

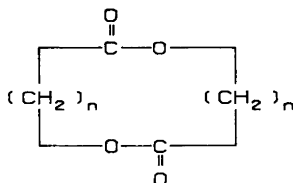
## Short Communications

### The Crystal Structure of 1,8-Dioxa-cyclotetradecane-2,9-dione at $-150\text{ }^{\circ}\text{C}$

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The alternation of melting points and dipole moments in the series of dilactones has been explained on conformational grounds.<sup>1</sup> The crystal structure determination of the first



member with odd  $n$ , ( $n=5$ ), was undertaken in order to verify that this molecule has a compact strain-free conformation consisting of straight parallel polymethylene chains linked by two ester bridges with the carbonyl groups oriented in opposite directions.

The crystals of  $\text{C}_{12}\text{H}_{20}\text{O}_4$  are monoclinic with space group  $P2_1/c$ , cell dimensions  $a=8.552(1)$ ,  $b=10.111(2)$ ,  $c=7.533(2)$  Å,  $\beta=113.83(1)^\circ$ , and  $Z=2$  ( $D_x=1.27\text{ g cm}^{-3}$ ,  $D_m=1.25\text{ g cm}^{-3}$ ). With  $2\theta_{\text{max}}=50^\circ$ ,  $\text{MoK}_\alpha$ -radiation, and an observed-unobserved cutoff at  $2.5\sigma(I)$ , 946 independent reflections were recorded as observed on an automatic fourcircle diffractometer at *ca.*  $-150\text{ }^{\circ}\text{C}$ . No corrections for absorption or secondary extraction were

Table 1. Final fractional coordinates with estimated standard deviations.

ATOM	X	Y	Z
O2	.2674( 1)	.0580( 1)	.2589( 1)
C1	.2290( 1)	.1091( 1)	.3804( 2)
C2	.3485( 2)	.1369( 1)	.5868( 2)
C3	.2918( 2)	.0682( 1)	.7326( 2)
C4	.2787( 2)	-.0810( 1)	.7029( 2)
C5	.2316( 2)	-.1574( 1)	.8496( 2)
C6	-.0601( 2)	.1202( 1)	.1512( 2)
O1	.0688( 1)	.1482( 1)	.3448( 1)
H21	.351( 2)	.232( 2)	.605( 2)
H22	.461( 2)	.106( 1)	.603( 2)
H31	.183( 2)	.105( 1)	.722( 2)
H32	.376( 2)	.090( 1)	.867( 2)
H41	.389( 2)	-.115( 1)	.711( 2)
H42	.195( 2)	-.101( 1)	.571( 2)
H51	.317( 2)	-.141( 1)	.982( 2)
H52	.231( 2)	-.254( 2)	.823( 2)
H61	-.054( 2)	.027( 1)	.123( 2)
H62	-.033( 2)	.173( 1)	.057( 2)

Table 2. Bond distances and angles and dihedral angles with estimated standard deviations. The primes correspond to symmetry operation  $-x, -y, 1-z$ .

DISTANCE		(Å)	DISTANCE		(Å)
O2 - C1	1.206	( 1)	C1 - C2	1.502	( 2)
C1 - O1	1.346	( 1)	C2 - C3	1.534	( 2)
C3 - C4	1.523	( 2)	C4 - C5	1.529	( 2)
C5' - C6	1.512	( 2)	C6 - O1	1.457	( 1)

ANGLE		(°)	ANGLE		(°)
O2 - C1 - C2	125.9	( 1)	O2 - C1 - O1	122.8	( 1)
C2 - C1 - O1	111.3	( 1)	C1 - C2 - C3	112.1	( 1)
C2 - C3 - C4	111.7	( 1)	C3 - C4 - C5	114.8	( 1)
C4 - C5 - C6	114.2	( 1)	C5' - C6 - O1	107.2	( 1)
C1 - O1 - C6	116.1	( 1)			

DIHEDRAL ANGLE		(°)
C2 - C1 - O1 - C6	-177.1	( 1)
O1 - C1 - C2 - C3	58.4	( 1)
C1 - C2 - C3 - C4	58.1	( 1)
C2 - C3 - C4 - C5	177.0	( 1)
C3 - C4 - C5 - C6'	60.8	( 2)
C4 - C5 - C6' - O1'	60.2	( 1)
C5 - C6 - O1 - C1	173.6	( 1)

applied (crystal size  $0.3 \times 0.5 \times 0.2$  mm). The structure was solved by direct methods<sup>2</sup> and refined by full-matrix least squares technique.<sup>3</sup> All programs used (except those for phase determination) are included in Ref. 3. Anisotropic temperature factors were used for oxygen and carbon atoms. Weights in least squares were calculated from the standard deviations in intensities,  $\sigma(I)$ , taken as  $\sigma(I) = [C_T + (0.02 C_N)^2]^{1/2}$  where  $C_T$  is the total number of counts and  $C_N$  the net count. The final  $R$ -values were  $R = 3.2\%$  and  $R_w = 3.9\%$  for 946 observed reflections. Maximum r.m.s. amplitudes range from 0.17 to 0.22 Å.

Final fractional coordinates with estimated standard deviations are given in Table 1. Bond distances and angles and dihedral angles may be found in Table 2. Fig. 1 is a perspective drawing of the centrosymmetric molecule showing the numbering of atoms. The

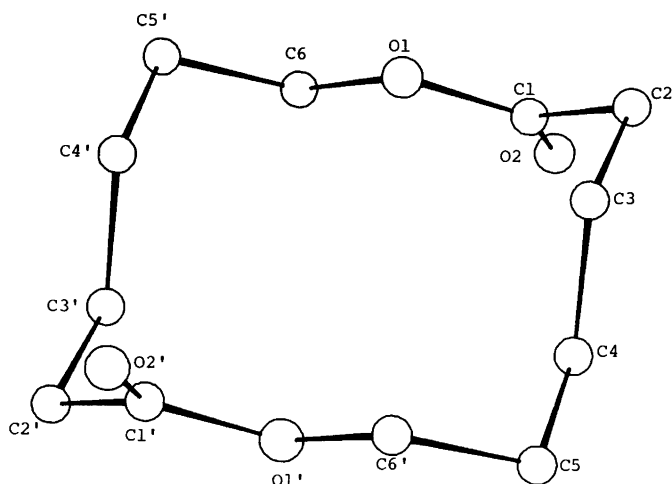
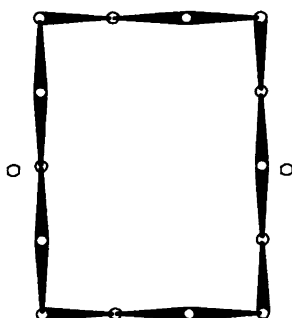


Fig. 1. Perspective drawing of the molecule showing the numbering of atoms.



[3434]

torsion angles of Table 2 show that the ring conformation may be described as [3 4 3 4].<sup>4</sup> This conformation was also found in cyclotetradecanone<sup>5</sup> and cyclotetradecane.<sup>6</sup>

It should be pointed out that the ester groups are situated at the long "edge" of the rectangular ring rather than acting as bridges between straight parallel polymethylene chains.<sup>1</sup> The center of inversion demands exact opposite orientation of the ester dipoles.

Bond distances and angles are normal within error limits.

Lists of thermal parameters and observed and calculated structure factors are available from the author.

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