Crystal structure of 2',4'-dimethoxy-3-hydroxyflavone, C₁₇H₁₄O₅

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Received December 11, 2000, CCDC-No. 1267/583

Abstract

C₁₇H₁₄O₅, triclinic, $P\bar{1}$ (No. 2), $a = 7.377(3)$ Å, $b = 9.619(3)$ Å, $c = 11.901(3)$ Å, $\alpha = 107.70(1)^\circ$, $\beta = 100.06(1)^\circ$, $\gamma = 108.77(1)^\circ$, $V = 726.2$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.037$, $wR_{\text{all}}(F) = 0.043$, $T = 293$ K.

Source of material

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

Discussion

The X-ray crystal structure of the title compound was determined to show the effect of the repulsion of the lone-pair electrons of the methoxy O2' and the hydroxy O3 atoms, which will cause a twist between the phenyl ring and the γ -pyrone portion of the molecule. The torsion angle for C3–C2–C1'–C2' is $-113.2(3)^\circ$. This repulsion was also observed in 3-hydroxy-2'-methoxyflavone with a torsion angle of $72.2(4)^\circ$ [1]. In the title compound, the C2–C1' bond distance is $1.473(4)$ Å. A weak bifurcated hydrogen bond is formed as a result of an *inter-* ($d(\text{O4}\cdots\text{H3}) = 2.31$ Å) and an *intra-* molecular ($d(\text{O4}\cdots\text{H3}) = 2.05$ Å) attraction between the molecules. We have reported the same type of hydrogen bond for 3-hydroxy-6-methoxy and 3-hydroxy-7-methoxyflavone with torsion angles of $11.3(5)^\circ$ and $1.2(6)^\circ$, respectively [2, 3]. In conclusion, the methoxy group in the C2' position in the presence of a C3 hydroxy group will cause a twist around the C2–C1' bond, and pack the molecule in a non-planar conformation.

Table 1. Data collection and handling.

Crystal:	pale yellow needle, size $0.3 \times 0.3 \times 0.4$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	1.1 cm^{-1}
Diffractometer, scan mode:	CAD4, $\omega/2\theta$
$2\theta_{\text{max}}$:	49.94°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	2736, 2545
Criterion for F_{obs} , $N(hkl)_{\text{gt}}$:	$F_{\text{obs}} > 3 \sigma(F_{\text{obs}})$, 1989
$N(\text{param})_{\text{refined}}$:	255
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')	2i	0.355(3)	0.987(3)	0.122(2)	0.044(7)
H(3')	2i	0.608(4)	1.246(3)	0.550(3)	0.059(8)
H(8)	2i	0.547(4)	0.529(3)	0.285(2)	0.063(8)
H(5')	2i	0.220(4)	1.160(3)	0.225(2)	0.053(7)
H(5)	2i	0.862(4)	0.538(3)	−0.027(2)	0.061(8)
H(7)	2i	0.639(4)	0.320(3)	0.191(3)	0.074(9)
H(3)	2i	0.875(4)	1.061(3)	0.072(3)	0.063(9)
H(6)	2i	0.791(4)	0.314(3)	0.036(3)	0.077(9)
H(21')	2i	0.943(4)	1.268(4)	0.615(3)	0.068(9)
H(22')	2i	0.791(4)	1.133(3)	0.639(3)	0.070(9)
H(23')	2i	1.001(5)	1.118(4)	0.621(3)	0.09(1)
H(41')	2i	0.209(5)	1.394(4)	0.350(3)	0.09(1)
H(42')	2i	0.050(5)	1.257(4)	0.367(3)	0.09(1)
H(43')	2i	0.169(5)	1.430(4)	0.479(3)	0.11(1)

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Table 3. 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)	2i	0.5924(3)	0.7706(2)	0.2380(2)	0.055(1)	0.0394(9)	0.046(1)	0.0207(8)	0.0278(8)	0.0218(8)
C(3)	2i	0.7474(3)	0.9243(3)	0.1317(2)	0.042(1)	0.037(1)	0.040(1)	0.018(1)	0.016(1)	0.020(1)
C(4)	2i	0.8115(3)	0.8040(3)	0.0678(2)	0.038(1)	0.042(1)	0.041(1)	0.018(1)	0.016(1)	0.020(1)
O(3)	2i	0.7973(3)	1.0614(2)	0.1110(2)	0.061(1)	0.045(1)	0.058(1)	0.0292(9)	0.036(1)	0.0315(9)
C(10)	2i	0.7548(3)	0.6627(3)	0.0968(2)	0.039(1)	0.039(1)	0.047(1)	0.017(1)	0.017(1)	0.020(1)
C(2)	2i	0.6417(3)	0.9051(3)	0.2118(2)	0.041(1)	0.037(1)	0.037(1)	0.016(1)	0.014(1)	0.017(1)
C(9)	2i	0.6521(4)	0.6537(3)	0.1835(2)	0.043(1)	0.036(1)	0.043(1)	0.016(1)	0.015(1)	0.017(1)
O(4)	2i	0.9101(3)	0.8232(2)	-0.0048(2)	0.062(1)	0.051(1)	0.062(1)	0.0288(9)	0.040(1)	0.0313(9)
C(1')	2i	0.5630(3)	1.0173(3)	0.2779(2)	0.042(1)	0.038(1)	0.041(1)	0.017(1)	0.019(1)	0.019(1)
C(5)	2i	0.8053(4)	0.5350(3)	0.0418(3)	0.055(2)	0.048(2)	0.069(2)	0.028(1)	0.033(2)	0.028(1)
C(4')	2i	0.4038(4)	1.2211(3)	0.4030(2)	0.051(2)	0.041(1)	0.055(2)	0.022(1)	0.027(1)	0.020(1)
C(2')	2i	0.6401(3)	1.0970(3)	0.4072(2)	0.039(1)	0.039(1)	0.041(1)	0.014(1)	0.015(1)	0.018(1)
C(8)	2i	0.6077(4)	0.5247(3)	0.2197(3)	0.059(2)	0.044(2)	0.059(2)	0.019(1)	0.028(2)	0.028(1)
O(2')	2i	0.7937(3)	1.0662(2)	0.4618(2)	0.047(1)	0.060(1)	0.043(1)	0.0268(9)	0.0084(8)	0.0165(9)
O(4')	2i	0.3358(3)	1.3216(2)	0.4743(2)	0.073(1)	0.063(1)	0.068(1)	0.044(1)	0.031(1)	0.019(1)
C(3')	2i	0.5602(4)	1.1972(3)	0.4689(3)	0.051(2)	0.043(1)	0.039(1)	0.018(1)	0.016(1)	0.011(1)
C(5')	2i	0.3282(4)	1.1465(3)	0.2752(3)	0.049(2)	0.053(2)	0.054(2)	0.027(1)	0.020(1)	0.027(1)
C(6)	2i	0.7612(5)	0.4077(3)	0.0767(3)	0.066(2)	0.047(2)	0.090(2)	0.031(2)	0.038(2)	0.033(2)
C(7)	2i	0.6659(5)	0.4053(3)	0.1675(3)	0.066(2)	0.044(2)	0.084(2)	0.023(1)	0.032(2)	0.038(2)
C(6')	2i	0.4093(4)	1.0448(3)	0.2145(2)	0.052(2)	0.049(2)	0.039(1)	0.023(1)	0.017(1)	0.020(1)
C(22')	2i	0.8895(5)	1.1511(4)	0.5923(3)	0.058(2)	0.063(2)	0.048(2)	0.019(2)	0.004(2)	0.017(2)
C(44')	2i	0.1777(6)	1.3524(5)	0.4125(4)	0.076(3)	0.069(2)	0.098(3)	0.047(2)	0.044(2)	0.035(2)

References

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