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Crystal structure of 2′-hydroxyflavanone, $C_{15}H_{12}O_3$

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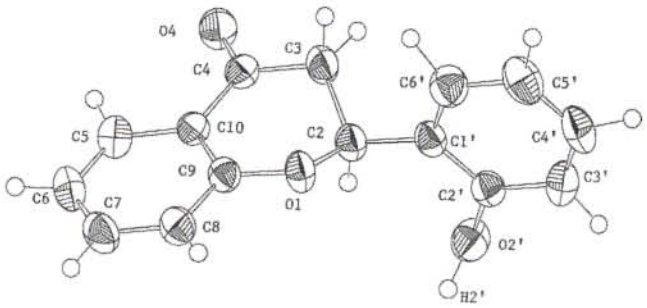

Abstract

$C_{15}H_{12}O_3$, monoclinic, $C12/c1$ (No. 15), a = 24.021(5) Å, b = 4.978(4) Å, c = 20.996(2) Å, β = 112.69(1)°, V = 2316.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.060, $R_w(F)$ = 0.070, T = 293 K.

Source of material

Indofine Chemical Company. Crystals grown from petroleum ether, mp 437 K – 438 K.

Discussion

The planar phenyl ring is in the equatorial position. The twist of this ring relative to the rest of the molecule, as characterized by the O1–C2–C1′–C2′ torsion angle, is 114.1(5)°. The dihedral angle between the plane of the chromon and the phenyl ring is 88.9(2)°. In contrast to flavone, where the range of the dihedral angles for the same rings can vary from 1° to 72° [1, 2], due to inter- and intra-molecular H-bonding and other crystal packing effects, flavanone crystal structures exhibit only a narrow range of dihedral angles, 70° – 90° [3, 4]. The crystal structure of 4′-hydroxy-5,7-dimethoxyflavanone is the only exception with a reported range of 36° and 69°, possibly due to the unusual packing of two molecules in the asymmetric unit [5].

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.2 x 0.2 x 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	50.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4806, 2038
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 1574
$N(param)_{refined}$:	211
Program:	Xtal3.4 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	8*f*	0.488(2)	0.182(9)	0.313(2)	0.04(1)
H(5′)	8*f*	0.678(2)	0.71(1)	0.499(2)	0.05(1)
H(4′)	8*f*	0.737(2)	0.46(1)	0.450(2)	0.05(1)
H(5)	8*f*	0.289(2)	0.405(9)	0.278(2)	0.04(1)
H(32)	8*f*	0.481(2)	0.70(1)	0.291(2)	0.06(2)
H(31)	8*f*	0.481(2)	0.74(1)	0.363(2)	0.06(2)
H(8)	8*f*	0.432(2)	−0.09(1)	0.460(2)	0.04(1)
H(6′)	8*f*	0.574(2)	0.68(1)	0.455(2)	0.06(1)
H(6)	8*f*	0.258(2)	0.09(1)	0.343(3)	0.07(2)
H(3′)	8*f*	0.689(2)	0.10(1)	0.367(2)	0.06(2)
H(7)	8*f*	0.333(2)	−0.18(1)	0.436(2)	0.07(2)
H(2′)	8*f*	0.569(3)	−0.16(1)	0.326(3)	0.26(3)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	8*f*	0.4837(1)	0.2068(7)	0.4065(2)	0.031(2)	0.053(2)	0.040(2)	−0.001(2)	0.015(1)	0.011(2)
C(9)	8*f*	0.4226(2)	0.1784(9)	0.3877(2)	0.036(2)	0.037(3)	0.035(2)	−0.002(2)	0.018(2)	−0.004(2)
C(10)	8*f*	0.3806(2)	0.3279(9)	0.3340(2)	0.035(2)	0.033(2)	0.036(2)	0.000(2)	0.016(2)	−0.002(2)
C(4)	8*f*	0.4009(2)	0.5330(9)	0.2981(2)	0.038(2)	0.035(2)	0.038(2)	0.001(2)	0.017(2)	−0.003(2)
O(4)	8*f*	0.3661(1)	0.6710(7)	0.2510(2)	0.043(2)	0.051(2)	0.049(2)	0.006(2)	0.016(2)	0.016(2)
C(1′)	8*f*	0.5689(2)	0.3476(9)	0.3830(2)	0.034(2)	0.038(3)	0.037(2)	−0.003(2)	0.014(2)	0.006(2)
C(2′)	8*f*	0.6040(2)	0.1913(9)	0.3571(2)	0.037(2)	0.038(3)	0.035(2)	−0.001(2)	0.012(2)	0.005(2)
C(8)	8*f*	0.4038(2)	−0.007(1)	0.4245(3)	0.048(3)	0.046(3)	0.043(3)	0.001(2)	0.024(2)	0.007(2)
O(2′)	8*f*	0.5758(1)	0.0118(8)	0.3052(2)	0.041(2)	0.058(2)	0.052(2)	−0.001(2)	0.010(2)	−0.015(2)
C(3)	8*f*	0.4679(2)	0.575(1)	0.3250(3)	0.036(3)	0.044(3)	0.056(3)	0.001(2)	0.021(2)	0.013(3)
C(5)	8*f*	0.3190(2)	0.282(1)	0.3176(2)	0.038(3)	0.054(3)	0.042(3)	0.000(2)	0.016(2)	0.000(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(3′)	8*f*	0.6661(2)	0.226(1)	0.3830(3)	0.036(3)	0.062(4)	0.047(3)	0.006(3)	0.019(2)	0.008(3)
C(2)	8*f*	0.5014(2)	0.317(1)	0.3530(2)	0.035(2)	0.042(3)	0.041(2)	–0.002(2)	0.018(2)	0.002(2)
C(7)	8*f*	0.3433(2)	–0.048(1)	0.4074(3)	0.053(3)	0.053(3)	0.054(3)	–0.008(3)	0.032(3)	0.004(3)
C(6′)	8*f*	0.5974(2)	0.530(1)	0.4350(3)	0.049(3)	0.053(3)	0.050(3)	–0.003(3)	0.021(2)	–0.005(3)
C(5′)	8*f*	0.6596(2)	0.566(1)	0.4609(3)	0.049(3)	0.067(4)	0.046(3)	–0.019(3)	0.011(2)	–0.009(3)
C(6)	8*f*	0.3006(2)	0.096(1)	0.3537(3)	0.039(3)	0.060(3)	0.056(3)	–0.009(3)	0.024(2)	–0.002(3)
C(4′)	8*f*	0.6931(2)	0.413(1)	0.4341(3)	0.034(3)	0.065(4)	0.049(3)	–0.013(3)	0.010(2)	0.009(3)

References

1. Shoja, M.; Sullivan, P.; Athanasopoulos, D.; Kabbani, R.: Crystal structure of 3-hydroxy-7-methoxyflavone. Z. Kristallogr. NCS **213** (1998) 375-376.
2. Wallet, J.-C.; Gaydou, E. M.; Baldy, A.: Structure of 2-(2,6-dimethoxy phenyl)-4*H*-1-benzopyran-4-one(2′,6′-dimethoxyflavone). Acta Crystallogr. C**45** (1989) 512-515.
3. Shoja, M.; Samuel, K.; Athanasopoulos, D.: Crystal structure of 6-hydroxy flavanone. Z. Kristallogr. NCS **213** (1998) 373-374.
4. Shoja, M.: The crystal structure of 5-hydroxy-7-methoxyflavanone. Z. Kristallogr. **189** (1989) 89-93.
5. Shoja, M.: Crystal structure of 4′-hydroxy-5,7-dimethoxyflavanone. Z. Kristallogr. NCS **212** (1997) 127-128.
6. Hall, S. R.; King, G. S. D.; Stewart, J. M. (Eds.): Xtal3.4 User's Manual. University of Western Australia, Lamb, Perth 1995.