

Ranges of h , k , l : 0 to 17, 0 to 12, -24 to 24, respectively. Three standard reflections monitored every half an hour: variation on $I < 3\%$. No correction for absorption. 3617 unique reflections, 1111 observed with $I > 2\sigma(I)$. $R = 4.86\%$, $wR = 2.90\%$, $S = 1.72$. Weighting scheme from counting statistics. The structure solution was attempted with direct methods both in $C2/c$ and Cc using the *MULTAN* program and solved in Cc with 339 highest E^2 's, 93 smallest E^2 's and 4486 Σ_2 relationships. The space group was then changed to $C2/c$ owing to the correlation of the two molecules in the asymmetric unit. $\Sigma w(\Delta F)^2$ minimized. H atoms found in difference Fourier map after isotropic refinement and then refined. $(\Delta/\sigma)_{\max} = 0.87$. Peaks in final difference Fourier map $< \pm 0.18 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: NRCC SDP PDP-11 package (Gabe & Lee, 1981), *MULTAN* and *ORTEP* from Enraf-Nonius (1979) *SDP*.

Atomic parameters are given in Table 1,* bond distances and angles in Table 2. A drawing of the molecule is shown in Fig. 1.

Related literature. The bond distances and angles of the (3,4-methylenedioxy)benzyl(idene) moieties are quite similar to those of piperine (Grynpas & Lindley, 1975) and other derivatives (Herbstein, Schwotzer, Addae-Mensah, Torto & Woode, 1981; Begley, Crombie,

* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43679 (26 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 (+)-Calocedrin

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Abstract. 5-Hydroxy-4-piperonyl-3-piperonylidene-tetrahydrofuran-2-one, $C_{20}H_{16}O_7$, $M_r = 368$, monoclinic, $P2_1/c$, $a = 10.974(2)$, $b = 21.045(4)$, $c = 7.325(2) \text{ \AA}$, $\beta = 92.03(2)^\circ$, $V = 1690(2) \text{ \AA}^3$, $Z = 4$, $D_m = 1.43$, $D_x = 1.45 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$, $\mu = 1.0 \text{ cm}^{-1}$, $F(000) = 768$, $T = 298 \text{ K}$, final $R = 0.045$ for 1503 observed reflections. The structure contains three planar parts as in (-)-savinin [Wang, Cheng, Jan & Cheng (1987). *Acta Cryst.* C43, 1005–1006]: two (3,4-methylenedioxy)benzyl(idene)

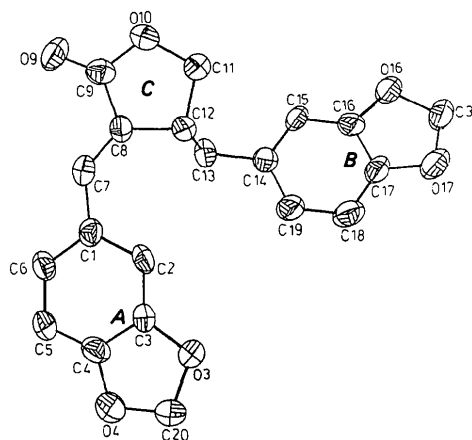


Fig. 1. ORTEP drawing of the molecule with 50% probability thermal ellipsoids.

Havard & Reynolds, 1977; Desiraju, Kamala, Kumari & Sarma, 1984).

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moieties (A and B) and a 5-hydroxytetrahydrofuran-2-one ring (C). The dihedral angles between them are A&B: 8.00(2), A&C: 7.77(2), B&C: 13.88(1)°. The compound can be reduced to (+)-savinin. There is intermolecular hydrogen bonding through the hydroxyl H atom and the ketone O atom, with an O...O distance of 2.721(6) Å.

Experimental. This is a new compound isolated from the wood of *Calocedrus formosana*. It was charac-

terized by spectroscopic methods (Fang, Jan & Cheng, 1985).

Crystal $0.1 \times 0.2 \times 0.5$ mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range 18.98 to 24.54° . D_m by flotation (n -hexane/ CCl_4). $2\theta_{\text{max}} = 50^\circ$. Ranges of h, k, l : 0 to $13, 0$ to $25, -8$ to 8 , respectively. Three standard reflections monitored every half an hour: variation on $I < 3\%$. No correction for absorption. 3153 unique reflections, 1503 observed with $I > 2\sigma(I)$. $R = 4.53\%$, $wR = 4.98\%$, $S = 1.72$. Weight $w = 1/[\sigma^2(F_o) + 0.01F_o^2]$. Structure solved by direct methods using the *MULTAN* program with 284 highest E 's, 89 smallest E 's and 3691 \sum_2 relationships. $\sum w(\Delta F)^2$ minimized. H atoms found in difference Fourier map after isotropic refinement and then refined. $(\Delta/\sigma)_{\text{max}} = 0.14$. Peaks in final difference Fourier map $< \pm 0.16 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: NRCC *SDP* PDP-11 package (Gabe & Lee, 1981), *MULTAN* and *ORTEP* from Enraf-Nonius (1979) *SDP*.

Atomic parameters are given in Table 1,* bond distances and angles in Table 2. A drawing of the molecule is shown in Fig. 1.

Related literature. The molecular structure is comparable with that of the reduced form (Wang *et al.*, 1987, and references therein). Similar intermolecular hydrogen bonding was observed in the planar structure of (3,4-methylenedioxy)cinnamic acid through the ethylenic H atom and the carboxylic O atom (Desiraju, Kamala, Kumari & Sarma, 1984).

* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43680 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

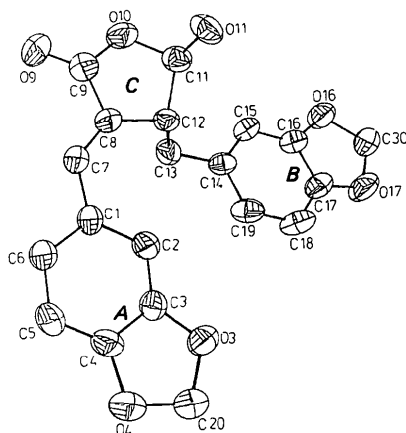


Fig. 1. *ORTEP* drawing of the molecule with 50% probability thermal ellipsoids.

Table 1. *Atomic fractional coordinates and equivalent isotropic temperature factors* (\AA^2)

$$B_{\text{eq}} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
C1	0.4007 (3)	0.1603 (2)	0.7750 (5)	3.6 (2)
C2	0.4061 (3)	0.0948 (2)	0.8138 (5)	3.8 (2)
C3	0.2982 (3)	0.0652 (2)	0.8421 (5)	3.5 (2)
C4	0.1883 (3)	0.0970 (2)	0.8394 (5)	3.9 (2)
C5	0.1805 (3)	0.1606 (2)	0.8074 (6)	4.9 (3)
C6	0.2890 (3)	0.1918 (2)	0.7728 (6)	4.3 (2)
C7	0.5071 (3)	0.1984 (2)	0.7331 (5)	3.8 (2)
C8	0.6245 (3)	0.1823 (2)	0.7229 (5)	3.5 (2)
C9	0.7135 (3)	0.2311 (2)	0.6816 (5)	4.4 (2)
O10	0.8277 (2)	0.2064 (1)	0.6872 (4)	4.9 (2)
C11	0.8250 (3)	0.1430 (2)	0.7717 (5)	4.0 (2)
C12	0.6917 (3)	0.1208 (2)	0.7455 (5)	3.2 (2)
C13	0.6753 (3)	0.0776 (2)	0.5741 (5)	3.9 (2)
C14	0.7142 (3)	0.0096 (2)	0.6069 (5)	3.6 (2)
C15	0.8346 (3)	-0.0092 (2)	0.5796 (5)	3.7 (2)
C16	0.8644 (3)	-0.0714 (2)	0.6147 (5)	3.8 (2)
C17	0.7817 (3)	-0.1142 (2)	0.6759 (5)	4.2 (2)
C18	0.6633 (4)	-0.0975 (2)	0.7027 (6)	5.0 (2)
C19	0.6311 (3)	-0.0345 (2)	0.6678 (5)	4.4 (2)
C20	0.1525 (3)	-0.0050 (2)	0.9096 (6)	4.7 (2)
C30	0.9602 (4)	-0.1643 (2)	0.6611 (6)	5.6 (3)
O3	0.2793 (2)	0.0020 (1)	0.8775 (4)	4.8 (2)
O4	0.0958 (2)	0.0550 (1)	0.8711 (4)	5.6 (2)
O9	0.6980 (2)	0.2868 (1)	0.6429 (4)	5.8 (2)
O11	0.8656 (2)	0.1483 (1)	0.9513 (4)	4.9 (2)
O16	0.9756 (2)	-0.1009 (1)	0.5940 (4)	5.2 (2)
O17	0.8361 (2)	-0.1728 (1)	0.6969 (4)	5.6 (2)

Table 2. *Bond lengths* (\AA) *and bond angles* ($^\circ$) *of* $\text{C}_{20}\text{H}_{16}\text{O}_7$

C1	C2	1.408 (4)	C1	C6	1.393 (4)		
C1	C7	1.459 (4)	C2	C3	1.361 (4)		
C3	C4	1.379 (4)	C3	O3	1.372 (4)		
C4	C5	1.360 (5)	C4	O4	1.373 (4)		
C5	C6	1.392 (5)	C7	C8	1.337 (4)		
C8	C9	1.457 (4)	C8	C12	1.496 (4)		
C9	O10	1.356 (4)	C9	O9	1.216 (4)		
O10	C11	1.471 (4)	C11	C12	1.542 (4)		
C11	O11	1.378 (4)	C12	C13	1.555 (4)		
C13	C14	1.510 (4)	C14	C15	1.400 (4)		
C14	C19	1.386 (4)	C15	C16	1.372 (4)		
C16	C17	1.366 (4)	C16	O16	1.383 (4)		
C17	C18	1.367 (5)	C17	O17	1.378 (4)		
C18	C19	1.394 (5)	C20	O3	1.427 (4)		
C20	O4	1.431 (4)	C30	O16	1.434 (4)		
C30	O17	1.408 (4)					
C2	C1	C6	120.1 (3)	C2	C1	C7	123.5 (2)
C6	C1	C7	116.4 (2)	C1	C2	C3	116.6 (3)
C2	C3	C4	122.7 (3)	C2	C3	O3	127.6 (3)
C4	C3	O3	109.6 (2)	C3	C4	C5	122.0 (3)
C3	C4	O4	109.6 (3)	C5	C4	O4	128.3 (3)
C4	C5	C6	116.5 (3)	C1	C6	C5	122.0 (3)
C1	C7	C8	130.7 (3)	C7	C8	C9	119.1 (3)
C7	C8	C12	133.3 (3)	C9	C8	C12	107.6 (2)
C8	C9	O10	110.4 (2)	C8	C9	O9	129.7 (3)
O10	C9	O9	119.9 (3)	C9	O10	C11	109.2 (2)
O10	C11	C12	104.8 (2)	O10	C11	O11	108.6 (2)
C12	C11	O11	114.6 (3)	C8	C12	C11	102.3 (2)
C8	C12	C13	112.0 (2)	C11	C12	C13	111.1 (2)
C12	C13	C14	113.7 (2)	C13	C14	C15	120.5 (2)
C13	C14	C19	120.0 (3)	C15	C14	C19	119.5 (3)
C14	C15	C16	117.6 (3)	C15	C16	C17	122.3 (3)
C15	C16	O16	127.9 (3)	C17	C16	O16	109.8 (3)
C16	C17	C18	121.6 (3)	C16	C17	O17	109.7 (3)
C18	C17	O17	128.6 (3)	C17	C18	C19	117.0 (3)
C14	C19	C18	122.0 (3)	O3	C20	O4	107.3 (2)
O16	C30	O17	108.0 (2)	C3	O3	C20	106.6 (2)
C4	O4	C20	106.4 (2)	C16	O16	C30	105.4 (2)

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Structure of an Antigelling Agent, L-Phenylalanyl-glycyl-glycyl-D-phenylalanine Trihydrate

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Abstract. L-Phenylalanyl-glycyl-glycyl-D-phenylalanine trihydrate, $C_{22}N_4O_5H_{26} \cdot 3H_2O$, $M_r = 480.5$, monoclinic, $P2_1$, $a = 5.787$ (1), $b = 11.787$ (2), $c = 17.610$ (2) Å, $\beta = 104.52$ (1)°, $V = 1162.7$ (1) Å³, $Z = 2$, $D_x = 1.372$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 7.62$ cm⁻¹, $F(000) = 512$, $T = 283$ K, $R = 0.058$ for 1212 unique observed reflections. The molecule has adopted a compact and amphipathic conformation. Peptide torsion angles: L-Phe1: $\psi = -120.6$ (9), $\omega = -171.7$ (8), $\chi^1 = 172.4$ (9), $\chi^{2.1} = 55.5$ (10); Gly2: $\phi = -109.6$ (9), $\psi = -12.9$ (9), $\omega = 180.0$ (8); Gly3: $\phi = -92.1$ (7), $\psi = 149.8$ (8), $\omega = -175.6$ (7); D-Phe4: $\phi = 73.2$ (8), $\psi_T = -34.2$ (7), $\chi^1 = 62.7$ (7), $\chi^{2.1} = 52.9$ (9)°. Intramolecular edge-to-face interaction between phenyl rings: phenyl(L-Phe1)—phenyl(D-Phe4) centroid separation = 5.12 (1) Å and dihedral angle = 76.9 (7)°. Intermolecular hydrogen bonds: N(L-Phe1)—H...O(Gly2) = 2.853 (10), N(L-Phe1)—H...O(1)(D-Phe4') = 2.787 (10), N(L-Phe1)—H...O(W2) = 3.042 (10), O(L-Phe1)...H—O(W3) = 2.801 (12), N(Gly2)—H...O(W3) = 2.918 (13), N(Gly3)—H...O(W3) = 2.979 (13), N(D-Phe4)—H...O2(D-Phe4') = 2.900 (10), O(2)(D-Phe4)...H—O(W2) = 2.610 (12), and O(W2)—O(W3) =

2.770 (12) Å. Intermolecular edge-to-face interaction between phenyl rings: phenyl(L-Phe1)—phenyl(D-Phe4') centroid separation = 4.78 (1) Å and dihedral angle = 54.3 (7)°. Finally, there is evidence of static disorder and/or increased thermal motion of waters 1 and 2, and the atoms N(Gly2), CA(Gly2), C(Gly2) and O(Gly2), which may be due to dehydration of water 3 [refined occupancy = 0.31 (2)]. The atoms CA(Gly2) and C(Gly2) make unreasonably short contacts with water 3, and the hydrogen-bonding network in the polar region of the crystal is only partially satisfied.

Experimental. Thin-plate, pseudo-hexagonal crystal by vapor diffusion from 10% 2-methyl-2,4-pentanediol at neutral pH, 0.5 × 0.4 × 0.08 mm, Nicolet P3 diffractometer, Ni-filtered radiation, ω -scan method, $(\sin\theta)/\lambda < 0.58$ Å⁻¹, lattice parameters from the 2θ values of 15 reflections with $34 < 2\theta < 45^\circ$, no absorption correction, $h = -6$ to 6, $k = 0$ to 10, $l = 0$ to 20, reflections 202, 036, 141, 115 and $\bar{2}06$ as intensity standards, intensity variation < 3%. 2074 unique reflections measured, 862 excluded during refinement [$F < 3\sigma(F)$]. Structure solved by direct methods (MULTAN; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), first E map revealed the positions of all but four non-H atoms, successive Fourier syntheses located a C atom and three H₂O molecules to

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