

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

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## Key indicators

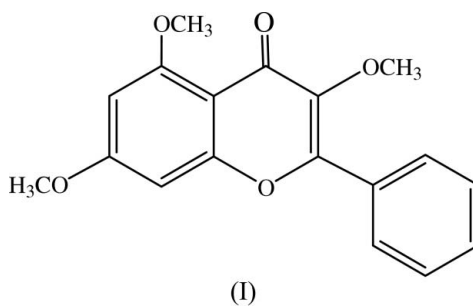
Single-crystal X-ray study  
*T* = 100 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
*R* factor = 0.042  
*wR* factor = 0.111  
Data-to-parameter ratio = 20.5

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

The title compound,  $\text{C}_{18}\text{H}_{16}\text{O}_5$ , a flavone, was isolated from the rhizomes of *Kaempferia parviflora*. The phenyl substituent is twisted away from the plane of the benzopyran-4-one ring system by  $31.05 (4)^\circ$ . The crystal structure is stabilized by  $\pi-\pi$  stacking interactions between the benzopyran-4-one ring system of inversion-related molecules stacked along the *a* axis and  $\text{C}-\text{H}\cdots\text{O}$  interactions between the screw-related molecules.

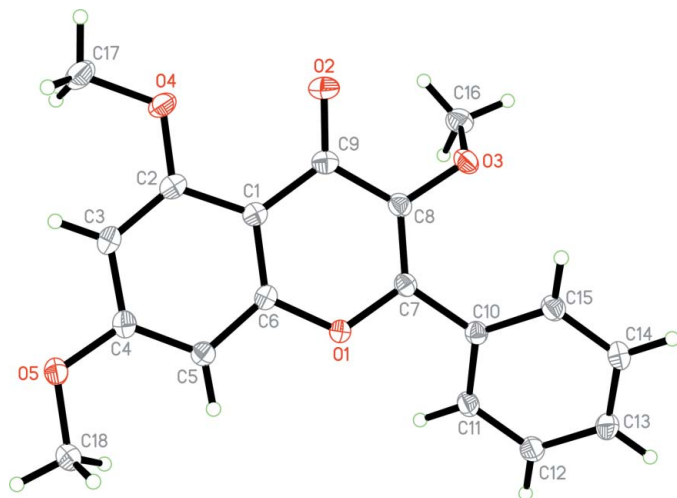
## Comment

The title compound, (I), a flavone, is a secondary metabolite occurring in plants. Some flavones show antimycobacterial activity (Yenjai *et al.*, 2004). We have previously reported the structure of the flavone, 5-hydroxy-3,7-dimethoxy-2-phenyl-4*H*-1-benzopyran-4-one, (II) (Fun *et al.*, 2005), isolated from the rhizomes of *Kaempferia parviflora*. Now, we have isolated the title compound, (I), from the rhizomes of *Kaempferia parviflora*, which were collected from Loei province in the northeastern part of Thailand. It was previously isolated from the black rhizomes of *Boesenbergia pandurata* (Jaipetch *et al.*, 1983). Compound (I) does not exhibit antiplasmodium, anti-fungal, antimycobacterial and cytotoxic activities (Yenjai *et al.*, 2004). As part of our ongoing studies on the phytochemistry and biological activities of Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Cheenpracha *et al.*, 2005; Fun *et al.*, 2005; Ng *et al.*, 2005*a*, 2005*b*), we have undertaken the X-ray crystal structure analysis of (I) in order to establish its molecular structure and relative stereochemistry.

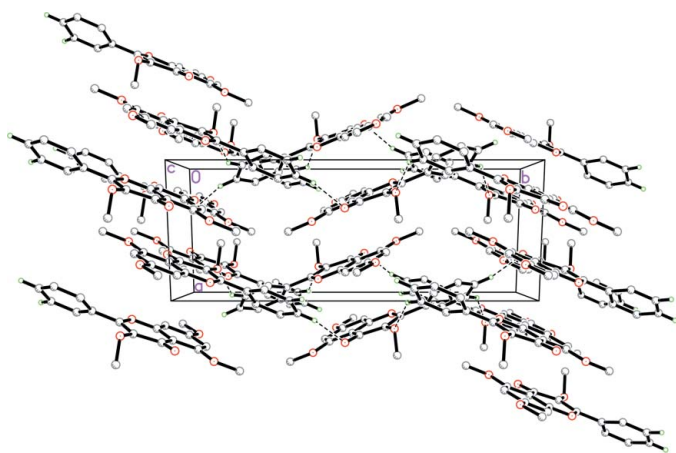


Bond distances and angles in (I) are comparable to those observed in (II) and show normal values (Allen *et al.*, 1987). The benzopyran-4-one (C1–C9/O1) ring system is essentially planar, with a maximum deviation of 0.058 (1) Å for atom C8. The phenyl substituent is twisted away from the benzopyran-4-one plane, the dihedral angle between the C1–C9/O1 and C10–C15 planes being  $31.05 (4)^\circ$  [ $38.00 (3)^\circ$  in molecule *A* and  $13.99 (3)^\circ$  in molecule *B* in (II); Fun *et al.*, 2005]. The two

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



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

methoxy groups attached at atoms C2 and C4 are almost coplanar with the benzopyran-4-one ring system while the third methoxy group at C8 is twisted away from it (Table 1).

The crystal structure is stabilized by  $\pi$ - $\pi$  stacking interactions between the benzopyran-4-one ring system of inversion-related molecules stacked along the *a* axis (Fig. 2). The ring system is stacked in such a way that the centroid-centroid distance between the O1/C1/C6-C9 ring at (*x*, *y*, *z*) and the C1-C6 ring at ( $2 - x$ ,  $1 - y$ ,  $1 - z$ ) is 3.4363 (6) Å, while the centroid-centroid distance between the C1-C6 rings at (*x*, *y*, *z*) and ( $1 - x$ ,  $1 - y$ ,  $1 - z$ ) is 3.3402 (7) Å. In addition, the crystal packing is stabilized by C-H...O interactions (Table 2) between the screw-related molecules.

## Experimental

Air-dried rhizomes of *K. parviflora* were ground and extracted with hexane and  $\text{CHCl}_3$  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%  $\text{CH}_2\text{Cl}_2$ -hexane

to afford seven fractions (F1-F7). Fraction F4 was subjected to column chromatography (CC) with 10% acetone-hexane to give three fractions (F4A-F4C). Fraction F4B was recrystallized from  $\text{CHCl}_3$ - $\text{CH}_3\text{OH}$  (4:1 *v/v*) to give colourless block-shaped single crystals of (I) after several days (m.p. 476-477 K).

## Crystal data

$\text{C}_{18}\text{H}_{16}\text{O}_5$   
 $M_r = 312.31$   
Monoclinic,  $P2_1/c$   
 $a = 7.4026$  (2) Å  
 $b = 18.4985$  (4) Å  
 $c = 11.4109$  (2) Å  
 $\beta = 109.466$  (1)°  
 $V = 1473.26$  (6) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.408$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 5703 reflections  
 $\theta = 2.2$ - $30.1$ °  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 100.0$  (1) K  
Block, colourless  
 $0.58 \times 0.30 \times 0.22$  mm

## Data collection

Bruker SMART APEX-2 CCD  
area-detector diffractometer with  
Oxford Cyrosystems Cobra  
low-temperature attachment  
 $\omega$  scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2005)  
 $T_{\min} = 0.964$ ,  $T_{\max} = 0.978$   
27408 measured reflections

4323 independent reflections  
3718 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$   
 $\theta_{\text{max}} = 30.1$ °  
 $h = -10 \rightarrow 10$   
 $k = -26 \rightarrow 26$   
 $l = -16 \rightarrow 16$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.111$   
 $S = 1.06$   
4323 reflections  
211 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.5096P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.44$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

O1-C6	1.3686 (12)	O4-C2	1.3594 (12)
O1-C7	1.3695 (11)	O4-C17	1.4308 (14)
O2-C9	1.2331 (12)	O5-C4	1.3574 (12)
O3-C8	1.3737 (12)	O5-C18	1.4352 (14)
O3-C16	1.4436 (14)	C7-C8	1.3535 (14)
C8-O3-C16	113.77 (8)	C4-O5-C18	116.65 (8)
C2-O4-C17	117.34 (9)		
C17-O4-C2-C3	0.76 (15)	C16-O3-C8-C7	117.65 (11)
C17-O4-C2-C1	179.03 (10)	C16-O3-C8-C9	-67.07 (12)
C18-O5-C4-C5	-1.80 (15)	O1-C7-C10-C11	32.40 (13)
C18-O5-C4-C3	177.47 (9)	C8-C7-C10-C15	33.91 (16)

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> -H... <i>A</i>	<i>D</i> -H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> -H... <i>A</i>
C12-H12...O3 <sup>i</sup>	0.93	2.55	3.3419 (15)	144
C13-H13...O2 <sup>ii</sup>	0.93	2.37	3.2077 (14)	150
C15-H15...O3 <sup>iii</sup>	0.93	2.41	2.9072 (14)	114
C16-H16B...O2 <sup>iii</sup>	0.96	2.44	2.9952 (13)	117

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

H atoms were placed in calculated positions, with C-H = 0.93 or 0.96 Å. The  $U_{\text{iso}}$  values were constrained to be  $1.5U_{\text{eq}}$  of the carrier atom for methyl H atoms and  $1.2U_{\text{eq}}$  for the remaining H atoms.

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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