# Crystal structure of ethyl [6-(2-fluorobenzoyl)-2-oxo-benzothiazolin-3-yl]-butanoate, $C_{20}H_{18}FNO_4S$

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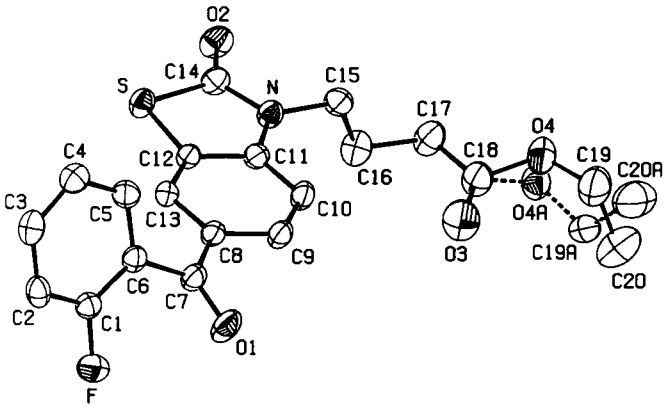


**Abstract**

$C_{20}H_{18}FNO_4S$, monoclinic, $P12_1/n1$ (no. 14), $a = 12.537(1)$ Å, $b = 11.902(1)$ Å, $c = 12.573(1)$ Å, $\beta = 93.229(4)°$, $V = 1873.0$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.135$, $T = 293$ 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 $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 2.08 cm$^{-1}$ |
| Diffractometer, scan mode: | Enraf-Nonius TurboCAD4, $\omega$ |
| $2\theta_{max}$: | 52.58° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 3949, 3779 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 2668 |
| $N(param)_{refined}$: | 272 |
| Programs: | SHELXS-97 [6], SHELXL-97 [7], ORTEP-3 [8], WinGX [9] |

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

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

| 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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