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Structure of a Triterpene Extracted from *Austroplenckia populnea* (Celastraceae), Methyl 3-Hydroxy-2-oxofriedelan-3-en-20α-oate

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Abstract. $C_{31}H_{48}O_4$, M_r = 484·73, monoclinic, $P2_1$, a = 6·697 (2), b = 14·714 (7), c = 13·866 (3) Å, β = 103·53 (2)°, V = 1328 (1) Å^3, Z = 2, D_x = 1·211 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·726 cm^{-1}, F(000) = 532, T = 298 K, R = 0·049 for 1430 observed reflections. Rings B, C, D and E adopt chair conformations and ring A is in a half-boat conformation. The bond angles are as expected within experimental error and the mean sp^2 and sp^3 bond angles are 120·0 (5) and 111·0 (4)°, respectively.

Experimental. The data-collection and refinement parameters are summarized in Table 1.

The crystal structure was solved using direct methods and difference Fourier techniques. In the final cycles of block-matrix least-squares refinement all non-H atoms were refined anisotropically. H atoms were located geometrically, all with the common isotropic temperature factor U = 0·08 Å^2.

Scattering factors for non-H atoms from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965), with corrections for anomalous dispersion from Cromer & Liberman (1970). Programs used: *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick 1976), *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/870 computer at the Instituto de Física e Química de São Carlos.

Table 1. *Experimental details*

(a) Data collection[i,ii]	
Mode	ω–2θ
Scan rate (°min^{-1})	2·88–6·7
θ range (°)	0–25
Range of hkl	$-7 < h < 7$, $0 < k < 16$, $0 < l < 17$
Total reflections measured	2538
Unique reflections	2428
Approx. crystal dimensions (mm)	0·23 × 0·23 × 0·25
(b) Structure refinement[iii]	
Reflections used [$I > 3\sigma(I)$]	1430
No. of variables	318
R, wR	0·049, 0·052
Max. shift/e.s.d.	0·07
Max., min. density in final difference map (e Å^{-3})	0·16, −0·21
S	4·17

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 23 reflections with $4·0 < \theta < 15·24°$. (ii) Enraf–Nonius CAD-4 diffractometer with graphite monochromator. One standard reflection (107) measured every hour showed no significant variation. No absorption correction. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma^2(F_o) + 0·001F_o^2]^{-1}$.

Table 2. *Final positional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
C(1)	−0·0823 (9)	0·7490 (4)	0·6300 (4)	3·5 (2)
C(2)	−0·2791 (9)	0·7946 (4)	0·5774 (4)	3·8 (2)
C(3)	−0·4215 (8)	0·7450 (4)	0·5021 (4)	3·3 (2)
C(4)	−0·3846 (8)	0·6628 (4)	0·4705 (4)	3·5 (2)
C(5)	−0·1809 (8)	0·6138 (4)	0·5138 (4)	2·7 (2)
C(6)	−0·2101 (8)	0·5104 (4)	0·5140 (4)	3·2 (2)
C(7)	−0·0202 (8)	0·4598 (4)	0·5693 (3)	3·1 (2)
C(8)	0·0449 (8)	0·4912 (3)	0·6770 (4)	2·6 (2)
C(9)	0·0973 (8)	0·5949 (3)	0·6833 (4)	2·5 (2)
C(10)	−0·0987 (8)	0·6450 (4)	0·6237 (4)	2·9 (2)
C(11)	0·1356 (8)	0·6217 (4)	0·7935 (4)	3·4 (2)
C(12)	0·2951 (9)	0·5632 (4)	0·8633 (4)	3·2 (2)
C(13)	0·2382 (7)	0·4604 (3)	0·8581 (4)	2·4 (2)
C(14)	0·2073 (8)	0·4284 (3)	0·7462 (4)	2·5 (2)
C(15)	0·1247 (9)	0·3305 (4)	0·7432 (4)	3·8 (2)
C(16)	0·281 (1)	0·2653 (4)	0·8065 (4)	4·2 (2)
C(17)	0·4001 (8)	0·2969 (4)	0·9119 (4)	3·3 (2)
C(18)	0·4214 (8)	0·4047 (3)	0·9201 (4)	2·6 (2)
C(19)	0·5026 (8)	0·4320 (4)	1·0330 (4)	2·9 (2)
C(20)	0·4199 (8)	0·3836 (4)	1·1153 (4)	3·3 (2)
C(21)	0·4024 (9)	0·2807 (4)	1·0967 (4)	4·1 (2)
C(22)	0·2960 (8)	0·2567 (4)	0·9902 (4)	3·4 (2)
C(23)	−0·5314 (9)	0·6206 (5)	0·3834 (4)	4·3 (2)
C(24)	−0·0390 (9)	0·6374 (5)	0·4450 (5)	4·1 (2)
C(25)	0·2894 (8)	0·6229 (4)	0·6466 (5)	3·9 (2)
C(26)	0·4087 (8)	0·4257 (4)	0·7103 (4)	3·6 (2)
C(27)	0·0400 (8)	0·4505 (4)	0·8962 (4)	3·2 (2)
C(28)	0·617 (1)	0·2568 (5)	0·9273 (5)	4·4 (2)
C(29)	0·5823 (9)	0·4040 (6)	1·2147 (4)	4·9 (2)
C(30)	0·217 (1)	0·4215 (5)	1·1298 (4)	3·9 (2)
C(31)	0·055 (1)	0·5542 (5)	1·1671 (6)	6·1 (3)
O(C2)	−0·3160 (7)	0·8744 (3)	0·5959 (3)	5·5 (2)
O(C3)	−0·5977 (6)	0·7919 (3)	0·4658 (3)	5·3 (2)
O(C30)	0·2299 (6)	0·5117 (3)	1·1433 (3)	4·3 (2)
O′(C30)	0·0687 (7)	0·3774 (3)	1·1353 (3)	4·8 (2)

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Table 3. *Bond distances* (Å) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·506 (8)	C(13)—C(14)	1·588 (7)
C(1)—C(10)	1·535 (8)	C(13)—C(18)	1·556 (7)
C(2)—C(3)	1·437 (8)	C(13)—C(27)	1·546 (7)
C(2)—O(C2)	1·239 (8)	C(14)—C(15)	1·540 (8)
C(3)—C(4)	1·329 (9)	C(14)—C(26)	1·544 (8)
C(3)—O(C3)	1·358 (7)	C(15)—C(16)	1·535 (9)
C(4)—C(5)	1·536 (8)	C(16)—C(17)	1·563 (8)
C(4)—C(23)	1·502 (8)	C(17)—C(18)	1·594 (7)
C(5)—C(6)	1·534 (8)	C(17)—C(22)	1·539 (8)
C(5)—C(10)	1·563 (7)	C(17)—C(28)	1·536 (9)
C(5)—C(24)	1·536 (8)	C(18)—C(19)	1·584 (7)
C(6)—C(7)	1·518 (8)	C(19)—C(20)	1·553 (8)
C(7)—C(8)	1·526 (7)	C(20)—C(21)	1·536 (9)
C(8)—C(9)	1·564 (7)	C(20)—C(29)	1·572 (8)
C(8)—C(14)	1·571 (7)	C(20)—C(30)	1·525 (9)
C(9)—C(10)	1·563 (7)	C(21)—C(22)	1·523 (8)
C(9)—C(11)	1·540 (7)	C(30)—O(C30)	1·340 (9)
C(9)—C(25)	1·546 (8)	C(30)—O′(C30)	1·204 (9)
C(11)—C(12)	1·528 (8)	C(31)—O(C30)	1·433 (9)
C(12)—C(13)	1·557 (8)		

Atomic coordinates are listed in Table 2* and bond lengths in Table 3. Fig. 1 is an *ORTEP* perspective drawing of the molecule showing the atom labelling.

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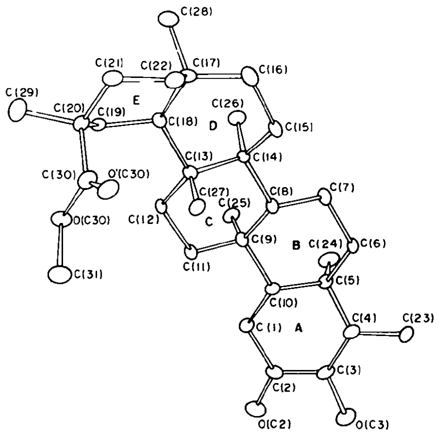

Fig. 1. Perspective view of the molecule showing the atom labelling.

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* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52165 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Methyl 3-Hydroxy-3′,3′-dimethylspiro-[1,2-benzo-1-cyclohexene-4,1′-cyclopropane]-2′-carboxylate*

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Abstract. $C_{16}H_{20}O_3$, $M_r = 260·3$, monoclinic, $P2_1/n$, $a = 10·739\ (1)$, $b = 22·750\ (3)$, $c = 11·631\ (1)$ Å, $\beta = 97·99\ (1)°$, $V = 2814·0\ (4)$ Å^3, $Z = 8$, D_m (flotation in KI solution) $= 1·24$, $D_x = 1·23$ g cm^{-3}, λ(Mo $K\alpha$) $= 0·7107$ Å, $\mu = 0·901$ cm^{-1}, $F(000) = 1120$, room temperature, $R = 0·066$ for 1850 observed reflections. The cyclohexene moiety has a 'half-chair' conformation with the hydroxy O atom in the β conformation. The molecules are held together by bifurcated hydrogen bonds [$O(1)\cdots O(3')$ $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 3·014\ (8)$; $O(1)\cdots O(1')$ $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 2·858\ (8)$ Å].

Experimental. The title compound belongs to the class of synthetic agricultural pyrethroids. Crystals were grown from petroleum ether solution. Crystal approx. 0·55 × 0·5 × 0·22 mm, Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta < 23·5°$, h 0 to 12, k 0 to 25, l −13 to 13, 4125 unique reflections collected but less than 50% (1850) judged

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