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N-[2-(Aminosulfonyl)-5,6-dihydro-6-methyl-7,7-dioxo-4*H*-thieno[2,3-*b*]-thiopyran-4-yl]acetamide

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Key indicators

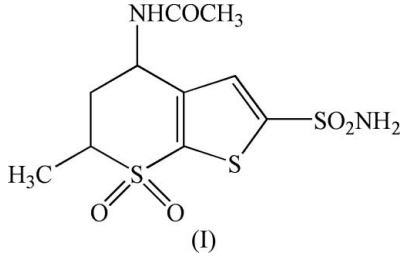
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.113
Data-to-parameter ratio = 15.2

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

The title compound, $C_{10}H_{14}N_2O_5S_3$, has been synthesized as a key intermediate in the synthesis of topically active carbonic anhydrase inhibitor MK-0507. N—H···O hydrogen bonds are responsible for the formation of centrosymmetric dimers and zigzag molecular chains. The intermolecular non-bonded S···O separation in the crystal structure is 3.415 (2) Å, indicating a strong intermolecular interaction between the aminosulfonyl and *S*-dioxide groups.

Comment

The title compound, (I), is a key intermediate in the total synthesis of topically active carbonic anhydrase inhibitor MK-0507 (Blacklock *et al.*, 1993; Thomas *et al.*, 1997).



(I)

The molecular structure and atom-labelling scheme are shown in Fig. 1. The shortest intermolecular S···O contact, between S1 and O3^{i}, is 3.415 (2) Å [symmetry code: (i) $-x+\frac{3}{2}$, $y+\frac{1}{2}$, z], indicating a strong intermolecular interaction between these two atoms. In addition, there are also weak S···C interactions [non-bonded separation = 3.858 (3) Å], as well as S···S short contacts [3.949> (4) Å] (Lan *et al.*, 2005).

Further examination of the crystal structure of (I) reveals possible N—H···O and C—H···O interactions (Table 1). Intermolecular N2—H2*B*···O3^{i} hydrogen bonds [symmetry code: (*i*) $-x+\frac{3}{2}$, $y+\frac{1}{2}$, z] form zigzag molecular chains propagating in the *b*-axis direction, while N2—H2*A*···O2^{ii} hydrogen bonds [symmetry code: (ii) $-x+2$, $-y+2$, $-z+1$] form centrosymmetric dimers, as shown in Fig. 2.

Experimental

The title compound was synthesized from methyl 3-(*p*-toluenesulfonyloxy)butyrate according to a literature method (Blacklock *et al.*, 1993; Ponticello *et al.*, 1987, 1988). Single crystals of (I) were grown by slow evaporation, in air, of a THF solution. Selected analytical data: pale-yellow solid, yield 89%; m.p. 524–525 K; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 8.62 (*d*, 1H), 8.05 (*s*, 2H), 7.42 (*s*, 1H), 5.19 (*m*, 1H), 3.89 (*m*, 1H), 2.45 (*m*, 1H), 2.29(*m*, 1H), 1.86 (*s*, 3H),

1.33 (*d*, 3H); IR (KBr) *v*: 3337, 3240, 3080, 2939, 1654, 1523, 1449, 1334, 1303, 1170, 1138, 1035, 926, 873, 694 cm^{-1};

Crystal data

$C_{10}H_{14}N_2O_5S_3$
M_r = 338.41
Orthorhombic, *Pbca*
a = 9.842 (3) Å
b = 15.290 (5) Å
c = 19.434 (6) Å
V = 2924.5 (15) $Å^3$
Z = 8
D_x = 1.537 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1543 reflections
θ = 2.7–21.2°
μ = 0.53 mm^{-1}
T = 273 (2) K
Prism, yellow
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.902, T_{max} = 0.949
13578 measured reflections
2865 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.0°
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 0.96
2865 reflections
189 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.32 e $Å^{-3}$

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O3^{i}	0.81 (4)	2.14 (4)	2.876 (4)	150 (3)
N2—H2A···O2^{ii}	0.85 (3)	2.14 (3)	2.962 (4)	161 (3)
N1—H1D···O1^{iii}	0.86	2.07	2.861 (3)	152
C5—H5A···O1^{iii}	0.98	2.52	3.346 (3)	142
C4—H4A···O2^{iv}	0.97	2.66	3.623 (4)	174
C3—H3A···O4^{v}	0.98	2.53	3.276 (3)	132

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

H atoms were included using a riding model and were constrained to have C—H = 0.95 Å, N—H = 0.86 Å and $U_{iso} = 1.2U_{eq}$ of the parent atom. H atoms attached to N2 were refined freely.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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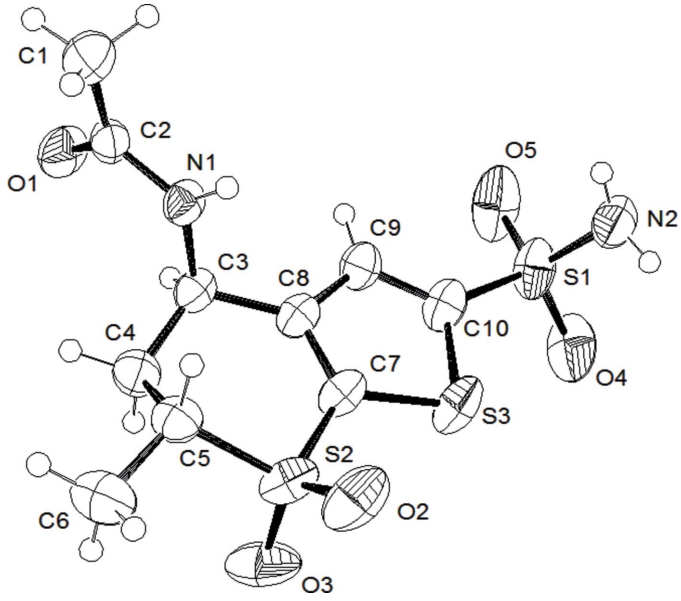

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

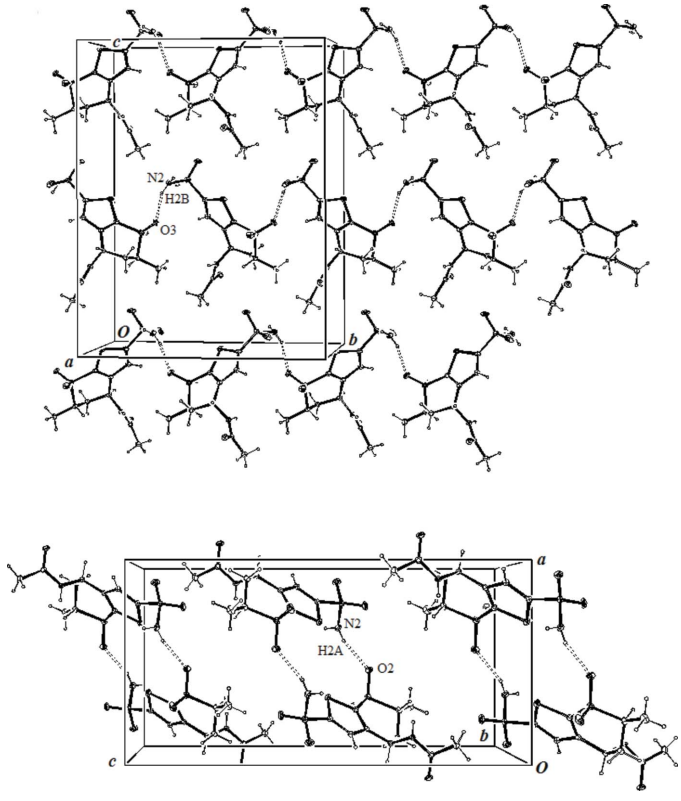

Figure 2
The crystal structure of (I), viewed in two directions. Dashed lines indicate N—H···O hydrogen bonds.

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