= 0·0011. In the last cycle $(\Delta/\sigma)_{max} = 0·18$; $\Delta\rho$ from −0·21 to +0·20 e Å^{-3}; final $R = 0·066$, $wR = 0·067$, $S = 1·242$; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with *SHELXTL* system of programs.

Discussion. A perspective drawing of the molecule (1) is shown in Fig. 1. Table 1 gives the final atomic parameters.* The bond lengths for non-H atoms are listed in Table 2.

The molecule consists of three six-membered rings and one five-membered ring, all *trans* fused. Rings *A* and *C* adopt distorted chair and twist-chair conformations, respectively, and ring *B* has a 1,2-diplanar conformation. The methyl substituents at C(10), C(13) and C(14) are axial.

The five-membered *D* ring has Δ and φ_m values of 5·2 (6) and −46·5 (6)°, respectively, indicating a half-chair conformation. (Altona, Geise & Romers, 1968).

The stereochemistry of (1) is as follows: C(5)-αH is *trans* to C(10)-βCH₃; C(9)-αH is *trans* to C(10)-βCH₃; C(13)-αCH₃ is *cis* to C(17)-α(side chain) and *trans* to C(14)-βCH₃. Bond lengths and angles are normal.

The molecular arrangement, consisting of layers of molecules perpendicular to **a**, is shown in Fig. 2. Owing to the stretched form adopted by the ring system and the extended conformation of the side chain at C(17),

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates, bond angles and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43673 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
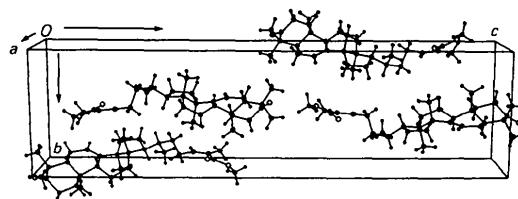

Fig. 2. A perspective drawing of the contents of one unit cell.

the molecule has an elongated form. The molecules in the crystal are packed at normal van der Waals distances. No unusually short intermolecular contacts occur.

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Structure of Fulvoplumierin at 138 K

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Abstract. (*E,E*)-7-(2-Butenylidene)-1,7-dihydro-1-oxo-cyclopenta[*c*]pyran-4-carboxylic acid methyl ester, $C_{14}H_{12}O_4$, $M_r = 244·2$, triclinic, $P\bar{1}$, $a = 4·812$ (1), $b = 9·929$ (3), $c = 12·492$ (4) Å, $\alpha = 93·77$ (3), $\beta = 98·78$ (3), $\gamma = 96·66$ (3)°, $V = 583·8$ Å³, $Z = 2$, $D_x = 1·389$ g cm^{-3} at 138 K, $F(000) = 256$, μ(Mo $K\alpha$) = 0·6 cm^{-1}. The structure was determined by direct methods from 2395 diffractometer data measured with Mo $K\alpha$ (graphite monochromator; $\lambda = 0·71069$ Å) and refined to a final $R = 0·034$ for 1942 observed reflections. The entire molecule is essentially planar. The r.m.s. deviation from the mean plane through all the non-hydrogen atoms of the molecule is 0·037 Å. The fulvene bond distances have alternate double- and

single-bond values reflecting a non-aromatic cyclo-pentadiene-type structure. The alkene chain is all *trans*.

Introduction. Fulvoplumierin (I), an antibacterial pigment from the bark of *Plumiera acutifolia* and *Plumiera rubra* var., was first isolated by Grumbach, Schmid & Bencze (1952). Its structure was established by chemical methods (Schmid & Bencze, 1953; Albers-Schönberg, Phillipsborn, Jackman & Schmid, 1962) and subsequently a chemical synthesis of the compound was reported by Buchi & Carlson (1969). Recently Sanduja, Weinheimer, Euler & Alam (1985) reported the presence of fulvoplumierin in the marine mollusk *Nerita albicilla*. We report here the crystal structure of fulvoplumierin at 138 K.

(I)
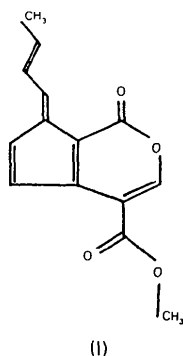
Experimental. Yellow needle-shaped crystals of the compound, grown from benzene, were kindly supplied by Dr M. Alam of the University of Houston. All X-ray measurements on an Enraf–Nonius CAD-4 diffractometer fitted with a low-temperature device, intensity statistics and subsequent structure refinement confirmed the triclinic space group $P\bar{1}$; crystal dimensions $0.08 \times 0.10 \times 0.65$ mm; cell parameters from $\pm 2\theta$ of 40 reflections $(18 < 2\theta < 27^\circ)$ measured at 138 K with Mo $K\alpha_1$ radiation; all unique reflections with $2\theta < 53^\circ$ measured at 138 (2) K with Mo $K\alpha$ (graphite monochromator), 2395 total reflections, $-6 < h < 6$, $-12 < k < 12$, $0 < l < 15$; 1942 observed reflections on the basis $I > 2\sigma(I)$; θ–2θ scan technique with variable scan width of $(0.80 + 0.20\tan\theta)^\circ$, three standard reflections monitored every 2 h of X-ray exposure showed maximum variation of 3%; Lorentz and polarization correction, but no absorption or extinction corrections; attempt to solve the structure by direct-methods program *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1976) failed, finally solved by using the direct-methods program *MITHRIL* (Gilmore, 1983); refined on F by a full-matrix least-squares routine, H atoms from a difference Fourier map and refined isotropically, the refinement converged to a final $R = 0.034$, $wR = 0.043$ for 1942 observed reflections, $w = 1/\sigma^2(F)$, $S = 1.5$ for 211 variables; $(\Delta/\sigma)_{max} = 0.01$, maximum peak in the final difference map

$+0.2$ e Å^{-3}. All Fourier and least-squares calculations with the use of *SHELX*76 (Sheldrick, 1976). Atomic scattering factors those of *SHELX*.

Table 1. *Final atomic parameters with e.s.d.'s for the last digit within parentheses*

$U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$; for hydrogen, $U_{eq} = B/8\pi^2$.

|	x	y	z	U_{eq}(Å^2)
C(1)	0.4872 (3)	0.5557 (1)	0.1359 (1)	0.0202 (4)
O(2)	0.2404 (2)	0.48319 (9)	0.07392 (7)	0.0228 (3)
C(3)	0.1394 (3)	0.3548 (1)	0.0899 (1)	0.0219 (4)
C(4)	0.2650 (3)	0.2832 (1)	0.1680 (1)	0.0201 (4)
C(5)	0.5197 (3)	0.3495 (1)	0.2349 (1)	0.0196 (4)
C(6)	0.6269 (3)	0.4795 (1)	0.2167 (1)	0.0193 (4)
C(7)	0.8852 (3)	0.5227 (1)	0.2942 (1)	0.0203 (4)
C(8)	0.9210 (3)	0.4078 (1)	0.3602 (1)	0.0247 (4)
C(9)	0.7067 (3)	0.3062 (1)	0.3255 (1)	0.0242 (4)
O(10)	0.5467 (2)	0.67059 (9)	0.11398 (8)	0.0268 (3)
C(11)	0.1429 (3)	0.1439 (1)	0.1837 (1)	0.0227 (4)
O(12)	0.2413 (2)	0.0774 (1)	0.25410 (9)	0.0362 (4)
O(13)	−0.0889 (2)	0.0983 (1)	0.11122 (1)	0.0329 (4)
C(14)	−0.2110 (4)	−0.0402 (2)	0.1213 (2)	0.0368 (6)
C(15)	1.0539 (3)	0.6437 (1)	0.3018 (1)	0.0213 (4)
C(16)	1.3107 (3)	0.6858 (1)	0.3774 (1)	0.0226 (4)
C(17)	1.4555 (3)	0.8095 (1)	0.3795 (1)	0.0246 (4)
C(18)	1.7216 (3)	0.8626 (2)	0.4542 (1)	0.0323 (5)
H(3)	−0.026 (4)	0.323 (2)	0.041 (1)	0.032 (4)
H(8)	1.081 (4)	0.405 (2)	0.421 (1)	0.034 (4)
H(9)	0.681 (4)	0.217 (2)	0.354 (1)	0.033 (4)
H(14)1	−0.071 (4)	−0.100 (2)	0.109 (1)	0.044 (5)
H(14)2	−0.362 (4)	−0.060 (2)	0.061 (2)	0.053 (6)
H(14)3	−0.280 (4)	−0.047 (2)	0.190 (2)	0.052 (6)
H(15)	0.999 (3)	0.710 (2)	0.254 (1)	0.027 (4)
H(16)	1.378 (3)	0.622 (1)	0.428 (1)	0.026 (4)
H(17)	1.384 (4)	0.871 (2)	0.329 (1)	0.034 (4)
H(18)1	1.698 (4)	0.938 (2)	0.503 (2)	0.062 (6)
H(18)2	1.874 (5)	0.898 (2)	0.413 (2)	0.067 (7)
H(18)3	1.783 (5)	0.797 (2)	0.497 (2)	0.064 (7)
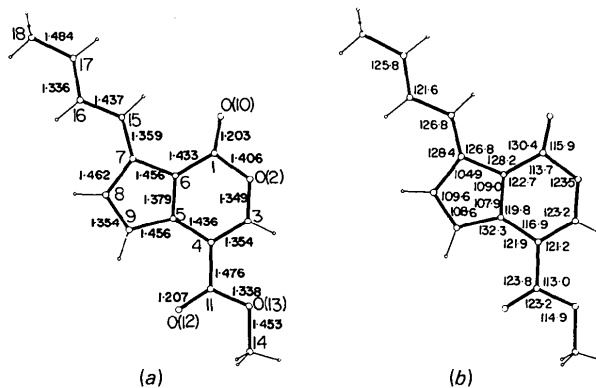
Fig. 1. (a) Bond distances (Å) are shown on a perspective drawing of the molecule. E.s.d. range: 0.001–0.002 Å. Atom-numbering scheme is arbitrary. (b) Bond angles (°). E.s.d.'s for all the angles: 0.1°.
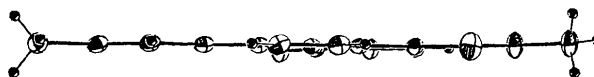

Fig. 2. An *ORTEP* (Johnson, 1965) drawing showing a side view of the molecule of fulvoplumierin.

Discussion. The final atomic parameters are listed in Table 1.* Bond distances and angles are shown in Figs. 1(*a*) and 1(*b*) respectively. The entire molecule of fulvoplumierin is essentially planar as can be seen in a side view in Fig. 2. The r.m.s. deviation of individual atoms from the least-squares plane through all the 18 non-hydrogen atoms in the molecule is 0·037 Å. The methyl ester has the maximum deviation, 0·081 Å. The present structure determination confirms the spectroscopically predicted structure in that the alkene chain is all *trans*.

The five-membered ring is perfectly planar, with the r.m.s. deviation of the ring atoms from the mean plane being 0·001 Å. The bond distances in the fulvene moiety have the characteristics of a non-aromatic butadiene-type structure with alternate double and single $C(sp^2)$–$C(sp^2)$ bond values as seen in pentafulvene (Baron, Brown, Burden, Domaille & Kent, 1972), 6-6 dimethylpentafulvene (Chiang & Bauer, 1970) and octachloropentafulvene (Ammon, Wheeler & Agranat, 1973). The three single bonds $C(5)$–$C(9) = 1·456$ (2), $C(6)$–$C(7) = 1·456$ (2) and $C(7)$–$C(8) = 1·462$ (2) Å are all slightly shorter than the 'pure' $C(sp^2)$–$C(sp^2)$ single bond, 1·485 Å (Dewar & Schmeising, 1960), and the three double bonds $C(5)$–$C(6) = 1·379$ (2), $C(8)$–$C(9) = 1·354$ (2) and $C(7)$–$C(15) = 1·359$ (2) Å are all a little longer than the $C(sp^2)$–$C(sp^2)$ double bond as in ethylene, 1·337 Å (Bartell, Roth, Hollowell, Kuchitsu & Young, 1965). $C(5)$–$C(6)$, being the bridging bond, is the longest. The fusion of the fulvene system with the lactone ring seems to have no additional effect on the bond distances.

In the unsaturated δ-lactone ring, the two C—O distances $C(1)$–$O(2)$ [1·406 (2)] and $C(3)$–$O(2)$ [1·349 (1) Å] are, as expected, significantly different from each other. Other distances and angles in the pyran ring agree well with those observed in related structures (Charles, Griffith, Murtiashaw, Burke & Amma, 1982; Dall'acqua, Benetollo & Bombieri, 1981). The C—H distances range between 0·92 (2) and 1·00 Å.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43643 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Despite the planarity of the molecule there is very little overlap of the rings in the crystal structure of fulvoplumierin. The packing is entirely due to van der Waals interactions. However, there are a few short non-bonded C···O and O···O contacts: $C(1)$···$O(2)$ $(1-x, 1-y, -z)$ [2·957 (2)], $O(2)$···$O(2)$ $(-x, 1-y, -z)$ [2·979 (2)], $O(2)$···$O(10)$ $(1-x, 1-y, -z)$ [2·982 (2)] and $C(3)$···$C(10)$ $(1-x, 1-y, -z)$ [2·972 (2) Å]. Atom $O(12)$ has a short contact with $H(17)$, $O(12)$···$H(17)$ $(x-1, y-1, z)$ [2·36 (2) Å].

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