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### References

CODY, V. (1987). *Acta Cryst.* C43, 1325–1328.
CODY, V., SUWIŃSKA, K. & WOJTCZAK, A. (1991). *Acta Cryst.* C47, 156–159.
CODY, V. & WOJTCZAK, A. (1991). *Acta Cryst.* C47, 787–790.
DE TITTA, G. T., EDMONDS, J. W., LANGS, D. A. & HAUPTMAN, H. A. (1975). *Acta Cryst.* A31, 472–479.
MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

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# Structure of 2′,4′-Dihydroxy-4,6′-dimethoxy-α,β-dihydrochalcone

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**Abstract.** $C_{17}H_{18}O_5$, $M_r = 302$, monoclinic, $P2_1/c$, $a = 4{\cdot}856\ (3)$, $b = 28{\cdot}896\ (7)$, $c = 10{\cdot}776\ (3)$ Å, $\beta = 98{\cdot}04\ (4)°$, $V = 1497\ (1)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}34$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}922$ cm$^{-1}$, $F(000) = 640$, room temperature, $R = 0{\cdot}068$, $wR = 0{\cdot}074$ for 792 observed reflections [$I \geq 2{\cdot}5\sigma(I)$]. The phenyl rings are planar within experimental accuracy; the angle between the normals to the best least-squares planes of the phenyl rings is 81·49 (9)°. Intermolecular and intramolecular hydrogen bonds are present: O1···H3′—O3′ ($x-1$, $\frac{1}{2}-y$, $\frac{1}{2}+z$), 2·77 (1) Å, O2—H2···O1, 2·50 (1) Å; this interaction may contribute to the low value of the O1—C1—C2—C3 [−5·42 (9)°] and O1—C1—C11—C12 [5·63 (9)°] torsion angles. The molecular structure found is in total agreement with spectroscopic results.

**Experimental.** Transparent colourless flat prism of dimensions 0·20 × 0·08 × 0·15 mm, Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator. Unit-cell parameters were determined by least squares from 25 reflections with $3 \leq \theta \leq 13°$; 5191 reflections collected, 2632 unique, 792 considered observed with $I \geq 2{\cdot}5\sigma(I)$; $\omega$–$2\theta$ scan mode, $hkl$ range: $-5 \leq h \leq 5$, $-34 \leq k \leq 34$, $0 \leq l \leq 12$, $\theta \leq 25°$. Three standard reflections monitored every 50 measurements showed no significant decay in intensity. Lorentz–polarization corrections, but no absorption correction. Structure solved by direct methods (*MULTAN*11/84; Main, Germain & Woolfson, 1984). Anisotropic full-matrix least-squares refinement (on $F$) for non-H atoms (*SHELX*76; Sheldrick, 1976). Hydroxy H atoms were found by difference Fourier synthesis and included in the refinement, and the remaining H atoms were included at calculated positions in the final cycle and refined; two different isotropic temperature factors, one for methyl and one for non-methyl H atoms, were refined to final values $U = 0{\cdot}106\ (16)$ and $0{\cdot}121\ (13)$ Å$^2$, respectively; $R = 0{\cdot}068$, $wR = 0{\cdot}074$, $w = 1{\cdot}2689/[\sigma^2(F) + 0{\cdot}002575F^2]$ for 792 observed reflections and 207 variables. Final $(\Delta/\sigma)_{max} = 0{\cdot}11$, $S = 1{\cdot}17$. Largest peaks in final $\Delta F$ map: −0·24 and 0·19 e Å$^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Plots were made with *PLUTO* (Motherwell & Clegg, 1978). All calculations were performed on a MicroVAX II computer.

The atomic parameters are given in Table 1.† A view of the molecule with the atomic numbering scheme is shown in Fig. 1; the molecular packing is shown in Fig. 2. Table 2 gives bond distances and angles.

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† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53935 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å$^2$)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| C11 | 0·4923 (20) | 0·2231 (3) | 1·0850 (8) | 3·4 |
| C12 | 0·4386 (20) | 0·2717 (4) | 1·1085 (9) | 3·5 |
| O2 | 0·2713 (14) | 0·2856 (2) | 1·1892 (6) | 4·4 |
| C13 | 0·5595 (19) | 0·3062 (3) | 1·0450 (9) | 3·7 |
| C14 | 0·7372 (21) | 0·2944 (3) | 0·9613 (9) | 3·9 |
| O3 | 0·8586 (19) | 0·3292 (2) | 0·9016 (7) | 4·9 |
| C15 | 0·7927 (22) | 0·2485 (3) | 0·9340 (9) | 3·9 |
| C16 | 0·6728 (20) | 0·2134 (4) | 0·9947 (8) | 3·8 |
| O4 | 0·7162 (15) | 0·1681 (2) | 0·9739 (6) | 4·9 |
| C17 | 0·8647 (25) | 0·1564 (3) | 0·8739 (10) | 5·4 |
| C1 | 0·3570 (22) | 0·1887 (4) | 1·1577 (10) | 4·2 |
| O1 | 0·1870 (13) | 0·2019 (2) | 1·2280 (6) | 4·6 |
| C2 | 0·4151 (22) | 0·1385 (3) | 1·1503 (9) | 4·2 |
| C3 | 0·2743 (24) | 0·1091 (3) | 1·2419 (11) | 5·7 |
| C31 | 0·3907 (24) | 0·0597 (4) | 1·2516 (11) | 4·7 |
| C32 | 0·307 (4) | 0·0270 (5) | 1·1630 (19) | 12·3 |
| C33 | 0·406 (4) | −0·0180 (5) | 1·1688 (17) | 10·5 |
| C34 | 0·5955 (24) | −0·0309 (4) | 1·2638 (12) | 4·8 |
| O5 | 0·6813 (19) | −0·0761 (3) | 1·2653 (8) | 7·0 |
| C35 | 0·698 (3) | 0·0017 (5) | 1·3469 (14) | 9·6 |
| C36 | 0·585 (4) | 0·0454 (5) | 1·3399 (14) | 10·0 |
| C37 | 0·885 (3) | −0·0903 (5) | 1·3642 (13) | 8·0 |
| H2 | 0·208 (27) | 0·256 (5) | 1·236 (12) | 9·6 |
| H3 | 1·005 (27) | 0·319 (5) | 0·871 (12) | 9·6 |

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| C2—C1 | 1·48 (1) | C2—C3 | 1·53 (1) |
| C1—O1 | 1·25 (1) | | |
| C1—C11 | 1·48 (1) | C3—C31 | 1·53 (1) |
| C11—C12 | 1·46 (1) | C31—C32 | 1·36 (2) |
| C12—C13 | 1·39 (1) | C32—C33 | 1·38 (2) |
| C13—C14 | 1·38 (1) | C33—C34 | 1·33 (2) |
| C14—C15 | 1·39 (1) | C34—C35 | 1·35 (2) |
| C15—C16 | 1·38 (1) | C35—C36 | 1·37 (2) |
| C16—C11 | 1·42 (1) | C36—C31 | 1·31 (2) |
| C12—O2 | 1·33 (1) | | |
| C14—O3 | 1·37 (1) | | |
| C16—O4 | 1·39 (1) | C34—O5 | 1·37 (1) |
| C17—O4 | 1·42 (1) | C37—O5 | 1·41 (1) |
| O1—C1—C2 | 118·5 (9) | C3—C2—C1 | 113·8 (8) |
| C11—C1—O1 | 119·5 (9) | | |
| C2—C1—C11 | 122·0 (9) | C2—C3—C31 | 111·5 (9) |
| C1—C11—C12 | 117·0 (8) | C3—C31—C32 | 121 (1) |
| C1—C11—C16 | 126·2 (9) | C3—C31—C36 | 124 (1) |
| C11—C12—C13 | 120·6 (9) | C31—C32—C33 | 123 (2) |
| C12—C13—C14 | 119 (1) | C32—C33—C34 | 120 (1) |
| C13—C14—C15 | 122·1 (9) | C33—C34—C35 | 118 (1) |
| C14—C15—C16 | 119·5 (9) | C34—C35—C36 | 120 (1) |
| C15—C16—C11 | 121·3 (9) | C35—C36—C11 | 125 (1) |
| C11—C12—O2 | 123·0 (8) | | |
| C13—C14—O3 | 118·5 (9) | | |
| C15—C16—O4 | 123·2 (8) | C33—C34—O5 | 117 (1) |
| C16—O4—C17 | 117·8 (7) | C34—O5—C37 | 118 (1) |

**Related literature.** In the course of systematic studies of Colombian natural products, the title compound was isolated from leaves of a specimen of *Iryanthera tricornis* and recrystallized from acetone–hexane (m.p. 444–445 K). Braz-Filho, Da Silva & Gottlieb (1980) and Garzon, Cuca, Martínez, Yoshida & Gottlieb (1987) reported the presence of 2′,4′-dihydroxy-4,6′-dimethoxydihydrochalcone in the woody trunk and in the fruit of *Iryanthera laevis* (Myristicaceae). The structure of the compound was determined by these authors on the basis of MS, UV, $^1$H and $^{13}$C NMR. The $^1$H and $^{13}$C NMR spectra of the title compound are consistent. Recently, the activity of 2′,4′-dihydroxy-4,6′-dimethoxydihydrochalcone against Gram (+) bacteria (*Bacilus*

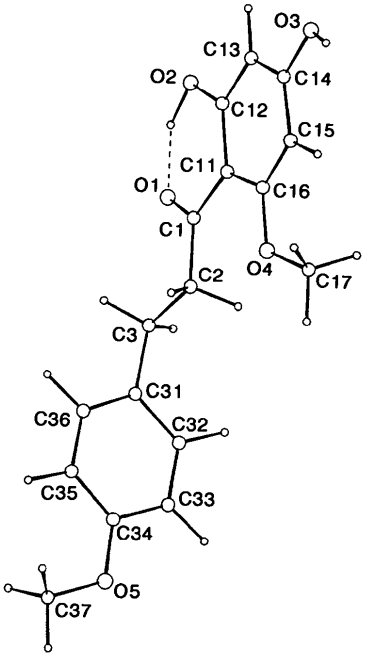


Fig. 1. View of the molecule with atom-numbering scheme.

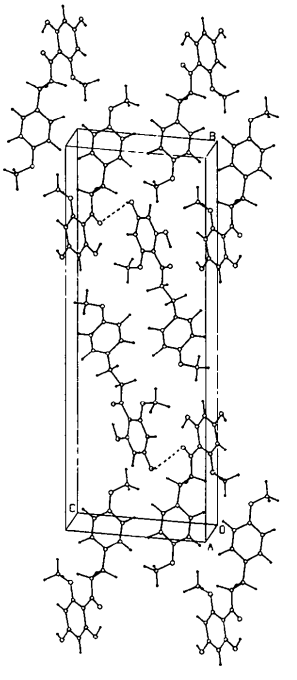

Fig. 2. Unit-cell packing diagram viewed down the *a* axis.

*anthasis*) was reported (Villamil, Cuca & Martínez, 1988).

The crystal structure determination was undertaken in order to confirm the assignment and to learn the three-dimensional configuration of the molecule.

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**References**

BRAZ-FILHO, R., DA SILVA, M. S. & GOTTLIEB, O. R. (1980). *Phytochemistry*, **19**, 1195–1197.

GARZON, L., CUCA, L. E., MARTÍNEZ, J. C., YOSHIDA, M. & GOTTLIEB, O. R. (1987). *Phytochemistry*, **26**, 2835–2837.

MAIN, P., GERMAIN, G. & WOOLFSON, M. M. (1984). *MULTAN*11/84. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*78. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

VILLAMIL, E., CUCA, L. E. & MARTÍNEZ, J. C. (1988). *Spectroscopy*, **6**, 157–165.

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# Structure of 1-(2′,4′-Dihydroxyphenyl)-3-(3″,4″-methylenedioxyphenyl)propane hemihydrate

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**Abstract.** 4-[(Benzo-1,3-dioxol-5-yl)propyl]-*m*-resorcinol hemihydrate, $C_{16}H_{16}O_4.\frac{1}{2}H_2O$, $M_r = 281{\cdot}3$, monoclinic, $C2/c$, $a = 41{\cdot}01\ (6)$, $b = 4{\cdot}842\ (3)$, $c = 14{\cdot}11\ (3)$ Å, $\beta = 93{\cdot}28\ (8)°$, $V = 2798\ (7)$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}33$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}908$ cm$^{-1}$, $F(000) = 1184$, room temperature, $R = 0{\cdot}090$ for 863 observed reflections and 88 parameters. Each pair of molecules of diarylpropane, related by a crystallographic twofold axis, are linked through hydrogen bonds to a disordered water molecule located on the twofold axis [O*w*···O2 = 2·77 (1) Å]. Other important intermolecular contacts are O1···O2′($x$, $1-y$, $0{\cdot}5+z$) at 2·67 (1) Å and O1···O*w*″($2-x$, $-y$, $2-z$) at 2·74 (1) Å. The phenyl rings 1 and 3 are planar to within standard deviations of 0·006 and 0·008 Å, respectively; the methylenedioxy atoms O3, O4 and C37 are −0·009, −0·021 and −0·047 Å out of the 3-phenyl ring plane; the angle between normals to phenyl-ring planes is 100·3 (9)°. The results of the X-ray study are in full agreement with the structure proposed on the basis of spectral and analytical data.

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**Experimental.** Benzenic extract of trunk wood of a specimen of *Iryanthera tricornis* collected in the Amazonic region of Colombia affords 1-(2′,4′-dihydroxyphenyl)-3-(3″,4″-methylenedioxyphenyl)-propane and also two additional 1,3-diarylpropanes.

Transparent colourless needles of the title compound were obtained from a benzene–*n*-hexane solution (m.p. 373–374 K), a crystal suitable for X-ray study with dimensions 0·10 × 0·06 × 0·02 mm was mounted on an Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator. Unit-cell parameters by least squares from 25 reflections with $3 \le \theta \le 17°$; 2071 reflections collected, 863 considered observed with $I \ge 2{\cdot}5\sigma(I)$; $\omega$–$2\theta$ scan mode, $hkl$ range: $-11 \le h \le 11$, $0 \le k \le 5$, $0 \le l \le 13$, $\theta \le 23°$; three standard reflections monitored every 50 measurements showed no significant decay in intensity. Lorentz–polarization corrections, but no absorption correction. Structure solved by direct methods (*MULTAN*11/84; Main, Germain & Woolfson, 1984). The poor diffracting quality of the crystals did not allow measurement of sufficient intensity data to perform anisotropic refinements; isotropic full-matrix least-squares refinement (on $F$)

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