Crystal structure of 4-(α-hydroxy-*p*-fluorobenzyl)isochroman-1,3-dione, $C_{16}H_9FO_4$

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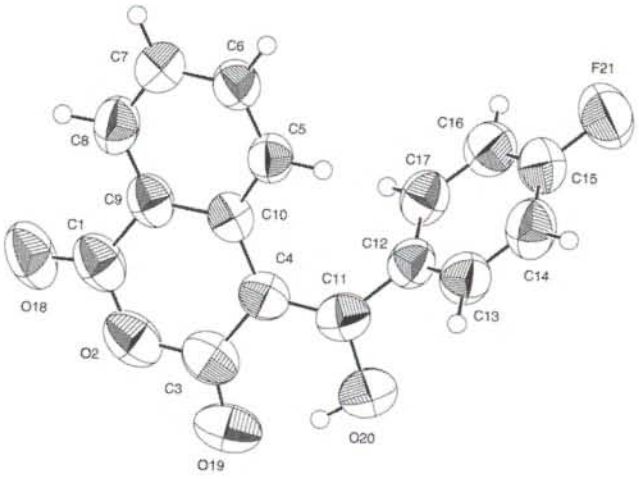

Abstract

$C_{16}H_9FO_4$, monoclinic, $P12_1/c1$ (No. 14), $a = 15.173(1)$ Å, $b = 6.069(1)$ Å, $c = 14.344(1)$ Å, $\beta = 110.074(1)°$, $V = 1240.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.074$, $wR(F) = 0.174$, $T = 298$ K.

Source of material

The compound is obtained by reaction of 200 ml of THF with 40 mmol of benzoyl chloride, 0.12 mol of triethylamine and HCl solution diluted, the organic phase is washed, neutralized, dried and evaporated [1]. The compound was crystallised in CH_2Cl_2.

Discussion

From IR data it's supposed that the isochroman-1,3-diones have dicarbonyl structure [2] but in solution as in crystal only the exocyclic enolic tautomer exist [1, 3]. So, we present the crystal and molecular structure of 4-(α-hydroxy-*p*-fluorobenzyl)-isochroman-1,3-dione. The packing of the molecules reveals strong intramolecular bond (H20–O19 = 1.59(3) Å) and intermolecular bonds (F21–H14 = 2.73(1) Å, O18–H6 = 2.538(1) Å, O2–H5 = 2.599(1) Å). The atoms of cycle P1(C1O2C3C4C10C5C6C7C8C9) are coplanar, only the atom C4 is slightly outside (0.123(1) Å). The angle between P1 and P2(C12C13C14C15C16C17) cycles is 57.93(4)°. The pseudo-cycle P3(H20O20C11C4C3O19) is also plane and have an angle of 9.72(4)° with P1.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.25 × 0.3 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.2 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	53.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2810, 2560
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 1\ \sigma(I_{obs})$, 1881
$N(param)_{refined}$:	190
Programs:	maXus [4], MULTAN [5], ORTEPII [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(6)	4*e*	0.6244	0.64314	0.17839	0.05
H(5)	4*e*	0.7019	0.32785	0.15906	0.05
H(8)	4*e*	0.54482	0.76674	–0.1153	0.05
H(17)	4*e*	0.88625	0.41214	0.15165	0.05
H(7)	4*e*	0.54929	0.87139	0.04177	0.05
H(13)	4*e*	0.80645	–0.17897	0.22704	0.05
H(14)	4*e*	0.87785	–0.09304	0.39505	0.05
H(16)	4*e*	0.96159	0.49613	0.31921	0.05
H(20)	4*e*	0.803(1)	–0.148(3)	–0.022(2)	0.050(7)

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}
F(21)	4*e*	0.96307(8)	0.2612(2)	0.46786(8)	0.1131(8)	0.1316(9)	0.0880(8)	–0.0082(6)	0.0081(6)	–0.0057(6)
O(2)	4*e*	0.65738(9)	0.2256(2)	–0.18285(8)	0.1112(9)	0.1079(9)	0.0657(7)	–0.0170(7)	0.0370(6)	–0.0120(6)
O(20)	4*e*	0.82839(8)	–0.1429(2)	0.04877(9)	0.1055(8)	0.0927(8)	0.1052(9)	0.0203(6)	0.0356(7)	–0.0127(7)
O(19)	4*e*	0.74085(9)	–0.0726(2)	–0.13078(9)	0.125(1)	0.1049(9)	0.1022(9)	–0.0060(7)	0.0578(8)	–0.0318(7)

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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(18)	4*e*	0.5745(1)	0.5199(2)	–0.24744(9)	0.143(1)	0.117(1)	0.0597(7)	–0.0240(7)	0.0157(7)	0.0152(6)
C(4)	4*e*	0.7296(1)	0.1697(2)	–0.0050(1)	0.0728(8)	0.0712(9)	0.0675(9)	–0.0061(7)	0.0304(7)	–0.0041(7)
C(10)	4*e*	0.67833(9)	0.3618(2)	0.0111(1)	0.0627(7)	0.0714(9)	0.0588(8)	–0.0081(6)	0.0212(6)	0.0008(6)
C(6)	4*e*	0.6249(1)	0.6060(3)	0.1135(1)	0.0746(9)	0.085(1)	0.0655(9)	0.0056(7)	0.0220(7)	–0.0027(7)
C(5)	4*e*	0.67230(9)	0.4209(2)	0.1029(1)	0.0670(8)	0.0742(9)	0.0612(8)	0.0065(7)	0.0207(6)	0.0041(7)
C(12)	4*e*	0.83883(9)	0.1068(3)	0.1736(1)	0.0629(8)	0.0697(9)	0.082(1)	0.0044(6)	0.0215(7)	0.0023(8)
C(8)	4*e*	0.5787(1)	0.6786(3)	–0.0588(1)	0.082(1)	0.078(1)	0.079(1)	0.0003(8)	0.0118(8)	0.0185(8)
C(9)	4*e*	0.6267(1)	0.4904(2)	–0.0711(1)	0.0723(8)	0.079(1)	0.0578(8)	–0.0112(7)	0.0185(7)	0.0045(7)
C(11)	4*e*	0.7948(1)	0.0490(3)	0.0683(1)	0.0756(9)	0.070(1)	0.091(1)	0.0007(7)	0.0351(8)	–0.0063(8)
C(15)	4*e*	0.9233(1)	0.2085(3)	0.3700(1)	0.0707(9)	0.093(1)	0.081(1)	0.0020(8)	0.0111(8)	–0.0015(9)
C(17)	4*e*	0.8852(1)	0.3080(3)	0.2016(1)	0.0692(9)	0.077(1)	0.089(1)	0.0014(7)	0.0254(8)	0.0110(8)
C(7)	4*e*	0.5802(1)	0.7390(3)	0.0330(1)	0.084(1)	0.078(1)	0.083(1)	0.0134(8)	0.0219(8)	0.0001(8)
C(3)	4*e*	0.7110(1)	0.0976(3)	–0.1057(1)	0.087(1)	0.093(1)	0.077(1)	–0.0156(8)	0.0379(9)	–0.0102(9)
C(1)	4*e*	0.6169(1)	0.4241(3)	–0.1718(1)	0.095(1)	0.094(1)	0.065(1)	–0.0255(9)	0.0237(8)	0.0004(9)
C(13)	4*e*	0.8378(1)	–0.0401(2)	0.2462(1)	0.0799(9)	0.068(1)	0.091(1)	–0.0045(7)	0.0182(8)	0.0025(8)
C(14)	4*e*	0.8791(1)	0.0114(3)	0.3453(1)	0.087(1)	0.089(1)	0.089(1)	–0.0033(8)	0.0180(9)	0.0182(9)
C(16)	4*e*	0.9286(1)	0.3591(3)	0.3003(1)	0.0688(9)	0.079(1)	0.100(1)	–0.0064(7)	0.0159(8)	–0.0019(9)

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