

C2—C1—C7	121.6 (11)	C2'—C1'—C7'	121.8 (11)
C6—C1—C7	117.8 (11)	C6'—C1'—C7'	121.1 (11)
C1—C2—C3	123.0 (11)	C1'—C2'—C3'	128.8 (11)
C1—C2—O27	117.5 (11)	C1'—C2'—O27'	115.7 (10)
C3—C2—O27	119.5 (10)	C3'—C2'—O27'	115.0 (10)
C2—C3—C4	116.3 (11)	C2'—C3'—C4'	112.4 (11)
C2—C3—C8	119.7 (10)	C2'—C3'—C8'	128.3 (11)
C4—C3—C8	123.6 (10)	C4'—C3'—C8'	119.1 (11)
C3—C4—C5	120.5 (12)	C3'—C4'—C5'	120.0 (13)
C4—C5—C6	121.2 (12)	C4'—C5'—C6'	125.4 (14)
C1—C6—C5	118.4 (12)	C1'—C6'—C5'	115.5 (12)
C3—C8—C9	107.4 (9)	C3'—C8'—C9'	104.6 (10)
C10—C9—C8	119.1 (9)	C10'—C9'—C8'	120.5 (10)
C10—C9—C14	118.4 (10)	C10'—C9'—C14'	119.7 (11)
C14—C9—C8	122.1 (10)	C14'—C9'—C8'	119.8 (10)
C11—C10—C9	120.8 (10)	C11'—C10'—C9'	123.5 (11)
C11—C10—O15	121.8 (10)	C11'—C10'—O15'	117.8 (10)
C9—C10—O15	117.4 (9)	C9'—C10'—O15'	118.6 (10)
C10—C11—C12	119.5 (11)	C10'—C11'—C12'	117.6 (11)
C11—C12—C13	120.2 (11)	C11'—C12'—C13'	118.9 (12)
C12—C13—C14	119.5 (11)	C12'—C13'—C14'	121.4 (12)
C13—C14—C9	121.4 (11)	C13'—C14'—C9'	118.7 (11)
C10—O15—C16	112.3 (9)	C10'—O15'—C16'	112.8 (9)
C17—C16—O15	105.7 (10)	C17'—C16'—O15'	106.1 (10)
C16—C17—O18	105.0 (10)	C16'—C17'—O18'	105.2 (10)
C17—O18—C19	111.8 (10)	C17'—O18'—C19'	110.0 (10)
C20—C19—O18	109.3 (13)	C20'—C19'—O18'	104.1 (13)
C19—C20—O21	102.1 (14)	C19'—C20'—O21'	92.8 (14)
C20—O21—C22	122.8 (21)	C20'—O21'—C22'	103.9 (15)
C23—C22—O21	123.7 (25)	C23'—C22'—O21'	98.5 (17)
C22—C23—O24	125.4 (22)	C22'—C23'—O24'	113.0 (19)
C26—C25—O24	116.0 (24)	C26'—C25'—O24'	115.8 (18)
C25—C26—O27	103.1 (19)	C25'—C26'—O27'	113.9 (13)
C23—O24—C25	150.0 (19)	C23'—O24'—C25'	127.6 (18)
C2—O27—C26	114.5 (10)	C2'—O27'—C26'	117.4 (9)

Table 3. Distances (Å) between O atoms of the etheral chains characterizing the aperture of the crowned calixarene

	A	A'
O15...O27	3.24 (4)	3.25 (4)
O18...O24'	5.58 (6)	5.39 (6)
O21...O21'	7.24 (8)	6.57 (8)

Symmetry code: (i) indicates the equivalent atom generated by the twofold axis.

As the crystals of the studied calixarene were of poor quality, half the reciprocal space was measured and averaged values were calculated to increase the measurement accuracy.

Data collection: *Structure Determination Package* (Enraf-Nonius, 1979). Cell refinement: *Structure Determination Package*. Data reduction: local program (CRMC2, France). Program(s) used to solve structure: *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEPII* (Johnson, 1976) and *GENMOL* (Pèpe & Siri, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 3-Oxo-2-phenyl-3a,4,5,7a-tetrahydro-5,7a-epoxyisoindoline-4-carboxylic Acid in Two Crystalline Phases, Solvated with DMSO and Unsolvated

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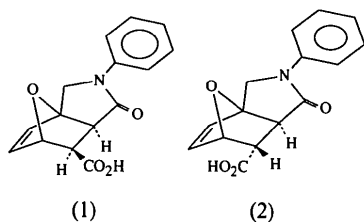
(Received 16 May 1994; accepted 10 October 1994)

## Abstract

The title compound is a precursor of optically active  $\gamma$ -lactams. Two crystalline forms, solvated with DMSO (C<sub>15</sub>H<sub>13</sub>NO<sub>4</sub>·C<sub>2</sub>H<sub>6</sub>OS) and unsolvated (C<sub>15</sub>H<sub>13</sub>NO<sub>4</sub>), were found and analyzed.

### Comment

The reaction of *N*-furfuryl amine with maleic anhydride leads to a tricyclic compound. Two different pathways may occur for this cyclization reaction: (a) an intermolecular Diels–Alder reaction between maleic anhydride and the furan nucleus followed by an intramolecular attack of the amino group on the anhydride function, or (b) condensation of the amine with maleic anhydride followed by an intramolecular Diels–Alder reaction. The mechanism of the reaction has been reinvestigated and fully elucidated; it was found to proceed by pathway (b) (Brun, Zylber, Pèpe & Reboul, 1994). However, the structure of the final adduct has been a matter of controversy and two different configurations, (1) and (2), have been proposed (Bilovic, 1968; Isaacs & van der Beeke, 1982).



The solvated crystals (I) were obtained from DMSO solution while the unsolvated crystals (II) were grown from diethyl ether solution. The X-ray analyses indicate the existence of configuration (1) only. The interatomic distances and the valence angles are equivalent in the two crystalline forms, but the phenyl ring occupies two symmetric positions with respect to the five-membered ring, characterized by the torsion angle C1—C6—N7—C11 [160.7(5) and -155.0(5)° for (I) and (II), respectively], while the position of the carboxyl group

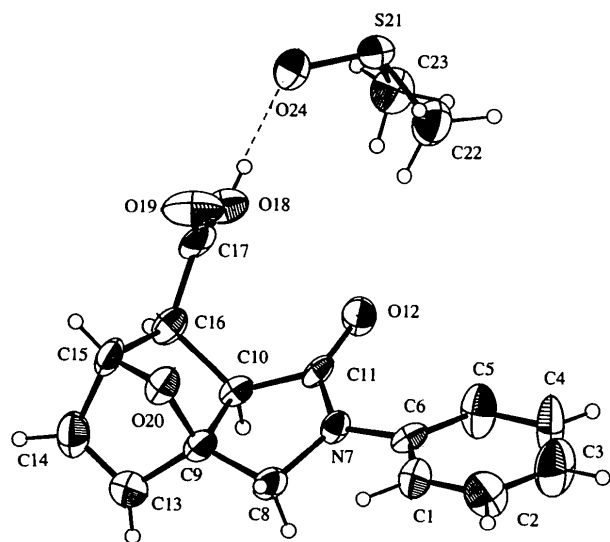


Fig. 1. ORTEP drawing (Johnson, 1976) of the solvated compound (I) with displacement ellipsoids at 50% probability.

plane with respect to the bridged ring, characterized by the torsion angle C10—C16—C17—O19, is almost the same [-91.8(5) and -93.1(4)° for (I) and (II), respectively].

In compound (I), a hydrogen bond between atom O18 of the carboxyl group and atom O24 of the DMSO molecule is observed, the O···O distance being 2.593(8) Å. The intermolecular contacts in the crystal are of van der Waals nature. In compound (II), a hydrogen bond between the exocyclic O12 atom and atom O18 of the closest molecule (related by a center of symmetry) is observed, the O···O distance being 2.632(7) Å.

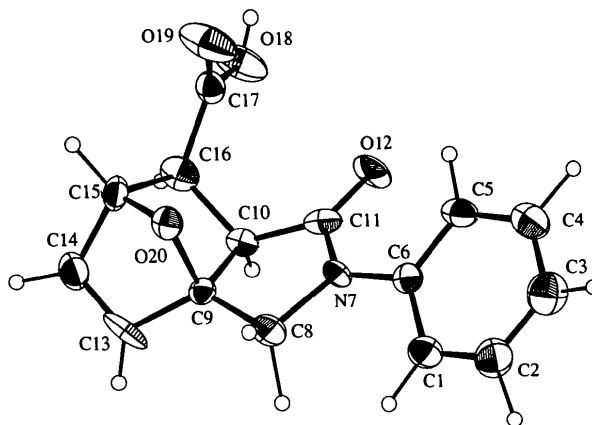


Fig. 2. ORTEP drawing (Johnson, 1976) of the unsolvated compound (II) with displacement ellipsoids at 50% probability.

### Experimental

#### Compound (I)

##### Crystal data

C<sub>15</sub>H<sub>13</sub>NO<sub>4</sub>·C<sub>2</sub>H<sub>6</sub>OS

*M<sub>r</sub>* = 349.35

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 7.813(2) Å

*b* = 11.830(2) Å

*c* = 19.022(6) Å

β = 100.61(3)°

*V* = 1728.1(4) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.343 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.35(2) Mg m<sup>-3</sup>

*D<sub>m</sub>* measured by flotation

Cu *K*α radiation

λ = 1.5418 Å

Cell parameters from 25

reflections

θ = 15–45°

μ = 1.78 mm<sup>-1</sup>

*T* = 293 K

Hexagonal prism

0.5 × 0.4 × 0.3 mm

Colourless

##### Data collection

Enraf–Nonius CAD-4

diffractometer

θ scans

Absorption correction:

none

3723 measured reflections

3449 independent reflections

2924 observed reflections

[*I* > 3.0σ(*I*)]

θ<sub>max</sub> = 45°

*h* = -8 → 8

*k* = 0 → 12

*l* = 0 → 20

4 standard reflections

frequency: 60 min

intensity decay: none

**Refinement****Refinement on  $F$**  $R = 0.044$  $wR = 0.044$  $S = 0.74$ 

2924 reflections

274 parameters

H-atom displacement

parameters not refined

Unit weights applied

 $(\Delta/\sigma)_{\max} = 0.15$  $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$ 

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV, Table

2.2B)

**Compound (II)****Crystal data** $\text{C}_{15}\text{H}_{13}\text{NO}_4$  $M_r = 271.27$ 

Monoclinic

 $A2/a$  $a = 16.961 (5) \text{ \AA}$  $b = 13.219 (2) \text{ \AA}$  $c = 11.598 (2) \text{ \AA}$  $\beta = 81.41 (2)^\circ$  $V = 2571.6 (7) \text{ \AA}^3$  $Z = 8$  $D_x = 1.402 \text{ Mg m}^{-3}$  $D_m = 1.41 (2) \text{ Mg m}^{-3}$  $D_m$  measured by flotation**Data collection**Enraf-Nonius CAD-4  
diffractometer $\theta$  scans

Absorption correction:

none

5489 measured reflections

2552 independent reflections

2356 observed reflections

 $[I > 3.0\sigma(I)]$ **Refinement****Refinement on  $F$**  $R = 0.041$  $wR = 0.041$  $S = 0.93$ 

2356 reflections

220 parameters

H-atom displacement

parameters not refined

Unit weights applied

Cu  $K\alpha$  radiation $\lambda = 1.5418 \text{ \AA}$ 

Cell parameters from 25

reflections

 $\theta = 15\text{--}45^\circ$  $\mu = 0.76 \text{ mm}^{-1}$  $T = 293 \text{ K}$ 

Square prism

 $0.5 \times 0.3 \times 0.3 \text{ mm}$ 

Colourless

 $\theta_{\max} = 45^\circ$  $h = -17 \rightarrow 17$  $k = 0 \rightarrow 14$  $l = 0 \rightarrow 12$ 

4 standard reflections

frequency: 60 min

intensity decay: none

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (I)

	$B_{\text{eq}} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j$			
	$x$	$y$	$z$	$B_{\text{eq}}$
C1	0.3214 (3)	0.1865 (2)	0.1433 (1)	3.4 (1)
C2	0.3234 (4)	0.1896 (2)	0.0720 (1)	4.5 (1)
C3	0.2704 (5)	0.0953 (3)	0.0307 (1)	5.6 (1)
C4	0.2164 (5)	0.0019 (2)	0.0625 (1)	5.5 (1)
C5	0.2144 (4)	-0.0023 (2)	0.1343 (1)	4.0 (1)
C6	0.2704 (2)	0.0911 (2)	0.1769 (1)	2.4 (1)
N7	0.2800 (5)	0.0904 (1)	0.2519 (1)	2.2 (1)
C8	0.4057 (3)	0.1634 (2)	0.2985 (1)	2.8 (1)
C9	0.3598 (2)	0.1473 (2)	0.3705 (1)	2.5 (1)

C10	0.2687 (2)	0.0307 (2)	0.3683 (1)	2.3 (1)
C11	0.1930 (2)	0.0189 (1)	0.2891 (1)	2.1 (1)
O12	0.0787 (2)	-0.0473 (1)	0.2637 (1)	3.3 (1)
C13	0.4812 (3)	0.1615 (2)	0.4406 (1)	3.6 (1)
C14	0.3806 (3)	0.1744 (2)	0.4881 (1)	3.9 (1)
C15	0.1958 (3)	0.1714 (2)	0.4473 (1)	3.0 (1)
C16	0.1496 (3)	0.0477 (2)	0.4246 (1)	2.7 (1)
C17	-0.0422 (3)	0.0325 (2)	0.3961 (1)	2.9 (1)
O18	-0.0932 (2)	-0.0737 (1)	0.4000 (1)	3.6 (1)
O19	-0.1416 (2)	0.1074 (1)	0.3725 (1)	4.6 (1)
O20	0.2167 (2)	0.2195 (1)	0.3799 (1)	2.9 (1)
S21	-0.4561 (1)	-0.1883 (1)	0.2725 (1)	3.9 (1)
C22	-0.3440 (4)	-0.1390 (3)	0.2048 (2)	5.1 (1)
C23	-0.3319 (4)	-0.3108 (3)	0.2993 (2)	5.5 (1)
O24	-0.4107 (2)	-0.1073 (2)	0.3343 (1)	4.8 (1)

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (II)

	$B_{\text{eq}} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j$			$B_{\text{eq}}$
	$x$	$y$	$z$	
C1	-0.0087 (1)	-0.1955 (1)	-0.4759 (2)	3.0 (1)
C2	0.0484 (1)	-0.1829 (2)	-0.4045 (2)	3.6 (1)
C3	0.1128 (1)	-0.1198 (2)	-0.4366 (2)	3.2 (1)
C4	0.1182 (1)	-0.0656 (1)	-0.5385 (2)	3.2 (1)
C5	0.0596 (1)	-0.0742 (1)	-0.6104 (1)	2.5 (1)
C6	-0.0043 (1)	-0.1400 (1)	-0.5801 (1)	2.0 (1)
N7	-0.0642 (1)	-0.1524 (1)	-0.6497 (1)	2.0 (1)
C8	-0.1442 (1)	-0.1923 (1)	-0.6012 (1)	2.6 (1)
C9	-0.1931 (1)	-0.1682 (1)	-0.6921 (1)	1.7 (1)
C10	-0.1348 (1)	-0.1663 (1)	-0.8076 (1)	1.8 (1)
C11	-0.0583 (1)	-0.1360 (1)	-0.7685 (1)	2.2 (1)
O12	0.0041 (1)	-0.1060 (1)	-0.8290 (1)	3.0 (1)
C13	-0.2709 (1)	-0.2161 (1)	-0.7113 (2)	3.1 (1)
C14	-0.3052 (1)	-0.1531 (2)	-0.7761 (2)	3.0 (1)
C15	-0.2512 (1)	-0.0624 (1)	-0.7945 (1)	2.6 (1)
C16	-0.1775 (1)	-0.0947 (1)	-0.8848 (1)	2.6 (1)
C17	-0.1285 (1)	-0.0056 (1)	-0.9375 (1)	2.1 (1)
O18	-0.0864 (1)	-0.0294 (1)	-1.0369 (1)	4.1 (1)
O19	-0.1283 (1)	0.0763 (1)	-0.8954 (1)	5.1 (1)
O20	-0.2166 (1)	-0.0624 (1)	-0.6875 (1)	2.2 (1)

Table 3. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (I)

C1—C2	1.360 (4)	C17—O18	1.324 (3)
C1—C6	1.391 (3)	C17—O19	1.209 (3)
C10—C9	1.549 (3)	C2—C3	1.383 (5)
C10—C11	1.520 (3)	C22—S21	1.784 (4)
C10—C16	1.556 (3)	C23—S21	1.766 (4)
C11—N7	1.362 (2)	C3—C4	1.363 (5)
C11—O12	1.219 (2)	C4—C5	1.370 (4)
C13—C9	1.497 (3)	C5—C6	1.392 (4)
C13—C14	1.311 (4)	C6—N7	1.415 (3)
C14—C15	1.509 (4)	C8—N7	1.475 (3)
C15—C16	1.549 (4)	C8—C9	1.490 (3)
C15—O20	1.439 (3)	C9—O20	1.444 (3)
C16—C17	1.508 (4)	O24—S21	1.507 (2)
C11—C10—C9	101.6 (4)	C3—C4—C5	122.2 (6)
C11—C10—C16	121.4 (5)	C4—C5—C6	119.4 (5)
C16—C10—C9	101.3 (4)	C1—C6—C5	117.5 (5)
C10—C11—N7	108.5 (4)	C1—C6—N7	120.1 (5)
C10—C11—O12	125.1 (4)	C5—C6—N7	122.3 (5)
N7—C11—O12	126.3 (4)	C9—C8—N7	103.4 (5)
C14—C13—C9	105.4 (5)	C10—C9—C8	106.0 (5)
C13—C14—C15	106.3 (5)	C10—C9—C13	109.7 (5)
C14—C15—C16	108.5 (5)	C10—C9—O20	99.7 (4)
C14—C15—O20	101.2 (5)	C13—C9—C8	125.7 (5)
C16—C15—O20	100.6 (5)	C13—C9—O20	101.6 (4)
C10—C16—C15	100.1 (5)	C8—C9—O20	111.2 (4)
C10—C16—C17	114.7 (5)	C11—N7—C6	126.1 (4)
C15—C16—C17	112.2 (5)	C11—N7—C8	113.1 (4)
C16—C17—O18	112.5 (6)	C6—N7—C8	120.6 (4)
C16—C17—O19	125.0 (6)	C15—O20—C9	95.4 (5)
O18—C17—O19	122.5 (7)	C22—S21—C23	99.2 (4)
C1—C2—C3	119.2 (8)	C22—S21—O24	106.1 (4)
C2—C3—C4	119.1 (7)	C23—S21—O24	105.0 (4)

Table 4. Selected geometric parameters (Å, °) for (II)

C1—C2	1.375 (4)	C16—C17	1.517 (3)
C1—C6	1.406 (3)	C17—O18	1.301 (3)
C10—C9	1.542 (3)	C17—O19	1.188 (3)
C10—C11	1.492 (3)	C2—C3	1.381 (4)
C10—C16	1.554 (3)	C3—C4	1.374 (4)
C11—N7	1.383 (3)	C4—C5	1.394 (3)
C11—O12	1.244 (3)	C5—C6	1.393 (3)
C13—C9	1.510 (3)	C6—N7	1.399 (3)
C13—C14	1.314 (4)	C8—N7	1.486 (3)
C14—C15	1.505 (3)	C8—C9	1.472 (3)
C15—C16	1.565 (3)	C9—O20	1.453 (3)
C15—O20	1.450 (3)		
C11—C10—C9	102.4 (3)	C2—C3—C4	119.7 (6)
C11—C10—C16	120.9 (4)	C3—C4—C5	120.7 (5)
C16—C10—C9	102.5 (3)	C4—C5—C6	119.8 (4)
C10—C11—N7	108.9 (4)	C1—C6—C5	118.8 (4)
C10—C11—O12	128.3 (4)	C1—C6—N7	119.0 (4)
N7—C11—O12	122.7 (4)	C5—C6—N7	122.2 (4)
C14—C13—C9	106.9 (5)	C9—C8—N7	103.4 (4)
C13—C14—C15	105.9 (5)	C10—C9—C8	105.7 (4)
C14—C15—C16	106.6 (4)	C10—C9—C13	109.9 (4)
C14—C15—O20	101.2 (4)	C10—C9—O20	98.8 (3)
C16—C15—O20	100.8 (3)	C13—C9—C8	127.9 (4)
C10—C16—C15	100.1 (3)	C13—C9—O20	99.7 (3)
C10—C16—C17	115.5 (4)	C8—C9—O20	111.3 (4)
C15—C16—C17	113.1 (4)	C11—N7—C6	127.3 (4)
C16—C17—O18	111.4 (4)	C11—N7—C8	111.1 (4)
C16—C17—O19	125.3 (4)	C6—N7—C8	121.6 (4)
O18—C17—O19	123.4 (4)	C15—O20—C9	96.5 (4)
C1—C2—C3	120.7 (6)		

For both compounds, data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Operations Manual*; data reduction: local program (CRMC2, France); program(s) used to solve structures: *MULTAN80* (Main *et al.*, 1980); program(s) used to refine structures: *SHELX76* (Sheldrick, 1976); molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1143). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A New Bioactive $\beta$ -Dihydroagarofuran Sesquiterpenoid

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### Abstract

The structure of a new sesquiterpene,  $1\beta,2\beta$ -diacetoxy- $9\alpha$ -(3-phenyl-2-oxiranylcarboxy)- $\beta$ -dihydroagarofuran, from *Celastraceae* has been determined and found to have a very interesting three-membered ring.

### Comment

Historically, bioactive  $\beta$ -dihydroagarofuran sesquiterpenoids of the plant genus *Celastraceae* have been used as insecticides in China. Recently, a new sesquiterpene,  $1\beta,2\beta$ -diacetoxy- $9\alpha$ -(3-phenyl-2-oxiranylcarboxy)- $\beta$ -dihydroagarofuran, (I), has been isolated from the seeds of *C. gemmatus loes*. Experiments show that for the cabbage worm, *Pieris rapae* (a main species of injurious insects), the death rate is about 50% at a concentration of 500 p.p.m.

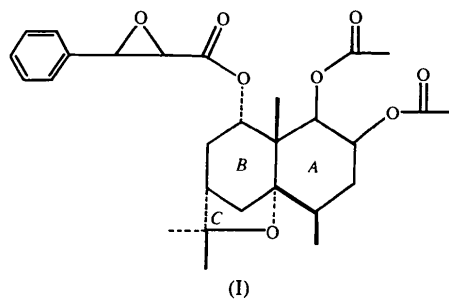


Fig. 1 shows the structure of the  $\beta$ -dihydroagarofuran sesquiterpenoid. Both rings A and B adopt the chair conformation and they are almost parallel. The C ring is virtually perpendicular to A and B. The mean dihedral angle between the C and A/B rings is  $83^\circ$ . The  $9\alpha$ -hydroxy group of the B ring is esterified by an epoxidized cinnamoyl residue. The presence of the epoxy group is difficult to distinguish by chemical means or magnetic resonance, because it is easily converted into cinnamic acid in aqueous solution.

Related studies on dihydroagarofuran sesquiterpenoids have been reported by Delle Monache, Marini Bettolo & Bernays (1984), Jacobson (1958), Yamada, Shizuri & Hirate (1978), Wakabayashi *et al.* (1988) and Rosza, Perjesi, Pelzer, Argay & Kálmán (1989).