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Key indicators

Single-crystal X-ray study
 $T = 273$ K
 Mean $\sigma(C-C) = 0.003$ Å
 R factor = 0.054
 wR factor = 0.156
 Data-to-parameter ratio = 17.6

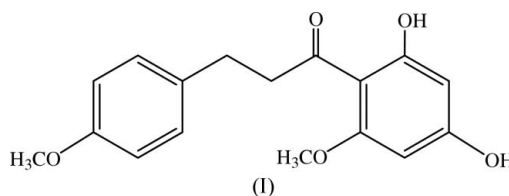
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

A second monoclinic polymorph of 1-(2,4-dihydroxy-6-methoxyphenyl)-3-(4-methoxyphenyl)propan-1-one

The title compound, $C_{17}H_{18}O_5$, was isolated from the leaves of *Rauwenhoffia siamensis* Scheff. There are two crystallographically independent molecules in the asymmetric unit. The dihedral angle between the two benzene rings is $80.81(7)^\circ$ in one molecule and $65.89(7)^\circ$ in the other. The symmetry-related molecules are linked *via* $O-H \cdots O$ intermolecular hydrogen bonds to form chains along [201].

Comment

Rauwenhoffia siamensis Scheff. belongs to the family of Annonaceae, which is widely distributed in Thailand, Malaysia and Indonesia. *R. siamensis* has a local Thai name, Nom Maew, and has been used for biofragrance (Chulalaksananukul *et al.*, 1998). The title compound, (I), was isolated from the leaves of *R. siamensis*, which were collected from Songkhla province in the southern part of Thailand. The naringin dihydrochalcone, which is a derivative of compound (I), was known as a sweetener (Shin *et al.*, 1995). As part of our systematic studies on chemical constituents of Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Thongdeeying *et al.*, 2005; Fun *et al.*, 2005), we have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and relative stereochemistry.



The crystal structure of (I) was previously reported by De Matheus *et al.* (1991) in the monoclinic space group $P2_1/c$, with $a = 4.856(3)$, $b = 28.896(7)$, $c = 10.776(3)$ Å, $\beta = 98.04(4)^\circ$ and $Z = 4$. In the present work, the compound crystallized again in the monoclinic space group $P2_1/c$, but with different cell parameters and $Z = 8$.

The asymmetric unit of (I) contains two molecules, *A* and *B*, in which the orientation of the C1–C6 benzene ring with respect to the central $-C7-C8-C9=O3$ linkage is different (Fig. 1). The corresponding bond lengths and angles of these two molecules agree with each other and show normal values (Allen *et al.*, 1987), and are comparable to those observed in the other monoclinic polymorph (De Matheus *et al.*, 1991) and a closely related structure (Shin *et al.*, 1995). In molecule *A*, the C1–C6 and C10–C15 benzene rings are oriented at angles of $83.79(8)$ and $8.69(11)^\circ$ [$65.15(9)$ and $5.74(12)^\circ$ in molecule *B*] with respect to the $C7-C8-C9=O3$ plane. The

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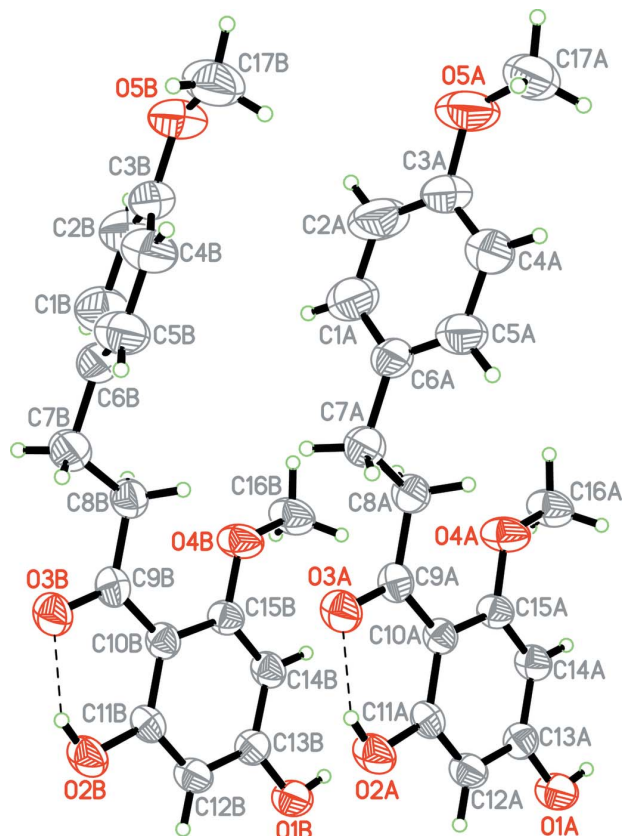


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate hydrogen bonds.

dihedral angle between the two benzene rings is $80.81(7)^\circ$ in molecule *A* and $65.89(7)^\circ$ in molecule *B*, compared to $81.49(9)^\circ$ in the other polymorph (De Matheus *et al.*, 1991). In both molecules *A* and *B*, the methoxy groups are coplanar with the attached benzene rings. The molecular structure is stabilized by intramolecular O—H...O hydrogen bonds (Table 2). The symmetry-related molecules are linked into chains along [201] by O—H...O intermolecular hydrogen bonds (Fig. 2).

Experimental

Air-dried leaves of *R. siamensis* were ground and extracted with CH_2Cl_2 at room temperature. The residue obtained after evaporation of the solvent was repeatedly subjected to column chromatography over silica gel to afford (I). Single crystals of (I) were obtained by recrystallization from a CH_2Cl_2 – CH_3OH (8:2 v/v) solvent system (m.p. 442–443 K).

Crystal data

$\text{C}_{17}\text{H}_{18}\text{O}_5$	$D_x = 1.342 \text{ Mg m}^{-3}$
$M_r = 302.31$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7172 reflections
$a = 9.7048(2) \text{ \AA}$	$\theta = 1.4$ – 27.9°
$b = 28.9048(5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 13.4920(2) \text{ \AA}$	$T = 273(2) \text{ K}$
$\beta = 127.770(1)^\circ$	Plate, colourless
$V = 2991.72(10) \text{ \AA}^3$	$0.50 \times 0.42 \times 0.07 \text{ mm}$
$Z = 8$	

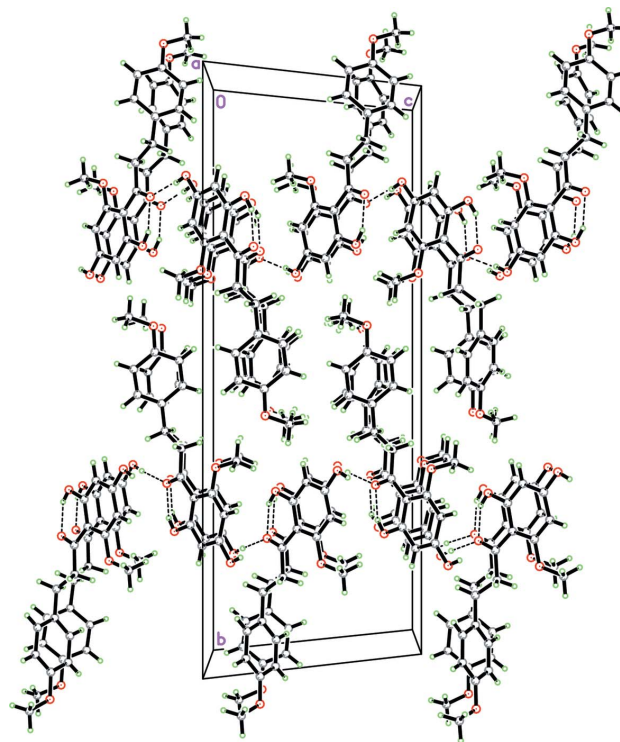


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	7172 independent reflections
ω scans	3709 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$R_{\text{int}} = 0.037$
$T_{\text{min}} = 0.952$, $T_{\text{max}} = 0.993$	$\theta_{\text{max}} = 27.9^\circ$
26484 measured reflections	$h = -12 \rightarrow 12$
	$k = -38 \rightarrow 29$
	$l = -14 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.2747P]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.156$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
7130 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
405 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1A—C13A	1.3498 (19)	O4A—C16A	1.429 (2)
O2A—C11A	1.3460 (19)	O5A—C3A	1.379 (2)
O3A—C9A	1.254 (2)	O5A—C17A	1.401 (3)
O4A—C15A	1.3540 (19)		
C6A—C7A—C8A	111.87 (15)	C8B—C7B—C6B	112.50 (16)
C9A—C8A—C7A	114.40 (15)	C9B—C8B—C7B	114.13 (15)
O3A—C9A—C8A	117.73 (15)	O3B—C9B—C8B	117.69 (15)
C10A—C9A—C8A	123.17 (15)	C10B—C9B—C8B	122.85 (15)
C17A—O5A—C3A—C4A	$-9.2(3)$	C17B—O5B—C3B—C4B	$6.5(3)$
C6A—C7A—C8A—C9A	$171.32(16)$	C6B—C7B—C8B—C9B	$168.49(17)$
C7A—C8A—C9A—C10A	$-171.88(16)$	C7B—C8B—C9B—C10B	$-177.31(17)$
C16A—O4A—C15A—C14A	$6.4(3)$	C16B—O4B—C15B—C14B	$9.6(3)$

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1A-H1A\cdots O3A^i$	0.82	1.94	2.755 (2)	175
$O2A-H2A\cdots O3A$	0.82	1.78	2.505 (2)	147
$O1B-H1C\cdots O3B^i$	0.82	1.97	2.783 (2)	173
$O2B-H2C\cdots O3B$	0.82	1.77	2.504 (2)	147

Symmetry code: (i) $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$.

H atoms were placed in calculated positions, with $O-H = 0.82$ Å and $C-H = 0.93-0.97$ Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINTE* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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