

Table 3. Deviations (Å) of non-H atoms from the least-squares planes through different parts of the molecules

Deviations of atoms which do not define the plane are listed in parentheses.

Molecule 1			Molecule 2		
C(1)	0.0543	-0.0315	C(11)	-0.0324	0.0352 (-0.0033)
C(2)	-0.0119	-0.0270	C(12)	0.0030	-0.0159 (-0.0078)
C(3)	-0.0342	-0.0023	C(13)	0.0210	-0.0015 (-0.0064)
C(4)	0.0897	(0.0864)	C(14)	-0.0434	0.0211 (-0.0427)
C(5)	-0.0834	(-0.1679)	C(15)	0.2974	(0.3360)
			C(15A)	-0.2018	(-0.2889)
O(1)	(0.1730)	0.0304	O(11)	(-0.0444)	(0.0490) 0.0085
O(2)	(0.0556)	(0.0539)	O(12)	(0.0355)	(-0.0493) (-0.0073)
O(3)	(-0.0823)	0.0206	O(13)	(0.0747)	(-0.0160) 0.0105

hydrogen bonds from two hydroxyl groups of two different neighboring molecules. In the TMRA structure, the carbonyl group from molecule 1 accepts hydrogen bonds from two hydroxyl groups of one of its neighbors while the carbonyl group of molecule 2 only accepts one hydrogen bond from the hydroxyl group of its neighboring molecule (Fig. 2). In addition, the hydroxyl groups in RA only act as hydrogen-bond donors while one of the hydroxyl groups in molecule 2 in TMRA acts as both a hydrogen-bond donor and acceptor.

Acta Cryst. (1990). **C46**, 1674–1676

Structure of a 5,6-Lactone*

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(Received 21 July 1989; accepted 17 October 1989)

Abstract. 6,6-Dimethyl-7-vinyl-8-oxatricyclo[5.2.2.0^{1,5}]undec-4-ene-9-one, C₁₄H₁₈O₂, $M_r = 216.30$, orthorhombic, $Pna2_1$, $a = 13.114$ (6), $b = 8.244$ (7), $c = 11.148$ (6) Å, $V = 1205$ (2) Å³, $Z = 4$, $D_m = 1.19$ (2) (floatation), $D_x = 1.189$ g cm⁻³, Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 5.889$ cm⁻¹, $F(000) = 472$, $T = 298$ K, final $R = 0.051$, $wR = 0.063$ for 943 reflections with $I > 3\sigma(I)$. The lactone ring exists in the boat conformation and the molecules in the crystal are stabilized by van der Waals interactions. Bond lengths and angles are similar to those observed in other lactones.

Introduction. Lactones like coumarins, xanthenes and flavones are found to occur in several plants and

This work was supported by US Public Health Service Grant GM 32134 from the National Institute of General Medical Sciences. We are grateful to Dr Lloyd Taylor of Polaroid Corporation for providing TMRA and for valuable discussions.

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fungi and they are found to possess antitumour activity (Bowman & Rand, 1980). The present paper reports the crystal structure of a lactone synthesized at the department of Organic Chemistry, University of Madras (Ganesh Raj, Janardhanam & Rajagopalan, 1988).

Experimental. D_m by floatation, white needles from benzene, $0.40 \times 0.30 \times 0.20$ mm, Nonius CAD-4 graphite-monochromated single-crystal diffractometer, $\omega/2\theta$ scan, Cu $K\alpha$ radiation, cell parameters refined from 25 accurately centered reflections, $30 \leq 2\theta \leq 60^\circ$, Lp correction, θ -dependent absorption correction using *DIFABS* from the *SDP* package (Frenz, 1978) (max., min. transmission 0.98, 0.90), 1080 unique reflections with $2\theta \leq 120^\circ$ and $0 \leq h \leq 14$, $0 \leq k \leq 9$, $0 \leq l \leq 12$, 943 reflections with $I > 3\sigma(I)$; three standard reflections monitored

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Table 1. *Positional parameters of non-H atoms with e.s.d.'s in parentheses*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3)]$.

	x	y	z	B(Å ²)
C1	0.3473 (5)	-0.0765 (6)	0.4494 (6)	4.5 (1)
C2	0.2543 (6)	-0.1862 (7)	0.4733 (9)	7.0 (2)
C3	0.2015 (7)	-0.1206 (8)	0.5842 (8)	7.3 (2)
C4	0.2546 (6)	0.0403 (8)	0.6055 (7)	5.2 (2)
C5	0.3309 (5)	0.0653 (6)	0.5331 (6)	3.8 (1)
C6	0.4058 (4)	0.2038 (6)	0.5194 (6)	3.7 (1)
C7	0.4826 (4)	0.1458 (7)	0.4222 (6)	3.8 (1)
O8	0.4228 (3)	0.1103 (4)	0.314	4.1 (1)
C9	0.3545 (5)	-0.0066 (6)	0.3234 (6)	4.1 (1)
O10	0.3033 (4)	-0.0456 (5)	0.2376 (5)	6.0 (1)
C11	0.4521 (6)	-0.1478 (7)	0.4748 (7)	5.8 (2)
C12	0.5334 (5)	-0.0158 (7)	0.4562 (6)	4.9 (1)
C13	0.5616 (6)	0.2687 (8)	0.3925 (7)	5.5 (2)
C14	0.5649 (7)	0.3510 (9)	0.2944 (9)	7.6 (2)
C15	0.3487 (5)	0.3573 (6)	0.4776 (7)	4.7 (1)
C16	0.4595 (6)	0.2388 (9)	0.6383 (8)	6.1 (2)

Table 2. *Bond distances (Å) and angles (°) with e.s.d.'s in parentheses*

C1—C2	1.541 (9)	C6—C15	1.542 (8)
C1—C5	1.511 (8)	C6—C11	1.530 (10)
C1—C9	1.522 (9)	C7—O8	1.470 (6)
C1—C11	1.522 (9)	C7—C12	1.537 (8)
C2—C3	1.517 (10)	C7—C13	1.487 (9)
C3—C4	1.517 (9)	O8—C9	1.320 (7)
C4—C5	1.302 (9)	C9—O10	1.212 (8)
C5—C6	1.514 (8)	C11—C12	1.538 (9)
C6—C7	1.554 (8)	C13—C14	1.288 (10)
C2—C1—C5	103.6 (6)	C7—C6—C15	110.9 (5)
C2—C1—C11	117.1 (5)	C7—C6—C16	111.4 (5)
C5—C1—C9	106.6 (4)	C15—C6—C16	109.3 (5)
C5—C1—C11	108.2 (5)	C6—C7—O8	106.8 (4)
C2—C1—C9	115.5 (6)	C6—C7—C12	112.1 (5)
C11—C1—C9	105.2 (6)	C6—C7—C13	113.4 (5)
C1—C2—C3	107.0 (6)	O8—C7—C12	105.2 (5)
C2—C3—C4	103.3 (6)	O8—C7—C13	108.9 (5)
C3—C4—C5	113.2 (6)	C12—C7—C13	110.1 (5)
C4—C5—C6	132.9 (6)	C7—O8—C9	116.2 (4)
C7—C13—C14	124.8 (7)	O8—C9—O10	120.4 (5)
C1—C5—C6	115.4 (5)	O8—C9—C1	113.1 (5)
C1—C5—C4	111.7 (5)	O10—C9—C1	126.4 (4)
C5—C6—C7	105.0 (4)	C1—C11—C12	109.1 (5)
C5—C6—C15	109.5 (5)	C7—C12—C11	110.3 (3)
C5—C6—C16	110.7 (5)		

every 100 reflections showed the change in the intensity to be less than 3%; structure solution by direct methods; five out of 18 hydrogen positions from ΔF map; full-matrix least-squares refinement on F , anisotropic temperature factors for all non-H atoms and isotropic for H atoms, final R factor 0.051, $wR = 0.063$ where $w = 1/[\sigma^2(F_o) + 0.04F_o^2]$, $S = 2.045$, final ΔF map featureless, $(\Delta/\sigma)_{\max} = 0.20$, final excursions $\pm 0.3 \text{ e } \text{Å}^{-3}$, no correction for secondary extinction, all calculations performed using the *SDP* package (Frenz, 1978) on Vax 11/730 computing system. Scattering factors were those incorporated in the *SDP* package.

Discussion. Final positional parameters of all non-H atoms are given in Table 1.* Bond lengths and bond angles are given in Table 2. A stereo diagram of the molecule (*PLUTO*; Motherwell & Clegg, 1978) is given in Fig. 1.

The values of bond lengths and angles are similar to those observed in other lactones (Cheng, Koo, Mellor, Nyburg & Young, 1970; Svinning & Sorum, 1979). The cyclopentene ring is in the envelope conformation (Bixon & Lifson, 1967) with atom C2 0.173 (3) Å from the least-squares plane ($\chi^2 = 4$) through the rest of the atoms. The cyclohexane ring C1—C5—C6—C7—C12—C11 exists in the boat conformation (Bixon & Lifson, 1967) and the atoms C1 and C7 deviate by 0.714 (7) and 0.694 (6) Å, respectively, from the least-squares plane ($\chi^2 = 3$) through the rest of the atoms. The lactone rings O8—C9—C1—C5—C6—C7 (ring I) and O8—C9—C1—C11—C12—C7 (ring II) exist in boat conformations. The asymmetry in bond lengths of O8—C9 = 1.320 (7) and O8—C7 = 1.470 (6) Å is observed in

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52747 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

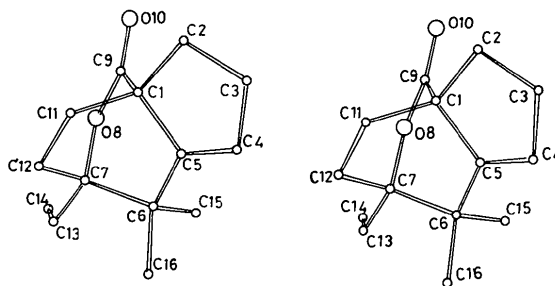
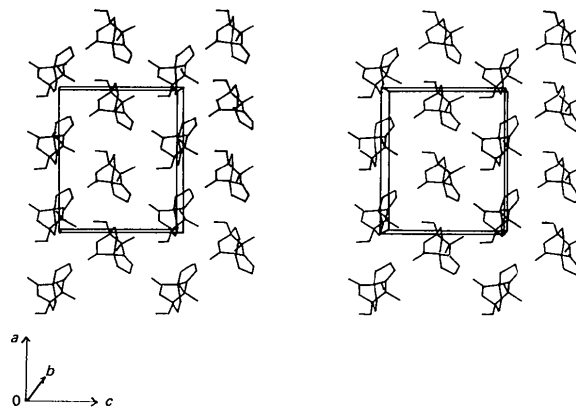


Fig. 1. Stereo diagram of the molecule.

Fig. 2. Stereo packing diagram of the contents of the unit cell viewed down b .

all lactones (Ruzic-Toros & Leban, 1978; Cheng, Koo, Mellor, Nyburg & Young, 1970; Svinning & Sorum, 1979; Berman, Carrell & Glusker, 1973). The cyclopentene ring makes an angle of 33.8 (4)° with the mean plane of the cyclohexane ring, and angles of 24.3 (5) and 92.7 (2)°, respectively, with the mean planes of the lactone rings I and II. The cyclohexane ring is fused to the lactone rings I and II making angles of 58.1 (2) and 58.9 (2)°, respectively.

Such bicyclic systems fused to each other impose considerable strain on the molecule (Sasaki & Hirata, 1973; Bucourt & Hainaut, 1965) and this contributes to the activity of the compound (Bowman & Rand, 1980). The stereo packing diagram down **b** is shown in Fig. 2. The molecules are stabilized by van der Waals forces.

We thank Professors R. Srinivasan and K. Rajagopalan for discussions. One of the authors (VG) thanks the CSIR for financial assistance.

Acta Cryst. (1990). **C46**, 1676–1678

Structure of Fluorescein Dipropionate Acetone Solvate

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(Received 25 August 1988; accepted 6 November 1989)

Abstract. 3-Oxospiro[isobenzofuran-1(3*H*),9'-[9*H*]-xanthene]-3',6'-diyl dipropionate-acetone (2/1), C₂₆H₂₀O_{7.5}, *M_r* = 473.38, monoclinic, *P*2/*c*, *a* = 14.917 (3), *b* = 11.964 (2), *c* = 13.633 (3) Å, β = 101.98 (5)°, *V* = 2380.0 Å³, *Z* = 4, *D_x* = 1.321 g cm⁻³, λ(Cu Kα) = 1.5418 Å, μ = 7.93 cm⁻¹, *T* = 298 K, *F*(000) = 992, *R* = 0.0556 for 2587 unique observed reflections. The title compound was prepared by esterification of fluorescein and on recrystallization from acetone it formed a crystalline inclusion compound with the solvent in the molar ratio 2:1.

Introduction. Fluorescein esters are highly sensitive agents for living biosubstances and much attention has been devoted to them recently. They can penetrate the cell membrane and can be hydrolyzed

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by the lipolase therein (Rotman & Papermaster, 1966), producing the fluorescent substance fluorescein and giving bright yellow-green fluorescence under fluorescent microscopy. Because of the relationship between cell vitality and lipolase content, the living state of the cell may be determined by monitoring variations in fluorescent strength as the lipolase hydrolyzes fluorescein esters within the cell (Wang, Wang, Wang & Ni, 1984). In order to understand the mechanism of fluorescence in different esters, it was thought useful to study their molecular structures. In this paper we present the structure of fluorescein dipropionate.

Experimental. Fluorescein dipropionate (FDP), m.p. 439 K, was prepared from propionic acid anhydride and fluorescein; the results of elemental analysis