

## Conformation of an Eleven-Membered Lactone: Structure of (*E*,13*R*,14*S*)-13-Hydroxy-17-nor-8,9-secolabd-9(11)-en-8,14-olide

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**Abstract.**  $C_{19}H_{32}O_3$ ,  $M_r = 308.49$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.072$  (3),  $b = 9.389$  (3),  $c = 27.888$  (11) Å,  $V = 1852$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.11$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.08$  mm<sup>-1</sup>,  $F(000) = 680$ ,  $T = 293$  K,  $R = 0.050$  for 1475 independent reflections with  $I > 2.5\sigma(I)$ . The conformation of the 11-membered lactone ring is essentially the same as the calculated minimum-energy conformation of *trans,trans*-cycloundeca-1,6-diene with differences between corresponding ring torsion angles of only 1–15° (mean 6°). This conformation is found in several cytochalasans and dolabellane diterpenoids but not in the pyrrolizidine alkaloids with 11-membered dilactone rings. The cyclohexane ring is distorted to alleviate 1,3-diaxial repulsion between methyl substituents [C(18)–C(20) = 3.291 (7) Å]; the smallest torsion angles [–47.4 (5), 48.1 (5)°] are at ring bonds between the methyl groups, the largest torsion angles [–55.6 (6), 56.4 (6)°] at bonds remote from these substituents.

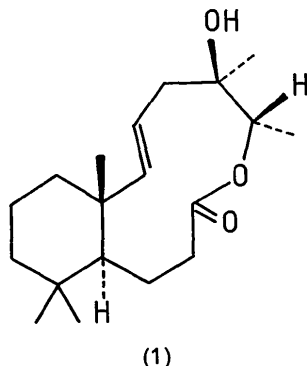
**Introduction.** Eleven-membered rings are features of several classes of natural products, *e.g.* the cytochalasans (Tamm, 1980), dolabellane diterpenoids (Matsuo, Kamio, Uohama, Yoshida, Connolly & Sim, 1988) and pyrrolizidine alkaloids (Robins, 1982). Examination of crystallographic results reveals two conformations for the *trans,trans*-cycloundeca-1,5-diene system in humulene derivatives (Khan, MacAlpine, Porte & Sim, 1983) and a number of dolabellane diterpenoids (Matsuo, Uohama, Yoshida, Nakayama, Hayashi, Connolly & Sim, 1985; Huneck, Baxter, Cameron, Connolly, Harrison, Phillips, Rycroft & Sim, 1986; Connolly, Sim & Matsuo, 1987), and essentially a single conformation for the *trans,trans*-cycloundeca-1,6-diene system in various cytochalasans (Tsukuda & Koyama, 1972; Beno, Cox, Wells, Cole, Kirksey & Christoph, 1977; Neupert-Laves & Dobler, 1982) and dolabellane diterpenoids (Ireland, Faulkner, Finer & Clardy, 1976; Gonzalez, Martin, Norte,

Perez, Weyler, Rafii & Clardy, 1983). Empirical force-field (molecular-mechanics) calculations have been employed to explore the conformations of cycloundecane (Bixon & Lifson, 1967; Dale, 1973; Engler, Andose & Schleyer, 1973; Anet & Rawdah, 1978; Russell, 1981; Saunders, 1987), cycloundecene (Russell, 1981) and the various *trans,trans*-cycloundecadienes (Russell, Sim & Haufe, 1989). The two lowest-energy conformations derived for cycloundeca-1,5-diene differ by only 3.1 kJ mol<sup>-1</sup> and prove to be those found in the various X-ray studies. The observation of both conformations is consistent with their small energy difference and the possible effects of molecular substituents and crystal packing. The calculated energy difference (5.6 kJ mol<sup>-1</sup>) between the two lowest-energy conformations of cycloundeca-1,6-diene is substantially higher than the result for the 1,5-diene: only the lowest-energy 1,6-diene conformation has been found in crystal structures of cytochalasans and dolabellane diterpenoids.

The CO—OR bond in esters and lactones has appreciable double-bond character, the geometry about the bond resembles that about the C=C bond (Mathieson & Taylor, 1961; McConnell, Mathieson & Schoenborn, 1962; Mathieson, 1963; Cheung, Overton & Sim, 1965). The eleven-membered dilactone rings in the pyrrolizidine alkaloids can therefore be regarded as analogues of cycloundeca-1,6-diene with both C=C bonds replaced by CO—OR bonds. Nevertheless, the conformations of the dilactone rings of the alkaloids in the solid state (Sussman & Wodak, 1973; Stoeckli-Evans, 1979, 1982; Stoeckli-Evans & Robins, 1983; Mackay, Sadek & Culvenor, 1984; Brown, Burton, Robins & Sim, 1986; Robins & Sim, 1987) do not correspond to the conformations of cycloundeca-1,6-diene derived by force-field calculations.

Compound (1) was prepared during synthesis of lactones related to natural diterpenes (Grant, Lai, Prasad & Yap, 1988). The eleven-membered ring in

(1) can be regarded as an analogue of cycloundeca-1,6-diene with one C=C bond replaced by a CO—OR bond and an X-ray crystallographic study of (1) was undertaken to characterize the conformation of the eleven-membered ring.



**Experimental.** Crystal dimensions  $0.2 \times 0.2 \times 0.4$  mm. Enraf-Nonius CAD-4 diffractometer, Mo  $K\alpha$  radiation, generator settings 50 kV, 20 mA. Cell dimensions from setting angles of 25 reflections with  $\theta$  11–18°, 4688 reflections  $hkl$  and  $\bar{h}\bar{k}l$  surveyed in range  $\theta$  1.5–27.0° with  $h_{\max}$  10  $k_{\max}$  12,  $l_{\max}$  36; scan width  $(1.0 + 0.3 \tan \theta)^\circ$ ; max. scan time 120 s; 2309 pairs of equivalent reflections averaged,  $R_{\text{int}} = 0.035$ ; 1475 independent reflections with  $I > 2.5\sigma(I)$ . Two reference reflections monitored periodically showed no significant variation in intensity. Corrections applied for Lorentz-polarization effects assuming ideally imperfect monochromator crystal. Structure elucidated by direct phasing using *MITHRIL* (Gilmore, 1984); H-atom positions located in difference Fourier syntheses and refined. Full-matrix least-squares calculations on  $F$  with anisotropic thermal parameters for C and O atoms and isotropic for H atoms. Convergence at  $R = 0.050$ ,  $wR = 0.056$ ,  $S = 3.31$  for 327 parameters,  $\Delta/\sigma < 0.3$  for C, O atoms,  $< 1.0$  for H atoms,  $w = 1/\sigma^2(|F_o|)$ , final  $\Delta\rho$  max. 0.16, min.  $-0.16 e \text{ \AA}^{-3}$ . Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations on a SEL 32/27 computer with the *GX* system of programs (Mallinson & Muir, 1985).

**Discussion.** Atomic coordinates are listed in Table 1\* and molecular dimensions in Table 2. Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular geometry. The atomic numbering is based on the

\* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52980 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	x	y	z	$U_{\text{eq}}(\text{\AA}^2)$
C(1)	0.8400 (10)	0.1962 (6)	0.0398 (2)	0.067
C(2)	0.8363 (14)	0.0749 (9)	0.0038 (2)	0.080
C(3)	0.9909 (10)	-0.0314 (6)	0.0140 (2)	0.071
C(4)	0.9845 (7)	-0.0937 (5)	0.0648 (2)	0.053
C(5)	0.9763 (6)	0.0311 (4)	0.1023 (1)	0.042
C(6)	0.9756 (7)	-0.0218 (6)	0.1541 (2)	0.049
C(7)	1.1504 (7)	0.0161 (5)	0.1837 (2)	0.052
C(8)	1.1400 (7)	0.1583 (5)	0.2080 (2)	0.049
C(9)	0.8694 (7)	0.2727 (5)	0.1249 (2)	0.050
C(10)	0.8275 (6)	0.1496 (5)	0.0920 (2)	0.048
C(11)	1.0106 (8)	0.3647 (5)	0.1213 (2)	0.058
C(12)	1.0485 (7)	0.4853 (5)	0.1553 (2)	0.054
C(13)	1.2461 (6)	0.4986 (4)	0.1755 (2)	0.048
C(14)	1.2850 (7)	0.3887 (5)	0.2152 (2)	0.051
C(15)	1.4705 (11)	0.4007 (8)	0.2402 (3)	0.075
C(16)	1.3960 (11)	0.4941 (8)	0.1367 (3)	0.068
C(18)	0.8198 (8)	-0.1999 (6)	0.0690 (3)	0.069
C(19)	1.1714 (8)	-0.1737 (6)	0.0734 (3)	0.071
C(20)	0.6207 (8)	0.1015 (7)	0.1019 (3)	0.072
O(1)	1.0204 (6)	0.1908 (4)	0.2366 (1)	0.079
O(2)	1.2753 (4)	0.2462 (3)	0.1940 (1)	0.047
O(3)	1.2632 (5)	0.6360 (3)	0.1975 (1)	0.061

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

C(1)—C(2)	1.520 (10)	C(1)—C(10)	1.522 (8)
C(2)—C(3)	1.508 (12)	C(3)—C(4)	1.534 (7)
C(4)—C(5)	1.570 (6)	C(4)—C(18)	1.538 (8)
C(4)—C(19)	1.539 (8)	C(5)—C(6)	1.528 (7)
C(5)—C(10)	1.558 (6)	C(6)—C(7)	1.528 (8)
C(7)—C(8)	1.499 (7)	C(8)—O(1)	1.203 (7)
C(8)—O(2)	1.323 (6)	C(9)—C(10)	1.506 (7)
C(9)—C(11)	1.325 (8)	C(10)—C(20)	1.555 (8)
C(11)—C(12)	1.501 (7)	C(12)—C(13)	1.511 (7)
C(13)—C(14)	1.540 (7)	C(13)—C(16)	1.515 (10)
C(13)—O(3)	1.434 (6)	C(14)—C(15)	1.490 (10)
C(14)—O(2)	1.465 (6)		
C(2)—C(1)—C(10)	114.6 (5)	C(1)—C(2)—C(3)	111.0 (7)
C(2)—C(3)—C(4)	114.0 (6)	C(3)—C(4)—C(5)	109.4 (4)
C(3)—C(4)—C(18)	109.8 (5)	C(3)—C(4)—C(19)	107.7 (5)
C(5)—C(4)—C(18)	113.9 (5)	C(5)—C(4)—C(19)	107.1 (5)
C(18)—C(4)—C(19)	108.8 (5)	C(4)—C(5)—C(6)	112.8 (4)
C(4)—C(5)—C(10)	115.8 (4)	C(6)—C(5)—C(10)	113.8 (4)
C(5)—C(6)—C(7)	115.6 (5)	C(6)—C(7)—C(8)	114.3 (5)
C(7)—C(8)—O(1)	124.1 (5)	C(7)—C(8)—O(2)	112.8 (4)
O(1)—C(8)—O(2)	123.1 (5)	C(10)—C(9)—C(11)	127.1 (5)
C(1)—C(10)—C(5)	110.0 (4)	C(1)—C(10)—C(9)	110.5 (4)
C(1)—C(10)—C(20)	107.9 (5)	C(5)—C(10)—C(9)	107.6 (4)
C(5)—C(10)—C(20)	113.3 (4)	C(9)—C(10)—C(20)	107.5 (5)
C(9)—C(11)—C(12)	125.4 (5)	C(11)—C(12)—C(13)	117.5 (5)
C(12)—C(13)—C(14)	112.2 (4)	C(12)—C(13)—C(16)	112.3 (5)
C(12)—C(13)—O(3)	108.2 (4)	C(14)—C(13)—C(16)	111.7 (5)
C(14)—C(13)—O(3)	106.2 (4)	C(16)—C(13)—O(3)	105.8 (5)
C(13)—C(14)—C(15)	116.4 (5)	C(13)—C(14)—O(2)	108.1 (4)
C(15)—C(14)—O(2)	107.5 (5)	C(8)—O(2)—C(14)	118.9 (4)

conventional system for the labdane family of diterpenoids.

The torsion angles for the eleven-membered ring (Table 3) establish that the conformation closely resembles the lowest-energy conformation of cycloundeca-1,6-diene (Russell, Sim & Haufe, 1989); differences between corresponding torsion angles in (1) and the force-field angles are 1–15° (mean 6°).

The results in Table 3 demonstrate that cycloundeca-1,6-diene rings in various cytochalasans and dolabellane diterpenoids also adopt this conformation. The agreement among corresponding torsion angles in Table 3 is generally good and the mean discrepancy between the experimental and force-field results is 6°; the principal discrepancy (row 2, column E) arises from the comparison of a torsion angle of the type C=C—CO—C with torsion angles of the type C=C—CH<sub>2</sub>—C. There is a notable contrast between the conformational behaviour of the lactone ring in (1) and the dilactone rings in the pyrrolizidine alkaloids.

The bond angles at the C(sp<sup>3</sup>) atoms in the eleven-membered ring are in the range 107.6 (4)–117.5 (5)°. The mean value, 112.7 (4)°, is larger than tetrahedral, a common feature of large rings (Ermer, Dunitz & Bernal, 1973; Sim, 1987).

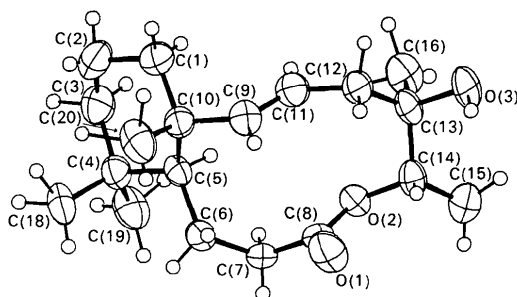


Fig. 1. Molecular structure and atomic numbering. The thermal ellipsoids of the C and O atoms are drawn at the 50% probability level and H atoms are represented by spheres of radius 0.1 Å.

Table 3. Torsion angles (°) in the 11-membered lactone ring of compound (1) and in derivatives of cycloundeca-1,6-diene: results are given consecutively around the ring

Column A: 11-membered lactone, in order C(8)—O(2), O(2)—C(14) etc.  
 Column B: 10-acetoxy-18-hydroxydolabella-2,7-diene (Ireland, Faulkner, Finer & Clardy, 1976).  
 Column C: 10,18-dihydroxy-7,8-epoxydolabella-2-ene (Gonzalez, Martin, Norte, Perez, Weyler, Rafii & Clardy, 1983).  
 Column D: cytochalasin D (Tsukuda & Koyama, 1972).  
 Column E: 17,18-di-O-acetylaspochohalasin C (Neupert-Laves & Dobler, 1982).  
 Column F: cytochalasin H (Beno, Cox, Wells, Cole, Kirksey & Christoph, 1977).  
 Column G: molecular-mechanics results for lowest-energy conformation of cycloundeca-1,6-diene (Russell, Sim & Haufe, 1989).

A	B	C	D	E	F	G
-179	175	176	178	172	-178	179
-115	-115	-122	-119	-146	-118	-116
64	69	74	52	79	61	70
-76	-73	-78	-69	-58	-71	-74
129	129	117	136	103	119	114
-179	-174	-158	-175	-171	-173	-178
74	67	64	76	79	85	85
59	68	76	66	68	65	64
-112	-127	-130	-131	-114	-118	-111
87	74	71	73	70	71	73
-119	-105	-102	-91	-95	-103	-111

The cyclohexane ring is distinctly distorted by repulsion between the axial methyl groups at C(4) and C(10). The C(18)···C(20) separation of 3.291 (7) Å involves the ring torsion angles about bonds C(4)—C(5), 48.1 (5), and C(5)—C(10), -47.4 (5)°, being substantially smaller than the other torsion angles in the ring: C(1)—C(2) -55.6 (6), C(2)—C(3) 56.4 (6), C(3)—C(4) -52.1 (6) and C(1)—C(10) 50.4 (6)°. The bond angles C(3)—C(4)—C(5) 109.4 (4) and C(1)—C(10)—C(5) 110.0 (4)° are smaller than other bond angles in the ring, and bonds C(4)—C(5) 1.570 (6) and C(5)—C(10) 1.558 (6) Å are longer than other ring bonds.

The molecules are linked in the crystal by a hydrogen bond O(3)—H···O(1) (2 - x, ½ + y, ½ - z) with O···O 2.768 (5) Å, O—H···O 158 (4)°.

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## Structure of 4,4',6,6'-Tetrakis(methylthio)-2,2'-bithieno[3,4-d][1,3]dithiolylidene

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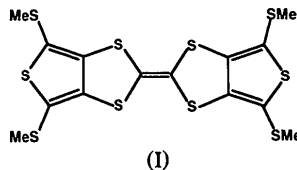
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**Abstract.** C<sub>14</sub>H<sub>12</sub>S<sub>10</sub>, *M<sub>r</sub>* = 500.91, triclinic, *P* $\bar{1}$ , *a* = 7.050 (2), *b* = 8.229 (2), *c* = 9.540 (2) Å,  $\alpha$  = 112.08 (2),  $\beta$  = 78.87 (2),  $\gamma$  = 100.97 (2)°, *V* = 499.0 (2) Å<sup>3</sup>, *Z* = 1, *D<sub>x</sub>* = 1.667 Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha)$  = 0.71069 Å,  $\mu$  = 1.059 mm<sup>-1</sup>, *F*(000) = 256, *T* = 293 K, *R* = 0.038 for 1984 observed reflections. The conformation of the molecular frame is that of a very shallow chair with the 1,3-dithiole rings of envelope type. One of the exocyclic methylthio groups is perpendicular to the frame while the other is approximately coplanar to the molecular plane. The molecules form a sheet-like network nearly parallel to the ( $\bar{1}20$ ) plane where the molecules are connected with van der Waals S...S contacts of 3.642 (2) and 3.753 (2) Å. The intersheet interaction is also van der Waals type and unfavorable to the intermolecular charge-transfer interaction.

**Introduction.** Recently many efforts have been made to obtain new types of organic donors and acceptors. The title compound, (I), a thiophene-fused tetrathiafulvalene (TTF) modified by methylthio groups, has been prepared to introduce interstack interactions (Kobayashi, 1985). Unfortunately, the molecule does not show a strong electron-donating character. The crystal structure analysis was carried out to investi-

gate relations between structure and donating character.



**Experimental.** Yellow needles, 0.70 × 0.35 × 0.35 mm, unit-cell parameters by least squares from 25 reflections (30 < 2θ < 47°), Rigaku AFC-4 diffractometer with graphite monochromator, 2 ≤ 2θ ≤ 55°, *h* = -9 → 9, *k* = 0 → 10, *l* = -12 → 12, ω-2θ scan, scan range (1.4 + 0.4 tan θ)°, scan speed 4° min<sup>-1</sup> in 2θ, three reflections (132, 211, 240) monitored every 50 reflections, variations within 3%. 2591 reflections measured, 2422 unique, *R<sub>int</sub>* = 0.02, 1984 observed, |*F<sub>o</sub>*| ≥ 3σ(*F*). No absorption corrections were applied. Structure solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms were found from the difference map. Block-diagonal least-squares refinement with anisotropic temperature factors for non-H atoms and isotropic ones for H.  $\sum w(|F_o| - k^{-1}|F_c|)^2$  was minimized. *w* = 1 for all reflections. Δρ<sub>max</sub> = 0.3 e Å<sup>-3</sup>, Δ/σ<sub>max</sub> = 0.08 for

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