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## Structure of 5,7-Dihydroxy-8-(2-methylbutyryl)-4-*n*-pentyl-3,4-dihydrocoumarin (Dihydromammea C/OB), C<sub>19</sub>H<sub>26</sub>O<sub>5</sub>\*

BY CARL H. SCHWALBE

*Department of Pharmacy, The University of Aston in Birmingham, Gosta Green, Birmingham B4 7ET, England*

AND PETER G. WATERMAN

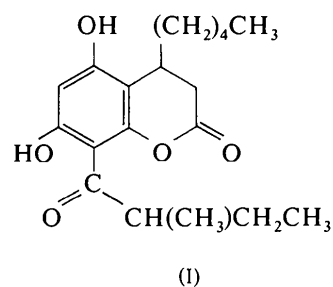
*Phytochemistry Research Laboratory, School of Pharmaceutical Sciences, University of Strathclyde, Glasgow G1 1XW, Scotland*

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**Abstract.**  $M_r = 334.38$ , orthorhombic,  $P2_12_12_1$ ,  $a = 8.737$  (1),  $b = 8.808$  (9),  $c = 24.009$  (4) Å,  $U = 1848$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.20$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.051$  mm<sup>-1</sup>,  $F(000) = 720$ ,  $T = 294$  (2) K. Final  $R = 0.041$  for 1315 observed reflections. The structure established chemically is confirmed. The attachment of the *n*-pentyl side chain to the dihydrocoumarin ring is found to be axial, and the non-equivalence to NMR of methylene protons in the 2-methylbutyryl side chain is attributed to the restricted rotation of this group.

**Introduction.** Mammea coumarin C/OB (I) is an unusual dihydrocoumarin derivative that has been isolated, as a minor component, from the seeds of the West African tree *Mammea africana* Sabine (Guttiferae) (Crichton & Waterman, 1978). Identification of (I) was achieved largely on the basis of <sup>1</sup>H and <sup>13</sup>C NMR data and comparison with that obtained for similarly substituted coumarins and simple dihydrocoumarins. These data indicated the presence of strong intramolecular hydrogen bonding in (I) but failed to yield information regarding the shape of the lactone ring. Furthermore the observed coupling between the C(3) protons and the C(4) proton (90 MHz,  $J_1 = 7$ ,  $J_2 = 3$  Hz), while suggesting an axial configuration for the C(4) *n*-pentyl substituent, left this assignment open to doubt. In order to clarify these matters and to gain

further insight into the striking spectral features of this compound, a crystal-structure determination was undertaken.



**Experimental.** Needle crystals of (I) obtained from a light petroleum solution, crystal  $0.8 \times 0.3 \times 0.04$  mm selected for examination on an Enraf-Nonius CAD-4 diffractometer; unit-cell dimensions obtained by least-squares analysis of setting angles of 25 diffraction maxima measured with graphite-monochromated Mo  $K\alpha$  radiation; intensities collected by  $\omega$ - $2\theta$  scans for  $\theta$  2–25°; 1931 reflections measured merged to give 1899 unique data ( $R_{\text{int}} = 0.030$ ), of which 1315 were considered observed ( $F > 3\sigma$ ); standard deviations assigned on basis of counting statistics and instrument instability; intensities corrected for Lorentz-polarization effects but not for absorption; structure solved by *MULTAN* (Germain, Main & Woolfson, 1971) and refined with *SHELX* (Sheldrick, 1976); after non-H coordinates and isotropic temperature factors

\* Contribution from the Crystallography Unit, Universities of Aston and Birmingham.

had been refined by least squares, both phenolic H atoms located in a difference electron density map and all other H-atom positions calculated; in subsequent refinements methyl groups treated as rigid bodies free to rotate with a refinable common isotropic temperature factor for the H atoms in each group; other H atoms kept fixed and assigned the isotropic temperature factor of the attached atom; final refinements based on  $F$  adjusted coordinates and anisotropic thermal parameters for all non-H atoms; reflections weighted by  $w = 1/[\sigma^2(F_o) + 0.000728 F_o^2]$  in final refinement cycle; final discrepancy indices for observed data are  $R = 0.041$  and  $R_w = 0.053$ ; in last cycle no parameter shifted by more than 0.13 e.s.d., goodness-of-fit ratio  $S = 0.49$ ; a difference electron density map showed no peak exceeding  $0.15 \text{ e } \text{Å}^{-3}$ .

**Discussion.** The structure of (I), which agrees with that deduced from spectral data, is depicted with its numbering scheme in Fig. 1. Atomic coordinates are given in Table 1,\* and bond distances, bond angles and selected torsion angles in Table 2. The lactone ring geometry is strongly affected by the considerable double-bond character of two bonds: C(4A)–C(8A) at 1.373 (4) Å and O(1)–C(2) at 1.357 (5) Å. Torsion angles around these bonds are within 3° of the expected 0 or 180° for doubly bonded systems. The adjacent bond O(1)–C(8A) is weaker with a distance of 1.408 (4) Å and 25° twisting. Other torsion angles around the lactone ring skeleton fall within the synperiplanar or synclinal range (Klyne & Prelog, 1960), but always with magnitudes less than 60°. Bond angles within the ring show no more than 3° distortion from the angles expected for  $sp^3$  hybrid C(3) and C(4) and  $sp^2$  hybrids otherwise.

The attachment of the *n*-pentyl side chain is axial with the enlarged C(9)–C(4)–C(4A)–C(8A) torsion angle of  $-83 (1)^\circ$  serving to bring the chain well clear of the ring system. Torsion angles along the chain are all antiperiplanar giving maximum extension. The

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{Å}^2 \times 10^3$ ) for non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} (U_{11} + U_{22} + U_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
O(1)	6653 (2)	4834 (3)	3147 (1)	58 (1)
C(2)	6003 (4)	6015 (5)	2872 (2)	62 (1)
C(3)	7028 (4)	7180 (5)	2627 (2)	56 (1)
C(4)	8441 (4)	6412 (4)	2383 (1)	45 (1)
C(4A)	9158 (3)	5524 (4)	2845 (1)	41 (1)
C(5)	10747 (4)	5492 (4)	2945 (1)	43 (1)
C(6)	11347 (4)	4726 (4)	3395 (1)	45 (1)
C(7)	10400 (4)	3928 (4)	3752 (1)	47 (1)
C(8)	8773 (4)	3909 (4)	3675 (1)	47 (1)
C(8A)	8251 (4)	4761 (4)	3219 (1)	45 (1)
O(2)	4632 (3)	5994 (5)	2847 (1)	94 (1)
O(5)	11622 (3)	6255 (3)	2573 (1)	58 (1)
O(7)	11084 (3)	3187 (3)	4164 (1)	65 (1)
C(9)	7998 (4)	5440 (5)	1876 (2)	54 (1)
C(10)	9317 (4)	4688 (5)	1577 (1)	55 (1)
C(11)	8790 (6)	3806 (5)	1060 (2)	69 (2)
C(12)	10065 (7)	3052 (6)	740 (2)	82 (2)
C(13)	9499 (9)	2108 (6)	251 (2)	109 (3)
C(14)	7835 (5)	2997 (5)	4063 (2)	58 (1)
O(14)	8479 (4)	2322 (4)	4447 (1)	90 (1)
C(15)	6107 (5)	2855 (5)	4030 (2)	65 (1)
C(16)	5568 (6)	1311 (6)	4239 (2)	77 (2)
C(17)	5998 (7)	33 (6)	3846 (2)	88 (2)
C(18)	5394 (7)	4147 (7)	4377 (2)	98 (2)

Table 2. Bond distances (Å), bond angles and selected torsion angles ( $^\circ$ ) (e.s.d.'s for torsion angles average 0.4°)

O(1)–C(2)	1.357 (5)	O(1)–C(8A)	1.408 (4)
C(2)–O(2)	1.199 (4)	C(4)–C(9)	1.538 (5)
C(2)–C(3)	1.484 (5)	C(9)–C(10)	1.511 (5)
C(3)–C(4)	1.525 (5)	C(10)–C(11)	1.535 (5)
C(4)–C(4A)	1.495 (5)	C(11)–C(12)	1.507 (7)
C(4A)–C(5)	1.409 (4)	C(12)–C(13)	1.521 (6)
C(5)–O(5)	1.354 (4)	C(8)–C(14)	1.478 (5)
C(5)–C(6)	1.377 (5)	C(14)–O(14)	1.233 (5)
C(6)–C(7)	1.383 (5)	C(14)–C(15)	1.517 (6)
C(7)–O(7)	1.327 (4)	C(15)–C(16)	1.524 (6)
C(7)–C(8)	1.434 (5)	C(15)–C(18)	1.542 (7)
C(8)–C(8A)	1.403 (5)	C(16)–C(17)	1.516 (6)
C(4A)–C(8A)	1.373 (4)		
C(8A)–O(1)–C(2)	120.6 (3)	C(8)–C(8A)–C(4A)	125.7 (3)
O(1)–C(2)–C(3)	118.1 (3)	C(4A)–C(8A)–O(1)	118.0 (3)
O(1)–C(2)–O(2)	115.5 (4)	C(3)–C(4)–C(9)	110.3 (3)
O(2)–C(2)–C(3)	126.4 (4)	C(4A)–C(4)–C(9)	113.7 (3)
C(2)–C(3)–C(4)	109.5 (3)	C(4)–C(9)–C(10)	115.4 (3)
C(3)–C(4)–C(4A)	106.6 (3)	C(9)–C(10)–C(11)	112.2 (3)
C(4)–C(4A)–C(5)	123.3 (3)	C(10)–C(11)–C(12)	114.4 (4)
C(4)–C(4A)–C(8A)	120.0 (3)	C(11)–C(12)–C(13)	113.2 (5)
C(8A)–C(4A)–C(5)	116.6 (3)	C(7)–C(8)–C(14)	118.4 (3)
C(4A)–C(5)–C(6)	121.2 (3)	C(8A)–C(8)–C(14)	127.0 (3)
C(4A)–C(5)–O(5)	115.7 (3)	C(8)–C(14)–C(15)	124.3 (4)
O(5)–C(5)–C(6)	123.1 (3)	C(8)–C(14)–O(14)	118.7 (4)
C(5)–C(6)–C(7)	120.5 (3)	O(14)–C(14)–C(15)	117.0 (4)
C(6)–C(7)–C(8)	121.3 (3)	C(14)–C(15)–C(16)	111.3 (4)
C(6)–C(7)–O(7)	116.3 (3)	C(14)–C(15)–C(18)	108.2 (4)
O(7)–C(7)–C(8)	122.5 (3)	C(16)–C(15)–C(18)	110.8 (4)
C(7)–C(8)–C(8A)	114.6 (3)	C(15)–C(16)–C(17)	112.4 (3)
C(8)–C(8A)–O(1)	116.3 (3)		
C(8A)–O(1)–C(2)–C(3)	3	C(5)–C(4A)–C(8A)–O(1)	177
C(8A)–O(1)–C(2)–O(2)	–178	C(4A)–C(8A)–O(1)–C(2)	–25
O(1)–C(2)–C(3)–C(4)	39	C(7)–C(8)–C(14)–O(14)	2
O(2)–C(2)–C(3)–C(4)	140	C(7)–C(8)–C(14)–C(15)	–179
C(2)–C(3)–C(4)–C(4A)	57	C(8)–C(14)–C(15)–C(16)	149
C(2)–C(3)–C(4)–C(9)	67	C(8)–C(14)–C(15)–C(18)	–89
C(3)–C(4)–C(4A)–C(8A)	39	O(14)–C(14)–C(15)–C(16)	–33
C(9)–C(4)–C(4A)–C(8A)	83	O(14)–C(14)–C(15)–C(18)	90
C(4)–C(4A)–C(8A)–O(1)	1	C(14)–C(15)–C(16)–C(17)	70
C(4)–C(4A)–C(8A)–C(8)	179	C(18)–C(15)–C(16)–C(17)	169
C(5)–C(4A)–C(8A)–C(8)	3		

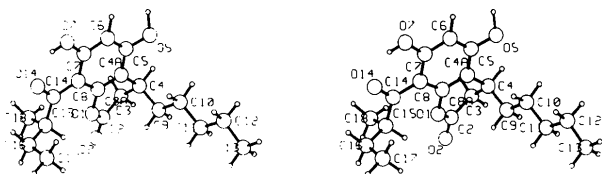


Fig. 1. PLUTO (Motherwell, 1972) stereo drawing of the molecule projected onto its least-squares plane with nomenclature indicated for the non-H atoms.

\* Lists of structure factors, anisotropic thermal parameters, H-atom positions, least-squares planes, and two projections of the molecule have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38284 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3.  $^1\text{H}$  NMR data for (I) at 250 MHz in  $\text{CDCl}_3$ 

$\delta$ (p.p.m.)	Multiplicity	$J$ (Hz)	Assignment
13.631 (1H)	s		H(O7)
6.196 (1H)	s		H(6)
6.133 (1H)	s		H(O5)
3.785 (1H)	ddq	$J_{15,18} = 6.4$ $J_{15,16A} = 6.5$ $J_{15,16B} = 5.9$	H(15)
3.301 (1H)	ddt	$J_{4,9} = 6.1$ $J_{4,3ax} = 6.1$ $J_{4,3eq} = 1.7$	H(4)
2.920 (1H)	dd	$J_{3eq,3ax} = 16.0$ $J_{3eq,4} = 1.7$	H(3)eq
2.700 (1H)	dd	$J_{3ax,3eq} = 16.0$ $J_{3ax,4} = 6.1$	H(3)ax
1.899 (1H)	ddq	$J_{16B,16A} = 14.8$ $J_{16B,15} = 5.9$ $J_{16B,17} = 7.4$	H(16B)
1.80–1.40 (3H)	m		H(9),H(16A)
1.40–1.20 (6H)	m		H(10)–H(12)
1.162 (3H)	d	$J_{18,15} = 6.4$	H(18)
0.986 (3H)	t	$J_{17,16} = 7.4$	H(17)
0.869 (3H)	t	$J_{13,12} = 6.8$	H(13)

torsion angles about C(3)–C(4) between the equatorial H(4) and the axial and equatorial H(3) are  $-55$  and  $65^\circ$  respectively. These angles may be compared with those obtained from a  $^1\text{H}$  NMR spectrum run at 250 MHz (Table 3) which showed the coupling constants between H(3) and H(4) to be 1.7 ( $J_{\text{eq,ax}}$ ) and 6.1 Hz ( $J_{\text{eq,ax}}$ ). Application of the Karplus equation (Williams & Fleming, 1980) gave calculated angles of  $61.1$  and  $30.0^\circ$ , respectively. The marked difference between observed and calculated  $J$  values is almost certainly attributable to the influence of the electro-negative carbonyl at C(2) being greater on one of the C(3) protons, a factor which can lead to large changes in  $J$  (Williams & Fleming, 1980).

H(9A) from the side chain is in van der Waals contact, 2.4 Å, with both H(3A) and H(4) from the ring. This proximity did not appear to significantly influence coupling patterns in the  $^1\text{H}$  spectrum, which revealed only the anticipated double doublets for the H(3) protons and an overlapping doublet of doublets of triplets for the interaction of H(4) with the H(3) and H(9) protons.

With respect to the least-squares plane through the benzene ring\* the directly attached O(1) and C(4) are almost coplanar, the C(2)–O(2) bond is parallel but displaced, C(3) is displaced further on the same side, and the *n*-pentyl side chain is displaced on the opposite side of the lactone ring.

The dihydroxyphenylketone unit is coplanar to within 0.08 (1) Å. Its C=O bond is long and weak [1.233 (5) Å], justifying the attribution (Crichton & Waterman, 1978) of the IR absorption at  $1650\text{ cm}^{-1}$  to it and the one at  $1780\text{ cm}^{-1}$  to the C(2)–O(2) bond [1.199 (4) Å]. The weakness of the C(14)–O(14) bond can be attributed both to hydrogen bonding and to

electron donation from the nearby phenolic hydroxyl at C(7). The extent of this donation from O(7) is seen both in the partial double-bond character of C(7)–O(7) at 1.327 (4) Å and in the extreme deshielding of H(O(7)) at  $\delta = 13.63$  p.p.m. in the  $^1\text{H}$  NMR spectrum. By contrast C(5)–O(5) is 1.354 (4) Å and H(O(5)) resonates at  $\delta = 6.13$  p.p.m. The intramolecular hydrogen bond O(7)···O(14) is short [2.494 (5) Å] and strong and the angle O(7)–H(O(7))···O(14) is  $144^\circ$ . A similar pattern of weak C=O bonding (average distance 1.250 Å) and strong intramolecular O–H···O=C hydrogen bonding (average O···O 2.550 Å) was noted in the two independent determinations of 2,4-dihydroxybenzophenone (Liebich, 1979; Schlemper, Hussain & Ross, 1981); its C=O stretch occurs near  $1630\text{ cm}^{-1}$  (Pouchert, 1970). In contrast, acetophenone, with no hydrogen-bonding OH groups, has a C=O distance (Tanimoto, Kobayashi, Nagakura & Saito, 1973) of 1.216 Å and a C=O stretch (Jones, Forbes & Mueller, 1957) at  $1691\text{ cm}^{-1}$ .

The presence of two separate resonances for the H(16) protons in the NMR spectrum (Table 3) implies that the C(16) methylene group cannot rotate freely. This unusual finding is probably a consequence of the crowding of the 2-methylbutyryl group against the dihydrocoumarin moiety (Fig. 1). The C(8)–C(14) bond is locked by the strong intramolecular O(7)–H···O(14) hydrogen bond which persists in solution. The C(14)–C(15) bond appears also to lack freedom to rotate. A very short H(15)···O(1) contact fulfils the geometrical criteria for a C–H···O hydrogen bond [C···O distance 2.786 (6), H···O 2.13 Å, C–H···O  $117^\circ$ ] although the presence of sufficient electrostatic interaction is unlikely. Any decrease in the C(8)–C(14)–C(15)–H(15) torsion angle would be strongly resisted by H(15) and O(1); any increase would diminish the methyl C(18)···O(1) contact distance from 3.209 (7) Å to unacceptable values. The magnitudes of the C(8)–C(14)–C(15)–C(18) and O(14)–C(14)–C(15)–C(18) torsion angles are near  $90^\circ$ . Thus any interference of C(18) with C(8) and O(14) is at a minimum. Rotation about the C(15)–C(16) bond is limited by the proximity of O(14) and C(18). The conformation adopted in the crystal structure evidently reduces hindrance of C(17) to the minimum possible. It places H(16B) within the deshielding influence of O(14) unlike H(16A). A less favourable possibility has C(18)–C(15)–C(16)–C(17) +synclinal with greater methyl-group interference. In this case, too, one H atom, now H(16A), is near O(14) and the other is not. However, the third alternative with C(18)–C(15)–C(16)–C(17) –synclinal is not available because C(17) is too bulky to fit between O(14) and C(18), which are only 3.143 (7) Å apart.

The packing of molecules (Fig. 2) allows an intermolecular O(5)–H(O(5))···O(2) hydrogen bond to form at an O···O distance of 2.721 (5) Å and an O–H–O angle of  $155^\circ$ . The *n*-pentyl side chain of a

\* See previous footnote.

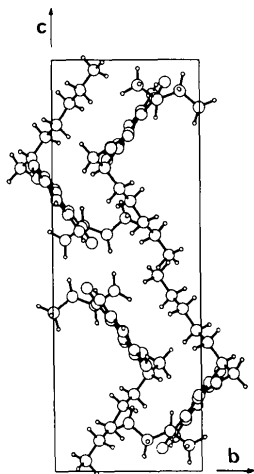


Fig. 2. PLUTO (Motherwell, 1972) drawing of the packing of molecules in the unit cell.

molecule related by  $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$  is almost parallel to the benzene ring with transformed C(12) 3.693 (7) Å above a point approximately midway between C(5) and C(7). The operation  $2 - x, -\frac{1}{2} + y, \frac{1}{2} - z$  produces

contacts on the opposite side of the ring between C(6) and transformed C(4) of 3.470 (5) Å, and between C(8A) and transformed O(5) of 3.628 (5) Å. Thus the 'L-shaped' molecules can fill space efficiently.

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### Structure of 2-Amino-3,5-dibromo-*N*-cyclohexyl-*N*-methylbenzenemethanamine-1,2-Benzisothiazol-3(2*H*)-one 1,1-Dioxide (1:1), C<sub>14</sub>H<sub>20</sub>Br<sub>2</sub>N<sub>2</sub>·C<sub>7</sub>H<sub>5</sub>NO<sub>3</sub>S

BY NOBUYUKI SHIMIZU AND SADA O NISHIGAKI

Pharmaceutical Institute, School of Medicine, Keio University, Shinjuku-ku, Tokyo 160, Japan

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**Abstract.**  $M_r = 559.3$ , orthorhombic,  $Pbca$ ,  $a = 15.374$  (2),  $b = 25.330$  (4),  $c = 11.630$  (13) Å,  $U = 4529.0$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.641$ ,  $D_m = 1.632$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 4.03$  mm<sup>-1</sup>,  $F(000) = 2256$ . Final  $R = 0.095$  for 2577 observed reflections. 2-Amino-3,5-dibromo-*N*-cyclohexyl-*N*-methylbenzenemethanamine (ABCMBMA) and 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (*o*-sulfobenzoimide, OSBI) are alternately linked by hydrogen bonds [N(1)–H(1)···O(2), N(2)–H···N(3)] and a permanent dipole–induced-dipole interaction to form an infinite chain in the *a* direction.

**Introduction.** ABCMBMA is an expectorant drug and OSBI is an artificial sweetener. This investigation was undertaken as part of a series of our structural studies of molecular complexes between different drugs.

**Experimental.** Suitable crystals (m.p. 459 K) for diffraction analysis grown *via* slow evaporation of an ethanol solution of equimolar quantities of ABCMBMA and OSBI,  $D_m$  measured by flotation in CCl<sub>4</sub>/CHBr<sub>3</sub>, crystal 0.2 × 0.3 × 0.4 mm; preliminary Weissenberg photographs revealed systematic absences  $0kl$  for  $k = 2n + 1$ ,  $h0l$  for  $l = 2n + 1$ ,  $hk0$  for  $h = 2n + 1$ , and indicated the centrosymmetric orthorhombic space group  $Pbca$  (No. 61); cell dimensions derived from least-squares treatment of the setting angles for 18 reflections, Rigaku four-circle diffractometer, Mo  $K\alpha$  radiation, graphite monochromator, no correction made for absorption; one independent octant of data out to  $2\theta = 55^\circ$  collected using  $\omega$ – $2\theta$  scan technique; three reference reflections (measured periodically) showed no significant variation in their intensities; 2577 reflections considered observed [ $F_o \geq 3\sigma(F_o)$ ]; structure