Crystal structure of 3-hydroxy-7-methoxyflavone, $C_{16}H_{12}O_4$

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$C_{16}H_{12}O_4$, monoclinic, $P2_1/c1$ (No. 14), $a = 14.780(3)$ Å, $b = 4.90(1)$ Å, $c = 17.686(5)$ Å, $\beta = 103.60(3)^\circ$, $V = 1243.9$ Å³, $Z = 4$, $R(F) = 0.049$, $R_w(F) = 0.030$.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless needle, size 0.2 x 0.2 x 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	1.1 cm ⁻¹
Diffraction:	Enraf-Nonius CAD4
Scan mode:	$\omega/2\theta$
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{unique}}$:	1315
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	229
Program:	Xtal3.4

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(111)	4e	0.159(3)	1.792(8)	0.598(2)	0.07(1)
H(112)	4e	0.145(3)	1.49(1)	0.653(2)	0.11(2)
H(113)	4e	0.234(3)	1.76(1)	0.678(3)	0.12(2)
H(2')	4e	0.445(2)	0.643(7)	0.435(2)	0.05(1)
H(3')	4e	0.571(2)	0.371(7)	0.418(2)	0.06(1)
H(4')	4e	0.545(2)	0.018(7)	0.323(2)	0.04(1)
H(5')	4e	0.399(2)	-0.069(8)	0.245(2)	0.06(1)
H(6')	4e	0.272(2)	0.237(7)	0.259(2)	0.050(9)
H(5)	4e	0.016(2)	1.148(7)	0.421(2)	0.06(1)
H(6)	4e	0.080(2)	1.451(7)	0.529(2)	0.07(1)
H(8)	4e	0.335(2)	1.136(6)	0.537(1)	0.033(7)
H(33)	4e	0.082(3)	0.35(1)	0.260(3)	0.26(2)

Source of material: Indofine Chemical Company. Crystals grown from toluene/ethanol, mp 450 K – 451 K.

An intramolecular H-bond exists between the oxygen of the hydroxyl and the H6' of the phenyl ring ($d(\text{O3} \cdots \text{H6}') = 2.15$ Å). In addition, a bifurcated H-bond is formed as a result of an inter ($d(\text{O4} \cdots \text{H33}) = 1.87$ Å) and an intramolecular ($d(\text{O4} \cdots \text{H33}) = 2.48$ Å) attraction between the 3-hydroxy group and the γ -pyrone carbonyl (see ref. 1). The C2–C1' is 1.462(5) Å and the C6'–C1'–C2–C3 torsion angle is equal to 1.2(6)°. This torsion angle is the lowest reported thus far for flavones (see refs. 2, 3). From AM1 calculations with strict convergence criteria the above torsion angle was found to be 28.8°, with a minimum in the heat of formation plot equal to -73.2 kcal. The same calculations were carried out for the four molecules at equivalent positions in the unit cell. The C6'–C1'–C2–C3 torsion angle was lowered to 5.4°. This drop in angle can be explained as the result of intermolecular interactions that supersede the intramolecular effects. In conclusion, the packing effects alter the configuration of the molecules in the unit cell with respect to their geometry as isolated molecules.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e	0.2960(1)	0.7985(5)	0.4275(1)	0.026(1)	0.051(2)	0.041(1)	0.006(1)	0.003(1)	-0.006(1)
C(4)	4e	0.1062(2)	0.7780(8)	0.3521(2)	0.033(2)	0.048(3)	0.045(2)	0.002(2)	0.005(2)	0.010(2)
C(10)	4e	0.1392(2)	0.9583(8)	0.4161(2)	0.029(2)	0.044(3)	0.040(2)	0.004(2)	0.005(1)	0.006(2)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(9)	4e	0.2346(2)	0.9671(8)	0.4519(2)	0.030(2)	0.044(3)	0.037(2)	0.007(2)	0.007(1)	0.005(2)
C(3)	4e	0.1754(2)	0.6086(8)	0.3294(2)	0.031(2)	0.047(3)	0.038(2)	−0.005(2)	0.005(2)	0.002(2)
C(2)	4e	0.2672(2)	0.6189(8)	0.3668(2)	0.031(2)	0.044(3)	0.032(2)	−0.003(2)	0.005(1)	0.002(2)
C(8)	4e	0.2705(2)	1.1383(9)	0.5137(2)	0.030(2)	0.050(3)	0.041(2)	0.003(2)	0.005(2)	0.002(2)
C(7)	4e	0.2113(2)	1.3065(8)	0.5424(2)	0.044(2)	0.048(3)	0.040(2)	0.008(2)	0.009(2)	0.006(2)
O(4)	4e	0.0231(1)	0.7550(6)	0.3169(1)	0.027(1)	0.074(2)	0.067(2)	0.002(1)	−0.008(1)	−0.001(1)
C(1')	4e	0.3449(2)	0.4577(8)	0.3528(2)	0.034(2)	0.040(3)	0.035(2)	−0.001(2)	0.010(1)	0.004(2)
O(3)	4e	0.1455(2)	0.4338(6)	0.2689(1)	0.033(1)	0.077(2)	0.048(1)	−0.008(1)	−0.001(1)	−0.013(2)
O(7)	4e	0.2516(2)	1.4644(6)	0.6036(1)	0.052(1)	0.064(2)	0.052(2)	0.014(2)	0.010(1)	−0.014(2)
C(6)	4e	0.1154(2)	1.3036(9)	0.5083(2)	0.041(2)	0.053(3)	0.057(3)	0.012(2)	0.013(2)	0.001(1)
C(5)	4e	0.0817(2)	1.1353(9)	0.4465(2)	0.029(2)	0.064(3)	0.059(3)	0.009(2)	0.008(2)	0.008(2)
C(2')	4e	0.4346(2)	0.492(1)	0.4000(2)	0.037(2)	0.061(3)	0.047(2)	0.005(2)	0.005(2)	−0.013(2)
C(6')	4e	0.3346(3)	0.2632(9)	0.2946(2)	0.042(2)	0.055(3)	0.050(2)	0.001(2)	0.006(2)	−0.006(2)
C(3')	4e	0.5078(3)	0.336(1)	0.3892(2)	0.034(2)	0.071(3)	0.056(3)	0.007(2)	0.007(2)	−0.011(2)
C(4')	4e	0.4958(3)	0.144(1)	0.3323(2)	0.044(2)	0.057(3)	0.058(3)	0.010(2)	0.023(2)	0.000(2)
C(5')	4e	0.4081(3)	0.108(1)	0.2835(2)	0.052(2)	0.062(3)	0.057(3)	0.002(2)	0.018(2)	−0.013(3)
C(11)	4e	0.1926(4)	1.639(1)	0.6379(3)	0.069(3)	0.060(4)	0.057(3)	0.021(3)	0.018(3)	−0.005(3)

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