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3-(6-Benzoyl-2-oxo-2,3-dihydro-2-benzothiazol-3-yl)propanoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.190
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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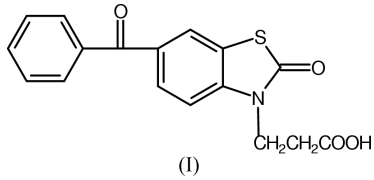
The molecule of the title compound, $C_{17}H_{13}NO_4S$, is non-planar. In the propanoic acid group, the average C—C bond length and the average C—C—C bond angle are 1.501 (1) Å and 110.9 (3)°, respectively.

Comment

The title compound, (I), shows higher analgesic activity than aspirin and has an anti-inflammatory activity as good as indometacin (Dündar *et al.*, 2003). In this study, the crystal structure of (I) has been determined by X-ray diffraction.



(I)

The atom-numbering scheme of (I) is shown in Fig. 1. Selected bond lengths and angles are listed in Table 1. The lengthening of the S1—C14 bond distance [1.781 (4) Å] *versus* the S1—C10 bond distance [1.743 (4) Å] is 0.034 (3) Å {S1—C7 = 1.776 (3) Å and S1—C6 = 1.742 (3) Å in methyl 3-[5-chloro-2-oxo-1.3-benzothiazol-3(2*H*)-yl]propanoate (Aydın *et al.*, 2002)}; this may be a consequence of steric interaction.

The maximum deviations from the mean plane through the benzothiazole (N1/C11/C12/C13/C8/C9/C10/S1/C14, which is almost planar) are −0.016 (3) and 0.017 (3) Å for N1 and C12, respectively. The dihedral angle between the benzothiazole and the phenyl ring (C1–C6) is 49.2 (1)°.

A quantum-chemical calculation was performed using the *PM*3 method; the charges at atoms N1, S1, O1, O2, O3 and O4 are 0.0353, 0.1836, −0.2321, −0.2989, −0.2958 and −0.2186 e^-, respectively. The heat of formation of the title compound is −61.49 kcal and its total energy is −3735.42 eV. The values of the HOMO and LUMO energies are −9.19104 and −0.83799 eV, respectively. The calculated molecular dipole moment is 4.641 Debye.

Hydrogen-bonding contacts are summarized in Table 2. The crystal structure is stabilized by C—H···O and O—H···O intermolecular hydrogen bonds.

Experimental

3-(6-Benzoyl-2-oxo-2-benzothiazolin-3-yl)propanenitrile (10.0 mmol) was added to an *N,N*-dimethylformamide–water–sulfuric acid (1:1:2) mixture (50 ml). After stirring at room temperature for 2 h, the mixture was refluxed for 4 h. The mixture, cooled to the room temperature, was poured into ice water (100 g). The resulting precipitate was filtered off by suction filtration, washed with water,

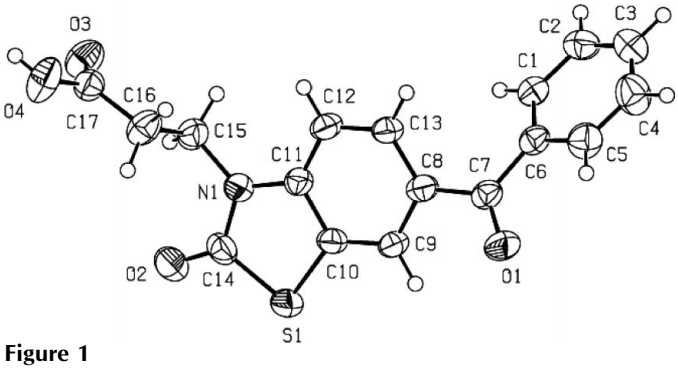


Figure 1
A displacement ellipsoid plot of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

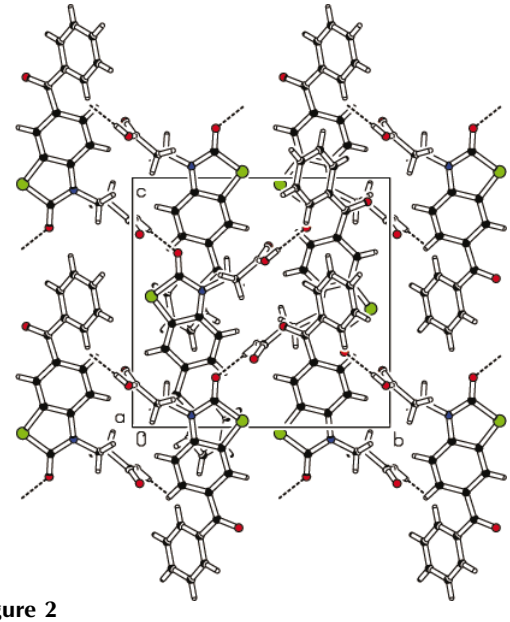


Figure 2
View of the crystal packing and hydrogen-bond contacts (dotted lines) along the *b* axis.

dried and crystallized from ethanol–water (yield 75%, m.p. 456–457 K).

Crystal data

$C_{17}H_{13}NO_4S$
$M_r = 327.35$
Monoclinic, $P2_1/c$
$a = 9.352$ (5) Å
$b = 12.921$ (5) Å
$c = 12.908$ (5) Å
$\beta = 104.370$ (5)°
$V = 1511.0$ (12) Å^3
$Z = 4$
$D_x = 1.439$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 37 reflections
$\theta = 9.4$–32.3°
$\mu = 0.23$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.25 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (Parkin *et al.*, 1995); cubic fit to $\sin(\theta)/\lambda$ – 24 parameters
$T_{min} = 0.944$, $T_{max} = 0.966$
4283 measured reflections
2818 independent reflections
1906 reflections with $I > 2\sigma(I)$
$R_{int} = 0.002$
$\theta_{max} = 26.3°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 0$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.190$
$S = 1.06$
2818 reflections
208 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1078P)^2 + 0.8574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C10	1.743 (4)	O4—C17	1.330 (5)
S1—C14	1.781 (4)	N1—C11	1.384 (5)
O1—C7	1.226 (4)	N1—C15	1.471 (5)
O2—C14	1.213 (5)	N1—C14	1.379 (5)
O3—C17	1.198 (5)		
C10—S1—C14	91.44 (16)	N1—C11—C10	112.2 (3)
C11—N1—C15	125.3 (3)	S1—C14—O2	125.1 (3)
C14—N1—C15	118.5 (3)	O2—C14—N1	126.0 (3)
C11—N1—C14	116.2 (3)	S1—C14—N1	108.9 (2)
O1—C7—C6	119.1 (3)	N1—C15—C16	112.2 (3)
O1—C7—C8	119.5 (3)	C15—C16—C17	111.0 (3)
S1—C10—C9	128.3 (2)	O3—C17—C16	124.4 (4)
S1—C10—C11	111.3 (3)	O4—C17—C16	112.8 (3)
N1—C11—C12	127.6 (3)	O3—C17—O4	122.8 (4)
C14—N1—C11—C10	1.3 (4)	C5—C6—C7—O1	−19.6 (4)
C15—N1—C14—O2	−1.1 (5)	O1—C7—C8—C9	−29.1 (4)
C11—N1—C15—C16	−92.9 (4)	O1—C7—C8—C13	148.0 (3)
C14—N1—C15—C16	87.2 (4)	N1—C15—C16—C17	−166.1 (3)
C11—N1—C14—O2	179.0 (3)	C15—C16—C17—O4	177.9 (3)
C1—C6—C7—O1	155.0 (3)	C15—C16—C17—O3	−0.7 (5)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H40···O2^{i}	0.82	1.90	2.723 (4)	176
C1—H1···O3^{ii}	0.93	2.54	3.181 (4)	127
C12—H12···O1^{iii}	0.93	2.38	3.291 (4)	168
C16—H16*B*···O4^{iv}	0.97	2.59	3.552 (6)	175

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $-x, 1-y, 1-z$; (iii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $1-x, 1-y, 1-z$.

All H atoms were placed in geometrically idealized positions, but not refined. Owing to the poor quality of the crystal, high-order reflections were very weak in intensity. The data collection was therefore stopped at $\theta_{max} = 26.3°$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *WinGX* (Farrugia, 1999).

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