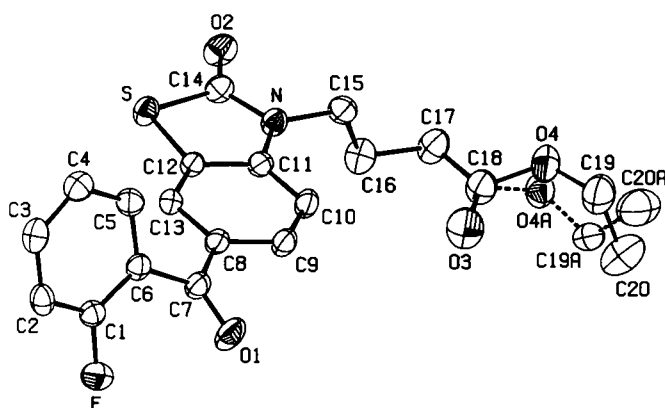


Crystal structure of ethyl [6-(2-fluorobenzoyl)-2-oxo-benzothiazolin-3-yl]-butanoate, C₂₀H₁₈FNO₄S

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Abstract

C₂₀H₁₈FNO₄S, monoclinic, *P*12₁/*n*1 (no. 14),
 $a = 12.537(1)$ Å, $b = 11.902(1)$ Å, $c = 12.573(1)$ Å,
 $\beta = 93.229(4)^\circ$, $V = 1873.0$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.044$,
 $wR_{\text{ref}}(F^2) = 0.135$, $T = 293$ K.

Source of material

2.0 mmol of sodium was dissolved in 30 ml of absolute ethanol and 2.0 mmol of 6-(2-fluorobenzoyl)-2-benzothiazolinone [1,2] was added to this solution. After 6-(2-fluorobenzoyl)-2-benzothiazolinone had dissolved, the solution was stirred for one hour and then evaporated to dryness. The solid residue was dissolved in dimethylformamide and 2.0 mmol of ethyl 4-chlorobutanoate was added. The final solution was refluxed at 353 K for 4 h. The solution was cooled to 273 K and then 100 g of ice-water was added and stirring continued for 1 h between 273 K and 283 K. The precipitate thus formed was collected by filtration, washed with water, dried and crystallized from ether.

Experimental details

The ethyl group was found to be disordered and resolved into two positions whose site occupancies were refined isotropically to 0.616(7)/0.384. The C_{sp2}—O, C_{sp3}—O and C—C bond lengths of the involved atoms were restrained to 1.340(5) Å, 1.450(5) Å and 1.530(5) Å, respectively. A split of O3 into two sites was omitted, the O—C—O angles are 122.6(4)° (O4) and 121.8(4)° (O4A).

Discussion

A comparison between the title structure and those of known thiazole derivatives [3–5] reveals no significant differences, either in bond lengths or in bond angles. Moreover, the derivatives

of the title compound show higher analgesic activity than aspirin and have an anti-inflammatory activity as good as indometacin [5]. In the butanoate group of the title structure, the freely refined C—C bond lengths range from 1.479(4) Å to 1.526(4) Å and the C—C—C bond angles are 110.6(2)° and 113.0(2)°. The C1—F distance 1.346(3) Å is in agreement with those of other compounds, such as 5-chloro-6-(2-fluorobenzoyl)-1,3-benzoxazol-2(3*H*)-one [3] (1.356(2) Å) and *trans,cis*-(±)-3'-(4-fluorophenyl)-2-phenylspiro[2*H*-1-benzothiopyran-3(4*H*),2'-oxiran]-4-one-1-oxide [4] (1.363(4) Å). The bond distances of S—C14 and S—C12 are 1.787(3) Å and 1.749(2) Å, respectively. These values are in agreement with those in 3-(6-benzoyl-2-oxo-2,3-dihydro-2-benzothiazol-3-yl)propanoic acid [5] (1.781(4) Å and 1.743(4) Å). The O1—C7—C8, O1—C7—C6, F—C1—C6 and F—C1—C2 bond angles are 121.2(2)°, 119.7(2)°, 118.8(2)° and 118.0(2)°, respectively. The C3—C4—C5—C6 and O2—C14—N—C11 torsion angles are 0.0(5)° and 179.8(3)°, respectively, and the double bond length of C14=O2 is 1.212(3) Å.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.30 × 0.35 × 0.35 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	2.08 cm ⁻¹
Diffraction, scan mode:	Enraf-Nonius TurboCAD4, ω
$2\theta_{\text{max}}$:	52.58°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3949, 3779
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2668
$N(\text{param})_{\text{refined}}$:	272
Programs:	SHELXS-97 [6], SHELXL-97 [7], ORTEP-3 [8], WinGX [9]

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

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4e		0.6245	0.0007	1.5616	0.067
H(3)	4e		0.7839	0.0930	1.6021	0.071
H(4)	4e		0.9240	0.0742	1.4920	0.072
H(5)	4e		0.9079	-0.0411	1.3444	0.066
H(9)	4e		0.8159	-0.3033	1.1258	0.064
H(10)	4e		0.8847	-0.2663	0.9636	0.065
H(13)	4e		0.7751	0.0271	1.1779	0.053
H(15A)	4e		0.9854	-0.2002	0.8359	0.067
H(15B)	4e		1.0082	-0.0966	0.7635	0.067
H(16A)	4e		0.8436	-0.1171	0.6723	0.084
H(16B)	4e		0.8124	-0.2140	0.7495	0.084
H(17A)	4e		0.8849	-0.2637	0.5734	0.087
H(17B)	4e		0.9959	-0.2446	0.6345	0.087

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(19A)	4e	0.62	1.0004	-0.5679	0.7528	0.134
H(19B)	4e	0.62	1.0749	-0.5912	0.6588	0.134
H(20A)	4e	0.62	0.9297	-0.7128	0.6462	0.187
H(20B)	4e	0.62	0.9252	-0.6328	0.5471	0.187
H(20C)	4e	0.62	0.8511	-0.6103	0.6412	0.187

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(19C)	4e	0.38	0.8887	-0.6070	0.5653	0.122
H(19D)	4e	0.38	0.8657	-0.5758	0.6827	0.122
H(20D)	4e	0.38	0.9992	-0.7111	0.6808	0.205
H(20E)	4e	0.38	1.0454	-0.6011	0.7342	0.205
H(20F)	4e	0.38	1.0700	-0.6300	0.6164	0.205

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S	4e		0.84627(5)	0.11911(5)	0.97117(5)	0.0668(4)	0.0393(3)	0.0532(4)	0.0023(3)	0.0052(3)	0.0038(3)
F	4e		0.5788(1)	-0.1259(1)	1.4064(1)	0.069(1)	0.075(1)	0.090(1)	-0.0159(8)	0.0241(8)	-0.0146(9)
O(1)	4e		0.7097(2)	-0.2660(2)	1.2872(2)	0.131(2)	0.045(1)	0.080(1)	-0.025(1)	0.052(1)	-0.0090(9)
O(2)	4e		0.9282(2)	0.1012(2)	0.7804(1)	0.091(1)	0.063(1)	0.053(1)	-0.010(1)	0.0116(9)	0.0110(9)
O(3)	4e		0.8728(2)	-0.4083(2)	0.7611(2)	0.142(2)	0.089(2)	0.070(1)	0.005(1)	0.039(1)	0.019(1)
N	4e		0.9045(2)	-0.0618(2)	0.8753(1)	0.062(1)	0.044(1)	0.045(1)	-0.0020(9)	0.0094(9)	-0.0018(8)
C(1)	4e		0.6730(2)	-0.0748(2)	1.4296(2)	0.056(1)	0.043(1)	0.053(1)	-0.001(1)	0.011(1)	0.003(1)
C(2)	4e		0.6817(2)	-0.0075(2)	1.5182(2)	0.071(2)	0.051(1)	0.047(1)	0.012(1)	0.018(1)	0.003(1)
C(3)	4e		0.7765(2)	0.0476(2)	1.5419(2)	0.081(2)	0.052(1)	0.044(1)	0.012(1)	-0.003(1)	-0.003(1)
C(4)	4e		0.8605(2)	0.0357(2)	1.4764(2)	0.062(2)	0.058(2)	0.059(2)	-0.001(1)	-0.005(1)	0.000(1)
C(5)	4e		0.8506(2)	-0.0332(2)	1.3876(2)	0.059(1)	0.052(1)	0.055(1)	0.001(1)	0.011(1)	0.003(1)
C(6)	4e		0.7555(2)	-0.0908(2)	1.3623(2)	0.059(1)	0.039(1)	0.043(1)	0.000(1)	0.012(1)	0.0035(9)
C(7)	4e		0.7475(2)	-0.1738(2)	1.2729(2)	0.069(2)	0.040(1)	0.055(1)	-0.006(1)	0.020(1)	-0.003(1)
C(8)	4e		0.7894(2)	-0.1426(2)	1.1689(2)	0.053(1)	0.040(1)	0.047(1)	-0.0052(9)	0.012(1)	-0.0049(9)
C(9)	4e		0.8216(2)	-0.2293(2)	1.1031(2)	0.071(2)	0.037(1)	0.054(1)	-0.005(1)	0.015(1)	-0.002(1)
C(10)	4e		0.8616(2)	-0.2079(2)	1.0056(2)	0.071(2)	0.038(1)	0.055(1)	-0.001(1)	0.016(1)	-0.009(1)
C(11)	4e		0.8669(2)	-0.0980(2)	0.9714(2)	0.049(1)	0.044(1)	0.044(1)	-0.0032(9)	0.0067(9)	-0.002(1)
C(12)	4e		0.8322(2)	-0.0102(2)	1.0350(2)	0.045(1)	0.036(1)	0.048(1)	-0.0007(9)	0.0019(9)	-0.0015(9)
C(13)	4e		0.7954(2)	-0.0315(2)	1.1346(2)	0.047(1)	0.038(1)	0.049(1)	0.0004(9)	0.0067(9)	-0.0056(9)
C(14)	4e		0.8995(2)	0.0524(2)	0.8584(2)	0.060(1)	0.050(1)	0.048(1)	-0.003(1)	0.003(1)	0.003(1)
C(15)	4e		0.9525(2)	-0.1368(2)	0.7982(2)	0.061(2)	0.057(2)	0.051(1)	-0.004(1)	0.012(1)	-0.001(1)
C(16)	4e		0.8715(2)	-0.1795(3)	0.7150(2)	0.085(2)	0.065(2)	0.060(2)	0.007(1)	-0.008(1)	-0.002(1)
C(17)	4e		0.9221(3)	-0.2655(2)	0.6432(2)	0.105(2)	0.064(2)	0.049(1)	-0.009(2)	0.016(1)	-0.006(1)
C(18)	4e		0.9188(3)	-0.3813(2)	0.6857(2)	0.101(2)	0.064(2)	0.056(2)	-0.001(2)	0.024(2)	-0.000(1)
O(4)	4e	0.62	0.9905(4)	-0.4495(4)	0.6401(4)	0.106(4)	0.063(2)	0.111(4)	0.007(3)	0.029(3)	-0.018(2)
C(19)	4e	0.62	1.0050(6)	-0.5643(4)	0.6762(7)	0.100(6)	0.076(5)	0.158(7)	0.011(4)	-0.002(5)	-0.016(5)
C(20)	4e	0.62	0.9201(7)	-0.6365(7)	0.6229(7)	0.159(9)	0.092(5)	0.121(6)	-0.033(6)	-0.002(5)	0.003(5)
O(4A)	4e	0.38	0.9352(6)	-0.4533(5)	0.6059(5)	0.095(5)	0.064(4)	0.073(4)	0.004(4)	0.025(4)	-0.008(3)
C(19A)	4e	0.38	0.9184(7)	-0.5705(6)	0.629(1)	0.062(6)	0.058(6)	0.19(1)	-0.007(5)	0.030(7)	-0.005(7)
C(20A)	4e	0.38	1.0173(8)	-0.634(1)	0.669(1)	0.090(8)	0.087(8)	0.23(2)	0.003(8)	-0.01(1)	0.05(1)

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