Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	−0.1279 (4)	0.0859	0.4901 (2)	0.0732 (10)
O2	0.1751 (3)	0.0775 (5)	0.5609 (2)	0.0470 (7)
O3	0.4391 (3)	0.3397 (5)	0.6391 (2)	0.0421 (6)
O4	−0.1159 (3)	−0.0561 (5)	0.2568 (2)	0.0527 (7)
N	0.1950 (4)	0.0000 (6)	0.3125 (2)	0.0373 (7)
C1	0.1505 (5)	0.1420 (6)	0.3727 (3)	0.0359 (8)
C2	0.0437 (5)	0.1009 (6)	0.4767 (3)	0.0446 (9)
C3	0.3745 (5)	0.1121 (6)	0.5268 (3)	0.0388 (8)
C4	0.3386 (4)	0.2095 (6)	0.4251 (2)	0.0338 (7)
C5	0.4956 (5)	0.2326 (7)	0.3408 (3)	0.0447 (9)
C6	0.4042 (6)	0.3085 (6)	0.2409 (3)	0.0584 (12)
C7	0.2139 (7)	0.3215 (7)	0.2218 (3)	0.0613 (11)
C8	0.0581 (5)	0.2596 (7)	0.2954 (3)	0.0501 (10)
C9	0.4863 (5)	0.1843 (7)	0.6229 (3)	0.0453 (9)
C10	0.0614 (5)	−0.0775 (6)	0.2501 (3)	0.0391 (8)
C11	0.1356 (5)	−0.1847 (6)	0.1650 (2)	0.0425 (8)
C12	0.0046 (6)	−0.2858 (7)	0.1164 (3)	0.0625 (11)
C13	0.0600 (10)	−0.3764 (7)	0.0296 (4)	0.085 (2)
C14	0.2418 (10)	−0.3702 (8)	−0.0085 (4)	0.090 (2)
C15	0.3724 (8)	−0.2712 (9)	0.0384 (3)	0.080 (2)
C16	0.3221 (6)	−0.1757 (7)	0.1248 (3)	0.0566 (11)

Table 2. *Selected geometric parameters (Å, °)*

O1—C2	1.206 (4)	O4—C10	1.240 (4)
O2—C2	1.359 (4)	N—C10	1.356 (4)
O2—C3	1.479 (4)	N—C1	1.487 (4)
O3—C9	1.423 (4)	C10—C11	1.505 (5)
C2—O2—C3	110.9 (2)	O2—C2—C1	109.5 (3)
C10—N—C1	123.5 (3)	O4—C10—N	122.5 (3)
O1—C2—O2	120.7 (3)	O4—C10—C11	119.9 (3)
O1—C2—C1	129.8 (3)	N—C10—C11	117.5 (3)
C3—O2—C2—C1	4.9 (4)	C5—C6—C7—C8	−1.4 (7)
C8—C1—C4—C3	160.0 (3)	C1—N—C10—C11	159.6 (3)
C2—C1—C4—C5	168.3 (3)		

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1992). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989). Software used to prepare material for publication: *SHELXL*93. Molecular geometry: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1053). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trinervinol†

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Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, 4,4a,4b,5,6,7,8,8a,9,10-decahydro-7-hydroxy-α-(hydroxymethyl)-4b,8,8-trimethyl-2,2(3*H*)-phenanthrenedimethanol, $C_{20}H_{34}O_4$. The molecule consists of three six-membered rings (*A*, *B* and *C*). The *A*/*B* and *B*/*C* rings are *trans*-fused. The *A*, *B* and *C* rings adopt a distorted chair $^1C_4$ conformation. The crystal structure is stabilized by a network of intra- and intermolecular hydrogen bonds.

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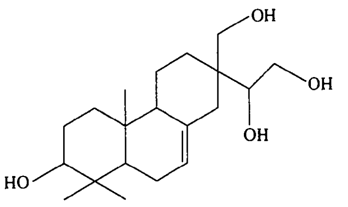
† Contribution No. 1409 of the Instituto de Química, UNAM.

Comment

Trinervinol (1) is a tetraol tricyclic diterpenoid which was isolated from the flowers and leaves of *Piqueria trinervia*, *Cav*, a member of the Compositae family found in Mexico. The chemical structure of (1) has been assigned from spectral (IR, UV and NMR) and chemical methods (Jiménez & González de la Parra, 1983). It is a new diterpene compound with an isopimarane skeleton with a hydroxyl group at C(17).



(1)

It was of interest to determine the crystal structure of this compound in order to ascertain its conformation and molecular geometry. The Cremer & Pople (1975) ring-puckering parameters for the *A*, *B* and *C* rings are $q^2 = 0.068(2)$, $q^3 = 0.534(2)$, $Q = 0.539(2)$ Å, $\theta = 7.3(2)$, $\varphi = 74(2)°$, $q^2 = 0.430(2)$, $q^3 = 0.319(2)$, $Q = 0.535(2)$ Å, $\theta = 53.4(2)$, $\varphi = -21.4(3)°$, and $q^2 = 0.062(2)$, $q^3 = 0.515(3)$, $Q = 0.518(3)$ Å, $\theta = 6.9(3)$, $\varphi = -157(3)°$, respectively. A similar conformation has been observed in (3*R*,5*R*,8*S*,10*R*,13*R*)-*ent*-pimara-9(11),15-dien-3-yl *p*-bromobenzoate (Soriano-García, Guerrero & Toscano, 1986), and closely resembles 11β-hydroxy-7-ketosandaracopimar-8(14),15-diene (Rao, Krishna & Suseela, 1985) for the C atoms in rings *A* and *B*. The arrangement of the molecules in the unit cell is shown in Fig. 2. The molecular packing is dominated by a three-dimensional hydrogen-bonding network. All hydroxyl groups in the molecule exhibit both donor and acceptor functions. There is one intramolecular hydrogen bond between the O(4)—H hydroxyl group and the O(3) carbonyl group. The molecules are connected by hydrogen bonds between hydroxyl groups, forming continuous parallel ribbons along the *c*-axis direction. The hydroxyl substituents at C(3) and C(16) are hydrogen bonded to O(2) and O(1) of adjacent molecules, while the hydroxyl substituent at C(15) is hydrogen bonded to O(4) of an adjacent molecule related by the twofold screw axis along the (010) direction with coordinates $(0, y, \frac{1}{4})$.

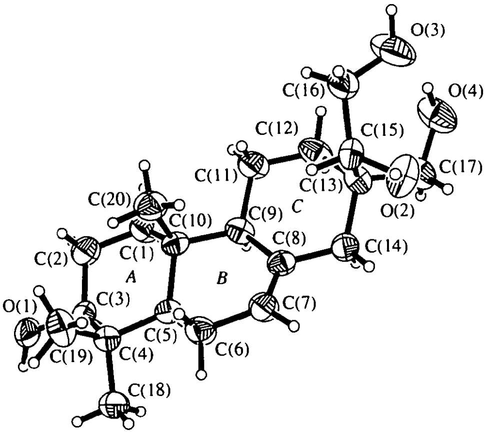


Fig. 1. The molecular structure of the title compound with the atom labelling; 50% probability displacement ellipsoids are shown.

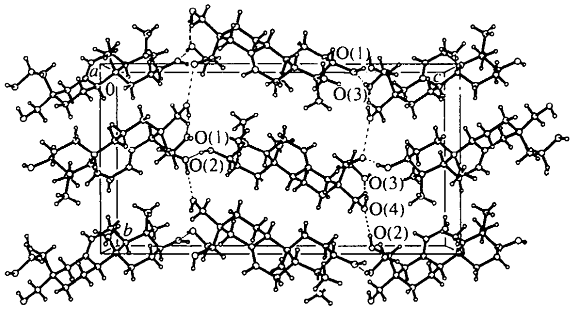


Fig. 2. A perspective drawing of the contents of one unit cell as viewed along **a**. Hydrogen bonds are shown as broken lines.

Experimental

The title compound was isolated from the flowers and leaves of *Piqueria trinervia*, *Cav*.

Crystal data

$C_{20}H_{34}O_4$
$M_r = 338.49$
Orthorhombic
$P2_12_12_1$
$a = 6.997(1)$ Å
$b = 11.887(2)$ Å
$c = 23.201(4)$ Å
$V = 1929.8(5)$ Å^3
$Z = 4$
$D_x = 1.163$ Mg m^{-3}
$D_m = 1.20$ Mg m^{-3}
D_m measured by pycnometry

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 6.5$–$15.8°$
$\mu = 0.596$ mm^{-1}
$T = 296$ K
Prism
0.38 × 0.38 × 0.20 mm
Colourless

Data collection

Nicolet *P*3/*F* diffractometer
$\theta/2\theta$ scans
Absorption correction: analytical
$T_{\min} = 0.781$, $T_{\max} = 0.987$
1415 measured reflections
1337 independent reflections
1295 observed reflections $[I > 2.8\sigma(I)]$

$\theta_{\max} = 55°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 24$
2 standard reflections monitored every 150 reflections
frequency: 90 min
intensity decay: 5%

Refinement

Refinement on *F*
$R = 0.037$
$wR = 0.056$

$(\Delta/\sigma)_{\max} = 0.139$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

$S = 1.256$
1295 reflections
217 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00191(F_o)^2]$

Extinction correction: Larson (1967)
Extinction coefficient: 0.012 (2)
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	1.0614 (4)	0.4048 (2)	−0.0659 (1)	0.050 (1)
C(2)	1.0640 (4)	0.4626 (2)	−0.1247 (1)	0.052 (1)
C(3)	0.8671 (3)	0.4995 (2)	−0.1423 (1)	0.041 (1)
C(4)	0.7758 (3)	0.5823 (2)	−0.0994 (1)	0.040 (1)
C(5)	0.7853 (3)	0.5291 (2)	−0.0381 (1)	0.036 (1)
C(6)	0.7056 (4)	0.6047 (2)	0.0095 (1)	0.046 (1)
C(7)	0.7008 (4)	0.5458 (2)	0.0662 (1)	0.038 (1)
C(8)	0.8007 (3)	0.4532 (2)	0.0780 (1)	0.049 (1)
C(9)	0.9412 (3)	0.4036 (2)	0.0359 (1)	0.039 (1)
C(10)	0.9797 (3)	0.4791 (2)	−0.0178 (1)	0.036 (1)
C(11)	1.1255 (4)	0.3681 (3)	0.0671 (1)	0.052 (1)
C(12)	1.0897 (4)	0.2992 (2)	0.1211 (1)	0.052 (1)
C(13)	0.9590 (4)	0.3569 (2)	0.1650 (1)	0.039 (1)
C(14)	0.7727 (4)	0.3884 (2)	0.1335 (1)	0.041 (1)
C(15)	1.0445 (4)	0.4677 (2)	0.1887 (1)	0.039 (1)
C(16)	1.2524 (4)	0.4689 (2)	0.2057 (1)	0.053 (1)
C(17)	0.9031 (4)	0.2737 (2)	0.2125 (1)	0.050 (1)
C(18)	0.5642 (4)	0.5955 (3)	−0.1157 (1)	0.063 (1)
C(19)	0.8701 (5)	0.6993 (2)	−0.1052 (1)	0.057 (1)
C(20)	1.1221 (4)	0.5730 (2)	−0.0022 (1)	0.055 (1)
O(1)	0.8805 (3)	0.5465 (2)	−0.1992 (1)	0.053 (1)
O(2)	0.9276 (3)	0.5053 (1)	0.2351 (1)	0.056 (1)
O(3)	1.2931 (4)	0.3898 (2)	0.2496 (1)	0.087 (1)
O(4)	1.0588 (4)	0.2225 (2)	0.2424 (1)	0.073 (1)

Table 2. *Selected geometric parameters (Å, °)*

C(1)—C(2)	1.530 (3)	C(2)—C(3)	1.502 (3)
C(3)—C(4)	1.539 (3)	C(4)—C(5)	1.557 (3)
C(5)—C(6)	1.530 (3)	C(6)—C(7)	1.491 (3)
C(7)—C(8)	1.332 (3)	C(8)—C(9)	1.507 (3)
C(1)—C(10)	1.533 (3)	C(5)—C(10)	1.557 (3)
C(9)—C(10)	1.559 (3)	C(9)—C(11)	1.538 (4)
C(11)—C(12)	1.518 (4)	C(12)—C(13)	1.530 (3)
C(8)—C(14)	1.513 (3)	C(13)—C(14)	1.541 (3)
C(13)—C(15)	1.548 (3)	C(15)—C(16)	1.508 (4)
C(13)—C(17)	1.532 (3)	C(4)—C(18)	1.536 (4)
C(4)—C(19)	1.545 (4)	C(10)—C(20)	1.539 (4)
C(2)—C(1)—C(10)	113.2 (2)	C(1)—C(2)—C(3)	111.3 (2)
C(2)—C(3)—C(4)	113.1 (2)	C(2)—C(3)—O(1)	107.7 (2)
C(4)—C(3)—O(1)	111.9 (2)	C(3)—C(4)—C(5)	108.3 (2)
C(3)—C(4)—C(18)	107.8 (2)	C(5)—C(4)—C(18)	107.9 (2)
C(3)—C(4)—C(19)	110.0 (2)	C(5)—C(4)—C(19)	115.4 (2)
C(18)—C(4)—C(19)	107.3 (2)	C(4)—C(5)—C(6)	113.9 (2)
C(4)—C(5)—C(10)	117.9 (2)	C(6)—C(5)—C(10)	108.9 (2)
C(5)—C(6)—C(7)	111.7 (2)	C(6)—C(7)—C(8)	123.8 (2)
C(7)—C(8)—C(9)	122.2 (2)	C(7)—C(8)—C(14)	121.8 (2)
C(9)—C(8)—C(14)	115.9 (2)	C(8)—C(9)—C(10)	113.9 (2)
C(8)—C(9)—C(11)	110.4 (2)	C(10)—C(9)—C(11)	113.0 (2)
C(1)—C(10)—C(5)	109.0 (2)	C(1)—C(10)—C(9)	108.2 (2)
C(5)—C(10)—C(9)	108.1 (2)	C(1)—C(10)—C(20)	110.3 (2)
C(5)—C(10)—C(20)	111.1 (2)	C(9)—C(10)—C(20)	110.0 (2)
C(9)—C(11)—C(12)	113.5 (2)	C(11)—C(12)—C(13)	113.9 (2)
C(12)—C(13)—C(14)	107.4 (2)	C(12)—C(13)—C(15)	112.7 (2)
C(14)—C(13)—C(15)	106.8 (2)	C(12)—C(13)—C(17)	110.0 (2)
C(14)—C(13)—C(17)	106.4 (2)	C(15)—C(13)—C(17)	113.1 (2)
C(8)—C(14)—C(13)	114.7 (2)	C(13)—C(15)—C(16)	118.3 (2)
C(13)—C(15)—O(2)	108.3 (2)	C(16)—C(15)—O(2)	110.7 (2)
C(15)—C(16)—O(3)	112.1 (2)	C(13)—C(17)—O(4)	115.4 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
O(4)—H···O(3)	2.583 (4)	1.68 (3)	164 (2)
O(1)—H···O(2$^{\rm i}$)	2.711 (3)	1.77 (3)	172 (3)
O(3)—H···O(1$^{\rm ii}$)	2.683 (3)	1.86 (3)	161 (3)
O(2)—H···O(4$^{\rm iii}$)	2.636 (3)	1.82 (2)	171 (3)

Symmetry codes: (i) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; (ii) $\frac{5}{2} - x, 1 - y, \frac{1}{2} + z$; (iii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The H atoms in the CH and CH_2 groups were allowed to ride on the bonded C atom and refined.

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*, *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1460–1462

Acetonide-3-trinervinone†

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(*Received 24 April 1995; accepted 17 November 1995*)

Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, 2,2,4b′,8′,8′-pentamethyl-6,10a′-epoxyspiro[1,3-dioxepane-5,2′-perhydrophenanthren]-7′-one, $C_{23}H_{36}O_4$, which consists of three

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† Contribution No. 1410 of the Instituto de Química, UNAM.