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2,3-Dihydro-7-hydroxy-3-[(4-methoxyphenyl)-methylene]-4*H*-1-benzopyran-4-one

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Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.106
Data-to-parameter ratio = 13.5

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

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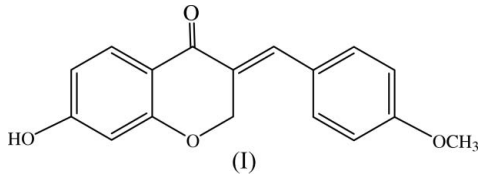
The title compound, $C_{17}H_{14}O_4$, a homoisoflavanoid, was isolated from the twigs and stems of *Caesalpinia digyna* Rottler. The pyran ring adopts an envelope conformation. The symmetry-related molecules are linked *via* O—H···O and C—H···O intermolecular hydrogen bonds to form a molecular network.

Comment

Caesalpinia digyna Rottler, locally known in Thailand as 'kamchai', belongs to the family Leguminosae-Caesalpinioideae (Smitinand, 2001). The genus *Caesalpinia* occurs mainly in the tropics and subtropics (Kinoshita *et al.*, 2005). Several members of the species *Caesalpinia* have been used traditionally for a wide variety of ethnomedical properties (Anonymous, 1992). We have isolated the title compound, (I), bonducellin (Fig. 1), for the first time from the twigs and stems of *C. digyna*, which were collected from Songkhla province in the southern part of Thailand. Compound (I) was previously isolated from *Caesalpinia pulcherrima* (Srinivas *et al.*, 2003). Our antimicrobial activity testing shows that (I) exhibits antimicrobial activities against BS (*Bacillus subsitilis*). In our continuing search for bioactive compounds from Thai medicinal plants (Chantrapromma *et al.*, 2003, 2004, 2005; Boonnak *et al.*, 2005; Fun *et al.*, 2005; Ng *et al.*, 2005; Pakhathirathien *et al.*, 2005; Teh *et al.*, 2005), we have determined the structure of (I) by X-ray analysis in order to establish its relative stereochemistry.



(I)

In the benzopyran-4-one (C1–C9/O1) ring system, the pyran ring (C1/C6–C9/O1) is in an envelope form, with

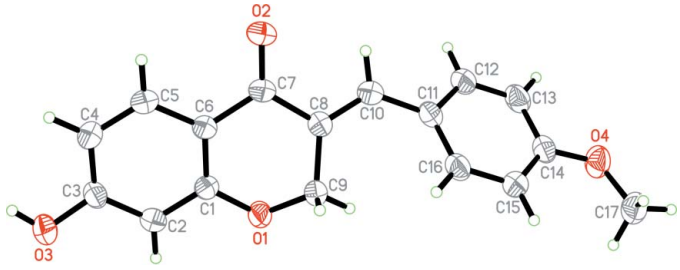


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

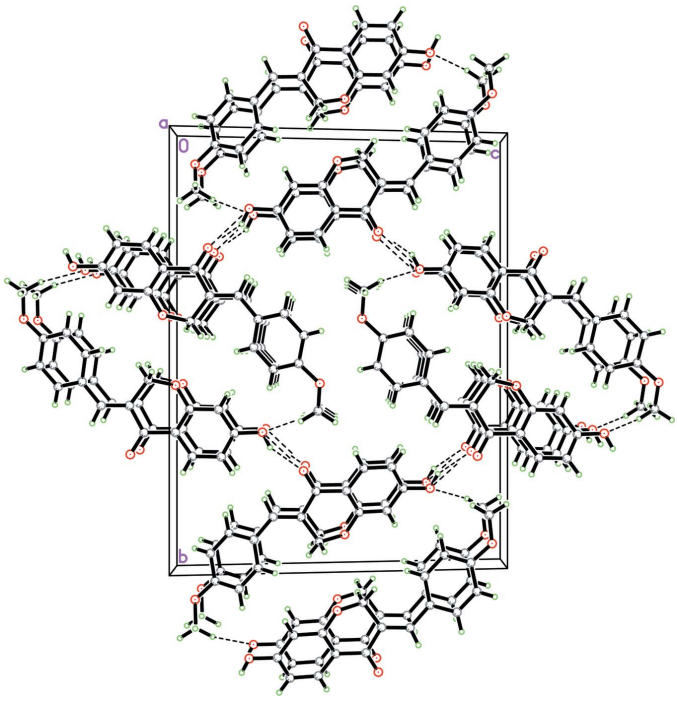


Figure 2
The crystal packing of (I), viewed down the *a* axis, showing the molecular network. Hydrogen bonds are shown as dashed lines.

puckering parameter (Cremer & Pople, 1975) Q = 0.377 (2) Å, θ = 118.9 (3)° and φ = 127.0 (3)°. The deviation of the puckered C9 atom from the C1/C6–C8/O1 plane is 0.519 (2) Å. The (4-methoxyphenyl)methylene substituent (C10–C17/O4) is attached to the pyran ring at atom C8, the torsion angle C7—C8—C10—C11 of 175.48 (18)° indicating an (+)-anti-periplanar conformation (Fig. 1). The dihedral angle between the O1/C1–C8 and C11–C16 planes is 54.15 (4)°. The methoxy group attached at atom C14 is slightly twisted away from the benzene ring [C17—O4—C14—C13 = 168.63 (17)° and C17—O4—C14—C15 = −10.9 (3)°]. All bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). Selected bond lengths and angles are given in Table 1.

In the crystal packing, atom O2 is involved in an intermolecular O—H···O hydrogen bond and intramolecular C—H···O weak interaction, while atoms O4 and O3 are involved in weak C—H···O interactions (Table 2). These hydrogen bonds link the symmetry-related molecules to form a molecular network (Fig. 2).

Experimental

Air-dried twigs and stems of *C. digyna* were extracted with CH_2Cl_2 (3 × 15 l) at room temperature. The residue obtained after evaporation of the solvent was separated by quick column chromatography (QCC) over silica gel and eluted with an acetone–hexane gradient system to give twelve fractions (F1–F12). Fraction F7 (1.25 g) was re-chromatographed on a silica gel column with 5% EtOAc/CH_2Cl_2 to afford nine subfractions (F7A–F7I). Compound (I) was obtained from subfraction F7D. Crystals of (I) suitable for single-crystal X-ray diffraction studies were obtained as colorless needles by recrystallization from $CHCl_3$–CH_3OH (4:1 *v*/*v*) after several days (m.p. 488–490 K).

Crystal data

$C_{17}H_{14}O_4$
M_r = 282.28
Monoclinic, $P2_1/c$
a = 3.9591 (11) Å
b = 20.939 (5) Å
c = 16.209 (4) Å
β = 99.844 (7)°
V = 1323.9 (6) Å^3
Z = 4
D_x = 1.416 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2593 reflections
θ = 1.6–26.0°
μ = 0.10 mm^{-1}
T = 297 (2) K
Needle, colorless
0.55 × 0.05 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.994, T_{max} = 0.996
13570 measured reflections
2593 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 26.0°
$h = -4 \rightarrow 4$
$k = -25 \rightarrow 25$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.106
S = 1.08
2593 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.4026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.364 (2)	O4—C14	1.364 (2)
O1—C9	1.444 (2)	O4—C17	1.429 (2)
O2—C7	1.236 (2)	C8—C10	1.340 (2)
O3—C3	1.351 (2)	C10—C11	1.460 (2)
C1—O1—C9	116.08 (13)	C10—C8—C9	124.45 (16)
C14—O4—C17	118.30 (15)	C7—C8—C9	115.68 (14)
O1—C1—C2	116.48 (15)	C8—C10—C11	130.10 (17)
O1—C1—C6	122.26 (15)	C16—C11—C12	117.16 (16)
O3—C3—C2	116.57 (16)	C16—C11—C10	123.33 (16)
O3—C3—C4	123.04 (16)	O4—C14—C13	115.61 (16)
O2—C7—C8	122.30 (16)	O4—C14—C15	124.56 (17)
C6—C7—C8	116.01 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	1.94	2.686 (2)	151
C10—H10···O2	0.93	2.47	2.826 (2)	103
C13—H13···O4^{ii}	0.93	2.59	3.506 (3)	168
C17—H17B···O3^{iii}	0.96	2.49	3.398 (2)	158

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

H atoms were placed in calculated positions, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.97 Å. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for hydroxy and methyl H atoms, and 1.2U_{eq} for the remaining H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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* and *PLATON* (Spek, 2003).

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