Crystal structure of methyl 3-oxo-3*H*-benzo[*f*]chromene-1-carboxylate, C₁₅H₁₀O₄

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Abstract

C₁₅H₁₀O₄, monoclinic, *P*12₁/*c*1 (No. 14), *a* = 12.246(2) Å, *b* = 10.541(2) Å, *c* = 8.912(2) Å, β = 96.205(4)°, *V* = 1143.7 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.058, *wR*_{ref}(*F*²) = 0.152, *T* = 110 K.

Source of material

Methyl 3-oxo-3*H*-benzo[*f*]chromene-1-carboxylate was synthesized in accordance with our new procedure [1]. The solution of title compound (0.3 g) in acetonitrile-benzene (1:1; 35 ml) left in

room temperature for six days. The reddish crystals were filtered off, washed with cold acetonitrile-benzene (1:1; 9 ml) and dried in vacuum over P₄O₁₀ (mp 413 K; yield 56%). Elemental analyses were consistent with the stoichiometry C₁₅H₁₀O₄ (found: C, 70.79%; H, 4.05%; calc.: C, 70.87%; H, 3.97%). Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer.

Discussion

Chromene derivatives are important heterocycles in bio-organic chemistry and are present in many natural products and pharmaceuticals. There have been reported many studies on the synthesis of the heterocyclic chromene ring structure [2–8]. In the recent years there has been increasing interest in the determination of X-ray crystal structures of biologically active compounds [9,10]. In the structure of title molecule (upper figure), the covalent bonds are found to be normal (relevant bond lengths: *d*(O1—C1) = 1.372(3) Å, *d*(O1—C5) = 1.376(3) Å, *d*(O2—C1) = 1.212(3) Å, *d*(O3—C14) = 1.203(3) Å, *d*(O4—C14) = 1.328(3) Å, *d*(O4—C15) = 1.457(3) Å, *d*(C1—C2) = 1.443(3) Å, *d*(C2—C3) = 1.348(3) Å, *d*(C3—C4) = 1.450(3) Å, *d*(C3—C14) = 1.503(3) Å; angles at the O atoms: 122.44(17)° at O1 and 114.55(18)° at O4) and are almost the same as in the previous reports [10,15,16]. The methyl group at O4 is approximately in the O4—C14—O3 plane (∠C15—O4—C14—O3 = −1.7(3)°) that agree well with a participation of O4 in the electron donation to the carbonyl group. The torsion angle ∠C4—C3—C14—O3 = 99.1(3)° shows clearly that the carbonyl group (C14—O3) is approximately perpendicular to the C4—C3—C14 plane, probably is as a result of steric intractions. π-π-Ring stacking is observed within the crystals, probably is as a result of the presence of parallel aromatic naphthalene rings in the crystal network that stabilize the crystal packing (lower figure).

Table 1. Data collection and handling.

Crystal:	reddish prism, size 0.10 × 0.20 × 0.35 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	1.08 cm ^{−1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
2θ _{max} :	54.08°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	6216, 2476
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1425
<i>N</i> (<i>param</i>) _{refined} :	212
Programs:	SHELXTL [11], SADABS [12]

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

Atom	Site	x	y	z	U _{iso}
H(2)	4e	0.053(2)	0.504(2)	0.257(2)	0.033(6)
H(8)	4e	0.308(2)	1.048(2)	−0.001(3)	0.040(7)
H(9)	4e	0.158(2)	1.013(2)	0.142(2)	0.035(7)
H(10)	4e	0.358(2)	0.548(2)	−0.023(2)	0.030(6)
H(11)	4e	0.504(2)	0.580(2)	−0.152(2)	0.031(6)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(12)	4e	0.557(2)	0.793(2)	−0.219(2)	0.026(6)
H(13)	4e	0.453(2)	0.963(2)	−0.136(2)	0.031(6)
H(15A)	4e	0.188(2)	0.237(3)	−0.019(3)	0.057(8)
H(15B)	4e	0.199(2)	0.293(3)	−0.195(4)	0.068(9)
H(15C)	4e	0.307(2)	0.302(2)	−0.060(3)	0.043(7)

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)	4e	0.0808(1)	0.8152(1)	0.2289(2)	0.0299(9)	0.0326(9)	0.0328(8)	0.0027(7)	0.0084(7)	−0.0014(7)
O(2)	4e	−0.0319(1)	0.7094(2)	0.3604(2)	0.032(1)	0.045(1)	0.044(1)	−0.0011(8)	0.0153(8)	−0.0033(8)
O(3)	4e	0.2766(1)	0.4038(2)	0.1898(2)	0.035(1)	0.0346(9)	0.0363(9)	0.0035(7)	0.0042(8)	0.0021(7)
O(4)	4e	0.1813(1)	0.4281(1)	−0.0382(2)	0.035(1)	0.0287(9)	0.0354(9)	0.0036(7)	0.0026(7)	−0.0033(7)
C(1)	4e	0.0399(2)	0.7029(2)	0.2772(2)	0.027(1)	0.034(1)	0.031(1)	0.001(1)	0.004(1)	0.000(1)
C(2)	4e	0.0877(2)	0.5889(2)	0.2230(2)	0.027(1)	0.030(1)	0.032(1)	−0.001(1)	0.005(1)	0.000(1)
C(3)	4e	0.1724(2)	0.5925(2)	0.1382(2)	0.025(1)	0.029(1)	0.026(1)	0.0016(9)	0.002(1)	−0.0013(9)
C(4)	4e	0.2185(2)	0.7121(2)	0.0954(2)	0.025(1)	0.026(1)	0.022(1)	0.0024(9)	0.0007(9)	−0.0006(9)
C(5)	4e	0.1670(2)	0.8199(2)	0.1421(2)	0.024(1)	0.031(1)	0.025(1)	0.0009(9)	0.002(1)	−0.0019(9)
C(6)	4e	0.3112(2)	0.7297(2)	0.0097(2)	0.027(1)	0.029(1)	0.023(1)	0.002(1)	−0.0008(9)	0.0003(9)
C(7)	4e	0.3428(2)	0.8556(2)	−0.0239(2)	0.029(1)	0.031(1)	0.025(1)	0.001(1)	−0.001(1)	0.0014(9)
C(8)	4e	0.2834(2)	0.9604(2)	0.0255(2)	0.038(1)	0.027(1)	0.031(1)	0.002(1)	0.004(1)	0.002(1)
C(9)	4e	0.1975(2)	0.9440(2)	0.1070(2)	0.037(1)	0.027(1)	0.029(1)	0.005(1)	0.004(1)	−0.002(1)
C(10)	4e	0.3746(2)	0.6307(2)	−0.0416(3)	0.033(1)	0.028(1)	0.034(1)	0.001(1)	0.006(1)	−0.001(1)
C(11)	4e	0.4622(2)	0.6525(2)	−0.1216(3)	0.030(1)	0.039(1)	0.034(1)	0.004(1)	0.005(1)	−0.003(1)
C(12)	4e	0.4920(2)	0.7767(2)	−0.1555(2)	0.027(1)	0.044(2)	0.030(1)	−0.002(1)	0.004(1)	0.004(1)
C(13)	4e	0.4329(2)	0.8756(2)	−0.1076(2)	0.034(1)	0.034(1)	0.029(1)	−0.002(1)	0.004(1)	0.006(1)
C(14)	4e	0.2171(2)	0.4649(2)	0.1010(2)	0.025(1)	0.027(1)	0.031(1)	−0.004(1)	0.006(1)	0.001(1)
C(15)	4e	0.2224(2)	0.3051(2)	−0.0810(3)	0.038(2)	0.030(1)	0.042(2)	0.004(1)	0.007(1)	−0.005(1)

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