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(11*SR*,13*RS*)-12-Oxa-9-azatetracyclo-[7.4.1.0^{2,7}.0^{11,13}]tetradeca-2(7),3,5-trien-8-one

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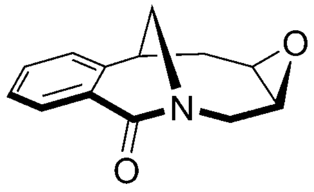
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 8.0.

The title compound, $C_{13}H_{13}NO_2$, was prepared by the epoxidation of its racemic precursor. Its structure was determined in order to establish which of two diastereomers would be favored in that reaction and in other reactions of interest. The presence of intermolecular C—H$\cdots$O interactions was considered probable, although H-atom positions were not refined. These interactions link the molecules into a two-dimensional network.

Related literature

For related chemistry, see: Greenberg (1988); Greenberg *et al.* (2000); Kirby *et al.* (1998); Paquette *et al.* (2006); Tani & Stoltz (2006); Winkler & Dunitz (1971). For related literature on geometry, see: Allen (1982); Allen *et al.* (1987); Nardelli (1995); Sheldrick (1997); Steiner & Saenger (1992); Taylor & Kennard (1983);



Experimental

Crystal data

$C_{13}H_{13}NO_2$
M_r = 215.24
Monoclinic, Cc
a = 7.1597 (10) Å
b = 16.533 (3) Å
c = 8.7277 (10) Å
β = 101.364 (11)°
V = 1012.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 (2) K
0.35 × 0.23 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min}$ = 0.933, $T_{\max}$ = 0.989
10172 measured reflections
1166 independent reflections
1069 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.01
1166 reflections
145 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.19 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C15—H15$B\cdots$O12$^{\rm i}$	0.99	2.45	3.379 (3)	156
C4—H4$\cdots$O12$^{\rm ii}$	0.95	2.54	3.345 (3)	142
C13—H13$\cdots$O16$^{\rm iii}$	1.00	2.58	3.278 (3)	127

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

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2141).

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supplementary materials

Acta Cryst. (2007). E**63**, o3417 [**doi:10.1107/S160053680703228X**]

(11*SR*,13*RS*)-12-Oxa-9-azatetracyclo[7.4.1.$0^{2,7}$.$0^{11,13}$]tetradeca-2(7),3,5-trien-8-one

J. C. Gallucci, **R. D. Dura** and **L. A. Paquette**

Comment

Chemical schemes of compounds (I), (II), (III) and (IV) are drawn in Fig. 2. The constrained placement of a lactam nitrogen at a bridgehead position in a relatively small bi- or tri-cyclic framework is known to give rise to an appreciable increase in solvolytic reactivity (Greenberg, 1988) (Greenberg *et al.*, 2000). This behavior has been properly attributed to the geometrically enforced loss of amide resonance (Tani & Stoltz, 2006), the cost of which can rise as high as 16–22 kcal/mol (Winkler & Dunitz, 1971). As a result, the carbonyl group in the most extreme cases (Kirby *et al.*, 1998) is decidedly ketone-like in its spectroscopic and chemical properties. The search for other unconventional behavior by compounds of this general class has more recently been directed to the consequences of photoactivation (Paquette *et al.*, 2006). The presence of a diene chromophore as in (I) was designed to enhance the absorption of light energy. When irradiated at 300 nm in hexane, (I) experiences stereoselective disrotatory cyclization to give initially (II), this pathway representing a significant departure from the response of the corresponding sultam, which undergoes SO_2–N bond homolysis and subsequent structural reorganization principally (Paquette *et al.*, 2006).

The synthetic protocol that gives rise to (I) proceeds *via* racemic 9-azatricyclo[7.4.1.$0^{2,7}$] tetradeca-2,4,6,11-tetraen-8-one, (III). This intermediate is characterized by the presence of an isolated double bond that is amenable in principle to electrophilic attack from the top and/or bottom surface. Since the conversion of (III) to (I) has to be accomplished by chemical modification of this sector of the lactam, it was considered advisable to clarify which approach to (I) is kinetically preferred. Epoxidation with *m*-chloroperbenzoic acid proved to be a suitable probe experiment, affording (IV) almost exclusively in an efficient manner. The stereochemistry of (IV) was established by X-ray crystallographic analysis as detailed herein.

The bond distances are in agreement with those selected in the critical evaluation of structures of organic molecules in the Cambridge data base (Allen *et al.*,1987). The phenyl ring is fused through C(2) and C(7) to the aliphatic remainder of the molecule. The geometry of epoxide molecules has been extensively discussed (Allen, 1982). The possible presence of C–H···O hydrogen bonds was considered although H-atom positions were not refined. The hydrogen positions were calculated using values provided by *SHELXL97* (Sheldrick, 1997). The shortest intermolecular value found was 2.45 Å with C–H···O 156° calculated using *PARST* (Nardelli, 1995) as given in *WinGX2005* (Farrugia, 1999). The value was normalized in *PARST* (Taylor & Kennard, 1983) to yield estimates of 2.37 Å and 155°, appropriate for comparison to neutron diffraction results. These values fall within the range of such numbers as discussed earlier (Steiner & Saenger, 1992). Table 2 contains these possible C–H···O hydrogen bonds and Fig.3 shows a portion of the H-bond two dimensional network.

Experimental

To a solution of (III) (0.19 g) in dichloromethane (20 ml) was added *m*-chloroperbenzoic acid (0.26 g) and $NaHCO_3$ (0.16 g). After being stirred overnight, the reaction mixture was poured into a saturated solution of Na_2SO_3 (10 ml) and dichloromethane (100 ml). The resulting organic phase was dried, freed of solvent under reduced pressure, and purified by column chromatography on silica gel (ether elution). Epoxide (IV) was isolated as a white solid (0.17 g, 83%) having a melting point of 129–131 C.

Refinement

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * U_{eq}(attached atom).

Figures

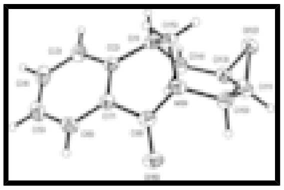


Fig. 1. The molecular structure of (IV), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

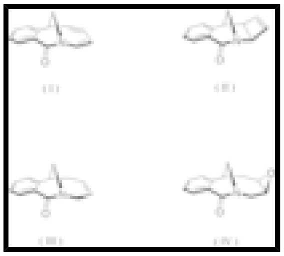


Fig. 2. Chemical schemes for (I), (II), (III) and (IV). Hydrogen atoms are not shown.

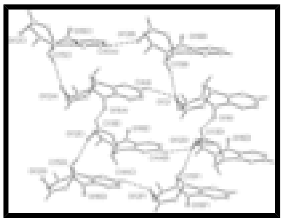


Fig. 3. A portion of the intermolecular hydrogen bond network.

(11*SR*,13*RS*)-12-Oxa-9- azatetracyclo[7.4.1.0^{2,7}.0^{11,13}]tetradeca-2(7),3,5-trien-8-one

Crystal data

$C_{13}H_{13}N_1O_2$

M_r = 215.24

Monoclinic, *Cc*

Hall symbol: C -2yc

a = 7.1597 (10) Å

b = 16.533 (3) Å

c = 8.7277 (10) Å

β = 101.364 (11)°

V = 1012.8 (3) Å^3

Z = 4

F_{000} = 456

D_x = 1.412 Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 1207 reflections

θ = 2.0–27.5°

μ = 0.10 mm^{-1}

T = 150 (2) K

Arrowhead-shaped chunk, colourless

0.35 × 0.23 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1166 independent reflections
Monochromator: graphite	1069 reflections with $I > 2\sigma(I)$
Detector resolution: 9 pixels mm^{-1}	$R_{\text{int}} = 0.032$
$T = 150(2)$ K	$\theta_{\text{max}} = 27.5°$
φ and ω scans	$\theta_{\text{min}} = 2.5°$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$h = -9\rightarrow9$
$T_{\text{min}} = 0.933$, $T_{\text{max}} = 0.989$	$k = -21\rightarrow21$
10172 measured reflections	$l = -11\rightarrow11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.2542P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{\text{max}} = 0.002$
1166 reflections	$\Delta\rho_{\text{max}} = 0.19$ e Å^{-3}
145 parameters	$\Delta\rho_{\text{min}} = -0.18$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. The data collection crystal was a clear, colorless arrowhead-shaped chunk. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 4.8, which means that 90% of the reflections were measured at least 4.8 times. A combination of phi and omega scans with a frame width of 1.0 degree was used. Data integration was done with *DENZO* (Otwinowski & Minor, 1997) and scaling and merging of the data was done with *SCALEPACK* (Otwinowski & Minor, 1997).

The structure was solved by the direct methods procedure in *SHELXS97* (Sheldrick, 1997) in space group *Cc*. Full-matrix least-squares refinements based on F^2 were performed in *SHELXL97* (Sheldrick, 1997), as incorporated in the *WinGX* package(Farrugia, 1999).

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
C8	0.2248 (4)	0.14357 (15)	0.0988 (3)	0.0241 (5)
C7	0.0275 (4)	0.12148 (14)	0.0165 (3)	0.0226 (5)
C6	−0.0335 (4)	0.13839 (14)	−0.1410 (3)	0.0277 (6)
H6	0.0542	0.1584	−0.2006	0.033*
C5	−0.2230 (4)	0.12614 (17)	−0.2118 (3)	0.0327 (6)
H5	−0.2647	0.1375	−0.3199	0.039*
C4	−0.3513 (4)	0.09735 (17)	−0.1245 (3)	0.0326 (6)
H4	−0.4808	0.089	−0.173	0.039*
C3	−0.2908 (4)	0.08056 (16)	0.0343 (3)	0.0281 (5)
H3	−0.3796	0.0611	0.0934	0.034*
C2	−0.1011 (3)	0.09220 (14)	0.1068 (3)	0.0221 (5)
C1	−0.0226 (3)	0.07768 (14)	0.2782 (3)	0.0222 (5)
H1	−0.1065	0.0387	0.3206	0.027*
C14	−0.0146 (3)	0.15923 (16)	0.3686 (3)	0.0268 (5)
H14A	−0.0397	0.2027	0.2891	0.032*
H14B	−0.1233	0.1592	0.4233	0.032*
C13	0.1604 (4)	0.18480 (16)	0.4879 (3)	0.0258 (5)
H13	0.1364	0.2332	0.5499	0.031*
C11	0.3621 (4)	0.17497 (15)	0.4812 (3)	0.0264 (5)
H11	0.4462	0.2181	0.5382	0.032*
C10	0.4408 (4)	0.13582 (16)	0.3511 (3)	0.0264 (5)
H10A	0.5112	0.177	0.3026	0.032*
H10B	0.5325	0.0932	0.396	0.032*
C15	0.1720 (3)	0.03971 (14)	0.2840 (3)	0.0231 (5)
H15A	0.2277	0.0228	0.3921	0.028*
H15B	0.16	−0.0086	0.2157	0.028*
N9	0.2944 (3)	0.10007 (12)	0.2303 (2)	0.0228 (5)
O16	0.3117 (3)	0.19990 (12)	0.0526 (2)	0.0329 (4)
O12	0.2746 (3)	0.12359 (12)	0.5828 (2)	0.0286 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.0247 (12)	0.0231 (12)	0.0263 (11)	0.0001 (9)	0.0092 (9)	−0.0015 (10)
C7	0.0261 (12)	0.0198 (11)	0.0230 (11)	0.0018 (9)	0.0076 (9)	−0.0024 (9)
C6	0.0373 (14)	0.0226 (12)	0.0245 (13)	0.0032 (10)	0.0094 (11)	0.0011 (10)
C5	0.0409 (16)	0.0318 (14)	0.0232 (12)	0.0083 (11)	0.0009 (11)	−0.0039 (10)
C4	0.0287 (14)	0.0347 (15)	0.0313 (14)	0.0049 (11)	−0.0014 (11)	−0.0058 (11)
C3	0.0261 (13)	0.0291 (13)	0.0295 (12)	0.0005 (10)	0.0064 (10)	−0.0061 (11)
C2	0.0262 (12)	0.0184 (11)	0.0225 (11)	0.0017 (9)	0.0069 (9)	−0.0011 (9)
C1	0.0226 (11)	0.0220 (12)	0.0231 (11)	−0.0013 (9)	0.0070 (9)	0.0008 (9)
C14	0.0260 (12)	0.0317 (13)	0.0236 (12)	0.0031 (10)	0.0072 (9)	−0.0047 (11)

C13	0.0303 (13)	0.0251 (12)	0.0228 (12)	0.0024 (10)	0.0070 (10)	0.0017 (9)
C11	0.0269 (12)	0.0229 (12)	0.0286 (12)	−0.0035 (10)	0.0033 (9)	0.0031 (10)
C10	0.0208 (12)	0.0280 (13)	0.0299 (13)	−0.0014 (9)	0.0039 (9)	0.0013 (10)
C15	0.0235 (12)	0.0177 (10)	0.0288 (12)	0.0001 (9)	0.0071 (9)	0.0026 (9)
N9	0.0214 (11)	0.0218 (10)	0.0259 (10)	−0.0003 (8)	0.0068 (8)	0.0022 (8)
O16	0.0335 (10)	0.0309 (10)	0.0369 (10)	−0.0076 (8)	0.0134 (8)	0.0052 (8)
O12	0.0306 (10)	0.0303 (10)	0.0243 (9)	−0.0009 (7)	0.0042 (7)	0.0059 (7)

Geometric parameters (Å, °)

C8—O16	1.231 (3)	C1—H1	1
C8—N9	1.363 (3)	C14—C13	1.523 (4)
C8—C7	1.498 (3)	C14—H14A	0.99
C7—C6	1.387 (4)	C14—H14B	0.99
C7—C2	1.411 (3)	C13—O12	1.453 (3)
C6—C5	1.391 (4)	C13—C11	1.465 (4)
C6—H6	0.95	C13—H13	1
C5—C4	1.389 (4)	C11—O12	1.456 (3)
C5—H5	0.95	C11—C10	1.510 (4)
C4—C3	1.396 (4)	C11—H11	1
C4—H4	0.95	C10—N9	1.457 (3)
C3—C2	1.394 (3)	C10—H10A	0.99
C3—H3	0.95	C10—H10B	0.99
C2—C1	1.510 (3)	C15—N9	1.465 (3)
C1—C15	1.520 (3)	C15—H15A	0.99
C1—C14	1.557 (3)	C15—H15B	0.99
O16—C8—N9	123.4 (2)	C1—C14—H14B	106.8
O16—C8—C7	121.1 (2)	H14A—C14—H14B	106.7
N9—C8—C7	115.4 (2)	O12—C13—C11	59.83 (16)
C6—C7—C2	120.7 (2)	O12—C13—C14	119.4 (2)
C6—C7—C8	120.6 (2)	C11—C13—C14	128.8 (2)
C2—C7—C8	118.3 (2)	O12—C13—H13	112.7
C7—C6—C5	120.0 (2)	C11—C13—H13	112.7
C7—C6—H6	120	C14—C13—H13	112.7
C5—C6—H6	120	O12—C11—C13	59.67 (16)
C4—C5—C6	119.9 (2)	O12—C11—C10	118.5 (2)
C4—C5—H5	120	C13—C11—C10	126.4 (2)
C6—C5—H5	120	O12—C11—H11	113.7
C5—C4—C3	120.3 (2)	C13—C11—H11	113.7
C5—C4—H4	119.9	C10—C11—H11	113.7
C3—C4—H4	119.9	N9—C10—C11	113.4 (2)
C2—C3—C4	120.5 (2)	N9—C10—H10A	108.9
C2—C3—H3	119.8	C11—C10—H10A	108.9
C4—C3—H3	119.8	N9—C10—H10B	108.9
C3—C2—C7	118.6 (2)	C11—C10—H10B	108.9
C3—C2—C1	124.5 (2)	H10A—C10—H10B	107.7
C7—C2—C1	116.8 (2)	N9—C15—C1	108.09 (19)
C2—C1—C15	104.98 (19)	N9—C15—H15A	110.1
C2—C1—C14	109.5 (2)	C1—C15—H15A	110.1

C15—C1—C14	113.5 (2)	N9—C15—H15B	110.1
C2—C1—H1	109.6	C1—C15—H15B	110.1
C15—C1—H1	109.6	H15A—C15—H15B	108.4
C14—C1—H1	109.6	C8—N9—C10	119.1 (2)
C13—C14—C1	122.0 (2)	C8—N9—C15	119.1 (2)
C13—C14—H14A	106.8	C10—N9—C15	115.81 (19)
C1—C14—H14A	106.8	C13—O12—C11	60.50 (16)
C13—C14—H14B	106.8		

O16—C8—C7—C6	26.4 (4)	C15—C1—C14—C13	−16.8 (3)
N9—C8—C7—C6	−156.8 (2)	C1—C14—C13—O12	−33.8 (3)
O16—C8—C7—C2	−146.0 (2)	C1—C14—C13—C11	39.6 (4)
N9—C8—C7—C2	30.7 (3)	C14—C13—C11—O12	−105.2 (3)
C2—C7—C6—C5	−0.3 (4)	O12—C13—C11—C10	104.8 (3)
C8—C7—C6—C5	−172.6 (2)	C14—C13—C11—C10	−0.4 (4)
C7—C6—C5—C4	0.3 (4)	O12—C11—C10—N9	66.7 (3)
C6—C5—C4—C3	0.0 (4)	C13—C11—C10—N9	−5.1 (4)
C5—C4—C3—C2	−0.2 (4)	C2—C1—C15—N9	65.8 (2)
C4—C3—C2—C7	0.2 (4)	C14—C1—C15—N9	−53.7 (3)
C4—C3—C2—C1	178.5 (2)	O16—C8—N9—C10	22.1 (4)
C6—C7—C2—C3	0.1 (3)	C7—C8—N9—C10	−154.6 (2)
C8—C7—C2—C3	172.5 (2)	O16—C8—N9—C15	173.8 (2)
C6—C7—C2—C1	−178.4 (2)	C7—C8—N9—C15	−2.9 (3)
C8—C7—C2—C1	−5.9 (3)	C11—C10—N9—C8	96.1 (3)
C3—C2—C1—C15	140.8 (2)	C11—C10—N9—C15	−56.4 (3)
C7—C2—C1—C15	−40.9 (3)	C1—C15—N9—C8	−45.9 (3)
C3—C2—C1—C14	−97.1 (3)	C1—C15—N9—C10	106.6 (2)
C7—C2—C1—C14	81.3 (3)	C14—C13—O12—C11	120.3 (2)
C2—C1—C14—C13	−133.7 (2)	C10—C11—O12—C13	−117.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15B···O12^{i}	0.99	2.45	3.379 (3)	156
C4—H4···O12^{ii}	0.95	2.54	3.345 (3)	142
C13—H13···O16^{iii}	1.00	2.58	3.278 (3)	127

Symmetry codes: (i) $x, -y, z-1/2$; (ii) $x-1, y, z-1$; (iii) $x-1/2, -y+1/2, z+1/2$.

Fig. 1

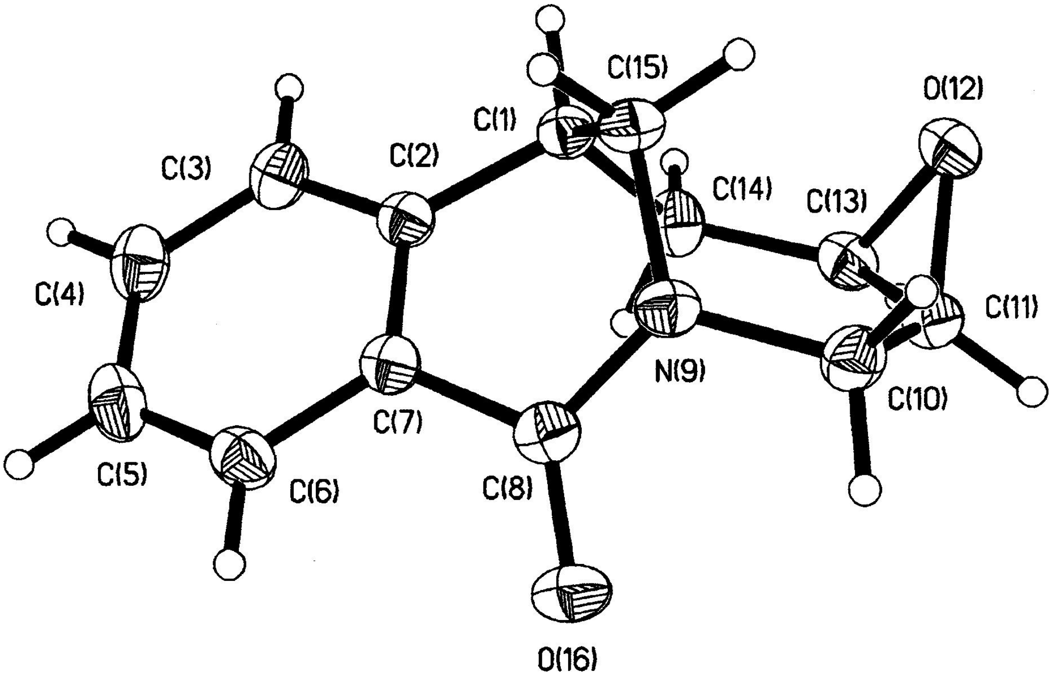
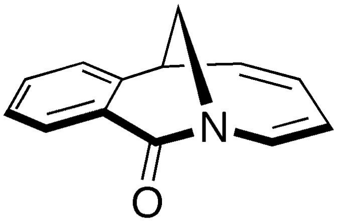
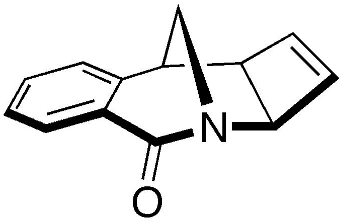


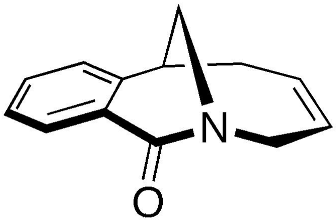
Fig. 2



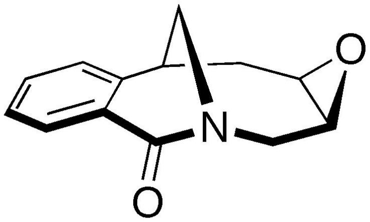
(I)



(II)

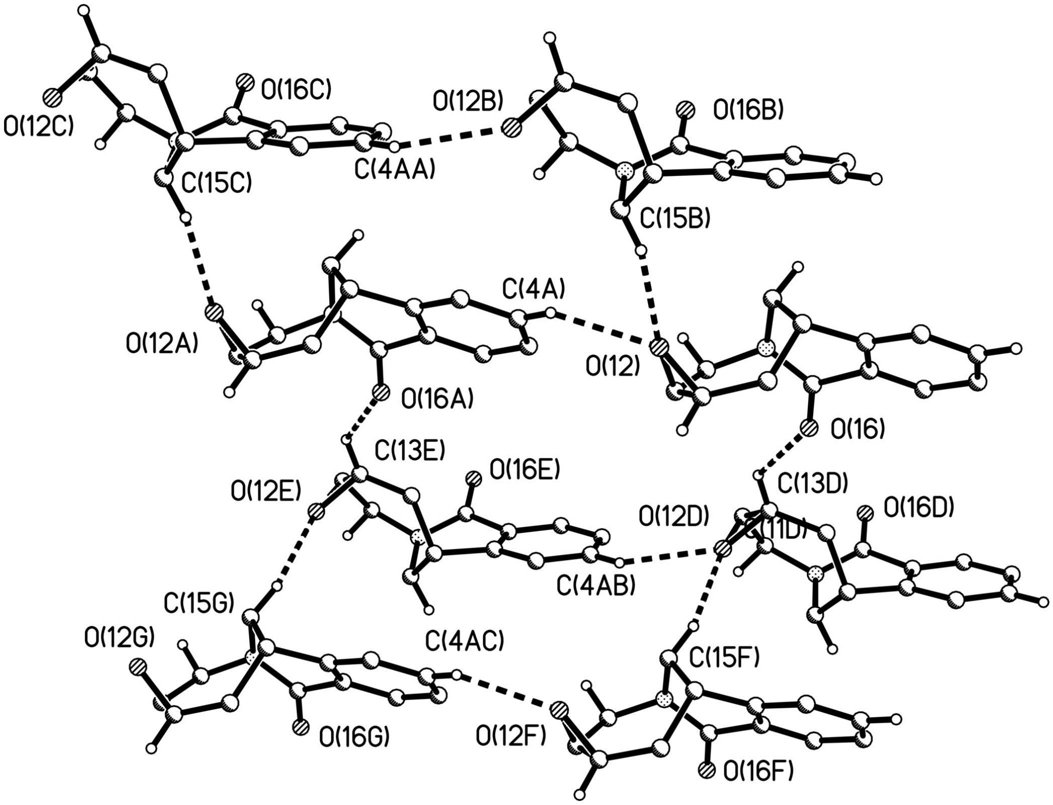


(III)



(IV)

Fig. 3


O(12C)
O(16C)
C(15C)
C(4AA)
O(12B)
O(16B)
C(15B)
C(4A)
O(12A)
O(12)
O(16A)
O(16)
C(13E)
O(16E)
O(12E)
C(13D)
O(12D)
O(16D)
C(11D)
C(4AB)
C(15G)
O(12G)
C(4AC)
C(15F)
O(16G)
O(12F)
O(16F)