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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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, $C_{16}H_{16}O_4.\frac{1}{2}H_2O$, $M_r = 281\cdot3$, monoclinic, $C2/c$, $a = 41\cdot01$ (6), $b = 4\cdot842$ (3), $c = 14\cdot11$ (3) Å, $\beta = 93\cdot28$ (8)°, $V = 2798$ (7) Å^3, $Z = 8$, $D_x = 1\cdot33$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot908$ cm^{-1}, $F(000) = 1184$, room temperature, $R = 0\cdot090$ 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 [$Ow\cdots O2 = 2\cdot77$ (1) Å]. Other important intermolecular contacts are $O1\cdots O2'(x, \ 1-y, \ 0\cdot5+z)$ at $2\cdot67$ (1) Å and $O1\cdots Ow''(2-x, -y, 2-z)$ at $2\cdot74$ (1) Å. The phenyl rings 1 and 3 are planar to within standard deviations of $0\cdot006$ and $0\cdot008$ Å, respectively; the methylenedioxy atoms O3, O4 and C37 are $-0\cdot009$, $-0\cdot021$ and $-0\cdot047$ Å out of the 3-phenyl ring plane; the angle between normals to phenyl-ring planes is $100\cdot3$ (9)°. The results of the X-ray study are in full agreement with the structure proposed on the basis of spectral and analytical data.

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 $0\cdot10 \times 0\cdot06 \times 0\cdot02$ 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 $I \ge 2\cdot5\sigma(I)$; ω–2θ 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)

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

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

	x	y	z	B_{eq}
C11	1·0782 (2)	0·6976 (20)	0·9128 (6)	3·3
C12	1·0551 (2)	0·4922 (21)	0·9038 (6)	3·3
C13	1·0394 (2)	0·3779 (21)	0·9790 (6)	3·3
C14	1·0477 (2)	0·4864 (22)	1·0692 (6)	3·5
C15	1·0697 (2)	0·6911 (21)	1·0831 (7)	4·1
C16	1·0852 (2)	0·7950 (23)	1·0050 (6)	4·3
O1	1·0476 (1)	0·3867 (14)	0·8124 (4)	3·9
O2	1·0322 (2)	0·3704 (15)	1·1456 (4)	4·8
C1	1·0963 (2)	0·8091 (21)	0·8318 (6)	3·6
C2	1·1238 (2)	0·6152 (21)	0·8009 (6)	3·6
C3	1·1433 (2)	0·7527 (22)	0·7235 (7)	4·5
C31	1·1676 (2)	0·5513 (21)	0·6851 (7)	3·9
C32	1·1630 (2)	0·4450 (21)	0·5940 (7)	4·3
C33	1·1857 (2)	0·2625 (24)	0·5644 (7)	4·6
C34	1·2122 (2)	0·1889 (22)	0·6204 (7)	4·4
C35	1·2177 (2)	0·2839 (24)	0·7100 (7)	5·0
C36	1·1948 (2)	0·4744 (24)	0·7410 (7)	4·6
O3	1·1857 (2)	0·1311 (19)	0·4768 (5)	7·1
C37	1·2139 (3)	−0·0421 (26)	0·4817 (8)	6·0
O4	1·2307 (2)	−0·0040 (17)	0·5706 (6)	6·1
Ow	1·0000	−0·0251 (24)	0·2500	4·8
Hw	1·008 (3)	0·087 (27)	1·205 (7)	11·4

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

C12—C11	1·38 (1)	C31—C3	1·52 (1)
C16—C11	1·40 (1)	C32—C31	1·39 (1)
C1—C11	1·50 (1)	C36—C31	1·38 (1)
C13—C12	1·39 (1)	C33—C32	1·37 (1)
O1—C12	1·40 (1)	C34—C33	1·36 (1)
C14—C13	1·40 (1)	O3—C33	1·39 (1)
C15—C14	1·35 (1)	C35—C34	1·35 (1)
O2—C14	1·40 (1)	O4—C34	1·42 (1)
C16—C15	1·40 (1)	C36—C35	1·40 (1)
C2—C1	1·55 (1)	C37—O3	1·42 (1)
C3—C2	1·54 (1)	O4—C37	1·41 (1)

C16—C11—C12	115·6 (8)	C31—C3—C2	110·7 (8)
C1—C11—C12	124·0 (8)	C32—C31—C3	119·4 (9)
C1—C11—C16	120·4 (8)	C36—C31—C3	119·7 (8)
C13—C12—C11	124·5 (9)	C36—C31—C32	119·6 (10)
O1—C12—C11	117·5 (8)	C33—C32—C31	117·6 (10)
O1—C12—C13	118·0 (8)	C34—C33—C32	121·9 (10)
C14—C13—C12	116·5 (9)	O3—C33—C32	126·5 (10)
C15—C14—C13	122·3 (9)	O3—C33—C34	111·2 (9)
O2—C14—C13	117·0 (8)	C35—C34—C33	123·0 (10)
O2—C14—C15	120·8 (9)	O4—C34—C33	108·5 (9)
C16—C15—C14	118·8 (9)	C36—C35—C34	115·8 (10)
C15—C16—C11	122·3 (9)	C37—O3—C33	105·4 (8)
C2—C1—C11	113·3 (8)	O4—C37—O3	108·7 (9)
C3—C2—C1	110·6 (8)	C37—O4—C34	106·3 (8)

of non-H atoms (*SHELX*76; Sheldrick, 1976). Water H atom was found by difference Fourier synthesis and included in the refinement; attempts to find the hydroxyl H atoms by the same method were unsuccessful; remaining H atoms were included in calculated positions, riding on the adjacent C atom with a common isotropic temperature factor, $U = 0.0834 \text{ Å}^2$; 863 reflections included, 88 parameters refined; $R = 0.090$, $wR = 0.094$, $w = 3.894/[\sigma^2(F) +$

$0.00033F^2$], $S = 2.98$, max. $(\Delta/\sigma) = 0.619$. Largest peaks in final ΔF map -0.50 and 0.47 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Drawing by *PLUTO* (Motherwell & Clegg, 1978). The atomic parameters are given in Table 1.* A view of the molecule with the atomic numbering scheme is shown in Fig. 1. Bond distances and angles are listed in Table 2.

Related literature. For the first time, Alves de Lima, Cavalcanti, Diaz & Gottlieb (1975) reported the isolation of one acetate derivative of the title 1,3-diarylpropane from the trunk wood of *Iryanthera coriacea* Ducke (Miristicaceae) and the corresponding IR, PMR and MS data were analysed. Subsequently, the isolation of 1-(2′,4′-dihydroxy-phenyl)-3-(3″,4″-methylenedioxyphenyl)propane from the trunk wood of *Iryanthera polineura* and *Iryanthera tricornis* was published (De Almeida, Braz-Filho, von Bülow, Correa, Gottlieb, Maia & Da Silva, 1979; Da Silva, Giesbrecht, De Alvarenga & Gottlieb, 1984). No agreement between their physical data with our data was found; however, our results agree well with those reported for the synthetic product by Morais, Braz-Filho & Gottlieb (1985). In order to ascertain the molecular conformation of the title compound, the present crystal structure determination was performed.
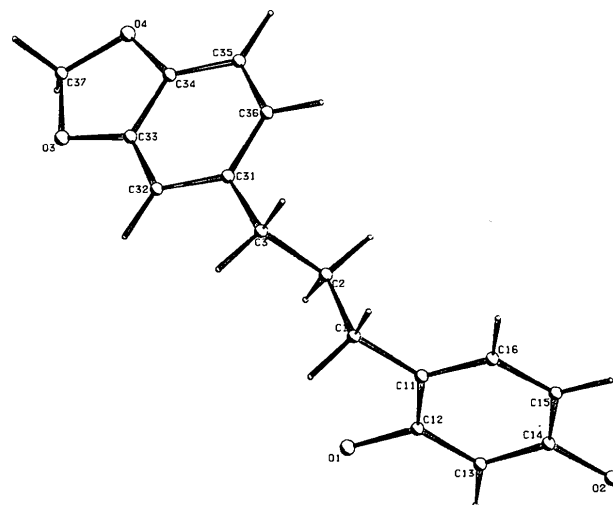
Fig. 1. View of the molecule with atom-numbering scheme.

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Structure of Ethyl 2-Amino-4-phenylthiophene-3-carboxylate

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Abstract. $C_{13}H_{13}NO_2S$, $M_r = 247\cdot3$, monoclinic, $P2_1/c$, $a = 10\cdot650$ (1), $b = 13\cdot942$ (2), $c = 9\cdot273$ (1) Å, $\beta = 115\cdot07$ (1)°, $V = 1247\cdot1$ (4) Å^3, $Z = 4$, $D_x = 1\cdot320$ (3) Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 2\cdot18$ mm^{-1}, $F(000) = 520$, $T = 295$ K, $R = 0\cdot05$ for 1664 observed reflections [$I > 2\sigma(I)$]. Molecular geometry is within the normal limits. The thiophene ring is planar. The steric repulsion between the phenyl ring and the ethoxycarbonyl group is minimized by (i) the rotation of the phenyl ring [dihedral angle 70·2 (1)° between the phenyl and thiophene rings] and (ii) the widening of the external angle at C(4) by 8·2 (4)° [the difference between the angles C(3)—C(4)—C(41) and C(5)—C(4)—C(41)]. The molecule exhibits both intra- and intermolecular N—H⋯O hydrogen bonding.

Experimental. The title compound was prepared by the following procedure: acetophenone was allowed to undergo a Cope reaction (Cope, Hoffmann, Wyckoff & Hardenbergh, 1941) with ethyl cyanoacetate, followed by a Gewald reaction (Gewald 1962, 1965; Gewald, Schinke & Bottcher, 1966) in the presence of sulfur to form ethyl 2-amino-4-phenylthiophene-3-carboxylate as a crystalline solid.

This was filtered, washed with ethanol and recrystallized from chloroform; pale-brown needles were obtained.

A specimen of dimensions 0·3 × 0·4 × 0·3 mm was chosen. Lattice parameters and their e.s.d.'s were derived from a least-squares treatment of 25 reflections ($6 < \theta < 33°$). Intensity data were collected on an Enraf–Nonius CAD-4 single-crystal X-ray diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode for $h - 10\rightarrow10$, $k0\rightarrow15$, l $0\rightarrow10$, $2\theta_{max} = 120°$. Three standard reflections monitored every 100 measurements showed no significant variation in the intensities. Of the 2076 unique reflections collected, 1664 were judged significant, $I > 2\sigma(I)$, $R_{int} = 0\cdot04$. Intensities were corrected for Lp effects. An empirical-absorption correction based on a series of ψ scans was applied. Transmission coefficient ranged from 0·847 to 0·994. Structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). 15 non-H atoms were located from an E map. The remaining atoms, including H atoms, were located from succeeding difference Fourier syntheses. Positional and thermal parameters of non-H atoms were refined by full-matrix least-squares refinement on F using *SHELX*76 (Sheldrick, 1976). The H atoms were refined with an individual isotropic temperature factor (fixed in the final cycles). At the final stage of

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