actions between O and N lone pairs and C–S bond lengths (Orrell & Wallis, 1984). In that study, a lengthening of the C–S bond to 1·880 (2) Å was attributed to the anomeric interaction between the N lone pair and the σ^* of the C–S. Nevertheless it was mentioned that this effect was combined with a conjugation of the S with the acrylic ester function that increased the polarization of the C–S bond.

A lengthening of the C–S bond up to 1·830 (5) Å was obtained for compounds in which an O lone pair was approximately antiperiplanar to the C–S bond (Soboleva, D'Yachenko, Atovmyan, Kharchenko & Klimenko, 1978). A corresponding shortening of the C–O bond to 1·425 (5) Å was also observed.

The particular *trans–cis* configuration of (IV) places a C–S bond in an axial orientation and susceptible to the anomeric effect. The torsion angle S(6)–C(7)–S(8)–C(9) of 73·4 (4)° observed in (IV) is such that the S lone pair of S(8) is antiperiplanar to the S(6)–C(7) bond. A resulting shortening of the S(8)–C(7) bond would be expected as well as an elongation of the S(6)–C(7) bond length. Observed values: 1·800 (6) and 1·822 (7) respectively are not significantly different from the 1·810 Å obtained for a standard C–S bond length; the differences are of the order of the standard deviations. No significant intermolecular interactions have been observed: packing is due to van der Waals contacts.

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Structure of Isomammeigin – a New Phenylcoumarin from Guttiferae Species

BY E. E. CASTELLANO AND J. ZUKERMAN-SCHPECTOR*

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 *São Carlos, SP, Brazil*

AND M. DE ABREU E SILVA AND T. J. NAGEM

Departamento de Química – ICEX, Universidade Federal de Minas Gerais, 31270 *Belo Horizonte, MG, Brazil*

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Abstract. $C_{25}H_{24}O_5$, $M_r = 404{\cdot}47$, triclinic, $P\bar{1}$, $a = 9{\cdot}487$ (2), $b = 9{\cdot}617$ (5), $c = 12{\cdot}397$ (3) Å, $\alpha = 77{\cdot}28$ (3), $\beta = 70{\cdot}04$ (2), $\gamma = 82{\cdot}43$ (2)°, $V = 1035{\cdot}1$ (7) Å^3, $Z = 2$, $D_x = 1{\cdot}30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·08 mm^{-1}, $F(000) = 428$, $T = 296$ K, $R = 0{\cdot}060$ for 1329 reflections. A new phenylcoumarin isolated from *Kilmeyera pumila* Pohl is shown to be 5-hydroxy-6-isovaleryl-2,2-dimethyl-10-phenyl-2*H*,8*H*-benzo[1,2-*b*:3,4-*b'*]dipyran-8-one. There is an intramolecular O(2)···O(5) hydrogen bond of 2·445 (5) Å.

* Present address: Laboratório de Cristalografia e Físico-Química dos Materiais/CCEN, Universidade Federal de Alagoas, 57000 Maceió, AL, Brazil.

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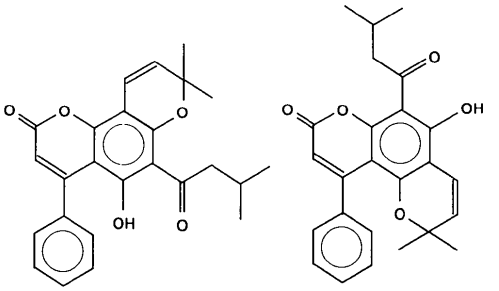
Introduction. Chemical investigation of the fruit of *Kilmeyera pumila* Pohl (family Guttiferae) collected in Minas Gerais, Brazil, led us to the isolation of two phenylcoumarins (I) and (II). Compound (I), named mammeigin, had already been isolated from *Mammea americana* L. (Finnegan & Mueller, 1964) and its structure established on spectroscopic evidence and on chemical correlation (Finnegan & Mueller, 1965). It was pointed out in that work that, from spectroscopic data alone, structures (I) and (II) were possible alternatives for mammeigin. From IR and NMR data (de Abreu e Silva, 1987) structure (II) was proposed for the new compound, named isomammeigin. To verify this hypothesis and to establish its molecular con-

formation unambiguously a crystal structure determination of the title compound was undertaken.



(I) (II)

Experimental. Irregular pale yellow crystals were obtained from acetone, max. and min. linear dimensions 0·30, 0·10 mm, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; cell parameters by least squares on setting angles for 20 reflections; $10 < \theta < 17°$; ω–2θ scans, scan width $(0{\cdot}80 + 0{\cdot}35\tan\theta)°$, max. scan speed 5° min^{-1}, range of hkl: $-9 < h < 9$, $-9 < k < 10$, $0 < l < 13$, standard reflection 500, varied ±3·2% of mean intensity over data collection; 2629 reflections measured, 2525 unique, $R_{int} = 0{\cdot}017$, 1329 observed above $3\sigma(I)$; Lp corrections. The structure was solved by direct methods; in final cycles of full-matrix least-squares refinement all non-H atoms anisotropic. H atoms located on difference maps, not refined; methyl H atoms refined with independent isotropic temperature factor. Function minimized $\sum w(|F_o| - |F_c|)^2$ with $w = 1/[\sigma^2(F_o) + (0{\cdot}0006|F_o|)^2]$, 275 parameters refined; unobserved reflections excluded; $R = 0{\cdot}060$, $wR = 0{\cdot}066$; inspection of F_c and F_o values indicated secondary-extinction correction required: $F_{corr} = F_c\,(1 - \chi F_c^2/\sin\theta)$, where χ refined to $1{\cdot}6 \times 10^{-6}$ in the final run, $(\Delta/\sigma)_{max} = 0{\cdot}05$, $\Delta\rho$ excursions within 0·22 and −0·25 e Å^{-3}. Scattering factors for non-H atoms from Cromer & Mann (1968) with anomalous dispersion from Cromer & Liberman (1970) and for H from Stewart, Davidson & Simpson (1965); programs used *SHELX*76 (Sheldrick, 1976), *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/780 computer from the Instituto de Física e Química de São Carlos.

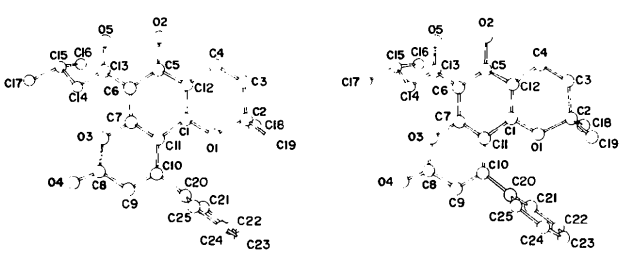


Fig. 1. Stereoscopic projection of the molecule showing the system of nomenclature.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*
C(1)	−0·0484 (6)	−0·2783 (5)	0·1010 (4)	2·9 (3)
C(2)	−0·0252 (6)	−0·0456 (5)	0·2647 (4)	3·4 (4)
C(3)	0·1143 (6)	−0·3997 (7)	0·2536 (5)	3·9 (4)
C(4)	0·1766 (6)	−0·2988 (7)	0·1623 (5)	3·8 (4)
C(5)	0·1671 (5)	−0·1596 (6)	−0·0321 (5)	2·9 (3)
C(6)	0·0886 (6)	−0·1014 (5)	−0·1130 (4)	2·7 (3)
C(7)	−0·0640 (6)	−0·1326 (6)	−0·0757 (5)	2·9 (3)
C(8)	−0·2922 (6)	−0·0834 (6)	−0·1249 (5)	3·8 (4)
C(9)	−0·3622 (6)	−0·1855 (7)	−0·0228 (5)	3·9 (4)
C(10)	−0·2887 (6)	−0·2517 (6)	0·0512 (5)	3·1 (3)
C(11)	−0·1370 (6)	−0·2215 (6)	0·0282 (5)	2·7 (3)
C(12)	0·0994 (6)	−0·2483 (6)	0·0756 (5)	3·1 (4)
C(13)	0·1736 (5)	−0·0219 (6)	−0·2284 (5)	3·2 (3)
C(14)	0·1147 (6)	0·0223 (6)	−0·3293 (5)	3·8 (4)
C(15)	0·2338 (6)	0·0610 (6)	−0·4470 (5)	4·1 (4)
C(16)	0·3183 (7)	−0·0735 (7)	−0·4849 (6)	6·8 (5)
C(17)	0·1629 (7)	0·1457 (7)	−0·5373 (5)	5·8 (4)
C(18)	0·0116 (7)	−0·5959 (6)	0·2075 (6)	5·8 (5)
C(19)	−0·1311 (6)	−0·4995 (6)	0·3888 (5)	5·1 (4)
C(20)	−0·3737 (6)	−0·3556 (7)	0·1524 (5)	3·5 (4)
C(21)	−0·3751 (7)	−0·4927 (8)	0·1411 (6)	5·5 (5)
C(22)	−0·4557 (8)	−0·5951 (7)	0·2360 (7)	6·5 (5)
C(23)	−0·5318 (7)	−0·5527 (9)	0·3395 (6)	5·9 (5)
C(24)	−0·5329 (7)	−0·4168 (8)	0·3499 (6)	5·2 (5)
C(25)	−0·4567 (6)	−0·3165 (6)	0·2584 (6)	4·2 (4)
O(1)	−0·1183 (3)	−0·3619 (3)	0·2061 (3)	3·6 (2)
O(2)	0·3099 (3)	−0·1305 (4)	−0·0549 (3)	3·6 (2)
O(3)	−0·1413 (3)	−0·0656 (3)	−0·1493 (3)	3·4 (2)
O(4)	−0·3493 (4)	−0·0120 (5)	−0·1946 (3)	5·5 (3)
O(5)	0·3036 (4)	0·0091 (4)	−0·2438 (3)	4·4 (2)

* $B_{eq} = \frac{4}{3}\sum_i\sum_j(\mathbf{a}_i.\mathbf{a}_j)B_{ij}$.

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non-hydrogen atoms with e.s.d.'s in parentheses*

C(1)–C(11)	1·414 (8)	C(8)–O(3)	1·384 (7)
C(1)–C(12)	1·383 (9)	C(8)–O(4)	1·213 (7)
C(1)–O(1)	1·369 (6)	C(9)–C(10)	1·340 (8)
C(2)–C(3)	1·493 (9)	C(10)–C(11)	1·425 (8)
C(2)–C(18)	1·517 (8)	C(10)–C(20)	1·481 (8)
C(2)–C(19)	1·514 (8)	C(13)–C(14)	1·496 (8)
C(2)–O(1)	1·480 (7)	C(13)–O(5)	1·248 (7)
C(3)–C(4)	1·335 (9)	C(14)–C(15)	1·513 (8)
C(4)–C(12)	1·463 (8)	C(15)–C(16)	1·508 (9)
C(5)–C(6)	1·424 (8)	C(15)–C(17)	1·529 (9)
C(5)–C(12)	1·407 (8)	C(20)–C(21)	1·36 (1)
C(5)–O(2)	1·341 (7)	C(20)–C(25)	1·386 (9)
C(6)–C(7)	1·413 (8)	C(21)–C(22)	1·42 (1)
C(6)–C(13)	1·475 (7)	C(22)–C(23)	1·36 (1)
C(7)–C(11)	1·390 (8)	C(23)–C(24)	1·34 (1)
C(7)–O(3)	1·363 (7)	C(24)–C(25)	1·372 (9)
C(8)–C(9)	1·435 (8)		

C(11)–C(1)–C(12)	124·4 (5)	C(9)–C(10)–C(20)	116·0 (5)
C(11)–C(1)–O(1)	116·7 (5)	C(11)–C(10)–C(20)	124·4 (5)
C(12)–C(1)–O(1)	118·8 (5)	C(1)–C(11)–C(7)	115·1 (5)
C(3)–C(2)–C(18)	111·3 (5)	C(1)–C(11)–C(10)	125·7 (5)
C(3)–C(2)–C(19)	114·1 (5)	C(7)–C(11)–C(10)	119·1 (5)
C(3)–C(2)–O(1)	110·0 (4)	C(1)–C(12)–C(4)	120·4 (5)
C(18)–C(2)–C(19)	111·2 (5)	C(1)–C(12)–C(5)	117·4 (5)
C(18)–C(2)–O(1)	107·6 (4)	C(4)–C(12)–C(5)	122·0 (5)
C(19)–C(2)–O(1)	102·2 (4)	C(6)–C(13)–C(14)	124·1 (5)
C(2)–C(3)–C(4)	121·6 (5)	C(6)–C(13)–O(5)	118·2 (5)
C(3)–C(4)–C(12)	117·9 (5)	C(14)–C(13)–O(5)	117·7 (5)
C(6)–C(5)–C(12)	122·2 (5)	C(13)–C(14)–C(15)	114·7 (5)
C(6)–C(5)–O(2)	121·1 (5)	C(14)–C(15)–C(16)	109·3 (5)
C(12)–C(5)–O(2)	116·7 (5)	C(14)–C(15)–C(17)	110·6 (5)
C(5)–C(6)–C(7)	115·6 (5)	C(16)–C(15)–C(17)	111·1 (5)
C(5)–C(6)–C(13)	118·1 (5)	C(10)–C(20)–C(21)	119·6 (6)
C(7)–C(6)–C(13)	126·2 (5)	C(10)–C(20)–C(25)	122·0 (6)
C(6)–C(7)–C(11)	125·0 (5)	C(21)–C(20)–C(25)	118·4 (6)
C(6)–C(7)–O(3)	115·0 (5)	C(20)–C(21)–C(22)	121·2 (7)
C(11)–C(7)–O(3)	120·0 (5)	C(21)–C(22)–C(23)	118·3 (7)
C(9)–C(8)–O(3)	117·0 (5)	C(22)–C(23)–C(24)	120·5 (7)
C(9)–C(8)–O(4)	127·4 (6)	C(23)–C(24)–C(25)	121·7 (7)
O(3)–C(8)–O(4)	115·5 (5)	C(20)–C(25)–C(24)	119·9 (6)
C(8)–C(9)–C(10)	121·6 (5)	C(1)–O(1)–C(2)	118·6 (4)
C(9)–C(10)–C(11)	119·6 (5)	C(7)–O(3)–C(8)	122·2 (4)

Discussion. Fig. 1 is a stereoscopic projection of the molecule showing the system of nomenclature. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Interatomic bond distances and angles are given in Table 2; all these values are within the expected range. Atoms O(2) and O(5) are involved in an intramolecular hydrogen bond as suggested by the O(2)···O(5) distance of 2·445 (5) Å. The shortest intermolecular contact between non-hydrogen atoms is O(2)···O(4^{i}) = 3·321 (5) Å [(i): $1+x, y, z$)].

The main result of the present study is that the three-dimensional structure of isomammeigin is now unambiguously determined.

* 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 51125 (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 Suriclone, a Benzodiazepine Receptor Agonist

BY PENELOPE W. CODDING,* MARIA B. SZKARADZINSKA AND NORMA E. DUKE

Departments of Chemistry and of Pharmacology and Therapeutics, University of Calgary, Calgary, Alberta, Canada T2N 1N4

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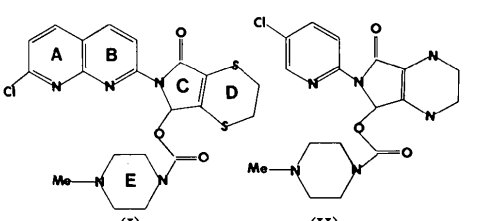
Abstract. 6-(7-Chloro-1,8-naphthyridin-2-yl)-2,3,6,7-tetrahydro-4-methyl-7-oxo-5*H*-1,4-dithiino[2,3-*c*]-pyrrol-5-yl-1-piperazinecarboxylic acid, $C_{20}H_{20}ClN_5O_3S_2$, $M_r = 477{\cdot}9$, triclinic, $P\bar{1}$, $a = 8{\cdot}7066$ (3), $b = 9{\cdot}7665$ (8), $c = 14{\cdot}2515$ (16) Å, $\alpha = 80{\cdot}986$ (9), $\beta = 75{\cdot}168$ (6), $\gamma = 65{\cdot}884$ (5)°, $V = 1067{\cdot}4$ (2) Å^3, $Z = 2$, $F(000) = 496$, room temperature, $D_m = 1{\cdot}482$, $D_x = 1{\cdot}487$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, Ni filter, $\mu = 36{\cdot}3$ cm^{-1}, $R = 0{\cdot}053$, $wR = 0{\cdot}070$ for the 3538 reflections included in the refinement. Comparisons of the structures of the two enantiomers of suriclone and the active conformer of the 1,4-benzodiazepine anxiolytics allow the identification of the active form of suriclone as the *R* isomer.

Introduction. The existence of specific benzodiazepine (BZD) receptors in various mammalian brain tissues is well documented. The receptors are linked to γ-aminobutyric acid (GABA) receptor sites which control chloride anion channels, and benzodiazepines elicit their biological actions *via* allosteric modulation of the GABA receptor. Other chemical classes of drugs different from the benzodiazepines bind tightly to the BZD receptor; examples include pyrazoloquinolinones, thiazolopyridazines, quinolines and the cyclopyrrolones, suriclone (I) and zopiclone (II). The cyclopyrrolones are the only non-1,4-benzodiazepine drugs that simultaneously exhibit high potency in displacing benzodiazepines from their binding sites and the diverse pharmacological properties of agonist benzodiazepine receptor ligands (Zundel, Blanchard & Julou, 1985).



(I) (II)

* Author to whom correspondence should be addressed.

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