

Fig. 2. Packing of the molecules in the unit cell. Atoms are identified by the following: N = large shaded circles, O = large open circles, H<sub>2</sub>O = large black circles, C = small open circles, Si = small black circles. Hydrogen bonds are indicated with narrow lines.

bond length is 1.658 (6) Å, which are in good agreement with previously published values for Si–X distances given in *International Tables for X-ray Crystallography* (1974).

The crystal structure is illustrated in Fig. 2. All available H atoms participate in hydrogen bonds [N6–H···O(W) = 2.929 (11), N7···H–O(W) = 2.775 (11) Å, O3'–H···O(W') = 2.838 (10) and N1···H–O(W') = 2.907 (11) Å], and there are no adenine–adenine base pairs. There is a stacking interaction between the phenyl ring and the base

[C5···C1(B) = 3.339 (11), C8···C6(B) = 3.377 (10), N9···C5(B) = 3.339 (11), N9···C6(B) = 3.435 (11) Å]. Finally, there is a close contact between C6 of the phenyl ring and O of the water of crystallization [C6(B)–H···O(W) = 3.197 (11) Å], which brings the δ(–) oxygen atom close to one of the δ(+) hydrogen atoms of the phenyl ring and is enthalpically favorable (Thomas, Smith, Thomas & Feldman, 1982).

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### Structure and Stereochemistry of the Methyl Ester of (5 $\alpha$ ,13 $\alpha$ ,14 $\beta$ ,17 $\alpha$ ,20S,24Z)-3-Oxolanosta-7,24-dien-26-oic Acid (Masticadienonic Acid)\*†

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**Abstract.** C<sub>31</sub>H<sub>48</sub>O<sub>3</sub>, *M<sub>r</sub>* = 468.7, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 7.118 (1), *b* = 10.165 (3), *c* =

38.244 (10) Å, *V* = 2767 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.125 Mg m<sup>-3</sup>, λ(Mo Kα) = 0.7107 Å, μ = 0.065 mm<sup>-1</sup>, *F*(000) = 1032, *T* = 293 K. Final *R* = 0.066 for 1636 observed reflections. The molecular structure determined from the X-ray data confirms the structure previously assigned on the basis of chemical and spectroscopic evidence. All the rings are *trans*

\* Contribution No. 838 of the Instituto de Química, UNAM.

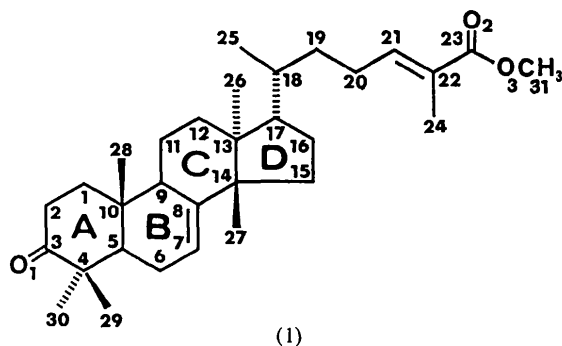
† Note that the crystallographic numbering differs from that used to name the compound.

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fused. The side chain adopts a fully extended *trans*-zigzag form and together with the stretched conformation of the ring system gives a long form of the molecule. The molecules in the crystal are held together by van der Waals interactions.

**Introduction.** The title compound (1) is a tetracyclic triterpene which was isolated from the bark of cuachalalá (*Amphyterygium adstringens* Schl., sin. *Julianania adstringens*), a plant widely distributed in Mexico; the sample was collected in Tejalpa (Estado de Puebla, Mexico). This compound exerts very marked physiological action, in the treatment of gastric ulcer and bile stones, for which it is much used in Mexico. Another point of interest concerning this compound is its antitumor activity (González & Delgado, 1962; González, McKenna & Delgado, 1962). The chemical and spectroscopic studies led to the proposal of the structure (1) for the title compound (Barton & Seoane, 1956; Domínguez, Franco, García, Porrás, Vázquez & Amezcuá, 1983).

The X-ray crystallographic structural determination of (1) was undertaken in order to establish the crystal structure and stereochemistry of this compound.



**Experimental.** Colorless crystal 0.30 × 0.44 × 0.40 mm. Nicolet R3 four-circle diffractometer. Lattice parameters from 25 machine-centered reflections with  $4.6 < 2\theta < 19.0^\circ$ . 2827 reflections with  $3 < 2\theta < 50^\circ$ ; 1636 independent with  $I > 2.5\sigma(I)$ ,  $R_{\text{int}} = 0.061$ ; 1191 reflections unobserved; index range  $h 0 \rightarrow 8$ ,  $k 0 \rightarrow 11$ ,  $l 0 \rightarrow 45$ ;  $\omega$ -scan mode, variable scan speed, scan width  $1.0^\circ$  ( $\theta$ ); two standard reflections (016, 023) monitored every 50 measurements, no intensity variation; Lp correction, absorption ignored; structure solved by combination of direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL* (Sheldrick, 1981); least-squares anisotropic refinement of all non-H atoms; H atoms riding on bonded C with fixed isotropic temperature factor,  $U = 0.06 \text{ \AA}^2$ ; function minimized  $\sum w(\Delta F)^2$ ,  $w = \{\sigma^2(F_o) + 0.001(F_o)^2\}^{-1}$ , where  $\sigma$  is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter  $X$

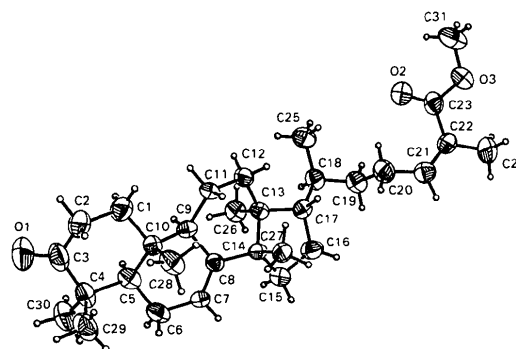


Fig. 1. The molecular conformation of (1), showing the atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	$U_{\text{eq}}$
O(1)	9530 (8)	10127 (6)	32 (1)	116 (3)
O(2)	3453 (6)	9565 (5)	3592 (1)	87 (2)
O(3)	1924 (7)	10396 (5)	4044 (1)	85 (2)
C(1)	7974 (9)	11049 (7)	854 (1)	59 (2)
C(2)	8607 (10)	11491 (7)	494 (1)	74 (3)
C(3)	8297 (10)	10444 (7)	222 (1)	71 (3)
C(4)	6317 (9)	9875 (6)	199 (1)	56 (2)
C(5)	5599 (8)	9540 (5)	577 (1)	47 (2)
C(6)	3587 (9)	9029 (6)	581 (1)	61 (2)
C(7)	2731 (8)	8914 (5)	940 (1)	45 (2)
C(8)	3527 (7)	9335 (5)	1229 (1)	40 (2)
C(9)	5508 (7)	9902 (5)	1220 (1)	41 (2)
C(10)	5899 (8)	10611 (5)	866 (1)	40 (2)
C(11)	6013 (8)	10700 (5)	1540 (1)	46 (2)
C(12)	5510 (8)	10040 (5)	1897 (1)	47 (2)
C(13)	3969 (8)	8982 (5)	1876 (1)	38 (2)
C(14)	2556 (7)	9351 (5)	1582 (1)	38 (2)
C(15)	957 (8)	8361 (6)	1643 (1)	54 (2)
C(16)	869 (8)	8230 (6)	2046 (1)	59 (2)
C(17)	2636 (8)	8873 (6)	2194 (1)	43 (2)
C(18)	3321 (8)	8186 (6)	2533 (1)	49 (2)
C(19)	1810 (9)	8196 (5)	2814 (1)	54 (2)
C(20)	1236 (9)	9551 (6)	2944 (1)	61 (2)
C(21)	-183 (10)	9494 (6)	3238 (2)	63 (2)
C(22)	106 (8)	9637 (6)	3579 (2)	53 (2)
C(23)	1997 (9)	9854 (6)	3726 (1)	57 (2)
C(24)	-1525 (9)	9607 (7)	3834 (2)	80 (3)
C(25)	5164 (9)	8744 (7)	2677 (1)	69 (2)
C(26)	4861 (8)	7631 (5)	1805 (1)	56 (2)
C(27)	1692 (8)	10741 (5)	1638 (1)	54 (2)
C(28)	4603 (9)	11810 (6)	823 (1)	60 (2)
C(29)	5129 (11)	10852 (7)	-7 (2)	77 (3)
C(30)	6383 (11)	8614 (6)	-16 (2)	82 (3)
C(31)	3685 (10)	10646 (7)	4210 (2)	90 (3)

Table 2. Bond lengths ( $\text{\AA}$ ) with e.s.d.'s in parentheses

O(1)–C(3)	1.185 (9)	O(2)–C(23)	1.194 (8)
O(3)–C(23)	1.335 (7)	O(3)–C(31)	1.428 (9)
C(1)–C(2)	1.516 (8)	C(1)–C(10)	1.543 (8)
C(2)–C(3)	1.505 (9)	C(3)–C(4)	1.526 (10)
C(4)–C(5)	1.572 (7)	C(4)–C(29)	1.523 (9)
C(4)–C(30)	1.523 (9)	C(5)–C(6)	1.523 (9)
C(5)–C(10)	1.566 (7)	C(6)–C(7)	1.506 (7)
C(7)–C(8)	1.316 (7)	C(8)–C(9)	1.524 (7)
C(8)–C(14)	1.514 (7)	C(9)–C(10)	1.560 (7)
C(9)–C(11)	1.512 (7)	C(10)–C(28)	1.537 (8)
C(11)–C(12)	1.561 (7)	C(12)–C(13)	1.538 (7)
C(13)–C(14)	1.556 (7)	C(13)–C(17)	1.547 (7)
C(13)–C(26)	1.537 (7)	C(14)–C(15)	1.537 (8)
C(14)–C(27)	1.557 (7)	C(15)–C(16)	1.547 (7)
C(16)–C(17)	1.527 (8)	C(17)–C(18)	1.550 (7)
C(18)–C(19)	1.521 (8)	C(18)–C(25)	1.532 (8)
C(19)–C(20)	1.520 (8)	C(20)–C(21)	1.515 (9)
C(21)–C(22)	1.326 (8)	C(22)–C(23)	1.476 (9)
C(22)–C(24)	1.516 (9)		

= 0.0011. In the last cycle  $(\Delta/\sigma)_{\max} = 0.18$ ;  $\Delta\rho$  from  $-0.21$  to  $+0.20$  e Å<sup>-3</sup>; 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  $\Delta$  and  $\varphi_m$  values of  $5.2(6)$  and  $-46.5(6)^\circ$ , respectively, indicating a half-chair conformation. (Altona, Geise & Romers, 1968).

The stereochemistry of (1) is as follows: C(5)- $\alpha$ H is *trans* to C(10)- $\beta$ CH<sub>3</sub>; C(9)- $\alpha$ H is *trans* to C(10)- $\beta$ CH<sub>3</sub>; C(13)- $\alpha$ CH<sub>3</sub> is *cis* to C(17)- $\alpha$ (side chain) and *trans* to C(14)- $\beta$ CH<sub>3</sub>. 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),

\* 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.

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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<sub>14</sub>H<sub>12</sub>O<sub>4</sub>,  $M_r = 244.2$ , triclinic,  $P\bar{1}$ ,  $a = 4.812(1)$ ,  $b = 9.929(3)$ ,  $c = 12.492(4)$  Å,  $\alpha = 93.77(3)$ ,  $\beta = 98.78(3)$ ,  $\gamma = 96.66(3)^\circ$ ,  $V = 583.8$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.389$  g cm<sup>-3</sup> at 138 K,  $F(000) = 256$ ,  $\mu(\text{Mo } K\alpha) = 0.6$  cm<sup>-1</sup>. The structure was determined by direct

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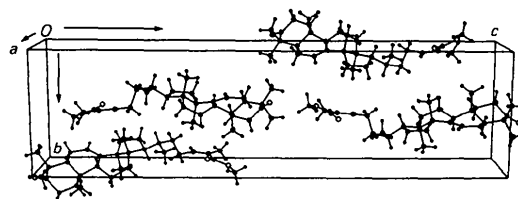


Fig. 2. A perspective drawing of the contents of one unit cell.

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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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