Crystal structure of 5,2′-dihydroxyflavone, $C_{15}H_{10}O_4$

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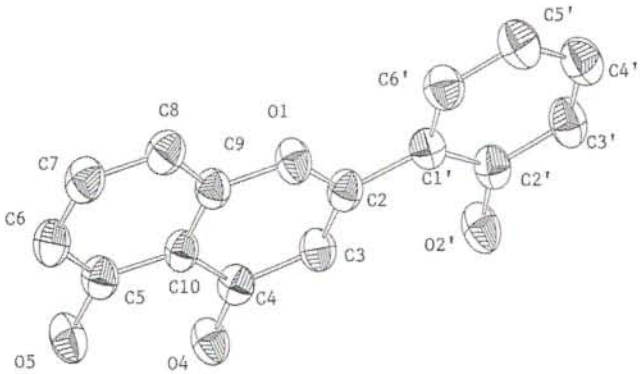

Abstract

$C_{15}H_{10}O_4$, monoclinic, $P12_1/c1$ (No. 14), a = 5.331(4) Å, b = 9.179(2) Å, c = 23.057(4) Å, β = 95.62(3)°, V = 1122.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{gt}(F)$ = 0.045, T = 293 K.

Source of material

Crystals of the title compound (Indofine Chemical Company) grown from ethyl acetate solution.

Discussion

The structure from X-ray data and the IR spectrum (C=O stretch at 1650 cm^{-1}) show no evidence of intermolecular H-bonding for the OH group at C5 [1]. An intramolecular H-bond (d(O4···H5—O5) = 1.94 Å) is formed between the hydrogen of the hydroxyl and the oxygen of the carbonyl group. The C2—C1′ is 1.462(4) Å and the C6′–C1′–C2–O1 torsion angle is equal to –0.5(5)°. These values are similar to the ones assigned to the 3-hydroxy-7-methoxyflavone with two intra and one inter-molecular H-bonds [2]. Comparing this torsion with that of –5.8(4)° for the 5-hydroxyflavone [3], we believe the planarity of the title compound is due to a weak intra- molecular (d(O2′···H3) = 2.79 A) attraction around the OH group at C2′.

Table 1. Data collection and handling.

Crystal:	pale yellow needle, size 0.2 × 0.3 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	49.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2425, 1980
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 1472
$N(param)_{refined}$:	262
Program:	Xta3.41 [4]

Table 2. 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}
C(1′)	4e	1.0095(6)	0.2618(4)	0.5147(1)	0.033(2)	0.044(2)	0.036(2)	–0.004(2)	0.004(1)	–0.002(2)
O(1)	4e	0.7945(4)	0.2389(3)	0.42185(9)	0.036(1)	0.052(2)	0.037(1)	0.006(1)	–0.004(1)	–0.004(1)
C(10)	4e	0.9071(6)	0.0850(4)	0.3440(1)	0.031(2)	0.041(2)	0.033(2)	–0.003(2)	–0.001(1)	0.003(2)
C(2)	4e	0.9976(6)	0.1905(4)	0.4578(1)	0.032(2)	0.042(2)	0.034(2)	–0.002(2)	–0.002(1)	0.003(2)
O(5)	4e	0.9944(6)	–0.0638(4)	0.2626(1)	0.045(2)	0.068(2)	0.038(2)	0.011(2)	–0.008(1)	–0.008(2)
C(9)	4e	0.7534(6)	0.1896(4)	0.3659(1)	0.035(2)	0.042(2)	0.034(2)	–0.004(2)	–0.002(1)	0.000(2)
C(3)	4e	1.1551(8)	0.0912(6)	0.4381(2)	0.034(2)	0.054(3)	0.033(2)	0.002(2)	–0.008(2)	0.003(2)
O(4)	4e	1.2653(4)	–0.0668(3)	0.36381(9)	0.037(1)	0.054(2)	0.037(1)	0.007(1)	–0.004(1)	–0.007(1)
C(4)	4e	1.1214(6)	0.0299(4)	0.3818(1)	0.032(2)	0.042(2)	0.035(2)	–0.003(2)	–0.001(1)	0.001(2)
C(3′)	4e	1.2108(8)	0.3088(6)	0.6115(2)	0.044(2)	0.070(3)	0.034(2)	–0.011(2)	–0.002(2)	–0.007(2)
C(5)	4e	0.8527(6)	0.0385(4)	0.2863(1)	0.036(2)	0.046(2)	0.038(2)	–0.001(2)	0.000(2)	–0.001(2)
C(8)	4e	0.5469(7)	0.2488(5)	0.3323(2)	0.041(2)	0.049(2)	0.045(2)	0.005(2)	–0.002(2)	0.002(2)
C(7)	4e	0.4976(7)	0.2000(5)	0.2759(2)	0.042(2)	0.054(3)	0.045(2)	0.005(2)	–0.010(2)	0.007(2)
C(2′)	4e	1.2024(6)	0.2327(4)	0.5597(1)	0.035(2)	0.047(2)	0.039(2)	–0.007(2)	0.004(1)	–0.003(2)
C(6′)	4e	0.8286(8)	0.3653(5)	0.5263(2)	0.046(2)	0.057(3)	0.042(2)	0.005(2)	0.002(2)	–0.002(2)
C(6)	4e	0.6457(7)	0.0959(5)	0.2525(2)	0.045(2)	0.055(3)	0.036(2)	0.002(2)	–0.010(2)	–0.001(2)
O(2′)	4e	1.3781(6)	0.1302(4)	0.5520(1)	0.044(2)	0.065(2)	0.043(2)	0.010(2)	–0.009(1)	–0.011(1)
C(5′)	4e	0.8381(9)	0.4381(5)	0.5780(2)	0.056(3)	0.059(3)	0.055(3)	0.007(2)	0.013(2)	–0.011(2)
C(4′)	4e	1.0314(8)	0.4114(5)	0.6214(2)	0.057(2)	0.063(3)	0.044(2)	–0.008(2)	0.012(2)	–0.015(2)

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Table 2. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
H(6)	4*e*	0.603(6)	0.064(4)	0.213(2)	0.03(2)	0.05(2)	0.05(2)	0.03(2)	0.00(2)	–0.03(2)
H(4')	4*e*	1.048(5)	0.464(4)	0.658(1)	0.02(2)	0.09(3)	0.03(2)	0.02(2)	0.00(2)	–0.05(2)
H(3)	4*e*	1.246(7)	0.064(4)	0.459(2)	0.05(3)	0.03(2)	0.03(3)	0.01(2)	0.02(2)	–0.02(2)
H(7)	4*e*	0.356(7)	0.243(4)	0.252(2)	0.11(3)	0.07(3)	0.03(3)	0.05(2)	0.02(2)	–0.01(2)
H(3')	4*e*	1.323(7)	0.288(4)	0.639(1)	0.07(3)	0.04(2)	0.02(2)	0.01(2)	0.02(2)	–0.02(2)
H(8)	4*e*	0.446(7)	0.330(4)	0.349(1)	0.09(3)	0.06(3)	0.05(2)	0.05(2)	–0.04(2)	–0.01(2)
H(5)	4*e*	1.086(9)	–0.098(5)	0.288(2)	0.07(4)	0.11(4)	0.05(3)	0.01(3)	0.03(3)	–0.06(3)
H(5')	4*e*	0.741(8)	0.511(5)	0.582(2)	0.08(3)	0.09(4)	0.03(2)	0.01(3)	–0.04(2)	–0.03(3)
H(6')	4*e*	0.723(7)	0.385(4)	0.497(2)	0.07(3)	0.04(3)	0.05(3)	0.03(2)	0.03(2)	–0.02(2)
H(2')	4*e*	1.43(1)	0.050(5)	0.537(2)	0.39(7)	0.33(8)	0.34(6)	–0.13(6)	–0.07(5)	0.06(5)

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