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5,7-Dimethoxy-2-phenyl-4*H*-1-benzopyran-4-one chloroform solvate

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Key indicators

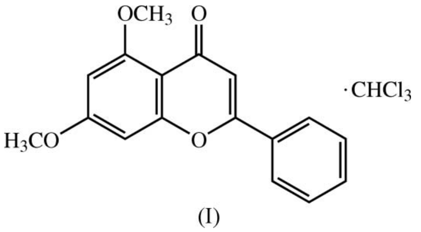
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.102
Data-to-parameter ratio = 22.8

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

The title compound, $C_{17}H_{14}O_4 \cdot CHCl_3$, was isolated from the rhizomes of *Kaempferia parviflora*. The benzopyran-4-one ring system and the phenyl substituent are approximately coplanar. The crystal structure is stabilized by π–π stacking interactions between the benzopyran-4-one ring system of inversion-related molecules stacked along the *c* axis, and C—H···O and C—H···π interactions.

Comment

We have previously reported the crystal structures of three flavonoids, namely 5-hydroxy-3,7-dimethoxy-2-phenyl-4*H*-1-benzopyran-4-one (Fun *et al.*, 2005), 3,5,7-trimethoxy-2-phenyl-4*H*-1-benzopyran-4-one (Teh, Fun, Razak, Boonnak *et al.*, 2005) and 5-hydroxy-7-methoxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one (Teh, Fun, Razak, Chantrapromma *et al.*, 2005) from *Kaempferia parviflora*, or 'Kra Chai Dam' in Thai, which is a medicinal plant growing in the north-eastern part of Thailand. Flavonoids are the main components of this plant. The title compound, (I), is another flavonoid which was isolated from the rhizomes of the same plant. Compound (I) was previously isolated from aerial parts of *Helichrysum nitens* (Francisco *et al.*, 1988). As part of our ongoing studies on the structure and biological activities of Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Fun *et al.*, 2005; Teh, Fun, Razak, Boonnak *et al.*, 2005; Teh, Fun, Razak, Chantrapromma *et al.*, 2005), we have undertaken the X-ray crystal structure determination of (I). The structure–activity relationship (SAR) of these flavonoids will be studied further.



(I)

Bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable to those observed in the closely related structures (Fun *et al.*, 2005; Teh, Fun, Razak, Boonnak *et al.*, 2005; Teh, Fun, Razak, Chantrapromma *et al.*, 2005). Selected bond lengths and angles are given in Table 1. The benzopyran-4-one (C1–C9/O1) ring system is essentially planar, with a maximum deviation 0.029 (1) Å for atom C8. The phenyl substituent at atom C9 is slightly twisted away from the benzopyran-4-one plane, the dihedral angle between the C1–C9/O1 and C10–C15 planes being 7.56 (5)°. The two

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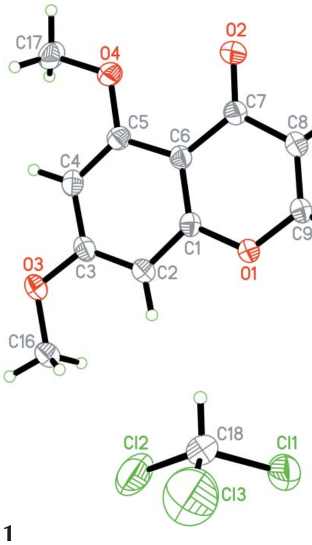

Figure 1
The structure of (I), showing 80% probability displacement ellipsoids and the atomic numbering.

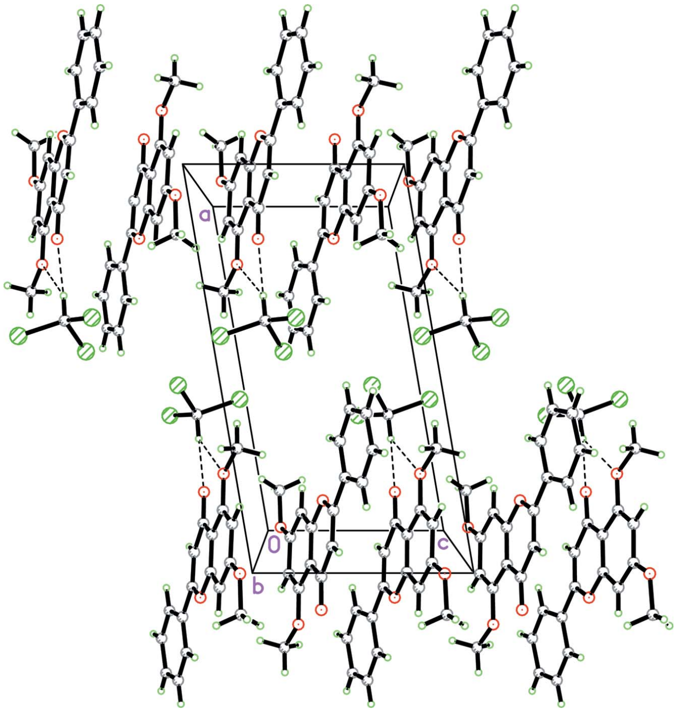

Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

methoxy groups attached at atoms C3 and C5 are almost coplanar with the benzopyran-4-one ring system (Fig. 1).

A weak intramolecular C15—H15*A*···O1 interaction generates an *S*(5) ring motif (Bernstein *et al.*, 1995). The crystal structure is stabilized by π-π stacking interactions between the benzopyran-4-one ring system of inversion-related molecules stacked along the *c* 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 ($-x$, $1-y$, $1-z$) is 3.544 (1) Å. The centroid–centroid distance between the C1–C6 rings at (x, y, z) and ($-x$, $1-y$, $-z$) is 3.602 (1) Å and that between the O1/C1/C6–C9 rings at (x, y, z) and ($-x$, $1-y$, $1-z$) is 3.606 (1) Å. In addition, the molecular stacking is stabilized by C—H···O interactions between the inversion-related molecules, and C—H···π interactions (Table 2) involving the C10–C15 phenyl ring.

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 F6 was subjected to column chromatography (CC) with CH_2Cl_2 to give three fractions (F6*A*–F6*C*). Fraction F6A was recrystallized from $CHCl_3$–CH_3OH (4:1 *v*/*v*) to afford, after several days, colourless block-shaped single crystals of (I) suitable for X-ray diffraction (m.p. 425–426 K).

Crystal data

$C_{17}H_{14}O_4 \cdot CHCl_3$
$M_r = 401.65$
Monoclinic, $P2_1/c$
$a = 13.4036$ (3) Å
$b = 18.8997$ (5) Å
$c = 7.1512$ (2) Å
$\beta = 99.675$ (1)°
$V = 1785.80$ (8) Å^3
$Z = 4$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9157 reflections
$\theta = 1.5$–30.0°
$\mu = 0.53$ mm^{-1}
$T = 100.0$ (1) K
Block, colourless
0.53 × 0.47 × 0.39 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.761$, $T_{max} = 0.812$
57712 measured reflections
5194 independent reflections
4765 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 30.0°$
$h = -18 \rightarrow 18$
$k = -26 \rightarrow 26$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.102$
$S = 1.06$
5194 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 2.0228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.03$ e Å^{-3}
$\Delta\rho_{min} = -1.12$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.7624 (16)	O2—C7	1.2370 (16)
Cl2—C18	1.7635 (17)	O3—C3	1.3580 (15)
Cl3—C18	1.7584 (18)	O3—C16	1.4382 (16)
O1—C9	1.3618 (15)	O4—C5	1.3569 (15)
O1—C1	1.3721 (15)	O4—C17	1.4316 (16)
O1—C1—C2	113.42 (11)	O2—C7—C6	124.97 (12)
C2—C1—C6	124.23 (12)	C8—C7—C6	114.81 (11)
O3—C3—C2	123.97 (12)	C8—C9—C10	126.15 (12)
O3—C3—C4	114.83 (11)	O1—C9—C10	112.20 (10)
O4—C5—C4	123.11 (11)	Cl3—C18—Cl1	109.41 (8)
O4—C5—C6	116.12 (11)	Cl3—C18—Cl2	109.88 (9)
C1—C6—C5	116.20 (11)	Cl1—C18—Cl2	110.45 (9)
C5—C6—C7	125.00 (11)		
C16—O3—C3—C4	176.10 (11)	C17—O4—C5—C6	175.83 (11)

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
C15—H15*A*···O1	0.93	2.37	2.7069 (16)	101
C18—H18*B*···O2$^{\mathrm{i}}$	0.98	2.08	2.9240 (18)	143
C18—H18*B*···O4$^{\mathrm{i}}$	0.98	2.51	3.3407 (18)	142
C12—H12*A*···*Cg*1$^{\mathrm{ii}}$	0.93	2.73	3.3887 (14)	129
C17—H17*B*···*Cg*1$^{\mathrm{i}}$	0.96	2.69	3.5547 (15)	151

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$. *Cg*1 is the centroid of the C10–C15 phenyl ring.

H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{\mathrm{eq}}$ of the carrier atom for methyl H atoms and $1.2U_{\mathrm{eq}}$ for the remaining H atoms. The highest peak and deepest hole are 0.78 and 0.67 Å, respectively, from atom Cl2. The data were collected with an Oxford Cyrosystem 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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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Boonnak, N., Chantrapromma, S., Fun, H.-K., Anjum Ali, S., Rahman, A. A. & Karalai, C. (2005). *Acta Cryst.* E**61**, o410–o412.

Bruker (2005). *APEX2* (Version 1.27), *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, K., Saewan, N., Fun, H.-K., Chantrapromma, S. & Rahman, A. A. (2004). *Acta Cryst.* E**60**, o312–o314.

Chantrapromma, S., Boonnak, N., Fun, H.-K., Anjum, S. & Rahman, A. A. (2005). *Acta Cryst.* E**61**, o2136–o2138.

Chantrapromma, S., Fun, H.-K., Razak, I. A., Laphookhieo, S. & Karalai, C. (2003). *Acta Cryst.* E**59**, o1864–o1866.

Francisco, A., Tomas, B., Jerome, D. M. & Kurt, H. (1988). *Phytochemistry*, **27**, 753–758.

Fun, H.-K., Razak, I. A., Boonnak, N., Laphookhieo, S. & Chantrapromma, S. (2005). *Acta Cryst.* E**61**, o3086–o3088.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Teh, J. B. J., Fun, H.-K., Razak, I. A., Boonnak, N., Chantrapromma, S. & Karalai, C. (2005). *Acta Cryst.* E**61**, o3653–o3655.

Teh, J. B. J., Fun, H.-K., Razak, I. A., Chantrapromma, S., Boonnak, N. & Karalai, C. (2005). *Acta Cryst.* E**61**, o3715–o3717.