Crystal structure of 4'-hydroxy-5,6-benzoflavone, C₁₉H₁₂O₃

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Abstract

C₁₉H₁₂O₃, monoclinic, *P*12₁/*c*1 (No. 14), *a* = 8.157(4) Å, *b* = 15.091(2) Å, *c* = 11.035(2) Å, β = 92.37(3)°, *V* = 1357.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.060, *wR*_{all}(*F*) = 0.088, *T* = 293 K.

Source of material

Crystals of the title compound (Indofine Chemical Company) were grown from ethyl acetate solution, mp 576 K – 578 K.

Discussion

X-ray analysis has shown that 5,6-benzoflavone is planar with a dihedral angle of 1.07°. The planarity of the molecule was considered to be due to the stabilization of the negative charge on the carbonyl O atom [1]. Potential energy calculations for the same molecule predicted a non-planar conformation. We have reported in the past the same type of disagreement between molecular quantum mechanical calculations and X-ray results [2]. The X-ray single crystal structure of 4'-hydroxy-β-naphthoflavone was undertaken to show that the conformation of these types of molecules are affected primarily by the crystal packing [3]. The title compound is in a non-planar conformation. The dihedral angle is 18.0(2)°, and *d*(C2–C1') = 1.440(8) Å.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.2 × 0.2 × 0.3 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	1.0 cm ⁻¹
Diffractometer, scan mode:	CAD4, ω/2θ
2θ _{max} :	49.94°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	2725, 2376
Criterion for <i>F</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>F</i> _{obs} > 3 σ(<i>F</i> _{obs}), 1351
<i>N</i> (<i>param</i>) _{refined} :	247
Program:	XTAL [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(6')	4e	0.120(6)	0.222(3)	0.914(5)	0.03(1)
H(3')	4e	−0.201(7)	0.220(4)	1.254(5)	0.05(2)
H(2')	4e	−0.073(7)	0.345(4)	1.202(5)	0.07(2)
H(13)	4e	0.565(7)	0.781(4)	0.905(5)	0.05(2)
H(8)	4e	0.222(7)	0.399(4)	0.693(5)	0.05(2)
H(5')	4e	−0.011(7)	0.095(4)	0.975(5)	0.06(2)
H(12)	4e	0.626(8)	0.766(4)	0.715(5)	0.07(2)
H(14)	4e	0.436(7)	0.674(4)	1.004(5)	0.05(2)
H(3)	4e	0.152(8)	0.441(4)	1.178(6)	0.07(2)
H(7)	4e	0.370(7)	0.506(4)	0.604(6)	0.06(2)
H(11)	4e	0.513(7)	0.644(4)	0.614(5)	0.05(2)
H(4')	4e	−0.17(1)	0.013(3)	1.132(7)	0.20(3)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(10)	4e	0.2978(6)	0.5217(3)	0.9303(4)	0.037(3)	0.033(3)	0.029(3)	0.004(2)	0.005(2)	0.001(2)
O(1)	4e	0.1580(5)	0.3814(2)	0.9053(3)	0.053(2)	0.041(2)	0.029(2)	−0.008(2)	0.005(2)	−0.001(2)
C(2)	4e	0.1217(7)	0.3784(4)	1.0250(4)	0.042(3)	0.043(3)	0.028(3)	0.005(3)	0.005(2)	0.005(2)
C(9)	4e	0.2403(7)	0.4518(3)	0.8603(4)	0.042(3)	0.040(3)	0.029(3)	0.002(3)	0.002(2)	0.004(2)
C(5)	4e	0.3878(7)	0.5910(3)	0.8719(5)	0.039(3)	0.037(3)	0.034(3)	0.005(3)	0.004(2)	0.003(2)
C(4)	4e	0.2630(7)	0.5202(4)	1.0609(5)	0.041(3)	0.038(3)	0.034(3)	0.008(3)	0.009(2)	0.001(3)
C(3)	4e	0.1723(7)	0.4454(4)	1.0991(5)	0.049(4)	0.045(3)	0.026(3)	0.001(3)	0.009(3)	−0.000(3)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(4)	4e	0.3120(5)	0.5784(3)	1.1331(3)	0.071(3)	0.039(2)	0.036(2)	-0.006(2)	0.011(2)	-0.010(2)
C(8)	4e	0.2626(8)	0.4456(4)	0.7350(5)	0.060(4)	0.042(4)	0.032(3)	-0.006(3)	0.001(3)	-0.002(3)
C(11)	4e	0.4968(9)	0.6509(5)	0.6865(6)	0.082(5)	0.062(5)	0.037(4)	-0.008(4)	0.014(3)	0.008(4)
C(1')	4e	0.0402(7)	0.2974(3)	1.0560(5)	0.037(3)	0.039(3)	0.030(3)	-0.001(3)	-0.001(2)	0.003(2)
C(14)	4e	0.4514(8)	0.6667(4)	0.9316(6)	0.047(4)	0.045(4)	0.038(3)	-0.001(3)	0.006(3)	-0.000(3)
C(6)	4e	0.4106(7)	0.5833(4)	0.7455(5)	0.050(4)	0.041(3)	0.035(3)	-0.002(3)	0.008(3)	0.005(3)
C(3')	4e	-0.1257(8)	0.2154(4)	1.1930(5)	0.049(4)	0.047(4)	0.034(3)	0.003(3)	0.006(3)	0.007(3)
C(13)	4e	0.5326(8)	0.7301(4)	0.8705(6)	0.062(5)	0.037(4)	0.060(4)	-0.007(3)	0.003(3)	-0.001(3)
C(2')	4e	-0.0456(7)	0.2908(4)	1.1638(5)	0.046(4)	0.038(3)	0.035(3)	0.004(3)	0.005(3)	-0.000(3)
C(7)	4e	0.3447(8)	0.5104(4)	0.6802(5)	0.066(4)	0.056(4)	0.029(3)	-0.006(3)	0.006(3)	0.002(3)
O(4')	4e	-0.2143(7)	0.0686(3)	1.1404(4)	0.098(4)	0.048(3)	0.045(2)	-0.020(3)	0.015(2)	0.002(2)
C(4')	4e	-0.1311(7)	0.1431(4)	1.1160(5)	0.056(4)	0.047(4)	0.034(3)	-0.008(3)	-0.003(3)	0.005(3)
C(12)	4e	0.5570(9)	0.7225(5)	0.7466(6)	0.069(5)	0.052(4)	0.055(4)	-0.009(4)	0.013(4)	0.013(3)
C(6')	4e	0.0373(8)	0.2239(4)	0.9807(5)	0.061(4)	0.049(4)	0.028(3)	-0.009(3)	0.009(3)	0.003(3)
C(5')	4e	-0.0432(9)	0.1477(4)	1.0096(5)	0.073(5)	0.047(4)	0.034(3)	-0.010(4)	0.008(3)	-0.004(3)

References

1. Rossi, M.; Cantrell, J. S.; Farber, A. J.; Dyott, T.; Carrell, H. L.; Glusker, J. P.: Molecular structures of 5,6- and 7,8-benzoflavones, inhibitors of aryl hydrocarbon hydroxylase. *Cancer Research* **40** (1980) 2774-2784.
2. Shoja, M.; Bisso, S.; Kabbani, R.; Athanasopoulos, D.: Crystal structure of 6-hydroxy-2'-methoxyflavone. *Z. Kristallogr. NCS* **214** (1999) 233-234.
3. Shoja, M.: Crystal structure of 3-hydroxy-6-methoxyflavone. *Z. Kristallogr. NCS* **213** (1998) 731-732.
4. Hall, S. R.; King, G. S. D.; Stewart, J. M. (Eds.): *Xtal3.4 User's Manual*. University of Western Australia: Lamb, Perth, Australia 1995.