but high and the observed value for (1) being statistically equivalent but low. Although some modification of MM parameters might be needed better to approximate this distance, nevertheless the calculated values are useful in analyzing observed bond-length variations.

The three hydroxyl groups participate in hydrogen bonding. In addition to the intramolecular hydrogen bond between O(9) and O(11) there are intermolecular hydrogen bonds involving O(11) and O(17): O(17)···O(9)($1-x$, $\frac{1}{2}+y$, $1-z$) = 2·818 (4) Å, H(O17)···O(9) = 1·71 (4) Å, O(17)–H(O17)···O(9) = 177·5 (8)°; O(11)···O(17)($1-x$, $\frac{1}{2}+y$, $1-z$) = 2·801 (5) Å, H(O11)···O(17) = 1·75 (4) Å, O(11)–H(O11)···O(17) = 171·5 (7)°.

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Structure of 1,1a,4,5,6,8,9,9a-Octahydro-3-methoxy-1,1,2-trimethyl-(1a*S*-*trans*)-7*H*-cyclopropa[3,4]cyclohept[1,2-*e*]inden-7-one

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Abstract. $C_{19}H_{24}O_2$, $M_r = 284{\cdot}40$, monoclinic, $P2_1$, $a = 15{\cdot}522$ (3), $b = 5{\cdot}196$ (1), $c = 10{\cdot}332$ (1) Å, $\beta = 107{\cdot}67$ (1)°, $V = 793{\cdot}9$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}19$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5{\cdot}54$ cm^{-1}, $F(000) = 308$, $T = 297$ K, final $R = 0{\cdot}043$ for 1548 unique observed reflections. Of interest in this structure is the novel *trans* ring junction between the three- and seven-membered rings. This stereochemical arrangement causes the carbonyl moiety and the cycloheptane ring to be twisted out of the plane of the aromatic ring. The cyclopropane is also twisted out of the plane of the aromatic system.

Introduction. The title compound was prepared as part of a program directed toward the total synthesis of jatropholones A and B (Smith, Liverton, Hrib, Sivaramakrishnan & Winzenberg, 1985), two architecturally novel diterpenes isolated from *Jatropha gossypiifolia* L. (Euphorbiaceae), the plant that also yields jatrophone and the hydroxyjatrophones A–C (Smith, 1984). An X-ray crystal determination was undertaken to define unambiguously the structure and relative stereochemistry of this substance. Of particular interest was the stereochemistry of the cyclopropane–cycloheptane ring fusion.

Experimental. Clear colorless crystal (0·30 × 0·20 × 0·15 mm), m.p. 397–398 K, $[\alpha]_D^{20\,°C} = +225°$ (0·052 g/100 ml in $CHCl_3$, 1 dm); Enraf–Nonius CAD-4 dif-

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[*] Camille and Henry Dreyfus Teacher-Scholar, 1978–1983; National Institutes of Health (National Cancer Institute) Career Development Award, 1980–1985.

fractometer with graphite-monochromated Cu $K\alpha$ radiation. Lattice parameters from 22 reflections with $44 \leq 2\theta \leq 65°$. 1780 reflections measured using ω–2θ scan technique within ranges $4 \leq 2\theta \leq 140°$, $-18 \leq h \leq 18$, $0 \leq k \leq 6$, $0 \leq l \leq 12$. Intensities of three standard reflections (401, $\bar{2}13$, 411) recorded every 3000 s of X-ray exposure varied by +0·9%. 1548 unique observed reflections [$I > 3\sigma(I)$], $R_{\text{int}} = 1{\cdot}5\%$. Data corrected for Lorentz and polarization effects, not for absorption. Structure solved by *MULTAN*11/82 (Main *et al.*, 1982) which revealed all 21 non-H atoms. H atoms found from subsequent difference Fourier syntheses. Refinement by full-matrix least squares to minimize $\sum w(|F_c - F_o|^2)|/\sum F_o^2$ led to $R = 0{\cdot}043$ and $wR = 0{\cdot}051$ for 261 variables (anisotropic thermal parameters for non-H atoms, positional parameters for H atoms refined with fixed isotropic thermal parameters of 6·0 Å^2) with $w = 1/\sigma^2(F_o)$ and $S = 2{\cdot}59$. Max. least-squares shift to e.s.d. ratio 0·14 in final refinement cycle. Largest residual electron density in final difference Fourier synthesis 0·17 e Å^{-3}. Atomic scattering factors from Cromer & Waber (1974); anomalous-dispersion terms from Ibers & Hamilton (1964). All computer programs from Enraf–Nonius *SDP* package (Frenz, 1978).*

Table 1. *Refined positional parameters*

	x	*y*	*z*	*B*(Å^2)†
C(1)	0·7759 (1)	0·7016*	0·4256 (2)	3·89 (5)
C(2)	0·7916 (2)	0·5637 (7)	0·5598 (2)	5·26 (6)
C(3)	0·8874 (2)	0·4604 (8)	0·5894 (2)	5·67 (6)
C(4)	0·9030 (1)	0·4276 (6)	0·4504 (2)	4·40 (5)
C(5)	0·8391 (1)	0·6229 (5)	0·3622 (2)	3·47 (4)
C(6)	0·8354 (1)	0·7214 (4)	0·2344 (2)	3·08 (4)
C(7)	0·9046 (1)	0·6301 (5)	0·1684 (2)	3·26 (4)
C(8)	0·9367 (1)	0·7949 (5)	0·0711 (2)	3·58 (4)
C(9)	0·8709 (1)	0·7817 (6)	−0·0761 (2)	3·92 (4)
C(10)	0·7783 (1)	0·7849 (5)	−0·0583 (2)	3·17 (4)
C(11)	0·6964 (1)	0·9581 (5)	−0·1070 (2)	3·39 (4)
C(12)	0·7643 (1)	0·9941 (4)	0·0342 (2)	3·02 (4)
C(13)	0·7665 (1)	0·9019 (4)	0·1723 (2)	3·08 (4)
C(14)	0·7051 (1)	0·9864 (5)	0·2381 (2)	3·53 (4)
C(15)	0·7104 (1)	0·8808 (5)	0·3646 (2)	3·86 (4)
C(16)	0·6377 (1)	1·1954 (6)	0·1775 (2)	4·77 (5)
C(17)	0·6061 (1)	0·8350 (6)	−0·1224 (2)	4·56 (5)
C(18)	0·6952 (2)	1·1625 (6)	−0·2105 (2)	4·75 (6)
C(19)	0·5775 (2)	0·797 (1)	0·4208 (3)	8·07 (9)
O(1)	0·64961 (9)	0·9650 (5)	0·4301 (2)	5·21 (4)
O(2)	0·9376 (1)	0·4159 (4)	0·1967 (2)	4·66 (4)
H(10)	0·758 (1)	0·611 (7)	−0·030 (2)	6
H(12)	0·799 (1)	1·146 (7)	0·033 (2)	6
H(2)	0·747 (1)	0·403 (7)	0·545 (2)	6
H(2′)	0·790 (1)	0·699 (7)	0·636 (2)	6
H(3)	0·897 (2)	0·285 (7)	0·646 (2)	6
H(3′)	0·931 (1)	0·600 (7)	0·642 (2)	6
H(4)	0·885 (1)	0·247 (7)	0·415 (2)	6
H(4′)	0·967 (2)	0·458 (7)	0·455 (2)	6
H(8)	0·999 (1)	0·727 (7)	0·077 (2)	6
H(8′)	0·943 (1)	0·969 (7)	0·102 (2)	6
H(9)	0·881 (1)	0·615 (7)	−0·121 (2)	6
H(9′)	0·884 (1)	0·926 (7)	−0·131 (2)	6
H(16)	0·576 (2)	1·123 (6)	0·147 (3)	6
H(16′)	0·662 (2)	1·272 (7)	0·109 (2)	6
H(16″)	0·628 (1)	1·321 (5)	0·244 (2)	6
H(17)	0·606 (1)	0·700 (7)	−0·051 (3)	6
H(17′)	0·580 (2)	0·743 (7)	−0·218 (2)	6
H(17″)	0·566 (1)	0·962 (7)	−0·116 (2)	6
H(18)	0·670 (2)	1·103 (7)	−0·307 (2)	6
H(18′)	0·754 (1)	1·254 (7)	−0·197 (2)	6
H(18″)	0·654 (1)	1·302 (6)	−0·203 (2)	6
H(19)	0·602 (2)	0·631 (7)	0·473 (2)	6
H(19′)	0·531 (1)	0·895 (6)	0·461 (2)	6
H(19″)	0·551 (1)	0·745 (8)	0·331 (2)	6

* The *y* coordinate of C(1) was not refined.

† Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:

$$\tfrac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha).$$

Discussion. Final positional parameters and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1. An *ORTEP* projection of the molecule appears in Fig. 1 and a stereoview of the unit cell in Fig. 2.

The stereochemistry about C(10)—C(12), where the cyclopropane ring is fused to the cycloheptane ring, is *trans*. This causes the carbonyl moiety of the cycloheptane ring to be twisted out of the plane of the aromatic ring system by 28·7 (10)° and the cyclopropane ring to be twisted out of the plane of the aromatic system by 49·7 (10)°. The absolute stereochemistry depicted derives from the method of synthesis.

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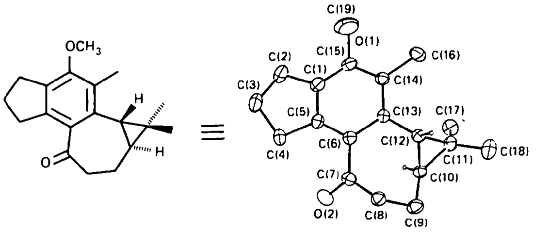

Fig. 1. *ORTEP* (Johnson, 1965) drawing (30% probability thermal ellipsoids) showing atom-numbering scheme. Stereochemically important H atoms are included.

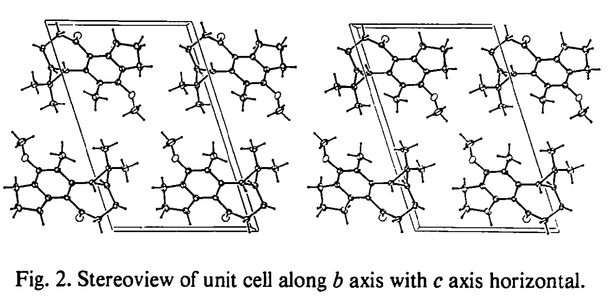

Fig. 2. Stereoview of unit cell along *b* axis with *c* axis horizontal.

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43125 (11 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 3*β*,20*β*-Diacetoxy-11*β*-hydroxymethyl-5α-pregnane: an Unusual Hydrogen-Bonding Scheme*

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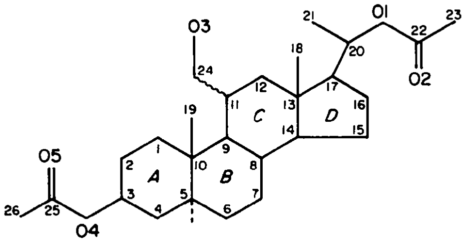
Abstract. $C_{26}H_{42}O_5$, $M_r = 434{\cdot}62$, monoclinic, $P2_1$, $a = 21{\cdot}776\,(5)$, $b = 6{\cdot}119\,(2)$, $c = 19{\cdot}864\,(8)$ Å, $\beta = 111{\cdot}69\,(5)^\circ$, $V = 2459{\cdot}4$ Å^3, $Z = 4$, $D_m = 1{\cdot}178$, $D_x = 1{\cdot}174$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha_1) = 1{\cdot}54056$ Å, $\mu = 0{\cdot}599$ mm^{-1}, $F(000) = 952$, $T = 297$ K, final $R = 0{\cdot}048$ for 3267 observed reflections. The two independent molecules of the asymmetric unit have the same 11*β*-hydroxymethyl configuration and the same absolute configuration. There are differences in conformation of the C(3) and, to a lesser extent, C(17) side chains in the two molecules, resulting from the intermolecular hydrogen bonding and different packing environments. Intermolecular hydrogen bonds O—H···O link the molecules into bundles of three interwound helices of pitch 3*b* along *y*.

Introduction. The title compound (1) and its derivatives are considered to be useful intermediates for the synthesis of products of biological interest. The compound was synthesized from an 11-methylene steroid, 3*β*,20*β*-diacetoxy-11-methylene-5α-pregnane, by oxidative hydroboration (Engel, Lourdusamy, Baroan, Ibrahim & Bończa-Tomasewski, 1984; Engel, Baroan, Ibrahim, Le Van, Lourdusamy & Mukherjee, 1985). It seemed important to determine unequivocally the configuration of the 11-hydroxymethyl substituent thus formed, not only because of the importance of the stereochemistry of 11-substituents on biological activities, but also because the catalytic reduction of the 11-methylene steroid which had afforded compound (1), and its 12-oxo derivative, had led, rather unexpectedly, not to 11*β*-methylated but to 11α-methylated products (Engel *et al.*, 1984).



(1)

Experimental. Crystals in the shape of thin needles were obtained from ether/hexane and their density was measured by flotation in aqueous KI solution. The X-ray measurements were carried out on an Enraf–Nonius CAD-4 diffractometer using Ni-filtered Cu radiation and a crystal 0·03 × 0·10 × 0·87 mm. The cell parameters were derived by a least-squares fit of the θ values for 17 reflections with $26 \le \theta \le 38^\circ$. Intensities up to $\theta = 65^\circ$ for $h = -25$ to 25, $k = 0$ to 7, $l = 0$ to 23, were measured by ω–2θ scans for $\Delta\omega = (1{\cdot}0 + 0{\cdot}14\tan\theta)^\circ$ plus 50% for the backgrounds. Three standard reflections measured every hour of radiation time varied within ±5% of the mean values. Of the 4626 unique reflections scanned, 3267 were observed with $I \ge 2{\cdot}0\sigma(I)$. The net intensities were

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