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4-Methoxy-7-phenyl-6,7-dihydrofuro[3,2-*g*]-chromen-5-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.122
Data-to-parameter ratio = 19.2

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

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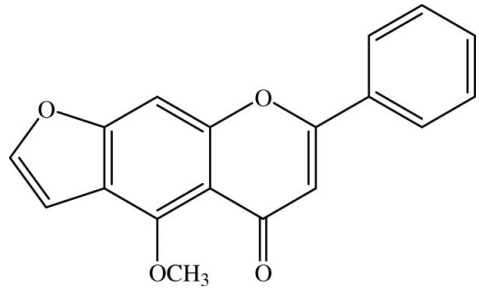
The title compound, $C_{18}H_{12}O_4$, known as pinnatin, is a furanoflavone isolated from *Derris indica* (Lam.). The furanoflavone nucleus is almost planar. The phenyl ring is axially attached to the furanoflavone skeleton. The methoxy group deviates slightly from the plane of the molecule. The molecules are linked in a zigzag manner through C—H···O interactions into molecular ribbons along the *b* axis. Further stabilization is provided by weak C—H···π and π–π interactions.

Comment

While screening for biologically active substances and chemically novel constituents of Asian medicinal plants and folk medicines (Boonnak *et al.*, 2005; Chantrapromma *et al.*, 2005; Pakhathirathien *et al.*, 2005; Chantrapromma, Boonnak *et al.*, 2006; Chantrapromma, Boonsri *et al.*, 2006), the title compound, (I), known as pinnatin, was isolated from *Derris indica* (Lam.), a mangrove plant belonging to the Leguminosae family, collected from Nakhon Si Thammarat province in southern Thailand. Different parts of this plant have been used in folk medicine for the treatment of bronchitis, whooping cough and rheumatic joints, and to reduce dipsia in diabetes (Yadav *et al.*, 2004). Investigation of the crude hexane and dichloromethane extracts of the stems and roots of *D. indica* (Lam.) showed moderate antimycobacterial activity against mycobacterium tuberculosis H37Ra. Pinnatin was previously isolated from the roots of *Gelonium multiflorum* (Das *et al.*, 1994) and *Fordia cauliflora* (Dai *et al.*, 2003). Its antifungal activity was reported previously (Pan *et al.*, 1985). The results of our antimycobacterial activity investigation show antimycobacterial activity of (I) against mycobacterium tuberculosis H37Ra with a minimum inhibition concentration of 12.5 μg ml^{-1}. It was inactive against NCI-H187 cell lines (human small-cell lung cancer).



(I)

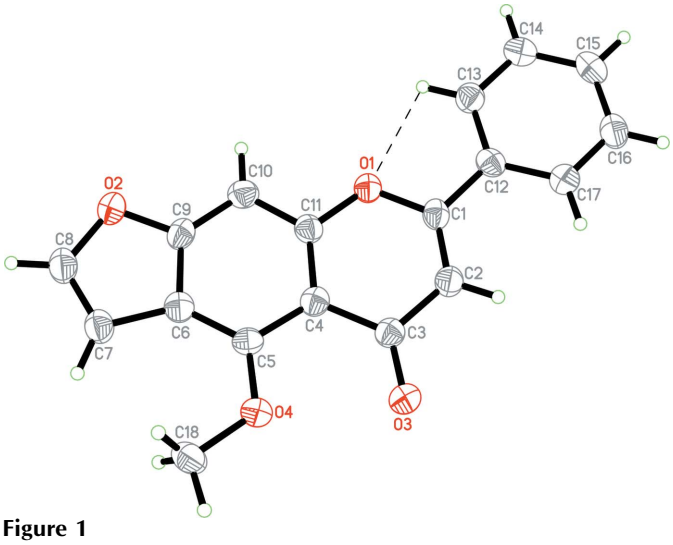


Figure 1
The asymmetric unit of (I), showing 80% probability displacement ellipsoids and the atomic numbering. The dashed line indicates the intramolecular hydrogen bond.

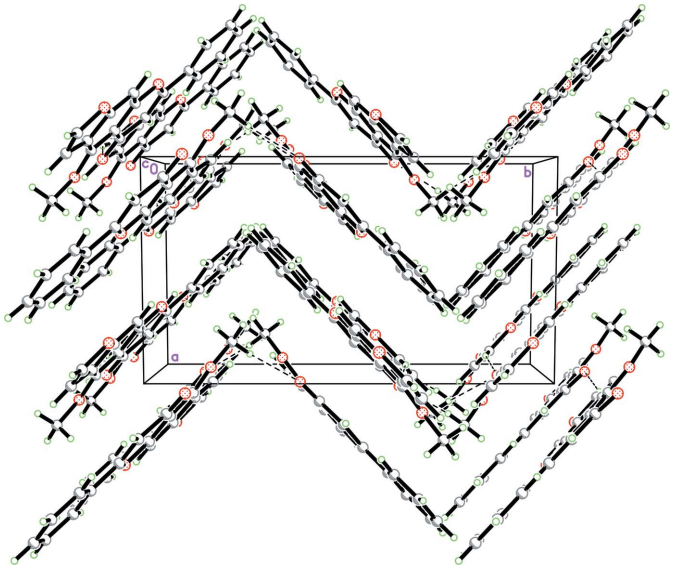


Figure 2
The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

In compound (I), the furanoflavone nucleus [O1–O3/C1–C11] is essentially planar, with a maximum deviation of 0.049 (2) Å for C2. The methoxy group deviates slightly from the C4–C6/C9–C11 ring plane [C18—O4—C5—C6 torsion angle 175.31 (13)°]. The C2—C1—C12—C13 torsion angle [−179.98 (15)°] indicates the axial attachment of the phenyl ring with respect to the furanoflavone nucleus. The dihedral angle between the least-squares planes of the furanoflavone and the C12–C17 phenyl ring is 3.36 (6)°. Bond lengths and angles (Table 1) show normal values (Allen *et al.*, 1987) and are comparable with those in closely related structures (Beale, 1973; El-Sayed *et al.*, 1988).

The methoxy group is involved in weak C—H···O intra and intermolecular interactions (Table 2). Molecules are linked into ribbons along the *b* axis in a zigzag manner through C8—H8···O3 and C18—H18*A*···O3 hydrogen bonds (Fig. 2, Table 2). Additional stability derives from weak C18—H18*C*···*Cg*1 interactions, where *Cg*1 is the centroid of the C12–C17 phenyl ring (Table 2). Furthermore, the centroid–centroid distance between the O2/C6–C9 and O1/C1–C4/C11 rings of adjacent molecules, at $(1-x,-y,1-z)$, is 3.5155 (9) Å and that between the O2/C6–C9 and C12–C17 rings of adjacent molecules, at $(2-x,-y,2-z)$, is 3.5556 (9) Å, indicating additional π–π interactions.

Experimental

The air-dried powdered stems of *Derris indica* (Lam.) (22.0 kg) were extracted with hexane (40 l) and CH_2Cl_2 (40 l) at room temperature. The extracts were combined and concentrated under vacuum to yield a brown gummy residue (87.51 g), which was subjected to flash column chromatography over silica gel and then eluted with hexane and three further solvents in order of increasing polarity, CH_2Cl_2, EtOAc and CH_3OH, to give 16 fractions (F1–F16). Fraction F12 (10.09 g) was further purified by column chromatography on Sephadex LH-20 to afford compound (I) (486.1 mg). Colourless plate-like single crystals of (I) were obtained by recrystallization from $CHCl_3$–hexane (4:1 *v*/*v*) after several days (m.p. 454–456 K).

Crystal data

$C_{18}H_{12}O_4$
M_r = 292.28
Monoclinic, $P2_1/c$
a = 8.6174 (1) Å
b = 15.5622 (3) Å
c = 9.8634 (1) Å
β = 96.945 (1)°
V = 1313.03 (3) Å^3
Z = 4
D_x = 1.479 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100.0 (1) K
Slab, colourless
0.39 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.960, T_{max} = 0.990
32598 measured reflections
3821 independent reflections
2950 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 30.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.122
S = 1.03
3821 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 1.1211P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3631 (18)	O3—C3	1.2382 (19)
O1—C11	1.3744 (18)	O4—C5	1.3473 (18)
O2—C9	1.3667 (18)	O4—C18	1.4331 (18)
O2—C8	1.3912 (19)	C7—C8	1.337 (2)
C5—O4—C18	119.46 (12)	C2—C1—C12	126.08 (14)
C1—C2—C3—O3	175.41 (15)	C18—O4—C5—C4	175.31 (13)
O3—C3—C4—C11	−176.22 (15)	C2—C1—C12—C13	−179.98 (15)
C18—O4—C5—C6	−2.9 (2)		

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

*Cg*1 is the centroid of the C12–C17 phenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O3^{i}	0.93	2.49	3.3772 (19)	159
C13—H13A···O1	0.93	2.37	2.7070 (19)	101
C18—H18A···O3^{ii}	0.96	2.49	3.450 (2)	174
C18—H18C···$Cg1^{iii}$	0.96	2.69	3.3621 (18)	128

Symmetry codes: (i) $x, y, z+1$; (ii) $x, -y-\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$.

H atoms were placed in calculated positions, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.96 Å. The U_{iso}(H) 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: *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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