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The Structure of 24-Methylene-9,19-cyclolanostan-3β-yl Acetate*

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Abstract. $C_{33}H_{54}O_2$, $M_r = 482.7$, monoclinic, $P2_1$, $a = 12.658$ (1), $b = 7.222$ (1), $c = 16.604$ (2) Å, $\beta = 97.52$ (1)°, $V = 1504.8$ Å^3, $Z = 2$, $D_m = 1.06$ (1), $D_x = 1.07$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 0.068$ mm^{-1}, $F(000) = 536$, $T = 293$ K, $R = 0.054$ for 1259 observed reflections. The four *trans*-fused rings A, B, C and D of the triterpene nucleus have chair, half-chair, 1:3 diplanar and half-chair conformations, respectively. Ring B is distorted by the cyclopropane ring fused to it. The molecules are loosely packed in the crystal as is evident from the very low density.

Introduction. Currently, a great deal of interest centres around the latex of the species *Euphorbia* as a possible petroleum substitute. The Himalayan plant *Euphorbia royleana* Boiss contains a large quantity of latex. The triterpene in the present study is obtained from source; it constitutes less than 0.3% of the latex and this precluded its structure determination by chemical methods and therefore X-ray crystal structure analysis was carried out.

Experimental. Compound isolated as its acetate by IDCC (inverted dry column chromatography) technique and crystallized from chloroform–acetone mixture, m.p. 386–387 K and $[\alpha]_D^{25°C} = 53.09°$. Crystal $\sim 0.60 \times 0.20 \times 1.00$ mm; D_m by flotation in NaCl solution; Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta \le 23.5°$, h 0 to 14, k 0 to 8, l −18 to 18. 2668 reflections measured, 1259 significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 20 reflections ($22 \le 2\theta \le 34°$), three standard reflections (020, 400 and 008) every 2000 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on F) of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms), H atoms in calculated positions (but not refined); convergence at $R = 0.054$, $wR = 0.054$, $S = 0.92$. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (3.5 + 1.0|F_o| + 0.014|F_o|^2)^{-1}$. $(\Delta/\sigma)_{max} = 0.1$. Final $\Delta\rho$ excursions $< |0.3|$ eÅ^{-3}. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *LALS* (Gantzel, Sparks & Trueblood, 1961) for refinement.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.‡ Bond lengths and angles are in Table 2. The chemical formula with the atomic numbering is shown in Fig. 1, and a perspective drawing of the molecule in Fig. 2.

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‡ Lists of structure factors, anisotropic thermal parameters, H-atom parameters and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42597 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta).$$

	x	y	z	B_{eq}
O(1)	4798 (4)	5358	8526 (3)	5·98
O(2)	6136 (6)	7328 (13)	8447 (4)	11·95
C(1)	3450 (10)	6162 (19)	6408 (7)	5·08
C(2)	3715 (10)	6394 (19)	7312 (6)	5·48
C(3)	4618 (11)	5168 (19)	7655 (6)	4·73
C(4)	4446 (9)	3089 (19)	7446 (7)	4·76
C(5)	4186 (9)	2999 (16)	6511 (6)	4·08
C(6)	4126 (10)	1038 (18)	6174 (7)	5·69
C(7)	3900 (10)	1123 (20)	5248 (7)	6·21
C(8)	2773 (9)	1815 (17)	4971 (6)	4·12
C(9)	2546 (8)	3694 (15)	5379 (6)	3·76
C(10)	3231 (9)	4162 (16)	6185 (6)	3·79
C(11)	2161 (9)	5289 (18)	4817 (7)	4·62
C(12)	1389 (9)	4825 (18)	4059 (6)	5·90
C(13)	1307 (8)	2723 (16)	3852 (6)	4·56
C(14)	2447 (8)	1888 (17)	4054 (6)	4·72
C(15)	2302 (11)	−62 (19)	3675 (7)	6·66
C(16)	1520 (10)	272 (18)	2886 (7)	6·89
C(17)	1030 (9)	2210 (18)	2938 (6)	5·05
C(18)	512 (9)	1831 (23)	4351 (6)	6·80
C(19)	2102 (7)	3514 (15)	6181 (5)	4·80
C(20)	−152 (8)	2296 (19)	2563 (6)	6·33
C(21)	−646 (10)	4146 (25)	2625 (7)	9·57
C(22)	−253 (8)	1678 (20)	1663 (6)	7·47
C(23)	−1336 (10)	1007 (29)	1322 (7)	11·35
C(24)	−1464 (10)	454 (28)	405 (9)	10·41
C(25)	−1422 (14)	1922 (31)	−205 (8)	12·19
C(26)	−2361 (19)	3177 (35)	−158 (14)	22·75
C(27)	−1471 (15)	1149 (31)	−1042 (9)	13·67
C(28)	5481 (7)	2059 (18)	7731 (6)	6·56
C(29)	3567 (8)	2280 (13)	7896 (6)	6·17
C(30)	3277 (7)	2917 (17)	3622 (5)	5·95
C(31)	−1546 (10)	−1404 (25)	264 (8)	13·08
C(32)	5531 (9)	6531 (15)	8839 (6)	7·57
C(33)	5584 (7)	6724 (14)	9752 (4)	8·93

The four rings of the triterpene nucleus are *trans* fused, similar to cyclograndisolide (Allen & Trotter, 1971). Ring *A* has a chair conformation. The presence of a *cis*-fused cyclopropane ring on *B* and of a *trans*-fused five-membered ring on *C* produces large distortions in rings *B* and *C*. The mean valence angle in ring *B* is 113·9 (9)° but angles C(5)—C(10)—C(9) and C(8)—C(9)—C(10) are 121·6 (9) and 117·1 (9)°, respectively, owing to the strain caused by the fusion of the cyclopropane ring. Ring *B* has a half-chair conformation while ring *C* has a 1:3 diplanar conformation. The five-membered ring *D* has a half-chair conformation.

The torsion angles* for rings *A*, *B* and *C* are in overall agreement with the theoretical values (Bucourt & Hainaut, 1965), and for the five-membered ring *D* with values for $\omega_1 = 15°$ (Hendrickson, 1961).

The C(sp^3)—C(sp^3) distances are in the range 1·50 (2)–1·56 (2) Å. The longer bonds join the more highly substituted atoms, *e.g.* C(13)—C(14) = 1·56 (1), C(17)—C(20) = 1·55 (1) Å, an effect noted in various other terpenoids (Birnbaum & Fergusson, 1969; Oh & Maslen, 1966). The side-chain atoms at C(17) generally have large thermal vibrations (Table 1) and this may be due to the absence of any strong intermolecular

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* See deposition footnote.

interactions, consistent with the observed low density. No evidence of disorder was found.

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

C(1)—C(2)	1·50 (2)	C(1)—C(10)	1·51 (2)
C(2)—C(3)	1·50 (2)	C(3)—C(4)	1·55 (2)
C(3)—O(1)	1·44 (1)	C(4)—C(5)	1·55 (2)
C(4)—C(28)	1·53 (2)	C(4)—C(29)	1·53 (2)
C(5)—C(6)	1·52 (2)	C(5)—C(10)	1·51 (2)
C(6)—C(7)	1·53 (2)	C(7)—C(8)	1·52 (2)
C(8)—C(9)	1·56 (2)	C(8)—C(14)	1·53 (1)
C(9)—C(10)	1·53 (1)	C(9)—C(11)	1·52 (2)
C(9)—C(19)	1·52 (1)	C(10)—C(19)	1·50 (1)
C(11)—C(12)	1·53 (2)	C(12)—C(13)	1·56 (2)
C(13)—C(14)	1·56 (1)	C(13)—C(17)	1·56 (1)
C(13)—C(18)	1·53 (2)	C(14)—C(15)	1·54 (2)
C(14)—C(30)	1·54 (1)	C(15)—C(16)	1·55 (2)
C(16)—C(17)	1·54 (2)	C(17)—C(20)	1·55 (2)
C(20)—C(21)	1·48 (2)	C(20)—C(22)	1·55 (1)
C(22)—C(23)	1·49 (2)	C(23)—C(24)	1·56 (2)
C(24)—C(25)	1·47 (3)	C(24)—C(31)	1·36 (3)
C(25)—C(26)	1·51 (3)	C(25)—C(27)	1·49 (2)
C(32)—C(33)	1·51 (1)	C(32)—O(1)	1·31 (1)
C(32)—O(2)	1·21 (1)		

|			
C(2)—C(1)—C(10)	111 (1)	C(1)—C(2)—C(3)	112 (1)
C(2)—C(3)—C(4)	114 (1)	C(2)—C(3)—O(1)	110 (1)
C(4)—C(3)—O(1)	108·4 (9)	C(3)—C(4)—C(5)	106 (1)
C(3)—C(4)—C(28)	108 (1)	C(3)—C(4)—C(29)	110 (1)
C(5)—C(4)—C(28)	110·6 (9)	C(5)—C(4)—C(29)	113·6 (9)
C(28)—C(4)—C(29)	108·1 (9)	C(4)—C(5)—C(6)	113·7 (9)
C(4)—C(5)—C(10)	113·0 (9)	C(6)—C(5)—C(10)	112·7 (9)
C(5)—C(6)—C(7)	109 (1)	C(6)—C(7)—C(8)	111 (1)
C(7)—C(8)—C(9)	111·9 (9)	C(7)—C(8)—C(14)	116 (1)
C(9)—C(8)—C(14)	111·2 (9)	C(8)—C(9)—C(10)	117·1 (9)
C(8)—C(9)—C(14)	117·0 (9)	C(8)—C(9)—C(19)	114·6 (8)
C(10)—C(9)—C(11)	117·9 (9)	C(10)—C(9)—C(19)	59·0 (7)
C(11)—C(9)—C(19)	118·5 (9)	C(1)—C(10)—C(5)	109·3 (9)
C(1)—C(10)—C(9)	119·4 (9)	C(1)—C(10)—C(19)	116·3 (9)
C(5)—C(10)—C(9)	121·6 (9)	C(5)—C(10)—C(19)	122·9 (9)
C(9)—C(10)—C(19)	59·9 (7)	C(9)—C(11)—C(12)	117 (1)
C(11)—C(12)—C(13)	114·3 (9)	C(12)—C(13)—C(14)	107·3 (9)
C(12)—C(13)—C(17)	116·5 (9)	C(12)—C(13)—C(18)	108·7 (9)
C(14)—C(13)—C(17)	101·6 (8)	C(14)—C(13)—C(18)	112·2 (9)
C(17)—C(13)—C(18)	110·4 (9)	C(8)—C(14)—C(13)	110·5 (9)
C(8)—C(14)—C(15)	112·2 (9)	C(8)—C(14)—C(30)	111·7 (9)
C(13)—C(14)—C(15)	102·0 (9)	C(13)—C(14)—C(30)	112·5 (9)
C(15)—C(14)—C(16)	107·6 (9)	C(14)—C(15)—C(16)	103 (1)
C(15)—C(16)—C(17)	108 (1)	C(13)—C(17)—C(16)	103·3 (9)
C(13)—C(17)—C(20)	117·8 (9)	C(16)—C(17)—C(20)	113 (1)
C(9)—C(19)—C(11)	61·0 (7)	C(17)—C(20)—C(21)	114 (1)
C(17)—C(20)—C(22)	109·3 (9)	C(21)—C(20)—C(22)	110 (1)
C(20)—C(22)—C(23)	114 (1)	C(22)—C(23)—C(24)	115 (1)
C(23)—C(24)—C(25)	119 (1)	C(23)—C(24)—C(31)	115 (1)
C(25)—C(24)—C(31)	127 (2)	C(24)—C(25)—C(26)	107 (2)
C(24)—C(25)—C(27)	112 (2)	C(26)—C(25)—C(27)	110 (2)
O(2)—C(32)—C(33)	123·0 (9)	O(1)—C(32)—C(33)	113·1 (8)
C(3)—O(1)—C(32)	118·0 (8)	O(1)—C(32)—O(2)	124 (1)
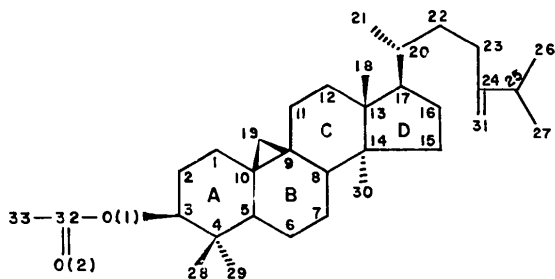
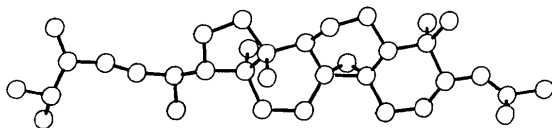Fig. 1. Crystallographic numbering. The numbers refer to C atoms unless otherwise indicated.

Fig. 2. A perspective view of the molecule.

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Structure of Aurmillone, an Isoflavone from *Millettia auriculata**

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Abstract. 5,7-Dihydroxy-8-methoxy-3-{4-[(3-methyl-2-butenyl)oxy]phenyl}-4H-1-benzopyran-4-one, $C_{21}H_{20}O_6$, $M_r = 368\cdot4$, monoclinic, $P2_1/n$, $a = 10\cdot494$ (1), $b = 15\cdot347$ (2), $c = 11\cdot456$ (2) Å, $\beta = 102\cdot86$ (1)°, $Z = 4$, $V = 1798\cdot7$ Å³, $D_m = 1\cdot33$, $D_x = 1\cdot359$ Mg m⁻³, $\lambda(Mo\,K\alpha) = 0\cdot7107$ Å, $\mu(Mo\,K\alpha) = 0\cdot107$ mm⁻¹, $F(000) = 776$, $T = 293$ K, $R = 0\cdot062$ for 1345 observed reflections. The benzopyrone moiety is almost planar with the angle between the rings 2·3 (9)°. The crystal structure is stabilized by a network of hydrogen bonds. There are no unusual bond distances or angles.

Introduction. The plant *Millettia auriculata* has insecticidal and pesticidal properties (Chopra, Nayar & Chopra 1956). A number of isoflavonoids have been isolated from its roots, leaves and stems. The title compound, aurmillone, was isolated from the seeds of *Millettia auriculata* (supplied by B. N. Dwivedi Nainital, India). Needle-shaped crystals were obtained from benzene. Since the melting point (448–449 K) of this compound (1) differed widely from the earlier reported value of 430–431 K (Subbaraju & Srimannarayana, 1978) and was rather close to the melting point reported for isoaurmillone (435–436 K) (Gupta, Bhattacharya, Mitra & Aditya-Chaudhury, 1983), it was decided to establish the structure unequivocally by X-ray diffraction.
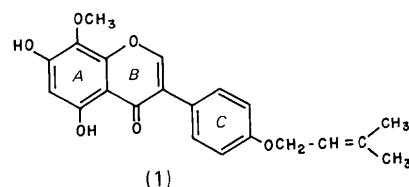
(1)

Experimental. Crystal approx. $0\cdot45 \times 0\cdot40 \times 0\cdot35$ mm. D_m by flotation in aqueous KI solution. Enraf–Nonius CAD-4F/11 M diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from 22 reflections ($6 < \theta < 15°$). $\omega/2\theta$ scan mode, scan speed 1° min⁻¹, $\theta \le 24°$; $h\,0$ to 12, $k\,0$ to 18, $l\,-13$ to 13. 2616 reflections measured, 1345 judged significant ($|F_o| \ge 3\sigma|F_o|$). No correction for absorption. Three standard reflections every 2000 s; 4% variation in intensity. Structure solved by direct methods, program *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement of scale factor, positional and

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