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5-Hydroxy-3,7-dimethoxy-2-phenyl-4*H*-1-benzopyran-4-one

Hoong-Kun Fun,[a]* Ibrahim Abdul Razak,[a] Nawong Boonnak,[b] Surat Laphookhieo[b]‡ and Suchada Chantrapromma[b]*

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

‡ Permanent address: School of Sciences, Mae Fah Luang University, Muang, Chaingrai 57100, Thailand.

Correspondence e-mail: hkfun@usm.my, suchada.c@psu.ac.th

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.142
Data-to-parameter ratio = 24.7

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

The title compound, $C_{17}H_{14}O_5$, a flavone, was isolated from the rhizomes of *Kaempferia parviflora*. There are two crystallographically independent molecules in the asymmetric unit. The molecular structure is stabilized by intramolecular O—H···O and C—H···O hydrogen bonds. C—H···O intermolecular hydrogen bonds and π–π stacking interactions link the molecules into a network.

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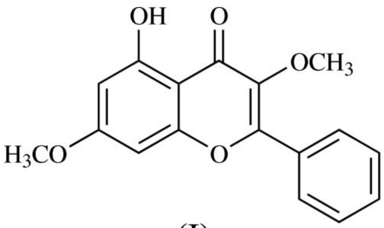
Comment

Kaempferia is small genus belonging to the *Zingiberaceae*, with at least 17 species distributed in Thailand. *Kaempferia parviflora* rhizomes were collected from Loei province in the northeastern part of Thailand. Among the local people, the rhizomes of *K. parviflora* have been known as health-promoting herbs, and are also frequently used for the treatment of colic disorder, and peptic and duodenal ulcers. In Thailand, a tonic drink made from the rhizomes of *K. parviflora* is commercially available and is believed to relieve impotence symptoms (Yenjai *et al.*, 2004). We have isolated the title compound, (I), from rhizomes of *K. parviflora*. It was previously isolated from the black rhizomes of *Boesenbergia pandurata* (Jaipetch *et al.*, 1983). Compound (I) does not exhibit antiplasmodium, antifungal, antimycobacterial and cytotoxic activities (Yenjai *et al.*, 2004). As part of our research on bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005), we have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and stereochemistry.



(I)

The asymmetric unit of (I) contains two molecules, *A* and *B*, which have similar bond lengths and angles (Fig. 1). The bond lengths in (I) show normal values (Allen *et al.*, 1987). The benzopyran-4-one (C7–C15/O1) ring system is essentially planar, with a maximum deviation of 0.052 (1) Å for atom C8*A* and 0.016 (1) Å for atom C10*B*. The phenyl substituent is twisted away from the benzopyran-4-one plane, the dihedral angle between the benzopyran-4-one and C1–C6 benzene planes being 38.00 (3)° in *A* and 13.99 (3)° in *B*. The methoxy group attached at atom C13 is almost coplanar with the

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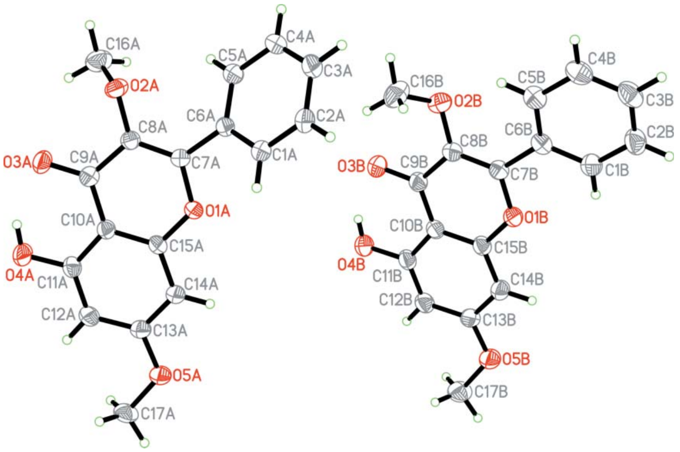

Figure 1
The asymmetric unit of (I), showing 80% probability displacement ellipsoids and the atomic numbering.

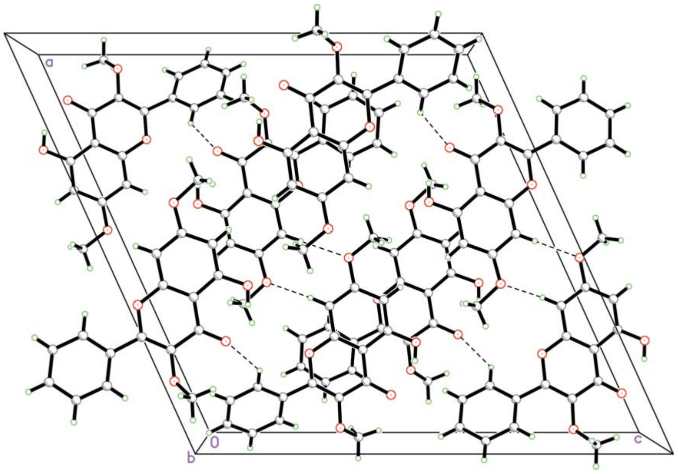

Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

benzopyran-4-one ring system [C17—O5—C13—C14 =174.03 (10)° in *A* and 168.57 (10)° in *B*] while the other methoxy group at C8 is twisted away from it [C16—O2—C8—C9 = −66.21 (12)° in *A* and 68.69 (12)° in *B*]. The molecular structure is stabilized by intramolecular O—H···O and C—H···O hydrogen bonds (Table 2).

C—H···O intermolecular hydrogen bonds link the molecules into a network (Fig. 2). In addition, the molecular packing is stabilized by π–π stacking interactions between the O1*A*/C7*A*–C10*A*/C15*A* pyran ring and C1*B*–C6*B* benzene ring of the symmetry-related molecule at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, the centroid–centroid distance being 3.6062 (6) Å.

Experimental

Air-dried rhizomes of *K. parviflora* were ground and extracted with hexane and $CHCl_3$ at room temperature. The residue obtained after evaporation of the solvent was separated by quick column chromatography (QCC) over silica gel and eluted with 3% CH_2Cl_2–hexane to afford seven fractions (F1–F7). Fraction F2 was subjected to column chromatography (CC) with 25% CH_2Cl_2–hexane to give five fractions (F2A–F2E). Fraction F2B was recrystallized from $CHCl_3$–CH_3OH (8:2 *v*/*v*) to give pale-yellow single crystals of (I) after several days (m.p. 419–421 K).

Crystal data

$C_{17}H_{14}O_5$
$M_r = 298.28$
Monoclinic, $P2_1/c$
$a = 20.0802\ (3)$ Å
$b = 7.1360\ (1)$ Å
$c = 20.7415\ (3)$ Å
$\beta = 114.399\ (1)°$
$V = 2706.66\ (7)$ Å^3
$Z = 8$

$D_x = 1.464$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9804 reflections
$\theta = 1.0$–$1.0°$
$\mu = 0.11$ mm^{-1}
$T = 100.0\ (1)$ K
Block, pale yellow
0.51 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.962$, $T_{max} = 0.979$
28245 measured reflections

9804 independent reflections
7907 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 32.5°$
$h = -29 \rightarrow 30$
$k = -10 \rightarrow 6$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.142$
$S = 1.02$
9804 reflections
397 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2 + 1.0297P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1*A*—C7*A*	1.3655 (11)	O1*B*—C15*B*	1.3640 (11)
O1*A*—C15*A*	1.3688 (11)	O1*B*—C7*B*	1.3675 (12)
O2*A*—C8*A*	1.3713 (11)	O2*B*—C8*B*	1.3666 (12)
O2*A*—C16*A*	1.4439 (14)	O2*B*—C16*B*	1.4479 (15)
O3*A*—C9*A*	1.2553 (12)	O3*B*—C9*B*	1.2562 (12)
O4*A*—C11*A*	1.3481 (12)	O4*B*—C11*B*	1.3504 (12)
O5*A*—C13*A*	1.3552 (12)	O5*B*—C13*B*	1.3539 (13)
O5*A*—C17*A*	1.4358 (12)	O5*B*—C17*B*	1.4369 (13)
C16*A*—O2*A*—C8*A*—C7*A*	123.20 (10)	C16*B*—O2*B*—C8*B*—C7*B*	−117.86 (11)
C16*A*—O2*A*—C8*A*—C9*A*	−66.21 (12)	C16*B*—O2*B*—C8*B*—C9*B*	68.69 (12)
C17*A*—O5*A*—C13*A*—C14*A*	174.03 (10)	C17*B*—O5*B*—C13*B*—C14*B*	168.57 (10)
C17*A*—O5*A*—C13*A*—C12*A*	−6.35 (15)	C17*B*—O5*B*—C13*B*—C12*B*	−12.86 (15)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4*A*—H4*AA*···O3*A*	0.82	1.87	2.6033 (11)	148
O4*B*—H4*BA*···O3*B*	0.82	1.85	2.5860 (12)	148
C1*A*—H1*AA*···O3*B*^{i}	0.93	2.49	3.2404 (14)	138
C4*A*—H4*AB*···O3*A*^{ii}	0.93	2.59	3.5201 (14)	177
C5*A*—H5*AA*···O2*A*	0.93	2.54	2.9530 (13)	107
C1*B*—H1*BA*···O1*B*	0.93	2.29	2.6419 (12)	102
C5*B*—H5*BA*···O2*B*	0.93	2.31	2.9067 (14)	121
C14*A*—H14*A*···O5*B*^{iii}	0.93	2.50	3.3486 (13)	153
C14*B*—H14*B*···O5*A*^{iv}	0.93	2.55	3.4535 (14)	163
C16*A*—H16*B*···O3*A*	0.96	2.38	2.9952 (15)	121
C16*B*—H16*F*···O3*B*	0.96	2.39	2.9975 (16)	121

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

H atoms were placed in calculated positions, with O—H distances of 0.82 Å and C—H distances in the range 0.93–0.96 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. The data were collected with an Oxford Cyrosystems Cobra low-temperature attachment.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (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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