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Structure of 4-Hydroxy-6-methoxy-5,9,13,13-tetramethyl-2-oxatetracyclo-[6.5.0.0^{1,8}.0^{8,12}]tridecan-7-one*

BY M. SORIANO-GARCÍA,† F. WALLS, F. YUSTE, R. SÁNCHEZ-OBREGÓN, B. ORTÍZ, E. DÍAZ, R. A. TOSCANO AND H. BARRIOS

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, *Mexico DF*

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Abstract. $C_{17}H_{24}O_4$, $M_r = 292{\cdot}4$, orthorhombic, $P2_12_12$, $a = 13{\cdot}533\ (4)$, $b = 14{\cdot}328\ (4)$, $c = 8{\cdot}047\ (2)$ Å, $V = 1560\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}24$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}082$ mm^{-1}, $F(000) = 632$, $T = 293$ K, $R = 0{\cdot}049$ for 991 observed reflections. The X-ray analysis establishes unequivocally the molecular structure of the title compound. The six-membered *A* ring is a distorted boat, and the five-membered *D* ring is a half-chair. The four-membered rings *B* (oxetane) and *C* (cyclobutane) exhibit puckering angles of 2·4 (4) and 12·5 (4)°, respectively. The *A*/*B*, *A*/*C* and *C*/*D* ring junctions are *cis*. The crystal structure is stabilized by intermolecular hydrogen bonding involving the O(1)—H hydroxyl group and the O(3) carbonyl group, O(1)—H···O(3)($-\frac{1}{2}+x$, $1{\cdot}5-y$, $1-z$) 2·800 (5) Å, and by two intermolecular C—H···O contacts < 3·4 Å: C(12)···O(1)($\frac{1}{2}+x$, $1{\cdot}5-y$, $1-z$) 3·30 (1) and C(16)···O(4)($\frac{1}{2}-x$, $\frac{1}{2}+y$, $1-z$) 3·38 (1) Å.

* Contribution No. 964 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

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

	x	y	z	U_{eq}
O(1)	219 (2)	7275 (3)	5743 (5)	61 (1)
O(2)	1214 (2)	9262 (2)	6575 (5)	56 (1)
O(3)	3758 (2)	6924 (2)	6264 (5)	54 (1)
O(4)	2635 (2)	6199 (2)	8826 (4)	44 (1)
C(1)	1633 (3)	8443 (3)	5792 (6)	36 (2)
C(3)	480 (4)	8708 (4)	7413 (8)	63 (2)
C(4)	893 (3)	7803 (3)	6690 (7)	42 (2)
C(5)	1339 (3)	7129 (3)	7882 (6)	36 (2)
C(6)	2304 (3)	6918 (3)	7856 (6)	33 (1)
C(7)	2998 (4)	7343 (4)	6659 (7)	37 (2)
C(8)	2766 (3)	8292 (3)	6021 (7)	35 (2)
C(9)	3371 (4)	9040 (3)	7011 (7)	49 (2)
C(10)	3350 (4)	9885 (4)	5844 (8)	71 (2)
C(11)	3470 (4)	9470 (4)	4124 (9)	65 (2)
C(12)	2932 (4)	8529 (4)	4155 (7)	48 (2)
C(13)	1789 (4)	8516 (4)	3858 (7)	44 (2)
C(14)	625 (4)	6621 (4)	9014 (8)	53 (2)
C(15)	3075 (4)	9203 (4)	8807 (9)	74 (2)
C(16)	1292 (4)	9344 (4)	3052 (8)	71 (2)
C(17)	1523 (4)	7620 (5)	2919 (8)	67 (2)
C(18)	3517 (4)	6355 (4)	9775 (8)	59 (2)

Experimental. The title compound was recrystallized from acetone–hexane and gave some colourless crystals and more abundant pale-yellow crystals. Crystal used for data collection was pale yellow, m.p. 393–394 K, with dimensions of 0·22 × 0·40 × 0·40 mm. Nicolet *R*3 four-circle diffractometer. Unit-cell parameters from 25 reflections, $4{\cdot}4 < 2\theta < 19{\cdot}1°$. 1206 unique reflections measured for an octant, $3 < 2\theta <$

Table 2. *Molecular geometry*

E.s.d.'s are given in parentheses.

(*a*) Bond lengths (Å)

O(1)—C(4)	1·409 (6)	O(2)—C(1)	1·447 (6)
O(2)—C(3)	1·439 (7)	O(3)—C(7)	1·232 (6)
O(4)—C(6)	1·367 (6)	O(4)—C(18)	1·435 (6)
C(1)—C(4)	1·538 (7)	C(1)—C(8)	1·560 (6)
C(1)—C(13)	1·574 (7)	C(3)—C(4)	1·527 (7)
C(4)—C(5)	1·489 (7)	C(5)—C(6)	1·341 (6)
C(5)—C(14)	1·514 (7)	C(6)—C(7)	1·477 (7)
C(7)—C(8)	1·487 (7)	C(8)—C(9)	1·566 (7)
C(8)—C(12)	1·556 (8)	C(9)—C(10)	1·532 (8)
C(9)—C(15)	1·518 (9)	C(10)—C(11)	1·515 (9)
C(11)—C(12)	1·533 (7)	C(12)—C(13)	1·565 (7)
C(13)—C(16)	1·509 (8)	C(13)—C(17)	1·533 (8)

(*b*) Valence angles (°)

C(1)—O(2)—C(3)	91·6 (3)	C(6)—O(4)—C(18)	117·3 (4)
O(2)—C(1)—C(4)	91·4 (3)	O(2)—C(1)—C(8)	116·5 (4)
C(4)—C(1)—C(8)	120·1 (4)	O(2)—C(1)—C(13)	115·4 (4)
C(4)—C(1)—C(13)	126·3 (4)	C(8)—C(1)—C(13)	89·7 (4)
O(2)—C(3)—C(4)	92·1 (4)	O(1)—C(4)—C(1)	119·2 (4)
O(1)—C(4)—C(3)	115·2 (4)	C(1)—C(4)—C(3)	84·9 (4)
O(1)—C(4)—C(5)	105·2 (4)	C(1)—C(4)—C(5)	115·2 (4)
C(3)—C(4)—C(5)	117·0 (5)	C(4)—C(5)—C(6)	122·1 (4)
C(4)—C(5)—C(14)	116·1 (4)	C(6)—C(5)—C(14)	121·5 (4)
O(4)—C(6)—C(5)	118·7 (4)	O(4)—C(6)—C(7)	118·3 (4)
C(5)—C(6)—C(7)	122·5 (4)	O(3)—C(7)—C(6)	119·9 (4)
O(3)—C(7)—C(8)	122·2 (5)	C(6)—C(7)—C(8)	117·9 (4)
C(1)—C(8)—C(7)	112·1 (4)	C(1)—C(8)—C(9)	118·6 (4)
C(7)—C(8)—C(9)	109·9 (4)	C(1)—C(8)—C(12)	89·8 (4)
C(7)—C(8)—C(12)	120·1 (4)	C(9)—C(8)—C(12)	105·4 (4)
C(8)—C(9)—C(10)	102·7 (4)	C(8)—C(9)—C(15)	116·8 (4)
C(10)—C(9)—C(15)	117·2 (5)	C(9)—C(10)—C(11)	104·3 (4)
C(10)—C(11)—C(12)	106·2 (5)	C(8)—C(12)—C(11)	196·0 (5)
C(8)—C(12)—C(13)	90·2 (4)	C(11)—C(12)—C(13)	118·5 (4)
C(1)—C(13)—C(12)	89·0 (4)	C(1)—C(13)—C(16)	114·7 (4)
C(12)—C(13)—C(16)	119·8 (4)	C(1)—C(13)—C(17)	113·6 (4)
C(12)—C(13)—C(17)	108·5 (4)	C(16)—C(13)—C(17)	110·0 (5)

(*c*) Selected torsion angles (°)

C(8)—C(1)—C(4)—C(5)	6·1 (6)	O(2)—C(1)—C(4)—C(3)	1·6 (4)
C(4)—C(1)—C(8)—C(7)	19·0 (6)	C(13)—C(1)—C(8)—C(12)	8·8 (3)
C(1)—C(8)—C(12)—C(13)	−8·9 (4)	C(9)—C(8)—C(12)—C(1)	−8·8 (5)
C(18)—O(4)—C(6)—C(5)	−134·5 (5)		

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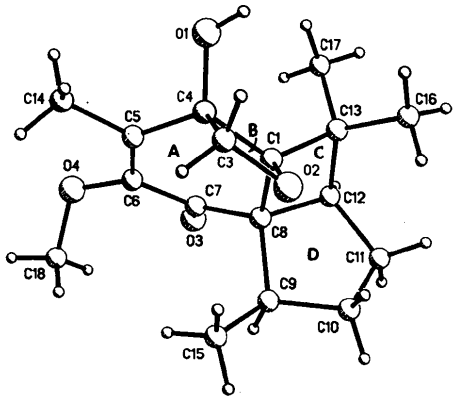

Fig. 1. Molecular structure of the title compound with atom numbering.

45°, of which 991 with $I > 2{\cdot}5\sigma(I)$ were used in the analysis. Index range $h\ 0 \rightarrow 14$, $k\ 0 \rightarrow 15$, $l\ 0 \rightarrow 8$, ω scan and variable scan speed. Two standard reflections ($0\bar{2}0$, $\bar{1}12$) monitored every 50 measurements, no significant variation. Lp correction, absorption ignored, $R_{int} = 0{\cdot}032$. Structure solved by direct methods using *SHELXTL5* (Sheldrick, 1985). Non-H atoms were treated anisotropically in least squares; H atoms in calculated positions riding on bonded C with a fixed isotropic temperature factor, $U = 0{\cdot}06 Å^2$, hydroxyl H-atom positions (from ΔF map) refined. $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}002(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter $X = 0{\cdot}0038$. In the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}080$; $\Delta\rho$ from $-0{\cdot}16$ to $0{\cdot}20$ e $Å^{-3}$, $S = 1{\cdot}07$; final $R = 0{\cdot}049$, $R_w = 0{\cdot}054$ and $wR = 0{\cdot}063$. 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 the *SHELXTL* system of programs.

The atomic coordinates are given in Table 1.* A perspective molecular drawing, together with the atom-numbering scheme, is displayed in Fig. 1. Bond distances, angles and selected torsion angles are listed in Table 2.

Related literature. As part of our studies on the ultraviolet irradiation of *O*-methylmethoxyperezone (Barrera, Barrios & Walls, 1980), the title compound was prepared. Its structure was investigated by chemical methods and could not be established unambiguously by ^{13}C NMR (Barrios, Salazar, Díaz, Walls & Joseph-Nathan, 1986).

We thank Mrs Cynthia Lesh de S. for technical assistance.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51835 (9 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-Amino-2-(6-methoxy-3-indolyl)propionic Acid Hemihydrate*

BY M. SORIANO-GARCÍA,† R. A. TOSCANO, M. RUBIO AND A. RODRÍGUEZ

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, *Mexico DF*

AND H. MUNGUIA-MEDINA

Departamento de Investigación del Hospital Civil 'Miguel Silva', Morelia, Mich. Mexico

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Abstract. $C_{12}H_{14}N_2O_3.0{\cdot}5H_2O$, $M_r = 243{\cdot}3$, orthorhombic, $Pna2_1$, $a = 9{\cdot}986\ (2)$, $b = 37{\cdot}117\ (7)$, $c = 6{\cdot}578\ (1)$ Å, $V = 2438\ (1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}32$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}780$ mm^{-1}, $F(000) = 1032$, $T = 293$ K, $R = 0{\cdot}044$ for 1588 observed reflections. The distances and angles of the indole rings agree well with those

* Contribution No. 965 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.

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