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6-Methylflavone

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.127
Data-to-parameter ratio = 16.8

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

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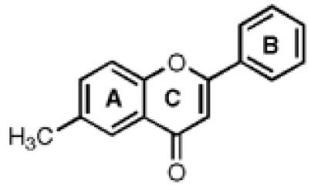
In the title molecule, $C_{16}H_{12}O_2$, the phenyl and quinone rings are effectively planar, with maximum deviations from planarity of 0.007 (2) and 0.008 (1) Å, respectively. The γ-pyrone ring makes a dihedral angle of 8.9 (3)° with the 2-phenyl substituent.

Comment

Flavones and related compounds are known to exhibit a wide range of interesting biological activities (Agullo *et al.*, 1997; Carlo *et al.*, 1993; Miksicek, 1993; Wang *et al.*, 1999). 6-Methylflavone is one of a number of flavones that have interesting modulatory activities at GABA–A receptors (Medina *et al.*, 1998; Chebib & Johnston (2000). The title compound, (I), was synthesized as part of an on-going structure–activity study to determine the properties of these compounds that confer this activity in order to aid the design of more active compounds.



(I)

All bond lengths and angles in (I) (Fig. 1) are as expected. Rings *AC* and *B* are planar; the maximum deviations are 0.008 (2) and 0.007 (1) Å from the ring planes *AC* and *B*, respectively.

The average C—C bond lengths for rings *A* and *B* are 1.394 (2) and 1.383 (2) Å, respectively. The dihedral angle between the phenyl and the γ-pyrone ring is small [8.9(3) Å] as expected in the generally preferred conformation of flavones. The small dihedral angle results in a relatively short C3—C10 bond length of 1.472 (2) Å which is consistent with bond lengths and dihedral angles found in other flavones. Flavone-3′-sulfonamide has a dihedral angle of 8.2 (3)° and the C3—C10 bond length of 1.478 (3) Å (Kendi *et al.*, 2000). In 5-hydroxyflavone, the dihedral angle is 5.2 (9)° and the C3—C10 bond length is 1.465 (4) Å (Shoja, 1990). 5,7–Dihydroxy-4′-methoxyflavone with a dihedral angle of 3.1° has a C3—C10 bond length of 1.453 (9) Å (Shoja, 1992). However, in 2′-methyl-3′-nitroflavone, the dihedral angle is 139.8 (2)° and the C3—C10 bond length is 1.491 (8) Å (Kendi *et al.*, 1996), and in 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone a large dihedral angle of 164.4 (6)° and a C2—C10 bond length of 1.503 (8) Å

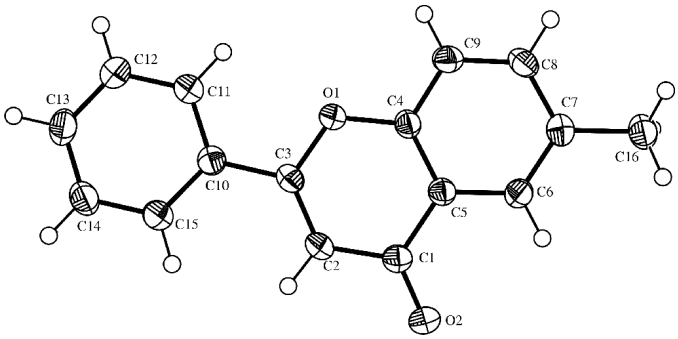


Figure 1
A general view of the molecular structure of (I). The ellipsoids are drawn at the 50% probability level (Farrugia, 1997).

are found (Vijayalakshmi *et al*., 1986).

The small dihedral angle between the phenyl and the γ-pyrone ring and shorter C1′—C2 bond length result in less delocalization of the π electrons in C3—C2—C1—O2, resulting in longer C2═C3 and C4═O2 bond lengths. 6-Methylflavone has a C2═C3 bond length of 1.348 (2) Å and a C4═O2 bond length of 1.237 (2) Å, a situation which is similar to flavone 3′-sulfonamide with a dihedral angle of 5.2 (9)°, and C2═C3 and C4═O2 bond lengths of 1.346 (3) and 1.247 (3) Å, respectively (Kendi *et al.*, 2000). Conversely, 2′-methyl-3′nitro-flavone with the larger dihedral angle of 139.8 (2)° and longer C3—C10 bond length, has shorter C2═C3 and C4═O2 bond lengths of 1.322 (9) and 1.227 (8) Å, respectively (Kendi *et al.*, 1996).

The widening of the O1—C4—C5 angle to 122.32 (13)° and the narrowing of the C2—C1—C5 angle to 114.51 (14)° in the γ-pyrone ring may be attributed to the ring strain caused by the neighbouring Csp^2—Csp^2 atoms.

Experimental

6-Methylflavone was obtained by the Baker–Venkataraman method and the structure confirmed by ^{1}H, and ^{13}C NMR, mass spectrometry and IR spectroscopy. The product was recrystallized by slow evaporation from methanol (1.64 g, 60%), m.p. 397–400. λ_{max}/cm^{-1} 2900 (ArC–H), 1640 (C═O), 1450, 1375, 1300, 1220, 1165, 1130, 1085, 1040, 1025, 975, 900, 850, 835, 815, 770, 720, 660; δH (300 MHz; $CDCl_3$) 2.48 (3H, *s*, CH3), 6.83 (1H, *s*, C3—H), 7.49–7.57 (5H, *m*, C2′—H, C3′—H, C4′—H), 7.92–7.96 (2H, *m*, C7—H, C8—H), 8.03 (1H, *symm. m*, C5—H); δC (75 MHz; $CDCl_3$) 21.01 (CH_3), 107.50 (C-8), 117.93 (C-3), 123.72 (C-5a), 125.14 (C-4), 126.33 (C-3′), 129.09 (C-2′), 131.56 (C-5), 132.00 (C-1′), 135.04 (C-7), 135.26 (C-6), 154.63 (C-2), 163.29 (C-8a), 178.55 (C-4); *m*/*z* 236 ([M]+, 100%), 235 (31), 208 (51), 134 (29), 106 (19), 105 (28), 78 (12), 77 (14).

Crystals of (I) were mounted using silicone oil which acted as both a coating and an adhesive.

Crystal data

$C_{16}H_{12}O_2$	D_x = 1.335 Mg m^{-3}
M_r = 236.26	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 999 reflections
a = 4.7103 (8) Å	θ = 1.9–28.3°
b = 11.684 (2) Å	μ = 0.09 mm^{-1}
c = 21.352 (4) Å	T = 150 (2) K
β = 90.043 (3)°	Prism, colourless
V = 1175.1 (3) Å^3	0.25 × 0.15 × 0.15 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD diffractometer	1644 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.033
Absorption correction: none	θ_{max} = 28.3°
7396 measured reflections	$h = -6 \rightarrow 6$
2758 independent reflections	$k = -10 \rightarrow 15$
	$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.127	$(\Delta/\sigma)_{max} < 0.001$
S = 0.93	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
2758 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
164 parameters	

H atoms were included in calculated positions (riding model) with U_{iso} set at 1.2(CH) and 1.5(CH_3) times the U_{eq} of the parent atoms.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 *for Windows* (Farrugia, 1997).

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References

Agullo, G., Gamet-Payrastre, L., Manenti, S., Viala, C., Remesy, C., Chap, H. & Payrastre, B. (1997). *Biochem. Pharmacol.* **53**, 1649–1657.
Carlo, G. D., Autore, G., Izzo, A. A., Maiolino, P., Mascolo, N., Viola, P., Diurno, M. V. & Capasso, F. (1993). *J. Pharm. Pharmacol.* **45**, 1054–1059.
Chebib, M. & Johnston, G. A. R. (2000). *J. Med. Chem.* **43**, 1427–1447.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kendi, E.,Özbey, S., Bozdag, O. & Ertan, R. (2000). *Acta Cryst.* C**56**, 457–458.
Kendi, E., Özbey, S., Tuncbilek, M. & Ertan, R. (1996). *Cryst. Res. Technol.* **5**, 457–458.
Medina, J. H., Viola, H., Wolfman, C., Marder, M., Wasowski, C., Calvo, D. & Paladini, A. C. (1998). *Phytomed.* **5**, 235–243.
Miksicek, R. J. (1993). *Mol. Pharmacol.* **44**, 37–43.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Shoja, M. (1990). *Acta Cryst.* C**46**, 517–519.
Shoja, M. (1992). *Acta Cryst.* C**48**, 2033–2035.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Vijayalakshmi, J., Rajan, S. S., Srinivasan, R. & Ramachandran Nair, A. G. (1986). *Acta Cryst.* C**42**, 1752–1753.
Wang, I.-K., Lin-Shiau, S.-Y. & Lin, J.-K. (1999). *Eur. J. Cancer*, **35**, 1517–1525.