

Cem Cüneyt Ersanlı,^{a*} Yasemin Dündar,^b Uğur Sarı,^c Ningur Noyanalpan,^b Mustafa Odabaşoğlu^d and Ahmet Erdönmez^a^aDepartment of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, ^bDepartment of Pharmaceutical Chemistry, Faculty of Pharmacy, Gazi University, 06330 Ankara, Turkey,^cDepartment of Computer Technology, Faculty of Education, Kırıkkale University, Yahşian Campus, 71450 Kırıkkale, Turkey, and^dDepartment of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey

Correspondence e-mail: ccersan@omu.edu.tr

Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.039

wR factor = 0.126

Data-to-parameter ratio = 21.8

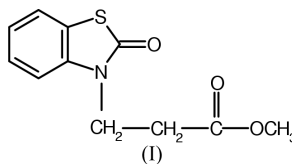
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Methyl 3-(2-oxobenzothiazolin-3-yl)-propanoate

In the title compound, $\text{C}_6\text{H}_4\text{SC}(\text{O})\text{NCH}_2\text{CH}_2\text{COOCH}_3$, the bicyclic benzothiazole system is planar within 0.025 \AA ; the displacements of the carbonyl oxygen and the β -carbon atom of the methylpropionate substituent from the benzothiazole mean plane are $-0.028 (2)$ and $0.002 (2) \text{ \AA}$, respectively. There is a short intermolecular $\text{C}-\text{H}\cdots\text{O}$ contact between the α atom of the methyl propionate substituent and the carbonyl oxygen of the oxobenzothiazole group [$\text{C}\cdots\text{O} 3.241 (2) \text{ \AA}$]. These contacts link the molecules into infinite chains, running along the b axis of the crystal.

Comment

As part of our studies of benzothiazole derivatives, which represent valuable starting materials for the design of new drugs and frequently exhibit important biological properties (Varkonda *et al.*, 1985), we have undertaken the X-ray structural study of the title compound, (I).



An *ORTEP*III (Farrugia, 1997) view of (I) and a packing diagram are shown in Figs. 1 and 2, respectively. The bicyclic benzothiazole system of (I) is planar within 0.025 \AA ; the displacements of the carbonyl oxygen and the β -carbon atom of the methyl propionate substituent are $-0.028 (2)$ and $0.002 (2) \text{ \AA}$, respectively.

The $\text{C7}-\text{N1}$, $\text{C7}-\text{S1}$, and $\text{C1}-\text{S1}$ bond lengths [$1.371 (2)$, $1.768 (2)$ and $1.742 (2) \text{ \AA}$, respectively] are approximately midway between the corresponding standard values for single and double carbon–nitrogen (1.49 and 1.27 \AA ; Clayden *et al.*, 2001) and carbon–sulfur (1.81 and 1.61 \AA ; Khan *et al.*, 1988) bonds. The $\text{C7}-\text{N1}$ bond length is in good agreement with the reported values of $1.365 (3) \text{ \AA}$ for 3-methyl-2(3*H*)-benzothiazolone (Rudd & Barany, 1984), $1.367 (2) \text{ \AA}$ for 4-(2-carboxybenzoyl)-2(3*H*)-benzothiazolone (Lamiot *et al.*, 1995), and $1.375 (3) \text{ \AA}$ for ethyl 4-(2-oxobenzothiazolin-3-yl)-butanoate (Baysen *et al.*, 2002). The difference between the $\text{C1}-\text{S1}$ and $\text{C7}-\text{S1}$ bonds may be attributed to the different degrees of conjugation of the electron lone pair of the S1 atom with the aromatic six-membered ring and the $\text{C}=\text{O}$ group. The $\text{C1}-\text{S1}$ and $\text{S1}-\text{C7}$ bond lengths [$1.742 (2)$ and $1.768 (2) \text{ \AA}$] agree well with the reported values of $1.743 (3)$ and $1.777 (3) \text{ \AA}$ for 3-methyl-2(3*H*)-benzothiazolone (Rudd & Barany, 1984), $1.734 (2)$ and $1.776 (3) \text{ \AA}$ for 4-(2-carboxy-

Received 21 July 2003

Accepted 24 September 2003

Online 7 October 2003

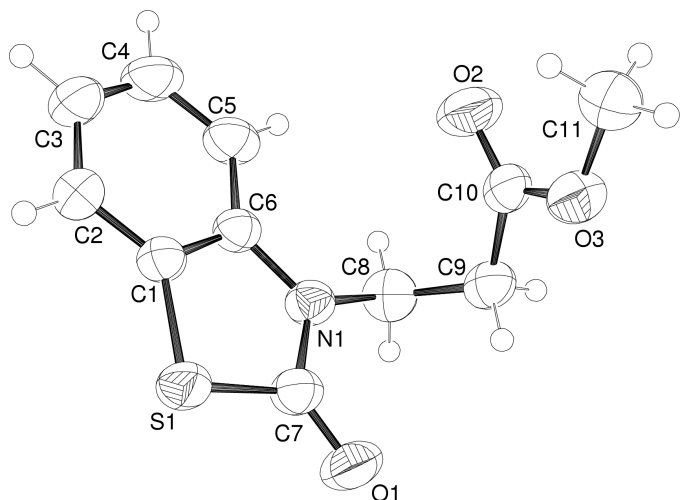


Figure 1
A view of the molecule of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level; H atoms are shown as small spheres of arbitrary radii.

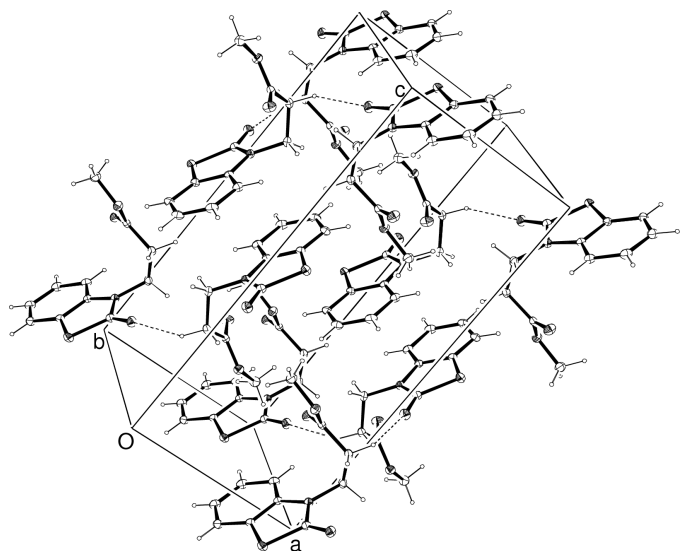


Figure 2
A packing diagram of the crystal structure of the title compound, showing the C—H...O contacts as dashed lines.

benzoyl)-2(3*H*)-benzothiazolone (Lamiot *et al.*, 1995), and 1.743 (3) and 1.772 (3) Å for ethyl 4-(2-oxobenzothiazolin-3-yl)butanoate (Baysen *et al.*, 2002).

There is a short intermolecular C9—H9B...O1ⁱ contact between the α atom of the methyl propionate substituent and the carbonyl oxygen of the oxobenzothiazole group [C9...O1ⁱ 3.241 (2) Å, H9B...O1ⁱ 2.47 Å; symmetry code (i): $-x, \frac{1}{2} + y, \frac{1}{2} - z$]. These contacts link the molecules into infinite chains along the *b* axis of the crystal.

Experimental

10.0 mmol (1.51 g) of 2(3*H*)-benzothiazolone and 11.0 mmol (1.11 g, 1.53 ml) of triethylamine were dissolved in 30 ml of methanol. 11.0 mmol (0.95 g, 0.99 ml) of methyl acrylate was added to the solution. The mixture was heated at 323–333 K for 6 h. It was then

cooled to room temperature and 100 g ice-water was added and stirred for 1 h. The precipitate was collected by filtration, dried and washed with 10% NaOH solution, followed by water until neutral, then dried again and crystallized from methanol; yield 1.66 g (70%).

Crystal data

C₁₁H₁₁NO₃S
M_r = 237.27
 Monoclinic, *P*2₁/*c*
a = 7.9109 (13) Å
b = 8.386 (3) Å
c = 16.4898 (16) Å
 β = 93.645 (12)°
V = 1091.8 (4) Å³
Z = 4

D_x = 1.443 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 3603 reflections
 θ = 20.1–26.4°
 μ = 0.29 mm⁻¹
T = 293 (2) K
 Prism, yellow
 0.90 × 0.60 × 0.25 mm

Data collection

Rigaku AFC-7S diffractometer
 ω -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
T_{min} = 0.812, *T_{max}* = 0.930
 3383 measured reflections
 3176 independent reflections
 2039 reflections with *I* > 2 σ (*I*)

R_{int} = 0.040
 θ_{\max} = 30.0°
h = 0 → 11
k = 0 → 11
l = -23 → 23
 3 standard reflections
 every 150 reflections
 intensity decay: 0.6%

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.039
wR(*F*²) = 0.126
S = 1.03
 3176 reflections
 146 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.2099P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.35 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL*
 Extinction coefficient: 0.085 (5)

Table 1

Selected geometric parameters (Å, °).

S1—C1	1.742 (2)	O1—C7	1.217 (2)
S1—C7	1.768 (2)	O2—C10	1.200 (2)
N1—C6	1.402 (2)	O3—C10	1.330 (2)
N1—C7	1.371 (2)	O3—C11	1.443 (2)
N1—C8	1.465 (2)		
C1—S1—C7	91.50 (8)	C1—C6—N1	112.5 (2)
C7—N1—C6	114.9 (2)	O1—C7—N1	125.7 (2)
C7—N1—C8	118.8 (2)	O1—C7—S1	124.4 (2)
C6—N1—C8	126.2 (2)	N1—C7—S1	109.9 (2)
C10—O3—C11	117.5 (2)	N1—C8—C9	112.6 (2)
C2—C1—C6	121.58 (16)	O2—C10—O3	123.7 (2)
C2—C1—S1	127.30 (14)	O2—C10—C9	125.3 (2)
C6—C1—S1	111.12 (13)	O3—C10—C9	111.1 (2)
C5—C6—N1	127.3 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C9—H9B...O1 ⁱ	0.97	2.47	3.241 (2)	136

Symmetry code: (i) $-x, \frac{1}{2} + y, \frac{1}{2} - z$.

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* for

Windows (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The authors thank the University of Kırıkkale, Turkey for the data collection.

References

- Baysen, F., Yabasan, R., Ide, S., Şahin, E., Çakır, B. & Dündar, Y. (2002). *Z. Kristallogr. New Cryst. Struct.* **217**, 529–530.
- Clayden, J., Greeves, N., Warren, S. & Wothers, P. (2001). *Organic Chemistry*, pp. 164–165. Oxford University Press.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Khan, M. A., Taylor, R. W., Lehn, J. M. & Dietrich, B. (1988). *Acta Cryst.* **C44**, 1928–1931.
- Lamiot, J., Baert, F., Taverne, T., Houssin, R. & Lesieur, I. (1995). *Acta Cryst.* **C51**, 1912–1915.
- Molecular Structure Corporation (1994). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1997). *TEXSAN for Windows* (Version 1.03) and *Single Crystal Structure Analysis Software* (Version 1.03). MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Rudd, S. & Barany, G. (1984). *Acta Cryst.* **C40**, 2118–2120.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Varkonda, S., Hýblová, O., Sutoris, V., Konecný, V. & Mikulášek, S. (1985). Czechoslovakian Patent No. 239.411.