Crystal structure of methyl (6-(2-fluorobenzoyl)-5-chloro-2-benzoxazolinon-3-yl)-acetate, $C_{17}H_{11}ClFNO_5$

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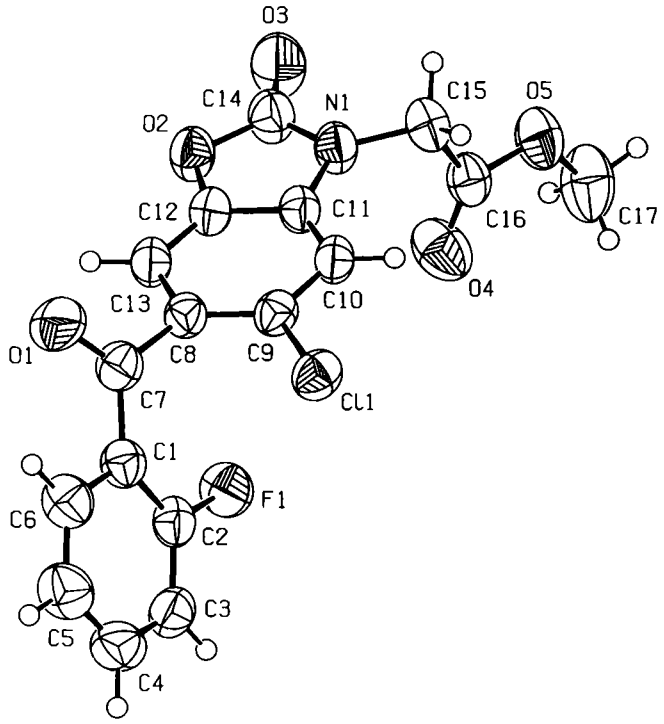

Abstract

$C_{17}H_{11}ClFNO_5$, monoclinic, $P2_1/c1$ (no. 14), a = 7.489(5) Å, b = 18.367(5) Å, c = 11.752(5) Å, β = 94.222(5)°, V = 1612.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.032, $wR_{ref}(F^2)$ = 0.083, T = 293 K.

Source of material

The mixture of potassium carbonate (5.75 mmol), methyl chloroacetate (5.75 mmol), the acetic acid and propanoic acid derivatives of 6-(2-fluorobenzoyl)-5-chloro-2-benzoxazolinone (5 mmol; 1:1, *v/v*) in 30 mL acetone were heated to reflux and stirred for 4 h. 100 g Ice water was added to the cooled (273-283 K) reaction mixture. After stirring for 1 h, the precipitate was collected by filtration, washed with water, dried, and recrystallized from ethanol (yield 81 %, m.p. 383-385 K). IR data are available in the CIF.

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Discussion

Benzothiazolinone/2-benzoxazolinone derivatives exhibit a variety of pharmacological effects, including analgesic and anti-inflammatory activity [1].

In the title compound the F1—C2 distance 1.350(2) Å is in agreement with that for ethyl[6-(2-fluorobenzoyl)-2-oxo-benzothiazolin-3-yl]-butanoate [2] (1.346(3) Å). The bond distance of N1—C14 is 1.365(2) Å and the double-bond length for C14=O3 is 1.199(2) Å. These values are also comparable to those of 1.343(2) Å and 1.204(2) Å observed in a related structure [3]. The Cl1—C9 bond distance is 1.742(2) Å and the C16–O5–C17 and F1–C2–C1 bond angles are 116.9(2)° and 119.6(1)°, respectively. The C17–O5–C16–C15 and C13–C8–C9–Cl1 torsion angles are 179.0(2)° and 176.9(1)°, respectively. According to our CNDO calculations [4], the dipole moment of title molecule is about 6.77 D. The HOMO and LUMO energy levels are –9.0565 eV and –0.7091 eV, respectively. The crystal structure is stabilized by the three-dimensional C–H···O type short hydrogen contacts.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.33 × 0.42 × 0.52 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	2.76 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	55.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14093, 3686
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2649
$N(param)_{refined}$:	227
Programs:	SIR97 [5], SHELXL-97 [6], ORTEP-3 [7], WinGX [8]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	1.2124	0.0411	0.3970	0.074
H(4)	4*e*	1.1438	0.1315	0.5231	0.084
H(5)	4*e*	0.8516	0.1663	0.5380	0.086
H(6)	4*e*	0.6265	0.1127	0.4246	0.075
H(10)	4*e*	0.7769	–0.0358	–0.0935	0.059
H(13)	4*e*	0.5124	–0.1203	0.2320	0.062
H(15A)	4*e*	0.7084	–0.1347	–0.2454	0.071
H(15B)	4*e*	0.6065	–0.2089	–0.2646	0.071
H(17A)	4*e*	1.0684	–0.3060	–0.3990	0.141
H(17B)	4*e*	1.0705	–0.3295	–0.2706	0.141
H(17C)	4*e*	1.1631	–0.2567	–0.3038	0.141

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(1)	4*e*	0.7808(2)	0.04513(8)	0.3379(1)	0.0521(7)	0.0524(7)	0.0500(7)	0.0051(6)	0.0076(6)	0.0001(6)
C(2)	4*e*	0.9583(2)	0.02521(8)	0.3332(1)	0.0535(7)	0.0583(8)	0.0486(7)	0.0059(6)	0.0110(6)	0.0030(6)
C(3)	4*e*	1.0946(2)	0.05607(9)	0.4017(1)	0.0518(8)	0.0715(9)	0.0615(9)	−0.0047(7)	0.0078(7)	0.0121(7)
C(4)	4*e*	1.0528(2)	0.1094(1)	0.4773(2)	0.076(1)	0.067(1)	0.0649(9)	−0.0179(8)	−0.0027(8)	0.0022(8)
C(5)	4*e*	0.8785(3)	0.1305(1)	0.4860(2)	0.087(1)	0.0597(9)	0.070(1)	−0.0028(8)	0.0071(9)	−0.0128(8)
C(6)	4*e*	0.7444(2)	0.09854(9)	0.4177(1)	0.0677(9)	0.0585(8)	0.0618(9)	0.0097(7)	0.0078(7)	−0.0063(7)
C(7)	4*e*	0.6269(2)	0.01238(8)	0.2700(1)	0.0487(7)	0.0624(8)	0.0560(8)	0.0086(6)	0.0094(6)	−0.0042(6)
C(8)	4*e*	0.6459(2)	−0.03441(7)	0.1674(1)	0.0430(6)	0.0551(7)	0.0463(7)	0.0054(5)	0.0064(5)	0.0016(6)
C(9)	4*e*	0.7234(2)	−0.01051(7)	0.0694(1)	0.0415(6)	0.0507(7)	0.0531(7)	0.0030(5)	0.0055(6)	0.0060(6)
C(10)	4*e*	0.7268(2)	−0.05271(8)	−0.0284(1)	0.0462(6)	0.0573(7)	0.0447(7)	0.0030(6)	0.0102(5)	0.0080(6)
C(11)	4*e*	0.6522(2)	−0.12079(7)	−0.0235(1)	0.0477(7)	0.0524(7)	0.0438(6)	0.0048(6)	0.0092(5)	0.0020(5)
C(12)	4*e*	0.5751(2)	−0.14444(7)	0.0732(1)	0.0511(7)	0.0494(7)	0.0485(7)	−0.0007(6)	0.0105(6)	0.0044(6)
C(13)	4*e*	0.5673(2)	−0.10339(8)	0.1687(1)	0.0513(7)	0.0594(8)	0.0458(7)	0.0008(6)	0.0124(6)	0.0057(6)
C(14)	4*e*	0.5445(2)	−0.23426(9)	−0.0536(1)	0.072(1)	0.0599(9)	0.0568(8)	−0.0058(7)	0.0149(7)	−0.0054(7)
C(15)	4*e*	0.6935(2)	−0.18310(9)	−0.2144(1)	0.0693(9)	0.0632(8)	0.0459(7)	0.0051(7)	0.0111(7)	−0.0009(6)
C(16)	4*e*	0.8704(2)	−0.22298(7)	−0.2104(1)	0.0692(9)	0.0457(7)	0.0494(7)	0.0011(6)	0.0121(7)	0.0041(6)
C(17)	4*e*	1.0639(3)	−0.2888(1)	−0.3221(2)	0.108(2)	0.090(1)	0.090(1)	0.038(1)	0.039(1)	0.006(1)
N(1)	4*e*	0.6274(2)	−0.17720(7)	−0.1023(1)	0.0641(7)	0.0579(7)	0.0493(6)	−0.0033(6)	0.0148(5)	−0.0034(5)
O(1)	4*e*	0.4755(2)	0.02155(8)	0.2987(1)	0.0513(6)	0.118(1)	0.0884(9)	0.0074(6)	0.0177(6)	−0.0362(8)
O(2)	4*e*	0.5074(2)	−0.21427(5)	0.05499(9)	0.0715(6)	0.0546(5)	0.0574(6)	−0.0095(5)	0.0180(5)	0.0007(5)
O(3)	4*e*	0.5075(2)	−0.29277(7)	−0.0939(1)	0.111(1)	0.0648(7)	0.0819(8)	−0.0208(7)	0.0271(8)	−0.0159(6)
O(4)	4*e*	0.9708(2)	−0.22780(8)	−0.1273(1)	0.0948(9)	0.0959(9)	0.0662(7)	0.0297(8)	−0.0028(7)	−0.0017(7)
O(5)	4*e*	0.8973(2)	−0.24982(7)	−0.3115(1)	0.0841(8)	0.0773(7)	0.0589(6)	0.0137(6)	0.0213(6)	−0.0070(5)
F(1)	4*e*	1.0020(1)	−0.02758(6)	0.26030(8)	0.0585(5)	0.0859(6)	0.0686(6)	0.0206(4)	0.0085(4)	−0.0136(5)
Cl(1)	4*e*	0.81124(5)	0.07720(2)	0.06594(3)	0.0585(2)	0.0540(2)	0.0671(2)	−0.0051(2)	0.0061(2)	0.0077(2)

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