The Crystal Structure of 1,7-Dioxa-Cyclododecane-2,8-dione and 1,7,13-Trioxa-Cyclooctadecane-2,8,14-trione at −150 °C

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The crystals of $C_{10}H_{16}O_4$, (I), belong to the monoclinic system with cell dimensions $a=15.179(2)$, $b=17.378(3)$, $c=15.356(2)$ Å, $\beta=96.28(1)°$, space group $P2_1/n$ and $Z=16$. Those of $C_{15}H_{24}O_6$, (II), are orthorhombic with cell dimensions $a=33.412(9)$, $b=4.732(1)$, $c=10.359(2)$ Å, space group $Pca2_1$ and $Z=4$. The structures were solved by direct methods. The final R-values were 6.7 % (4014 reflections) for (I) and 5.0 % (1279 reflections) for (II). Data were collected on an automatic four circle diffractometer at $ca.$ −150 °C. The conformation of the dilactone ring is the quadruangular [3 3 3 3] with the ester dipoles oriented roughly parallel. The trilactone ring skeleton adopts a nonangular conformation with approximately D_{3d} symmetry. The latter structure is disordered.

Crystal structure determinations of the title compounds, the syntheses of which will be published in the near future,[1] have been carried out for two reasons:

1; to verify the explanation (suggested on conformational grounds) of the alternating melting points and dipole moments in the series of dilactones.[2] The crystal structure determination of the lactone with $n=5$ has recently been reported.[3] (I) corresponds to $n=4$.

2; to explore whether the 18-membered ring of (II) adopts a conformation different from earlier findings.[4–9]

EXPERIMENTAL

Crystal and intensity data. Crystal data for (I) are: $a=15.179(2)$, $b=17.378(3)$, $c=15.356(2)$ Å, $\beta=96.28(1)°$, $Z=16$, space group $P2_1/n$, $D_x=1.32$ gcm^{-3}, $D_m=1.30$ gcm^{-3} (by flotation), $V=4026(1)$ Å^3, $\mu=1.09$ cm^{-1} (MoK$_\alpha$), λ(MoK$_\alpha$)=0.71069 Å. The crystal data for (II) are: $a=33.412(9)$, $b=4.732(1)$, $c=10.359(2)$ $Z=4$, space group $Pca2_1$, $D_x=1.22$ gcm^{-3}, $D_m=1.21$
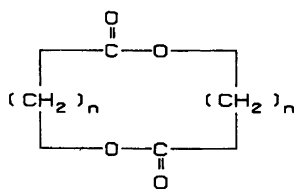
Table 1. Final fractional coordinates with estimated standard deviations for the non-hydrogen atoms of the dilactone.

ATOM	X	Y	Z
O2	-.0423(2)	.2108(2)	.5598(2)
O4	.2950(2)	.2052(2)	.5699(2)
C1	-.0240(3)	.1435(3)	.5750(3)
C2	-.0130(3)	.1065(2)	.6651(3)
C3	.0811(3)	.0768(2)	.6878(3)
C4	.1483(3)	.1425(3)	.7000(3)
C5	.2442(3)	.1154(3)	.6980(3)
O3	.2553(2)	.0873(2)	.6114(2)
C6	.2732(3)	.1398(3)	.5529(3)
C7	.2595(3)	.1066(2)	.4609(3)
C8	.1646(3)	.0812(3)	.4360(3)
C9	.1010(3)	.1480(3)	.4260(3)
C10	.0035(3)	.1236(3)	.4273(3)
O1	-.0087(2)	.0929(2)	.5126(2)
O6	.1518(2)	.0301(2)	-.0896(2)
O8	.0970(2)	.0376(2)	.2141(2)
C11	.1677(3)	.0971(3)	-.0726(3)
C12	.2574(3)	.1310(2)	-.0477(3)
C13	.2673(3)	.1599(2)	.0466(3)
C14	.2634(3)	.0945(2)	.1119(3)
C15	.2454(3)	.1207(2)	.2026(3)
O7	.1574(2)	.1548(2)	.1985(2)
C16	.0889(3)	.1051(3)	.1983(3)
C17	.0020(3)	.1465(3)	.1735(3)
C18	-.0068(3)	.1711(2)	.0767(3)
C19	-.0090(3)	.1025(2)	.0139(3)
C20	.0124(3)	.1240(2)	-.0769(3)
O5	.1040(2)	.1510(2)	-.0719(2)
O10	-.0431(2)	.2901(2)	.8044(2)
O12	.2934(2)	.2831(2)	.8138(2)
C21	-.0202(3)	.3556(2)	.8208(3)
C22	-.0081(3)	.3913(2)	.9100(3)
C23	.0878(3)	.4171(2)	.9362(3)
C24	.1525(3)	.3491(2)	.9462(3)
C25	.2481(3)	.3735(2)	.9448(3)
O11	.2608(2)	.4027(2)	.8583(2)
C26	.2756(3)	.3496(3)	.7964(3)
C27	.2659(3)	.3839(2)	.7066(3)
C28	.1705(3)	.4137(3)	.6813(3)
C29	.1035(3)	.3496(2)	.6723(3)
C30	.0091(3)	.3762(3)	.6720(3)
O9	-.0018(2)	.4062(2)	.7589(2)
O14	.6558(2)	.0415(2)	.6564(2)
O16	.6051(2)	.0328(2)	.9656(2)
C31	.6621(3)	.1087(3)	.6751(3)
C32	.7494(3)	.1502(2)	.6992(3)
C33	.7596(3)	.1755(3)	.7956(3)
C34	.7608(3)	.1070(2)	.8574(3)
C35	.7422(3)	.1290(2)	.9496(3)
O15	.6503(2)	.1544(2)	.9463(2)
C36	.5874(3)	.0989(3)	.9473(3)
C37	.4955(3)	.1310(3)	.9247(3)
C38	.4835(3)	.1589(3)	.8297(3)
C39	.4891(3)	.0943(2)	.7648(3)
C40	.5055(3)	.1206(3)	.6736(3)
O13	.5934(2)	.1562(2)	.6780(2)

Table 2. Bond distances and angles with estimated standard deviations for the dilactone.

DISTANCE	(Å)	DISTANCE	(Å)
O2 – C1	1.219(5)	O4 – C6	1.204(5)
C1 – C2	1.518(6)	C1 – O1	1.339(5)
C2 – C3	1.524(6)	C3 – C4	1.528(6)
C4 – C5	1.534(6)	C5 – O3	1.443(5)
O3 – C6	1.330(5)	C6 – C7	1.519(6)
C7 – C8	1.514(6)	C8 – C9	1.508(6)
C9 – C10	1.542(6)	C10 – O1	1.446(5)
O6 – C11	1.212(5)	O8 – C16	1.201(5)
C11 – C12	1.494(6)	C11 – O5	1.348(5)
C12 – C13	1.524(6)	C13 – C14	1.521(6)
C14 – C15	1.518(6)	C15 – O7	1.456(5)
O7 – C16	1.351(5)	C16 – C17	1.515(6)
C17 – C18	1.539(6)	C18 – C19	1.532(6)
C19 – C20	1.511(6)	C20 – O5	1.462(5)
O10 – C21	1.209(5)	O12 – C26	1.211(5)
C21 – C22	1.497(6)	C21 – O9	1.346(5)
C22 – C23	1.535(6)	C23 – C24	1.532(6)
C24 – C25	1.515(6)	C25 – O11	1.455(5)
O11 – C26	1.360(5)	C26 – C27	1.494(6)
C27 – C28	1.548(6)	C28 – C29	1.503(6)
C29 – C30	1.505(6)	C30 – O9	1.459(5)
O14 – C31	1.204(5)	O16 – C36	1.206(5)
C31 – C32	1.519(6)	C31 – O13	1.335(5)
C32 – C33	1.536(6)	C33 – C34	1.522(6)
C34 – C35	1.524(6)	C35 – O15	1.459(5)
O15 – C36	1.357(5)	C36 – C37	1.507(6)
C37 – C38	1.529(6)	C38 – C39	1.510(6)
C39 – C40	1.520(6)	C40 – O13	1.465(5)

ANGLE	(°)	ANGLE	(°)
O2 – C1 – C2	125.5(4)	O2 – C1 – O1	123.1(5)
C2 – C1 – O1	111.4(4)	C1 – C2 – C3	111.0(4)
C2 – C3 – C4	111.8(4)	C3 – C4 – C5	113.0(4)
C4 – C5 – O3	109.2(4)	C5 – O3 – C6	116.3(4)
O4 – C6 – O3	125.0(5)	O4 – C6 – C7	124.5(4)
O3 – C6 – C7	110.5(4)	C6 – C7 – C8	112.1(4)
C7 – C8 – C9	112.4(4)	C8 – C9 – C10	113.1(4)
C9 – C10 – O1	109.3(4)	C1 – O1 – C10	117.1(4)
O6 – C11 – C12	126.1(4)	O6 – C11 – O5	122.9(4)
C12 – C11 – O5	111.0(4)	C11 – C12 – C13	111.5(4)
C12 – C13 – C14	111.9(4)	C13 – C14 – C15	113.9(4)
C14 – C15 – O7	109.7(4)	C15 – O7 – C16	116.3(3)
O8 – C16 – O7	124.2(4)	O8 – C16 – C17	125.6(4)
O7 – C16 – C17	110.2(4)	C16 – C17 – C18	110.8(4)
C17 – C18 – C19	112.7(4)	C18 – C19 – C20	113.4(3)
C19 – C20 – O5	109.3(4)	C11 – O5 – C20	117.1(3)
O10 – C21 – C22	125.7(4)	O10 – C21 – O9	123.1(4)
C22 – C21 – O9	111.2(4)	C21 – C22 – C23	112.2(3)
C22 – C23 – C24	112.3(4)	C23 – C24 – C25	112.8(3)
C24 – C25 – O11	109.5(4)	C25 – O11 – C26	116.8(3)
O12 – C26 – O11	122.9(4)	O12 – C26 – C27	125.5(4)
O11 – C26 – C27	111.6(4)	C26 – C27 – C28	111.1(4)
C27 – C28 – C29	112.4(4)	C28 – C29 – C30	114.0(4)
C29 – C30 – O9	108.0(4)	C21 – O9 – C30	117.7(3)
O14 – C31 – C32	124.4(4)	O14 – C31 – O13	124.4(4)
C32 – C31 – O13	111.2(4)	C31 – C32 – C33	111.3(3)
C32 – C33 – C34	111.7(4)	C33 – C34 – C35	113.1(4)
C34 – C35 – O15	108.5(4)	C35 – O15 – C36	117.1(3)
O16 – C36 – O15	122.8(4)	O16 – C36 – C37	125.6(5)
O15 – C36 – C37	111.6(4)	C36 – C37 – C38	110.4(4)
C37 – C38 – C39	112.5(4)	C38 – C39 – C40	114.2(4)
C39 – C40 – O13	109.0(4)	C31 – O13 – C40	116.6(3)

Table 3. Dihedral angles with estimated standard deviations for the dilactone.

DIHEDRAL ANGLE				(°)
O2	C1	C2	C3	-117.4(5)
C2	C1	O1	C10	-166.0(4)
O1	C1	C2	C3	61.0(5)
C1	C2	C3	C4	67.5(5)
C2	C3	C4	C5	-164.3(4)
C3	C4	C5	O3	66.2(5)
C4	C5	O3	C6	84.7(5)
C5	O3	C6	C7	-165.2(4)
O4	C6	C7	C8	-117.1(5)
O3	C6	C7	C8	61.0(5)
C6	C7	C8	C9	68.1(5)
C7	C8	C9	C10	-163.4(4)
C8	C9	C10	O1	65.3(5)
C9	C10	O1	C1	84.5(5)
O6	C11	C12	C13	-113.7(5)
C12	C11	O5	C20	-167.5(4)
O5	C11	C12	C13	64.7(5)
C11	C12	C13	C14	65.1(5)
C12	C13	C14	C15	-162.8(3)
C13	C14	C15	O7	64.8(4)
C14	C15	O7	C16	81.3(4)
C15	O7	C16	C17	-167.4(4)
O8	C16	C17	C18	-109.7(5)
O7	C16	C17	C18	69.1(5)
C16	C17	C18	C19	63.0(5)
C17	C18	C19	C20	-161.2(3)
C18	C19	C20	O5	64.9(4)
C19	C20	O5	C11	83.9(4)
O10	C21	C22	C23	117.9(5)
C22	C21	O9	C30	166.5(3)
O9	C21	C22	C23	-61.9(5)
C21	C22	C23	C24	-65.3(5)
C22	C23	C24	C25	162.7(4)
C23	C24	C25	O11	-66.8(5)
C24	C25	O11	C26	-83.3(4)
C25	O11	C26	C27	166.3(3)
O12	C26	C27	C28	117.1(5)
O11	C26	C27	C28	-62.2(5)
C26	C27	C28	C29	-66.1(5)
C27	C28	C29	C30	164.4(4)
C28	C29	C30	O9	-67.6(5)
C29	C30	O9	C21	-84.8(4)
O14	C31	C32	C33	-113.0(5)
C32	C31	O13	C40	-168.1(4)
O13	C31	C32	C33	66.6(5)
C31	C32	C33	C34	62.2(5)
C32	C33	C34	C35	-162.3(3)
C33	C34	C35	O15	67.8(4)
C34	C35	O15	C36	82.5(5)
O16	C36	C37	C38	-115.0(5)
O15	C36	C37	C38	66.0(5)
C36	C37	C38	C39	64.4(5)
C37	C38	C39	C40	-162.4(4)
C38	C39	C40	O13	65.3(5)
C39	C40	O13	C31	83.6(5)
C35	O15	C36	C37	-168.0(4)

Determination and refinement of the structures. The structures were solved by direct methods [10] and refined by fullmatrix least-squares technique.[11] All programs used (except those for phase determination) are included in Ref. 11. For (II) the E-statistics and the $N(Z)$-plot strongly indicated an acentric space group. Since the centrosymmetric space group *Pbcm* (which is possible from the systematic absences) has eight equivalent positions and thus demands the molecule to possess symmetry, space group $Pca2_1$ was chosen. The E-map corresponding to the best figure of merit contain several split peaks indicating a disordered structure. The procedure from this map to the final structure was somewhat lengthy: repeated use of weighted Fourier calculations, intermediate introduction of anisotropic temperature factors (in order to calculate probable positions for partial
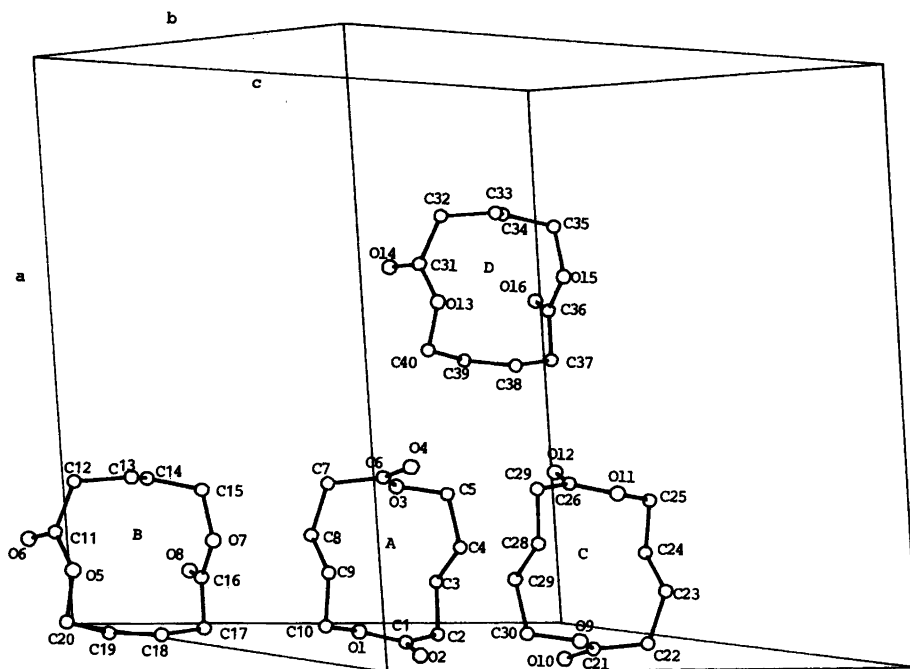
Fig. 1. Perspective drawing of the four independent dilactone molecules, (I), showing the numbering of atoms.
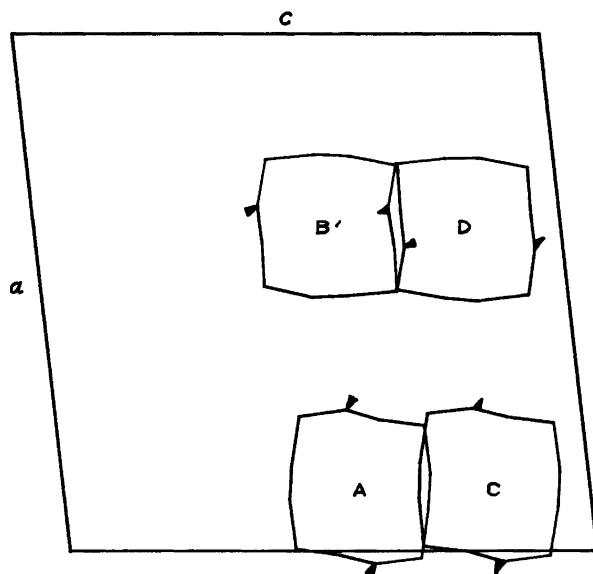

Fig. 2. Schematic drawing illustrating the pseudo symmetry in the dilactone, (I), unit cell.
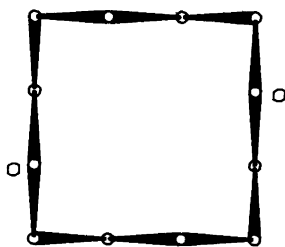
Table 4. Final fractional coordinates with estimated standard deviations for the non-hydrogen atoms of the trilactone.

ATOM	X	Y	Z
O1	.8041(1)	.2679(6)	.4765
O2A	.7680(7)	.5546(71)	.3542(27)
O3A	.9464(2)	.7475(14)	.5416(7)
O4A	.9957(2)	1.0346(15)	.6009(9)
O5A	.8872(2)	.1577(16)	.9053(7)
O6A	.8423(3)	.0955(39)	1.0691(14)
C1A	.8017(3)	.4811(24)	.3784(12)
C2A	.8388(3)	.5742(22)	.3178(10)
C3A	.8697(3)	.6988(20)	.4134(9)
C4A	.9086(3)	.7811(19)	.3436(9)
C5A	.9376(2)	.9341(19)	.4310(8)
C6A	.9763(3)	.8179(25)	.6201(13)
C7A	.9813(2)	.6195(19)	.7282(10)
C8A	.9420(3)	.5608(22)	.8011(11)
C9A	.9480(3)	.3946(21)	.9200(10)
C10A	.9109(3)	.3261(19)	.9930(9)
C11A	.8519(3)	.0632(22)	.9525(10)
C12	.8308(1)	-.0726(8)	.8531(5)
C13	.8148(1)	.1269(8)	.7496(4)
C14	.7863(1)	-.0195(9)	.6574(4)
C15	.7702(1)	.1786(9)	.5540(5)
O2B	.7586(7)	.5426(75)	.3771(28)
O3B	.9423(1)	.7241(12)	.4778(6)
O4B	.9856(2)	1.0753(14)	.5130(8)
O5B	.8892(2)	.2312(16)	.8815(8)
O6B	.8602(3)	.0730(36)	1.0586(14)
C1B	.7918(3)	.4425(21)	.3889(10)
C2B	.8275(3)	.4987(22)	.3043(10)
C3B	.8613(3)	.6441(18)	.3774(10)
C4B	.8986(2)	.6828(18)	.2956(8)
C5B	.9304(2)	.8618(17)	.3586(9)
C6B	.9708(3)	.8607(24)	.5483(13)
C7B	.9792(3)	.7000(22)	.6711(12)
C8B	.9426(2)	.6172(18)	.7483(9)
C9B	.9544(2)	.4542(22)	.8698(12)
C10B	.9200(3)	.3828(20)	.9577(10)
C11B	.8630(4)	.0881(28)	.9460(12)

atoms) [12], careful refinements of multiplicity factors (keeping thermal parameters constant), and calculations of partial hydrogen atom positions (to be used in structure factor calculations). Anisotropic temperature factors were introduced for all ordered non-hydrogen atoms and for some of the disordered ones of (II) (those for which the thermal analysis have physical meaningful results). Hydrogen atoms, the positions of which were calculated, were refined with isotropic temperature factors for (I). The H-atoms of (II) were not refined. Weights in least-squares were obtained from the standard deviations in intensities, $\sigma(I)$, taken as $\sigma(I)=[C_T+0.02C_N)^2]^{\frac{1}{2}}$ where C_T is the total number of counts and C_N the net count. Since attempts to refine the occupancy factors, G_A and G_B, of the disordered atoms in II) resulted in deviations from $G_A=G_B=0.5$ of less than three times the e.s.d.'s, a 50 % mixture of the two forms has been assumed. The final R-values were $R=6.7$ % $(R_w=6.1$ %) for the 4014 reflections of (I), and $R=5.0$ % $(R_w=4.7$ %) for the 1279 reflections of (II). Standard deviations in bond distances and angles and dihedral angles were calculated from the correlation matrices of the final least squares refinement cycles. Final fractional coordinates with estimated standard deviations for non-hydrogen atoms are given in Tables 1 and 4. No correlation between the refined parameters are larger than 0.60. The maximum r.m.s. amplitudes of (I) range from 0.17 to 0.23 Å, while those of (II) lay between 0.19 and 0.30 Å. Lists of hydrogen atom parameters, thermal parameters and observed and calculated structure factors are obtainable from the author.

DISCUSSION

The dilactone. A thorough reexamination of the intensity data (using a program that lists all reflections within thin shells in reciprocal space) revealed to additional symmetry to account for the unusual large number of four independent molecules in the asymmetric unit. Fig. 1 is a perspective drawing showing the numbering of atoms. Although no extra symmetry could be detected, the molecule pairs A,C and B',D of Fig. 2 (the prime means: $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$) are approximately related by the operation x, $\frac{1}{2}-y$, $\frac{1}{4}+z$, corresponding to a pseudo axial glide plane at $y=\frac{1}{4}$ with translation periode $c/4$. The fact that equivalent bond distances and angles (Table 2) and torsion angles (Table 3) of the four molecules are equal (and normal) within error limits provides an argument in favour of $Z=4$ rather than $Z=16$. An explanation must probably be sought in the packing conditions (although no short intermolecular distances are observed). Fig. 3 is a stereo view which shows the cell contents. The conformation of the ring skeleton is the strain-free quadrangular [3 3 3 3][13] with approximate C_4 symmetry.

[3333]

The ester dipoles (carbonyl groups) are oriented roughly parallel in accordance with predictions made to explain the alternation of melting points and dipole moments of dilactones[2].

The trilactone. Fig. 4 is a perspective drawing of the trilactone molecules illustrating the type of disorder and showing the numbering of atoms for the A-molecule. Eventual disorder of the molecule fragment C12−C13−C14−C15−O1 could not be handled computationally. The disorder of the rest of molecule arises from a rotation (*ca.* 7°) about an axis, normal to the "ring plane", through the mid point of the line between O1 and C12 (see Fig. 4). The bond distances and angles of Table 5 are normal within error limits except from those involving C1 and C11. A possible explanation to the rather ackward geometries arrived at in
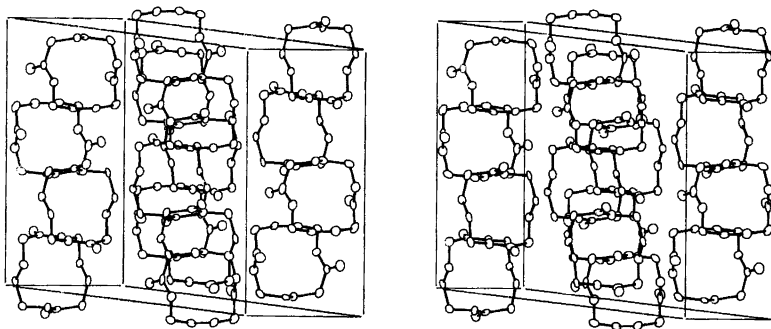

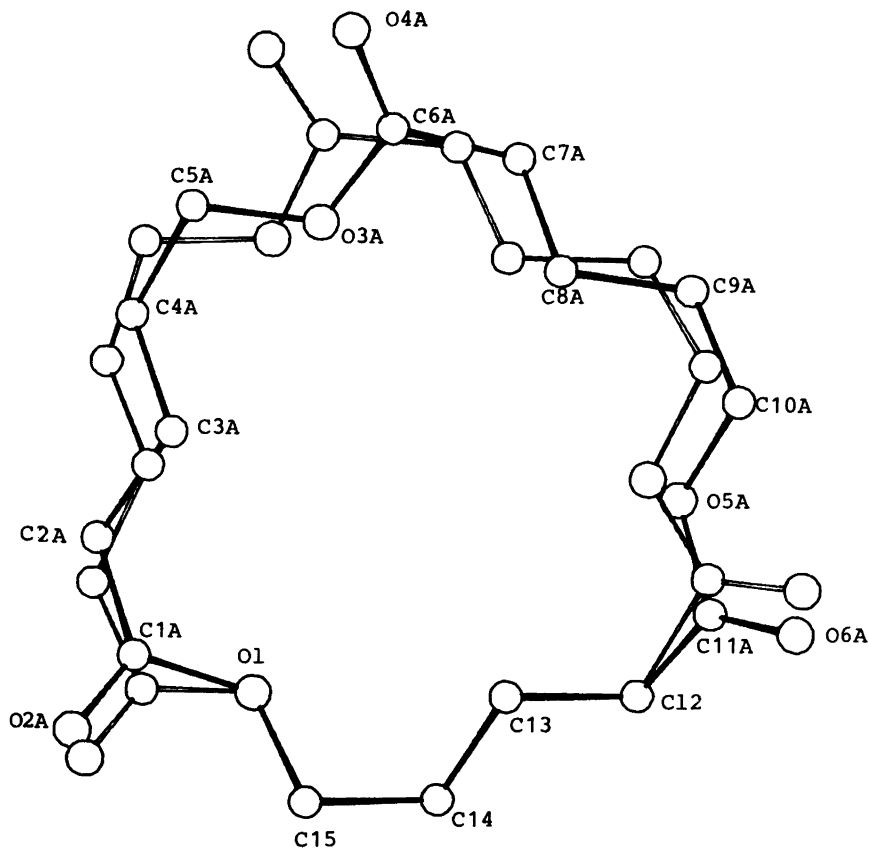*Fig. 3.* Stereo view showing the dilactone, (I), cell contents.

Fig. 4. Perspective drawing of the trilactone molecules, (II), illustrating the type of disorder and showing the numbering of atoms for the A-molecule.

these regions of the molecule could be that O1 and C12 in fact are disordered to some extent and, when treated as ordered atoms, prevent proper refinement of C1 and C11. On the other hand, the thermal parameters of all atoms in the fragment regarded as ordered have reasonable values, and attempts to introduce disorder were not successful. The torsion angles of Table 6 show that both ring skeletons adopt the nonangular conformation with approximate D_{3d} symmetry.
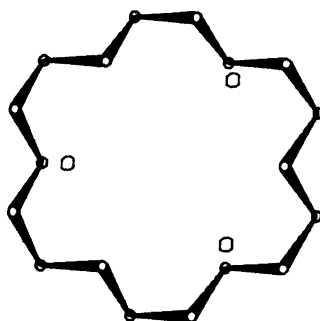
Table 5. Bond distances and angles with estimated standard deviations for the trilactone.

DISTANCE	(Å)	DISTANCE	(Å)
O2A - C1A	1.20(3)	O4A - C6A	1.23(1)
O6A -C11A	1.26(2)	O2B - C1B	1.21(3)
O4B - C6B	1.19(1)	O6B -C11B	1.17(2)
C1A - C2A	1.46(1)	C2A - C3A	1.55(1)
C3A - C4A	1.54(1)	C4A - C5A	1.51(1)
O3A - C5A	1.48(1)	C6A - O3A	1.33(1)
C6A - C7A	1.47(1)	C7A - C8A	1.54(1)
C8A - C9A	1.47(1)	C9A -C10A	1.49(1)
O5A -C10A	1.44(1)	O5A -C11A	1.35(1)
C12 -C11A	1.40(1)	C12 - C13	1.52(1)
C13 - C14	1.52(1)	C14 - C15	1.52(1)
O1 - C15	1.45(1)	O1 - C1A	1.43(1)
C1B - C2B	1.50(1)	C2B - C3B	1.52(1)
C3B - C4B	1.52(1)	C4B - C5B	1.51(1)
O3B - C5B	1.45(1)	C6B - O3B	1.36(1)
C6B - C7B	1.51(1)	C7B - C8B	1.51(1)
C8B - C9B	1.53(1)	C9B -C10B	1.50(1)
O5B -C10B	1.48(1)	O5B -C11B	1.29(2)
C12 -C11B	1.63(1)	O1 - C1B	1.29(1)

ANGLE	(°)	ANGLE	(°)
O1 - C1A - O2A	114.(1)	O2A - C1A - C2A	128.(2)
O4A - C6A - O3A	120.(1)	O4A - C6A - C7A	127.(1)
O6A -C11A - O5A	122.(1)	O6A -C11A - C12	129.(1)
O1 - C1B - O2B	128.(2)	O2B - C1B - C2B	127.(1)
O4B - C6B - O3B	122.(1)	O4B - C6B - C7B	128.(1)
O6B -C11B - O5B	127.(1)	O6B -C11B - C12	120.(1)
O1 - C1A - C2A	118.(1)	C1A - C2A - C3A	114.(1)
C2A - C3A - C4A	111.(1)	C3A - C4A - C5A	112.(1)
O4A - C6A - C7A	127.(1)	C6A - O3A - C5A	118.(1)
O3A - C6A - C7A	113.(1)	C6A - C7A - C8A	113.(1)
C7A - C8A - C9A	113.(1)	C8A - C9A -C10A	115.(1)
O5A -C10A - C9A	105.(1)	C10A - O5A -C11A	116.(1)
C12 -C11A - O5A	109.(1)	C13 - C12 -C11A	114.(1)
C12 - C13 - C14	112.(1)	C13 - C14 - C15	113.(1)
O1 - C15 - C14	107.(1)	C15 - O1 - C1A	123.(1)
O1 - C1B - C2B	106.(1)	C1B - C2B - C3B	112.(1)
C2B - C3B - C4B	113.(1)	C3B - C4B - C5B	114.(1)
O3B - C5B - C4B	108.(1)	C6B - O3B - C5B	116.(1)
O3B - C6B - C7B	110.(1)	C6B - C7B - C8B	115.(1)
C7B - C8B - C9B	111.(1)	C8B - C9B -C10B	114.(1)
O5B -C10B - C9B	108.(1)	C10B - O5B -C11B	117.(1)
C12 -C11B - O5B	113.(1)	C13 - C12 -C11B	111.(1)
C15 - O1 - C1B	109.(1)		

The same conformation has been found in several crystalline "adducts" or molecular complexes of 1,4,7,10,13,16-hexaoxacyclooctadecane (18-crown-6).[4-7] The stereo view showing the unit cell contents, Fig. 5, reveals no obvious reason for the observed disorder phenomenon of the present macrocycle. Neither a thorough examination of the inter-molecular distances between oxygen, carbon and hydrogen atoms could give an answer to the question. It is probably necessary to calculate energy profiles corresponding to rotational displacements[14] in order to throw light upon this peculiar type of disorder.

Table 6. Dihedral angles with estimated standard deviations for the trilactone.

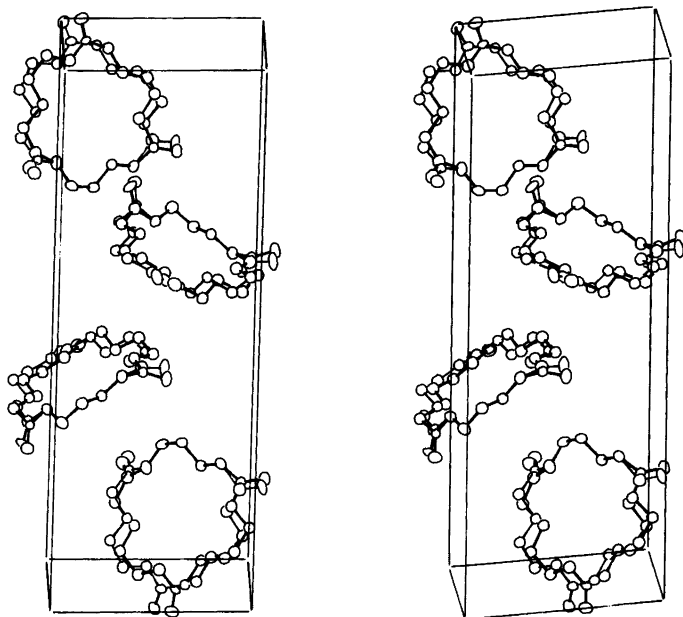| DIHEDRAL ANGLE	(°)
O1 - C1A - C2A - C3A	-59.(1)
C1A - C2A - C3A - C4A	178.(1)
C2A - C3A - C4A - C5A	173.(1)
C3A - C4A - C5A - O3A	58.(1)
C6A - O3A - C5A - C4A	169.(1)
C7A - C6A - O3A - C5A	178.(1)
O3A - C6A - C7A - C8A	-50.(1)
C6A - C7A - C8A - C9A	-172.(1)
C7A - C8A - C9A -C10A	-179.(1)
C8A - C9A -C10A - O5A	62.(1)
C11A - O5A -C10A - C9A	178.(1)
C10A - O5A -C11A - C12	174.(1)
C13 - C12 -C11A - O5A	-71.(1)
C11A - C12 - C13 - C14	-170.(1)
C12 - C13 - C14 - C15	180.(1)
C13 - C14 - C15 - O1	64.(1)
C1A - O1 - C15 - C14	-173.(1)
C15 - O1 - C1A - C2A	173.(1)
O1 - C1B - C2B - C3B	-64.(1)
C1B - C2B - C3B - C4B	176.(1)
C2B - C3B - C4B - C5B	172.(1)
C3B - C4B - C5B - O3B	60.(1)
C6B - O3B - C5B - C4B	-179.(1)
C7B - C6B - O3B - C5B	178.(1)
O3B - C6B - C7B - C8B	-50.(1)
C6B - C7B - C8B - C9B	180.(1)
C7B - C8B - C9B -C10B	176.(1)
C8B - C9B -C10B - O5B	54.(1)
C11B - O5B -C10B - C9B	162.(1)
C10B - O5B -C11B - C12	-179.(1)
C13 - C12 -C11B - O5B	-47.(1)
C11B - C12 - C13 - C14	177.(1)
C1B - O1 - C15 - C14	-179.(1)
C15 - O1 - C1B - C2B	-174.(1)

Fig. 5. Stereo view showing the trilactone, (II), unit cell contents.

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