IUCrData

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

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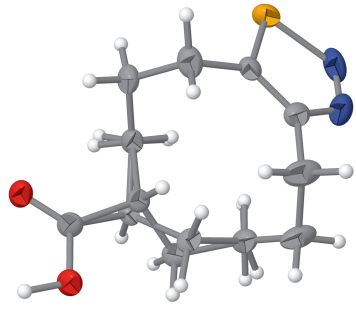
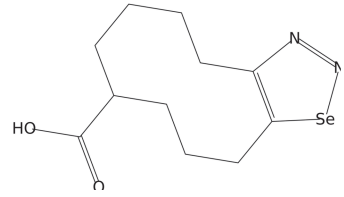
Structural data: full structural data are available from iucrdata.iucr.org

The chair-shaped molecules in the title crystal, $C_{11}H_{16}N_2O_2Se$, consist of two pairs of enantiomers with two conformations (ratio 1:3), differing only in the position of two ring carbon atoms. Two molecules are connected *via* carboxylic acid hydrogen bridges.

3D view



Chemical scheme



Structure description

The title compound, $C_{11}H_{16}N_2O_2Se$, was prepared in a project focusing on transannular cyclizations in medium-sized cycloalkynes (Detert *et al.*, 1992; Detert & Schollmeyer, 2021; Krämer *et al.*, 2009). The unit cell contains four chair-shaped molecules, two pairs of enantiomers with different conformations of the octamethylene chain in an approximate ratio of 1:3 (Fig. 1). The selenadiazole ring is planar to within 0.005 (2) Å; the adjacent carbon atoms are slightly below this plane [C5: −0.029 (2) Å, C12: −0.087 (2) Å]. Torsion angles in the octamethylene tether of the main conformer are C4—C5—C6—C7: −49.4 (3)°, C5—C6—C7—C8: −76.3 (3)°, C6—C7—C8—C9: 70.8 (4)°, C7—C8—C9—C10: 65.5 (5)°, C8—C9—C10—C11: −150.4 (3)°, C9—C10—C11—C12: 62.8 (3)°, and C10—C11—C12—C13: 49.3 (3)°. Interestingly, the positions of most atoms of both conformers are essentially identical, differing only in C8*A* and C9*A* and their attached H atoms. This change provokes a torsion angle C8*A*—C9*A*—C10—C11: −71.5 (9)°. Whereas the C9—H9 bond in the main conformer points in the direction of the selenadiazole plane, this is inverted in the minor conformer. In the extended structure, two molecules are connected *via* H-bridging carboxylic acids, the distances are O16—H16 = 0.94 (3) Å and O15—H15 = 1.69 (3) Å (Table 1, Fig. 2). With an angle of 174 (3)°, the hydrogen bond is slightly bent. The planes of selenadiazole and carboxylic acid dimer are close to parallel, the dihedral angle being only 7.1 (1)°. The disorder is the consequence of the flexibility of the large ring.

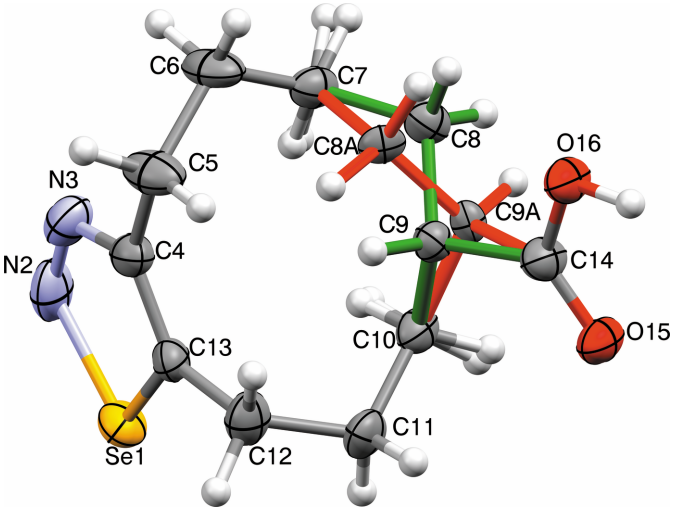


Figure 1
View of the title compound with displacement ellipsoids drawn at the 50% probability level and the minor component shown with red lines (Macrae *et al.*, 2020).

Synthesis and crystallization

The sample was prepared by G. Krämer (1996) from 6-hydroxymethyldecan-1-ol (Becker & Chappuis, 1979) *via* Jones oxidation, formation of the semicarbazone and reaction with selenous acid in 44% overall yield, m.p. 418 K. Crystallization by slow evaporation of a solution in methanol/dichlormethane. ^{1}H-NMR (400 MHz, $CDCl_3$): 11.0 (*vbs*, 1 H), 3.2 (*m*, 3H), 3.05 (*m*, 1H), 2.62 (*m*, 1 H), 2.22 (*m*, 1 H), 1.90 (*m*, 3 H), 1.80-1.50 (*m*, 3H), 1.30 (*m*, 2 H), 1.03 (*m*, 1 H). ^{13}C-NMR (100 MHz, $CDCl_3$): 181.6, 159.7, 159.6, 41.5, 29.4, 28.5, 27.4, 25.5, 22.5, 20.0.

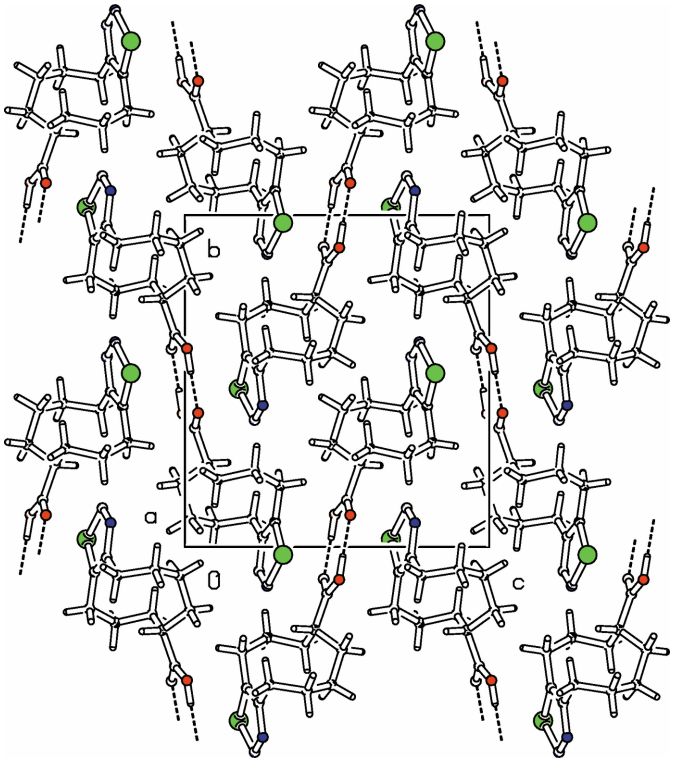


Figure 2
Part of the packing diagram viewed along the *a*-axis direction. Hydrogen bonds are indicated by dashed lines. Only the major disorder component is shown for clarity (Spek, 2009).

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

D—H···A	D—H	H···A	D···A	D—H···A
O16—H16···O15^{i}	0.94 (3)	1.69 (3)	2.630 (2)	174 (3)

Symmetry code: (i) $-x+1, -y, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{16}N_2O_2Se$
M_r	287.22
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	120
a, b, c (Å)	7.4231 (4), 13.2052 (5), 12.2802 (7)
β (°)	99.725 (4)
V (Å^3)	1186.45 (10)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.15
Crystal size (mm)	0.50 × 0.42 × 0.32
Data collection	
Diffractometer	Stoe Stadivari
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2020)
T_{min}, T_{max}	0.277, 0.408
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7535, 2993, 2458
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.671
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.085, 1.04
No. of reflections	2993
No. of parameters	168
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.61, −0.62

Computer programs: *X-AREA WinXpose*, *Recipe* and *Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2020).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Atoms C8 and C9 and their attached H atoms are disordered over two positions [0.759 (8):0.241 (8)].

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full crystallographic data

IUCrData (2025). **10**, x250242 [https://doi.org/10.1107/S2414314625002421]

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Crystal data

$C_{11}H_{16}N_2O_2Se$
M_r = 287.22
Monoclinic, $P2_1/n$
a = 7.4231 (4) Å
b = 13.2052 (5) Å
c = 12.2802 (7) Å
β = 99.725 (4)°
V = 1186.45 (10) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.608 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 13455 reflections
θ = 2.3–33.3°
μ = 3.15 mm^{-1}
T = 120 K
Block, brown
0.50 × 0.42 × 0.32 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)
T_{min} = 0.277, T_{max} = 0.408
7535 measured reflections
2993 independent reflections
2458 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.5°, θ_{min} = 2.3°
h = −9→8
k = −17→11
l = −13→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.085
S = 1.04
2993 reflections
168 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.4785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 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). Hydrogen atom H16 attached to O16 was refined with an isotropic displacement parameter.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Se1	0.42558 (3)	0.52341 (2)	0.82295 (2)	0.03699 (10)	
N2	0.5805 (3)	0.61713 (15)	0.77083 (18)	0.0457 (5)	
N3	0.7176 (3)	0.57253 (16)	0.74387 (17)	0.0414 (5)	
C4	0.7278 (3)	0.46859 (17)	0.75666 (18)	0.0318 (5)	
C5	0.8880 (3)	0.4149 (2)	0.7219 (2)	0.0459 (6)	
H5A	0.877920	0.341396	0.735378	0.055*	
H5B	1.002410	0.439400	0.767597	0.055*	
C6	0.8980 (3)	0.4323 (2)	0.5990 (2)	0.0451 (6)	
H6A	0.938296	0.502895	0.590196	0.054*	
H6B	0.993277	0.387084	0.578566	0.054*	
C7	0.7242 (3)	0.41505 (16)	0.51877 (19)	0.0330 (5)	
H7A	0.738068	0.451200	0.450044	0.040*	0.759 (8)
H7B	0.626215	0.450426	0.549313	0.040*	0.759 (8)
H7C	0.737146	0.428425	0.441144	0.040*	0.241 (8)
H7D	0.618827	0.452876	0.538069	0.040*	0.241 (8)
C8	0.6499 (5)	0.3088 (2)	0.4837 (3)	0.0323 (9)	0.759 (8)
H8A	0.551274	0.315954	0.419001	0.039*	0.759 (8)
H8B	0.749317	0.268862	0.459877	0.039*	0.759 (8)
C9	0.5750 (5)	0.2491 (2)	0.5736 (4)	0.0253 (8)	0.759 (8)
H9	0.672683	0.245793	0.640491	0.030*	0.759 (8)
C8A	0.7192 (13)	0.2967 (6)	0.5470 (10)	0.024 (3)	0.241 (8)
H8C	0.795335	0.258614	0.502232	0.028*	0.241 (8)
H8D	0.769474	0.285547	0.626060	0.028*	0.241 (8)
C9A	0.5227 (15)	0.2591 (6)	0.5220 (11)	0.020 (2)	0.241 (8)
H9A	0.465069	0.279025	0.445437	0.024*	0.241 (8)
C10	0.4030 (3)	0.29664 (15)	0.60785 (18)	0.0300 (4)	
H10A	0.405841	0.371012	0.598136	0.036*	0.759 (8)
H10B	0.292402	0.270147	0.559887	0.036*	0.759 (8)
H10C	0.419373	0.371049	0.608245	0.036*	0.241 (8)
H10D	0.276217	0.284916	0.569739	0.036*	0.241 (8)
C11	0.3948 (3)	0.27181 (16)	0.72804 (19)	0.0356 (5)	
H11A	0.392631	0.197255	0.736219	0.043*	
H11B	0.278436	0.298372	0.745791	0.043*	
C12	0.5532 (4)	0.31423 (17)	0.8131 (2)	0.0377 (5)	
H12A	0.525003	0.304052	0.888300	0.045*	
H12B	0.665876	0.275742	0.807827	0.045*	
C13	0.5875 (3)	0.42449 (15)	0.79660 (17)	0.0278 (4)	
C14	0.5316 (3)	0.14114 (16)	0.5328 (2)	0.0326 (5)	
O15	0.3937 (2)	0.09629 (11)	0.54373 (14)	0.0366 (4)	
O16	0.6672 (2)	0.09795 (12)	0.49285 (14)	0.0359 (4)	
H16	0.637 (4)	0.029 (2)	0.481 (3)	0.051 (9)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.03657 (15)	0.03565 (14)	0.04147 (16)	0.00471 (10)	0.01446 (10)	−0.00838 (10)
N2	0.0629 (15)	0.0245 (9)	0.0463 (12)	−0.0081 (10)	0.0000 (10)	−0.0051 (9)
N3	0.0413 (11)	0.0357 (11)	0.0465 (12)	−0.0110 (9)	0.0056 (9)	0.0039 (9)
C4	0.0290 (11)	0.0375 (12)	0.0290 (11)	−0.0022 (9)	0.0057 (8)	−0.0019 (9)
C5	0.0291 (12)	0.0719 (18)	0.0383 (13)	0.0079 (12)	0.0103 (10)	0.0043 (12)
C6	0.0314 (12)	0.0673 (17)	0.0408 (13)	0.0034 (12)	0.0181 (10)	0.0060 (13)
C7	0.0404 (12)	0.0226 (9)	0.0396 (12)	0.0003 (9)	0.0170 (10)	0.0017 (9)
C8	0.0418 (19)	0.0257 (14)	0.031 (2)	0.0011 (13)	0.0113 (16)	0.0014 (13)
C9	0.0288 (17)	0.0209 (13)	0.026 (2)	0.0001 (12)	0.0051 (14)	0.0003 (13)
C8A	0.026 (5)	0.017 (4)	0.028 (6)	0.005 (3)	0.006 (4)	0.010 (3)
C9A	0.026 (5)	0.014 (4)	0.018 (5)	0.005 (3)	−0.001 (4)	0.002 (4)
C10	0.0288 (10)	0.0206 (9)	0.0424 (12)	0.0009 (8)	0.0115 (9)	−0.0055 (8)
C11	0.0414 (13)	0.0234 (9)	0.0428 (13)	−0.0089 (9)	0.0093 (10)	−0.0023 (9)
C12	0.0459 (14)	0.0249 (10)	0.0408 (13)	−0.0033 (10)	0.0031 (10)	0.0002 (9)
C13	0.0297 (10)	0.0250 (9)	0.0293 (10)	−0.0014 (8)	0.0062 (8)	−0.0069 (8)
C14	0.0360 (12)	0.0235 (9)	0.0415 (12)	0.0014 (9)	0.0157 (9)	−0.0040 (9)
O15	0.0368 (9)	0.0240 (7)	0.0526 (10)	−0.0040 (7)	0.0184 (7)	−0.0080 (7)
O16	0.0399 (9)	0.0220 (7)	0.0501 (10)	−0.0016 (6)	0.0203 (7)	−0.0092 (7)

Geometric parameters (Å, º)

Se1—C13	1.840 (2)	C9—C10	1.544 (4)
Se1—N2	1.874 (2)	C9—H9	1.0000
N2—N3	1.267 (3)	C8A—C9A	1.522 (15)
N3—C4	1.382 (3)	C8A—H8C	0.9900
C4—C13	1.355 (3)	C8A—H8D	0.9900
C4—C5	1.507 (3)	C9A—C14	1.564 (9)
C5—C6	1.540 (3)	C9A—C10	1.570 (9)
C5—H5A	0.9900	C9A—H9A	1.0000
C5—H5B	0.9900	C10—C11	1.523 (3)
C6—C7	1.503 (3)	C10—H10A	0.9900
C6—H6A	0.9900	C10—H10B	0.9900
C6—H6B	0.9900	C10—H10C	0.9900
C7—C8	1.543 (4)	C10—H10D	0.9900
C7—C8A	1.603 (8)	C11—C12	1.540 (3)
C7—H7A	0.9900	C11—H11A	0.9900
C7—H7B	0.9900	C11—H11B	0.9900
C7—H7C	0.9900	C12—C13	1.498 (3)
C7—H7D	0.9900	C12—H12A	0.9900
C8—C9	1.536 (5)	C12—H12B	0.9900
C8—H8A	0.9900	C14—O15	1.210 (3)
C8—H8B	0.9900	C14—O16	1.321 (3)
C9—C14	1.527 (4)	O16—H16	0.94 (3)
C13—Se1—N2	87.26 (10)	C7—C8A—H8C	109.8

N3—N2—Se1	110.39 (15)	C9A—C8A—H8D	109.8
N2—N3—C4	117.57 (19)	C7—C8A—H8D	109.8
C13—C4—N3	115.9 (2)	H8C—C8A—H8D	108.3
C13—C4—C5	126.3 (2)	C8A—C9A—C14	106.4 (8)
N3—C4—C5	117.7 (2)	C8A—C9A—C10	113.1 (9)
C4—C5—C6	112.2 (2)	C14—C9A—C10	106.1 (6)
C4—C5—H5A	109.2	C8A—C9A—H9A	110.3
C6—C5—H5A	109.2	C14—C9A—H9A	110.3
C4—C5—H5B	109.2	C10—C9A—H9A	110.3
C6—C5—H5B	109.2	C11—C10—C9	110.3 (2)
H5A—C5—H5B	107.9	C11—C10—C9A	134.7 (5)
C7—C6—C5	116.2 (2)	C11—C10—H10A	109.6
C7—C6—H6A	108.2	C9—C10—H10A	109.6
C5—C6—H6A	108.2	C11—C10—H10B	109.6
C7—C6—H6B	108.2	C9—C10—H10B	109.6
C5—C6—H6B	108.2	H10A—C10—H10B	108.1
H6A—C6—H6B	107.4	C11—C10—H10C	103.5
C6—C7—C8	123.2 (3)	C9A—C10—H10C	103.5
C6—C7—C8A	93.1 (5)	C11—C10—H10D	103.5
C6—C7—H7A	106.5	C9A—C10—H10D	103.5
C8—C7—H7A	106.5	H10C—C10—H10D	105.3
C6—C7—H7B	106.5	C10—C11—C12	115.33 (19)
C8—C7—H7B	106.5	C10—C11—H11A	108.4
H7A—C7—H7B	106.5	C12—C11—H11A	108.4
C6—C7—H7C	113.1	C10—C11—H11B	108.4
C8A—C7—H7C	113.1	C12—C11—H11B	108.4
C6—C7—H7D	113.1	H11A—C11—H11B	107.5
C8A—C7—H7D	113.1	C13—C12—C11	112.92 (19)
H7C—C7—H7D	110.5	C13—C12—H12A	109.0
C9—C8—C7	115.0 (3)	C11—C12—H12A	109.0
C9—C8—H8A	108.5	C13—C12—H12B	109.0
C7—C8—H8A	108.5	C11—C12—H12B	109.0
C9—C8—H8B	108.5	H12A—C12—H12B	107.8
C7—C8—H8B	108.5	C4—C13—C12	128.7 (2)
H8A—C8—H8B	107.5	C4—C13—Se1	108.86 (16)
C14—C9—C8	109.0 (3)	C12—C13—Se1	122.37 (17)
C14—C9—C10	109.3 (2)	O15—C14—O16	122.81 (19)
C8—C9—C10	113.8 (3)	O15—C14—C9	123.8 (2)
C14—C9—H9	108.2	O16—C14—C9	113.1 (2)
C8—C9—H9	108.2	O15—C14—C9A	118.2 (4)
C10—C9—H9	108.2	O16—C14—C9A	115.0 (4)
C9A—C8A—C7	109.3 (8)	C14—O16—H16	107 (2)
C9A—C8A—H8C	109.8		

C13—Se1—N2—N3	−0.48 (17)	C9—C10—C11—C12	62.8 (3)
Se1—N2—N3—C4	0.2 (3)	C9A—C10—C11—C12	73.3 (6)
N2—N3—C4—C13	0.4 (3)	C10—C11—C12—C13	49.3 (3)
N2—N3—C4—C5	178.4 (2)	N3—C4—C13—C12	175.9 (2)

C13—C4—C5—C6	119.0 (3)	C5—C4—C13—C12	−1.8 (4)
N3—C4—C5—C6	−58.7 (3)	N3—C4—C13—Se1	−0.8 (2)
C4—C5—C6—C7	−49.4 (3)	C5—C4—C13—Se1	−178.56 (18)
C5—C6—C7—C8	−76.3 (3)	C11—C12—C13—C4	−107.2 (3)
C5—C6—C7—C8A	−64.3 (4)	C11—C12—C13—Se1	69.1 (2)
C6—C7—C8—C9	70.8 (4)	N2—Se1—C13—C4	0.68 (16)
C7—C8—C9—C14	−172.2 (3)	N2—Se1—C13—C12	−176.28 (19)
C7—C8—C9—C10	65.5 (5)	C8—C9—C14—O15	−136.1 (3)
C6—C7—C8A—C9A	154.6 (8)	C10—C9—C14—O15	−11.2 (5)
C7—C8A—C9A—C14	171.0 (5)	C8—C9—C14—O16	49.5 (4)
C7—C8A—C9A—C10	−72.8 (11)	C10—C9—C14—O16	174.5 (2)
C14—C9—C10—C11	87.5 (3)	C8A—C9A—C14—O15	162.0 (7)
C8—C9—C10—C11	−150.4 (3)	C10—C9A—C14—O15	41.2 (9)
C8A—C9A—C10—C11	−71.5 (9)	C8A—C9A—C14—O16	−39.9 (11)
C14—C9A—C10—C11	44.9 (10)	C10—C9A—C14—O16	−160.7 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O16—H16···O15^{i}	0.94 (3)	1.69 (3)	2.630 (2)	174 (3)

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