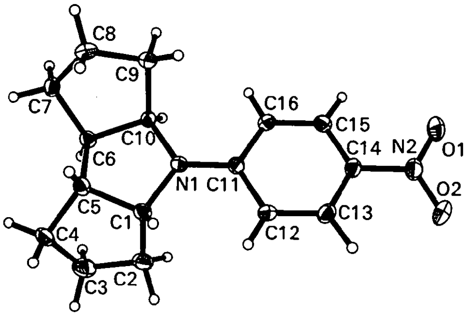
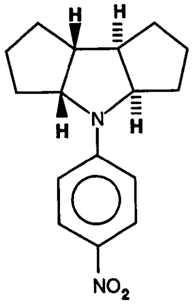


Fig. 1. Molecular structure of (1), with 30% probability ellipsoids, showing the atom-numbering scheme.

as a chiral analog of nitroaniline as a possible candidate for non-linear optical, second-harmonic generation (Chemla & Zyss, 1987).



(1)

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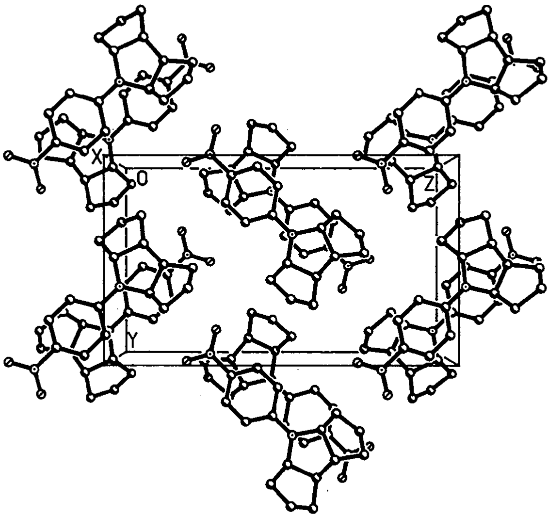

Fig. 2. Packing diagram of (1).

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Structure of 6-Hydroxymethyl-6,9-dimethylbicyclo[6.3.0]undeca-2,4-dien-1-ol

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Abstract. $C_{14}H_{22}O_2$, $M_r = 222{\cdot}33$, orthorhombic, $Pbca$, $a = 10{\cdot}207\,(3)$, $b = 9{\cdot}746\,(6)$, $c = 25{\cdot}533\,(5)$ Å, $V = 2540\,(3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}163$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}071$ mm^{-1}, $F(000) = 976$, $T = 293\,(1)$ K, $R = 0{\cdot}050$ for 1637 unique reflections with $I > 3\sigma(I)$. The molecular dimensions involving the eight-membered ring are normal while C(10) in the five-membered ring is disordered over two sites. The structure is stabilized by a short intermolecular distance O(2)···H(O1) of 1·94 Å. There is a short intramolecular contact O(1)···H(O2) of 2·00 Å.

Table 1. *Final fractional coordinates and equivalent isotropic themal parameters* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*	Occupancy
O1	−0·1406 (1)	0·3642 (2)	0·1270 (1)	0·052 (1)	
O2	−0·2318 (1)	0·1037 (1)	0·0955 (1)	0·055 (1)	
C1	−0·0009 (2)	0·3861 (2)	0·1217 (1)	0·042 (1)	
C2	0·0338 (2)	0·3965 (2)	0·0650 (1)	0·052 (1)	
C3	0·1074 (2)	0·3181 (3)	0·0342 (1)	0·058 (1)	
C4	0·1716 (2)	0·1864 (3)	0·0437 (1)	0·057 (1)	
C5	0·1316 (2)	0·0761 (2)	0·0697 (1)	0·052 (1)	
C6	0·0068 (2)	0·0532 (2)	0·1004 (1)	0·043 (1)	
C7	0·0137 (2)	0·1312 (2)	0·1528 (1)	0·042 (1)	
C8	0·0690 (2)	0·2761 (2)	0·1540 (1)	0·038 (1)	
C9	0·0723 (2)	0·3328 (3)	0·2108 (1)	0·055 (1)	
C10*a*	0·0273 (5)	0·4809 (4)	0·2070 (2)	0·072 (2)	0·60 (1)
C10*b*	0·1108 (6)	0·4901 (6)	0·1983 (3)	0·072 (3)	0·40 (1)
C11	0·0367 (3)	0·5200 (2)	0·1503 (1)	0·063 (1)	
C12	0·1898 (3)	0·2932 (4)	0·2410 (1)	0·105 (2)	
C13	−0·1124 (2)	0·0937 (2)	0·0668 (1)	0·049 (1)	
C14	−0·0062 (3)	−0·1000 (2)	0·1134 (1)	0·068 (2)	

* $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

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

O1—C1	1·448 (2)	C6—C13	1·540 (4)
O2—C13	1·425 (3)	C6—C14	1·535 (4)
C1—C2	1·494 (3)	C7—C8	1·521 (3)
C1—C8	1·530 (3)	C8—C9	1·551 (3)
C1—C11	1·545 (3)	C9—C10*a*	1·517 (5)
C2—C3	1·328 (3)	C9—C12	1·477 (5)
C3—C4	1·462 (4)	C10*a*—C10*b*	0·885 (8)
C4—C5	1·328 (3)	C10*a*—C11	1·500 (5)
C5—C6	1·512 (4)	C10*b*—C11	1·468 (8)
C6—C7	1·540 (4)	C9—C10*b*	1·610 (7)
O1—C1—C2	109·5 (2)	C6—C7—C8	119·5 (2)
O1—C1—C8	107·8 (2)	C1—C8—C7	117·8 (2)
O1—C1—C11	108·9 (2)	C1—C8—C9	105·3 (2)
C2—C1—C8	117·4 (2)	C7—C8—C9	111·1 (2)
C2—C1—C11	110·1 (2)	C8—C9—C10*a*	105·9 (2)
C8—C1—C11	102·8 (2)	C8—C9—C10*b*	99·2 (4)
C1—C2—C3	131·9 (3)	C8—C9—C12	114·3 (3)
C2—C3—C4	131·5 (2)		
C3—C4—C5	131·0 (2)	C10*a*—C9—C12	121·8 (3)
C4—C5—C6	129·6 (3)	C10*b*—C9—C12	98·9 (4)
C5—C6—C7	109·9 (2)	C9—C10*a*—C11	106·4 (3)
C5—C6—C13	109·8 (2)	C9—C10*b*—C11	103·2 (4)
C5—C6—C14	109·2 (2)	C1—C11—C10*a*	103·2 (2)
C7—C6—C13	113·2 (2)	C1—C11—C10*b*	110·8 (3)
C7—C6—C14	107·2 (3)		
C13—C6—C14	107·6 (2)	O2—C13—C6	114·0 (2)

Experimental. The preparation of the title compound is reported elsewhere (Feldman, Wu & Rotella, 1989). Colorless crystals were obtained by vapor-diffusion recrystallization from benzene–hexane. An elongated prism of approximate size 0·50 × 0·54 × 0·70 mm was used for data collection. Accurate cell dimensions and a crystal orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by least-squares refinement of the setting angles of 25 reflections in the θ range 10–15°. Intensity data were collected by the $\omega/2\theta$ scan method with an ω-scan width of $(0·70 + 0·35\tan\theta)°$ and a variable scan speed of 1·0–3·5° min^{-1} using graphite-monochromatized Mo $K\alpha$ radiation with indices h 0 to 12, k 0 to 11 and l −30 to 0 in the θ range 2–25°. The intensities of three standard reflections measured every 2 h showed no evidence of crystal decay. Intensities of 2230 reflections were measured of which 1637 had $I > 3\sigma(I)$, and were used in the structure solution and refinement. Data were corrected for Lorentz and polarization factors; absorption was ignored.

The structure was solved by direct methods using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement of the structure was by full-matrix least-squares calculations. C(10) was found to be disordered over two sites with occupancy factors of 0·60 (1) and 0·40 (1) which were determined through refinements in the initial cycles. At an intermediate stage in the refinement, a difference map revealed all H atoms except those for the disordered C atoms. H atoms were included in the subsequent cycles of refinement; C(10) H atoms were fixed over two sites at geometrically idealized positions (C—H 0·95 Å). The non-H atoms were refined anisotropically and an overall isotropic temperature factor was allowed for the H atoms. Refinement converged with $R = 0·050$ and $wR = (\sum w\Delta^2/\sum F_o^2)^{1/2} = 0·085$. Max. shift/e.s.d. was less than 0·1, $S = 1·612$, $w = 1/[\sigma^2(F_o) + 0·10 \times (F_o)^2]$; min. and max. electron density in the final difference map −0·35 and 0·41 e Å^{-3}, respectively. Scattering factors were those of Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). All computer programs used were part of the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985), and the figures were plotted using *ORTEP*II (Johnson, 1976).

The final fractional coordinates are given in Table 1* and bond distances and angles for non-H atoms are in Table 2. The molecular structure with the crystallographic numbering scheme is depicted in Fig. 1.

Related literature. Structures containing the bicyclo[6.3.0]undeca-2,4-diene moiety have been reported by Zimmerman & Bunce (1982), Feldman,

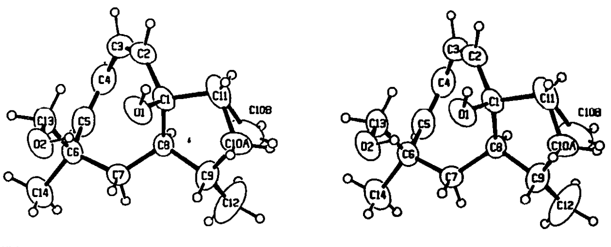

Fig. 1. Stereoview of the molecular structure of the title compound showing the atomic numbering scheme.

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* Lists of structure amplitudes, anisotropic temperature factors, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52160 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Tetracyclic Diketone

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Abstract. Ethyl 3,12-dioxotetracyclo[6.4.0.$0^{2,6}$.$0^{5,9}$]dodec-10-ene-11-carboxylate (2), $C_{15}H_{16}O_4$, M_r = 260·29, monoclinic, $A2/n$, a = 11·602 (1), b = 6·922 (1), c = 33·465 (3) Å, β = 106·89 (1)°, V = 2571·3 (4) Å^3, Z = 8, D_x = 1·345 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·91 cm^{-1}, $F(000)$ = 1104, T = 295 K, R = 0·0528 for 1351 reflections. Compound (2) consists of a norbornane moiety with a five-membered ring and a six-membered ring fused along each side. The five-membered ring contains a ketone functionality and is in a half-chair conformation while the cyclohexenone ring exhibits a 1,2-diplanar conformation. The two end bonds of the norbornane moiety, 1·580 (5) and 1·569 (4) Å, are significantly longer than other bonds in the structure. The two ketone groups and attached atoms are each planar (0·004 Å r.m.s.d.) and form an interplanar angle of 46·5 (4)°. The ester side chain is disordered.

Experimental. Studies of Lewis acid-promoted reactions of substituted pentacyclo[5.4.0.$0^{2,6}$.$0^{3,10}$.$0^{5,9}$]undecane-8,11-diones (PCUD-8,11-diones) with ethyl diazoacetate (EDA) have been reported recently (Marchand, Arney, Gilardi & Flippen-Anderson, 1987; Marchand, Annapurna, Reddy, Watson & Nagl, 1989). In one reaction a substituted tetracyclododecenedione, (2), was isolated in 17% yield (along with several other products) from the reaction of PCUD-8,11-dione (1) with EDA (1·3 equivalents) in the presence of boron trifluoride etherate (Scheme 1). A colorless crystal of (2) with dimensions 0·28 × 0·40 × 0·45 mm was mounted on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer; data collected in the ω mode ($3 \le 2\theta \le 45°$), variable scan rate of 4 to 29·3° min^{-1} using graphite monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($25{\cdot}25 \le 2\theta \le 28{\cdot}77°$), systematic extinctions (hkl, $k + l = 2n + 1$; $h0l$, $h + l = 2n + 1$) and statistics consistent with space group $A2/n$ (permutation of $C2/c$, *International Tables for Crystallography*, 1983); monitored reflections ($\bar{1}33$ and $0,0,\overline{18}$) showed variations of less than ±2%; 2405 reflections measured ($h = -12$, 11; k = 0, 7; l = 0, 36), 1687 unique ($R_{\rm int}$ = 0·009) and 1351 $\ge 3\sigma(I)$; Lorentz–polarization corrections and ψ-scan-based empirical absorption correction (transmission factors 0·711–0·990) applied; structure solved by direct-methods, block cascade (Nicolet Instrument Corporation, 1986) least-squares refinement, H atoms located in difference map, the side chain is disordered and two positions can be found for O(4) and C(14) [0·57 (1) and 0·43 (1) occu-

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