Crystal structure of 4-(α-hydroxy-*p*-nitrobenzyl)isochroman-1,3-dione, $C_{16}H_9NO_6$

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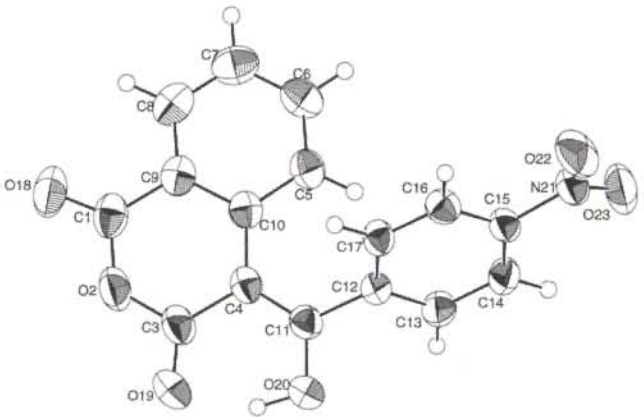

Abstract

$C_{16}H_9NO_6$, monoclinic, *P*1*c*1 (No. 7), $a = 5.553(1)$ Å, $b = 7.692(1)$ Å, $c = 15.676(1)$ Å, $\beta = 92.812(1)°$, $V = 668.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.037$, $wR(F) = 0.213$, $T = 298$ K.

Source of material

The compound is obtained according to [1]; 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 was washed, neutralized, dried and evaporated. The compound was crystallised from CH_2Cl_2.

Discussion

From the IR data, it's suposed that the isochroman-1,3 diones have dicarbonyl structure [2], but in solution [3] as in crystal only the exocyclic enolic tautomer exists. So, we present the crystal and molecular structure of 4-(α-hydroxy-*p*-nitrobenzyl)-isochroman-1,3-dione. The tautomer is stabilized by hard hydrogen intramolecular bond (H20–O19 = 1.63(3) Å) and intermolecular bonds (O22–H20 = 2.44(2)Å, O18–H14 = 2.482(2) Å). The atoms C1O2C3C4C10C5C6C7C8C9 are coplanar, only the atom C4 is slightly outside (0.104(1) Å). The angle between isochromane and the phenyl planes is 66.03(4)°.

Table 1. Data collection and handling.

Crystal:	yellow, prismatic, size 0.25 × 0.35 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.21 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	50.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1437, 1334
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 0.5\ \sigma(I_{obs})$, 1308
$N(param)_{refined}$:	208
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(13)	2*a*	0.98662	0.24051	0.42274	0.05
H(14)	2*a*	0.87177	0.36363	0.28933	0.05
H(16)	2*a*	0.26732	0.55970	0.39221	0.05
H(17)	2*a*	0.37372	0.42267	0.52213	0.05
H(7)	2*a*	–0.10193	–0.25952	0.47803	0.05
H(6)	2*a*	0.14037	–0.10656	0.38604	0.05
H(8)	2*a*	–0.00300	–0.26075	0.62432	0.05
H(5)	2*a*	0.47076	0.05461	0.43919	0.05
H(20)	2*a*	0.971(3)	0.251(3)	0.656(1)	0.050(5)

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}
C(15)	2*a*	0.5561(3)	0.4661(2)	0.3297(1)	0.0450(7)	0.0401(6)	0.0362(7)	–0.0029(5)	–0.0032(5)	0.0057(5)
O(20)	2*a*	0.9530(3)	0.3066(1)	0.6010(1)	0.0464(5)	0.0722(7)	0.0459(5)	–0.0068(5)	–0.0076(4)	0.0072(5)
C(13)	2*a*	0.8370(3)	0.3024(2)	0.4148(1)	0.0369(7)	0.0548(8)	0.0471(8)	0.0075(6)	0.0055(5)	0.0060(6)
C(10)	2*a*	0.4255(3)	0.0060(2)	0.5660(1)	0.0456(7)	0.0370(6)	0.0420(7)	0.0072(5)	0.0032(5)	0.0027(5)
C(14)	2*a*	0.7703(3)	0.3748(2)	0.3368(1)	0.0435(7)	0.0574(8)	0.0397(7)	0.0016(7)	0.0105(5)	0.0043(6)
O(18)	2*a*	0.2232(3)	–0.1751(2)	0.7632(1)	0.0732(8)	0.0794(8)	0.0624(8)	0.0066(6)	0.0235(6)	0.0252(6)
C(16)	2*a*	0.4099(3)	0.4892(2)	0.3975(1)	0.0411(7)	0.0434(7)	0.0456(7)	0.0063(5)	–0.0005(5)	0.0012(5)

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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(11)	2*a*	0.7550(3)	0.2351(2)	0.5663(1)	0.0391(6)	0.0482(7)	0.0390(7)	0.0045(6)	0.0009(5)	0.0013(6)
O(19)	2*a*	0.8649(3)	0.1414(2)	0.7343(1)	0.0653(7)	0.0811(7)	0.0458(6)	–0.0011(6)	–0.0137(5)	0.0119(5)
C(17)	2*a*	0.4746(3)	0.4125(2)	0.4743(1)	0.0433(7)	0.0472(7)	0.0370(7)	0.0063(6)	0.0060(5)	0.0000(5)
C(4)	2*a*	0.6249(3)	0.1074(2)	0.6056(1)	0.0466(7)	0.0429(6)	0.0361(7)	0.0067(6)	0.0019(5)	0.0033(5)
C(3)	2*a*	0.6907(3)	0.0781(2)	0.6950(1)	0.0536(9)	0.0526(8)	0.0414(8)	0.0094(7)	–0.0011(6)	0.0105(6)
O(23)	2*a*	0.5689(3)	0.4778(2)	0.1834(1)	0.096(1)	0.0869(9)	0.0386(6)	–0.0035(7)	0.0060(6)	0.0101(6)
O(2)	2*a*	0.5455(3)	–0.0220(2)	0.74292(9)	0.0694(7)	0.0647(6)	0.0396(6)	0.0037(6)	0.0010(5)	0.0166(5)
N(21)	2*a*	0.4745(3)	0.5347(2)	0.2462(1)	0.0587(7)	0.0552(7)	0.0423(7)	–0.0063(6)	–0.0061(5)	0.0113(6)
C(9)	2*a*	0.2828(3)	–0.0940(2)	0.6191(1)	0.0493(8)	0.0422(7)	0.0487(8)	0.0076(6)	0.0099(6)	0.0037(6)
C(1)	2*a*	0.3375(3)	–0.1010(2)	0.7113(1)	0.0589(9)	0.0496(8)	0.0531(9)	0.0113(7)	0.0141(7)	0.0123(6)
C(7)	2*a*	0.0355(4)	–0.1949(2)	0.4999(2)	0.0538(9)	0.0527(9)	0.077(1)	–0.0027(7)	0.0022(7)	–0.0112(7)
C(6)	2*a*	0.1782(3)	–0.1020(2)	0.4464(1)	0.0647(9)	0.0454(8)	0.0545(9)	0.0042(7)	–0.0078(7)	–0.0090(7)
C(12)	2*a*	0.6870(3)	0.3165(2)	0.4829(1)	0.0374(6)	0.0415(7)	0.0367(7)	–0.0006(5)	0.0012(5)	0.0022(5)
O(22)	2*a*	0.3173(3)	0.6448(2)	0.2437(1)	0.0784(9)	0.0852(8)	0.0667(8)	0.0198(8)	–0.0137(6)	0.0241(6)
C(8)	2*a*	0.0896(3)	–0.1929(2)	0.5863(1)	0.0562(9)	0.0503(8)	0.070(1)	–0.0027(7)	0.0154(7)	0.0004(7)
C(5)	2*a*	0.3710(3)	–0.0056(2)	0.4779(1)	0.0580(8)	0.0421(6)	0.0415(8)	0.0028(6)	0.0019(6)	–0.0022(5)

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