755 (3)	0.1703 (11)	0.344 (3)	0.034 (4)*
H3B	0.579 (3)	0.1750 (12)	0.279 (3)	0.034 (4)*
C4	0.6544 (3)	0.07597 (12)	0.3019 (3)	0.0389 (5)
H4A	0.687 (3)	0.0747 (13)	0.183 (4)	0.044 (5)*
H4B	0.730 (3)	0.0518 (13)	0.394 (4)	0.044 (5)*
C5	0.4957 (3)	0.04424 (11)	0.2800 (3)	0.0378 (5)
H5A	0.462 (3)	0.0536 (13)	0.390 (4)	0.046 (5)*
H5B	0.499 (3)	-0.0049 (14)	0.271 (4)	0.046 (5)*
C6	0.3781 (3)	0.06857 (11)	0.1084 (3)	0.0348 (4)
H6A	0.372 (3)	0.1168 (12)	0.112 (3)	0.034 (4)*
H6B	0.420 (3)	0.0588 (11)	0.000 (3)	0.034 (4)*
C7	0.2171 (3)	0.03777 (13)	0.0816 (3)	0.0470 (6)
H7A	0.221 (3)	-0.0077 (15)	0.071 (4)	0.058 (6)*
H7B	0.149 (3)	0.0529 (14)	-0.043 (4)	0.058 (6)*
C8	0.1357 (3)	0.05394 (12)	0.2377 (3)	0.0459 (5)
H8A	0.200 (3)	0.0389 (14)	0.359 (4)	0.056 (6)*
H8B	0.040 (3)	0.0265 (14)	0.224 (4)	0.056 (6)*
C9	0.0886 (2)	0.12727 (13)	0.2466 (3)	0.0388 (5)
H9A	0.151 (3)	0.1562 (12)	0.186 (4)	0.041 (5)*
H9B	-0.010 (3)	0.1333 (13)	0.172 (4)	0.041 (5)*
C10	0.0892 (2)	0.15211 (12)	0.4425 (3)	0.0356 (4)
H10A	0.049 (3)	0.1978 (14)	0.435 (3)	0.042 (5)*
H10B	0.016 (3)	0.1240 (13)	0.494 (3)	0.042 (5)*
C11	0.2460 (2)	0.15032 (10)	0.5800 (3)	0.0286 (4)
H11A	0.282 (3)	0.1034 (13)	0.606 (3)	0.036 (4)*
H11B	0.238 (3)	0.1680 (12)	0.694 (4)	0.036 (4)*
C12	0.36991 (19)	0.19034 (9)	0.5151 (2)	0.0235 (3)
H12	0.372 (2)	0.1780 (10)	0.388 (3)	0.019 (5)*
C13	0.52951 (19)	0.17624 (9)	0.6290 (2)	0.0229 (3)
N14	0.55574 (18)	0.18287 (8)	0.8195 (2)	0.0273 (3)
N15	0.6932 (2)	0.17459 (9)	0.9149 (2)	0.0337 (4)
O16	0.33526 (16)	0.26009 (7)	0.52548 (19)	0.0298 (3)
H16	0.385 (3)	0.2790 (13)	0.471 (4)	0.042 (7)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.02420 (11)	0.05973 (15)	0.02309 (11)	0.00772 (8)	0.00266 (7)	0.00148 (8)
C2	0.0242 (8)	0.0346 (9)	0.0195 (8)	0.0024 (7)	0.0041 (6)	0.0016 (6)
C3	0.0259 (9)	0.0447 (11)	0.0199 (8)	0.0060 (8)	0.0087 (7)	0.0013 (7)
C4	0.0465 (12)	0.0474 (12)	0.0247 (9)	0.0209 (10)	0.0122 (8)	-0.0004 (8)
C5	0.0617 (14)	0.0285 (10)	0.0255 (9)	0.0074 (9)	0.0147 (9)	0.0011 (7)

C6	0.0507 (12)	0.0313 (10)	0.0237 (9)	-0.0019 (8)	0.0116 (8)	-0.0038 (7)
C7	0.0634 (15)	0.0433 (13)	0.0350 (11)	-0.0173 (11)	0.0127 (10)	-0.0138 (10)
C8	0.0552 (13)	0.0454 (13)	0.0395 (12)	-0.0232 (11)	0.0162 (10)	-0.0096 (9)
C9	0.0289 (10)	0.0558 (13)	0.0286 (9)	-0.0081 (9)	0.0002 (8)	-0.0025 (9)
C10	0.0237 (9)	0.0506 (12)	0.0325 (10)	-0.0025 (8)	0.0063 (7)	-0.0029 (8)
C11	0.0244 (8)	0.0383 (10)	0.0235 (8)	-0.0032 (7)	0.0061 (7)	-0.0013 (7)
C12	0.0227 (8)	0.0293 (9)	0.0191 (7)	0.0020 (6)	0.0060 (6)	-0.0017 (6)
C13	0.0239 (8)	0.0269 (8)	0.0183 (7)	-0.0003 (6)	0.0058 (6)	-0.0006 (6)
N14	0.0288 (7)	0.0346 (8)	0.0187 (7)	0.0013 (6)	0.0059 (6)	-0.0008 (6)
N15	0.0319 (8)	0.0479 (10)	0.0204 (7)	0.0047 (7)	0.0042 (6)	0.0000 (6)
O16	0.0314 (7)	0.0297 (7)	0.0303 (7)	0.0044 (5)	0.0113 (5)	0.0029 (5)

*Geometric parameters (Å, °)*

Se1—C2	1.8467 (18)	C8—C9	1.526 (4)
Se1—N15	1.8723 (17)	C8—H8A	0.99 (3)
C2—C13	1.371 (2)	C8—H8B	1.00 (3)
C2—C3	1.502 (2)	C9—C10	1.528 (3)
C3—C4	1.540 (3)	C9—H9A	0.98 (3)
C3—H3A	0.95 (2)	C9—H9B	0.93 (3)
C3—H3B	0.97 (2)	C10—C11	1.529 (3)
C4—C5	1.525 (3)	C10—H10A	0.98 (3)
C4—H4A	0.99 (3)	C10—H10B	1.00 (3)
C4—H4B	0.97 (3)	C11—C12	1.527 (2)
C5—C6	1.526 (3)	C11—H11A	0.99 (2)
C5—H5A	0.94 (3)	C11—H11B	0.93 (3)
C5—H5B	0.98 (3)	C12—O16	1.430 (2)
C6—C7	1.533 (3)	C12—C13	1.505 (2)
C6—H6A	0.96 (2)	C12—H12	0.97 (2)
C6—H6B	0.97 (2)	C13—N14	1.378 (2)
C7—C8	1.532 (3)	N14—N15	1.276 (2)
C7—H7A	0.91 (3)	O16—H16	0.77 (3)
C7—H7B	1.03 (3)		
C2—Se1—N15	87.97 (7)	C9—C8—H8A	111.0 (17)
C13—C2—C3	128.55 (16)	C7—C8—H8A	109.7 (17)
C13—C2—Se1	107.90 (12)	C9—C8—H8B	106.9 (17)
C3—C2—Se1	123.55 (13)	C7—C8—H8B	110.7 (17)
C2—C3—C4	113.45 (16)	H8A—C8—H8B	103 (2)
C2—C3—H3A	107.5 (14)	C8—C9—C10	114.19 (19)
C4—C3—H3A	110.8 (14)	C8—C9—H9A	110.9 (14)
C2—C3—H3B	109.0 (14)	C10—C9—H9A	111.2 (16)
C4—C3—H3B	109.5 (14)	C8—C9—H9B	109.3 (16)
H3A—C3—H3B	106 (2)	C10—C9—H9B	108.2 (16)
C5—C4—C3	114.30 (16)	H9A—C9—H9B	102 (2)
C5—C4—H4A	110.8 (15)	C9—C10—C11	115.09 (17)
C3—C4—H4A	105.5 (15)	C9—C10—H10A	109.0 (15)
C5—C4—H4B	110.2 (15)	C11—C10—H10A	109.2 (15)

C3—C4—H4B	107.5 (15)	C9—C10—H10B	108.6 (15)
H4A—C4—H4B	108 (2)	C11—C10—H10B	108.1 (14)
C4—C5—C6	113.62 (17)	H10A—C10—H10B	107 (2)
C4—C5—H5A	107.3 (16)	C12—C11—C10	113.45 (16)
C6—C5—H5A	110.8 (16)	C12—C11—H11A	109.0 (14)
C4—C5—H5B	112.2 (16)	C10—C11—H11A	111.0 (14)
C6—C5—H5B	106.6 (15)	C12—C11—H11B	107.5 (15)
H5A—C5—H5B	106 (2)	C10—C11—H11B	109.8 (15)
C5—C6—C7	115.13 (19)	H11A—C11—H11B	106 (2)
C5—C6—H6A	108.9 (14)	O16—C12—C13	109.89 (14)
C7—C6—H6A	110.5 (14)	O16—C12—C11	108.02 (14)
C5—C6—H6B	107.3 (14)	C13—C12—C11	112.81 (14)
C7—C6—H6B	109.7 (14)	O16—C12—H12	110.3 (12)
H6A—C6—H6B	104.7 (19)	C13—C12—H12	105.6 (12)
C8—C7—C6	114.35 (18)	C11—C12—H12	110.2 (12)
C8—C7—H7A	107.9 (19)	C2—C13—N14	116.31 (15)
C6—C7—H7A	110.7 (19)	C2—C13—C12	125.62 (15)
C8—C7—H7B	109.3 (17)	N14—C13—C12	117.96 (14)
C6—C7—H7B	109.9 (16)	N15—N14—C13	117.79 (15)
H7A—C7—H7B	104 (2)	N14—N15—Se1	110.03 (12)
C9—C8—C7	114.6 (2)	C12—O16—H16	107 (2)
N15—Se1—C2—C13	0.38 (14)	C10—C11—C12—C13	-167.50 (16)
N15—Se1—C2—C3	-179.79 (17)	C3—C2—C13—N14	179.73 (18)
C13—C2—C3—C4	-96.5 (2)	Se1—C2—C13—N14	-0.5 (2)
Se1—C2—C3—C4	83.7 (2)	C3—C2—C13—C12	-4.2 (3)
C2—C3—C4—C5	72.5 (2)	Se1—C2—C13—C12	175.63 (14)
C3—C4—C5—C6	71.9 (2)	O16—C12—C13—C2	-108.7 (2)
C4—C5—C6—C7	-179.99 (18)	C11—C12—C13—C2	130.74 (19)
C5—C6—C7—C8	62.7 (3)	O16—C12—C13—N14	67.4 (2)
C6—C7—C8—C9	68.6 (3)	C11—C12—C13—N14	-53.2 (2)
C7—C8—C9—C10	-149.0 (2)	C2—C13—N14—N15	0.3 (3)
C8—C9—C10—C11	62.1 (3)	C12—C13—N14—N15	-176.12 (17)
C9—C10—C11—C12	57.0 (2)	C13—N14—N15—Se1	0.1 (2)
C10—C11—C12—O16	70.83 (19)	C2—Se1—N15—N14	-0.26 (15)

## Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O16—H16...N14 <sup>i</sup>	0.77 (3)	2.22 (3)	2.976 (2)	170 (3)

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