2',4'-Dihydroxy-5',6'-dimethoxychalcone

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The title compound, $C_{17}H_{16}O_5$, an (E)-1-(4,6-dihydroxy-2,3-dimethoxyphenyl)-3-phenylprop-2-en-1-one, was isolated from a plant of genus *Cryptocarya costata*. The molecule adopts a *trans* configuration with respect to the positions of the benzene ring and the dihydroxydimethoxybenzaldehyde fragment about the olefinic C=C double bond. The molecules are linked by intermolecular hydrogen bonds to form ribbons extending parallel to the *b* axis.

Comment

A number of chalcones containing hydroxyl groups at C(2'), C(2',4') or C(2',4) are known as potentially therapeutic agents against infections with methicilin-resistant *Staphylococcus aureus* strains (Alcaraz *et al.*, 2000). Therefore, some chalcone derivatives have been successfully synthesized, including 2',4'-dihydroxy-5',6'-dimethoxychalcone (I) (Bhaskar & Seshadri, 1974). However, only a few structures of naturally occurring chalcones isolated from plants have been reported. Some examples are 2',6'-dihydroxy-4,4'-dimethoxychalcone (II) from the plant *Pityrogramma chrysophylla* (Nilsson, 1961; Schmalle *et al.*, 1990), flemiculosin, (III), from the leaves of *Flemingia fruticolose* (Bhattacharyya *et al.*, 1999) and (E)-1-(2-hydroxy-3,4-dimethoxyphenyl)-3-(4-dimethoxyphenyl)prop-2-en-1-one, (IV) (Krishna *et al.*, 2005). In the course of our study of secondary metabolites from *Cryptocarya* species growing in Indonesia, crystals of the title compound, (I), suitable for X-ray investigation were obtained.

(I)
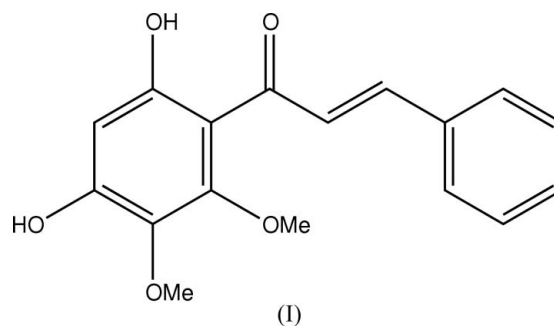
As in most substituted chalcones, the molecule of compound (I) is not planar. The substituted benzene ring (C1'–C6')/O2/O3/O4/O5 is essentially planar with a maximum deviation of 0.054 (2) Å for atom O2 from the least-squares plane. It is inclined by 28.22 (10)° to the propenylbenzene fragment (C1–C9) [maximum deviation 0.022 (2) Å for atom C8]. The O–Me bonds, O2–C10 and O3–C11, are perpendicular to the benzene C1'–C6' plane, with torsion angles C10–O2–C6'–C5' and C11–O3–C5'–C6' of 88.5 (3) and 65.93 (3)°,
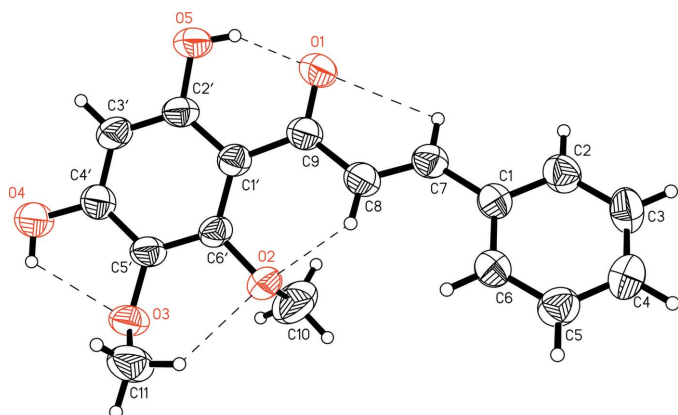
Figure 1
The molecular structure of the title compound, (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular O—H···O and C—H···O hydrogen bonds.

Figure 2
A packing diagram for (I). Intermolecular C—H···O and O—H···O hydrogen-bonding interactions are shown as dashed lines.

respectively. The carbonyl group, C9=O1, is in a *cis* configuration with respect to the olefenic C7=C8 double bond, typical of chalcone compounds.
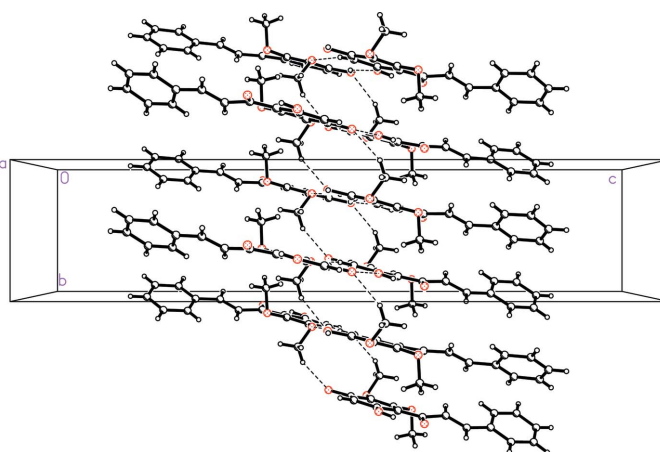
The bond lengths and angles of the molecule are in normal ranges (Table 1) and in agreement with all the analogues, with slight variations of the C=O bond length. The C9=O1 bond length is slightly shorter than that in (II) and (IV) [1.264 (3) and 1.2538 (17) Å, respectively]. The presence of five intramolecular hydrogen bonds (Table 2) may contribute to the stability and planarity of the molecule. In the crystal structure, the molecules are linked by intermolecular hydrogen bonds, forming ribbons extending parallel to the *b* axis (Fig. 2).

Experimental

Barks from the tree *Cryptocarya costata* Bl. were collected from The National Garden of Leralindu, Central Sulawesi, Indonesia. A voucher specimen (E-377L) was deposited at the herbarium of Faculty of Sciences, University of Tadulako, Central Sulawesi. The powdered dried tree barks (2.5 kg) of *C. costata* were macerated with methanol at room temperature for 24 h. The mixture was filtered and the methanol filtrate was evaporated under reduced pressure to give a concentrated methanol extract. To the extract, distilled water was added, and the aqueous methanol extract was partitioned into CHCl$_3$ and ethyl acetate to give CHCl$_3$- (140 g) and ethyl acetate- (40 g) soluble fractions. A portion (20 g) of the CHCl$_3$ fraction was fractionated using column vacuum chromatography (silica gel, eluted with *n*-hexane–ethyl acetate = 9:1–1:1) to give six major fractions, *A*−*F*. Repeated purification of fraction *C* (230 g) by radial chromatography (*n*-hexane–EtOAc = 7:3) gave compound (I). Recrystallization from methanol afforded yellow crystals (50 g) with melting point 440–442 K.

Crystal data

C$_{17}$H$_{16}$O$_5$
$M_r = 300.30$
Orthorhombic, *Pbca*
$a = 12.360$ (2) Å
$b = 7.1688$ (14) Å
$c = 33.275$ (6) Å
$V = 2948.3$ (10) Å^3
$Z = 8$
$D_x = 1.353$ Mg m^{-3}

Mo *K*α radiation
Cell parameters from 3566 reflections
$\theta = 2.0$–25.5°
$\mu = 0.10$ mm^{-1}
$T = 298$ (2) K
Block, yellow
$0.48 \times 0.45 \times 0.19$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.953$, $T_{\max} = 0.981$
14651 measured reflections

2747 independent reflections
2137 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$
$\theta_{\max} = 25.5°$
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 8$
$l = -33 \rightarrow 40$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.130$
$S = 1.11$
2747 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.9791P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.246 (2)	O3—C11	1.426 (3)
O2—C6′	1.371 (2)	O4—C4′	1.348 (2)
O2—C10	1.428 (3)	O5—C2′	1.350 (2)
O3—C5′	1.385 (2)	C7—C8	1.321 (3)
C10—O2—C6′—C1′	−92.0 (3)	C11—O3—C5′—C4′	−116.8 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···O3	0.82	2.29	2.741 (2)	115
O5—H5B···O1	0.82	1.77	2.496 (2)	147
C7—H7A···O1	0.93	2.46	2.780 (3)	100
C8—H8A···O2	0.93	2.26	2.786 (3)	115
C11—H11C···O2	0.96	2.36	2.971 (3)	121
O4—H4B···O5^i	0.82	2.15	2.790 (2)	135
C3′—H3B···O3^{ii}	0.93	2.57	3.462 (3)	161
C11—H11B···O4^{iii}	0.96	2.52	3.420 (4)	156

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $-x + 1, -y + 1, -z + 1$.

H atoms were located in a difference map and repositioned geometrically, with C—H = 0.93 − 0.96 Å and O—H = 0.86 Å. They were constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (1.5 for CH_3 and OH) times U_{eq}(C,O).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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