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N-[2-(Aminocarbonyl)phenyl]-4-hydroxy-2-methyl-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide dimethyl sulfoxide solvate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.105
Data-to-parameter ratio = 16.3

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

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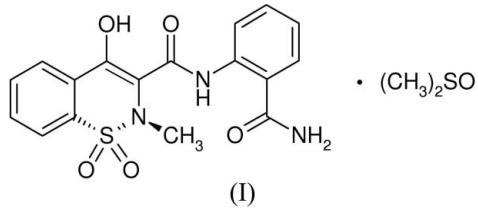
In the title compound, $C_{17}H_{15}N_3O_5S\cdot C_2H_6OS$, the thiazine ring adopts a distorted half-chair conformation. The enolic H atom is involved in both intramolecular and intermolecular O—H···O hydrogen bonds, the latter linking the molecules into centrosymmetric pairs. Both anthranilamide H atoms are involved in hydrogen bonding to O atoms of dimethyl sulfoxide molecules, linking the pairs of molecules into chains.

Comment

Owing to their application as non-steroidal anti-inflammatory agents (Turck *et al.*, 1996; Bihovsky *et al.*, 2004), considerable attention has been given to synthetic and structural investigations of 1,2-benzothiazine 1,1-dioxides and their precursor intermediates (Golič & Leban, 1987). During our syntheses of various benzothiazine derivatives (Rehman *et al.*, 2005; Rehman *et al.*, 2006) the crystal structure of the title compound, (I), has been determined.



(I)

In (I) (Fig. 1), the thiazine ring adopts a distorted half-chair conformation. The geometry at N1 is pyramidal, with the methyl group pointing approximately perpendicular to the thiazine ring. Atoms O3 and O1 lie approximately in the plane of the thiazine ring, while atom O2 lies approximately perpendicular to it. Atoms N1, C8, C9, O4 and N2 are coplanar to within 0.093 (2) Å. The S1—N1 bond length of 1.6427 (15) Å is as expected for a sulfonamide.

Like other 1,2-benzothiazine 1,1-dioxide molecules (Golič & Leban, 1987; Fabiola *et al.*, 1998), the enolic hydrogen on O3 is involved in intramolecular hydrogen bonding (Table 1), and there is a shortening of the C7—C8 bond [1.362 (3) Å] due to partial double-bond character. Two further intramolecular hydrogen bonds are also present in (I) that are not observed in related benzothiazine molecules such as piroxicam (Kojić-Prodić *et al.*, 1982) and meloxicam (Fabiola *et al.*, 1998). Specifically, atom H2 forms hydrogen bonds with both N1 and the anthranilamide atom O5. Atom H3 is also involved in intermolecular hydrogen bonding with atom O4 of an adjacent molecule (Table 1 and Fig. 2), linking the molecules into centrosymmetric pairs.

The O atoms of two symmetry-related dimethyl sulfoxide molecules link adjacent benzothiazine molecules through N—

H···O hydrogen bonds from H3*A* and H3*B* of the benzothiazine amino group (Table 1). These interactions link the centrosymmetric pairs of molecules into chains (Fig. 2).

Experimental

N-[2-(Aminocarbonyl)phenyl]-4-hydroxy-2-methyl-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide was synthesized according to a literature method (Rehman *et al.*, 2006). The compound was dissolved in a mixture of methanol and DMSO (80:20 *v*/*v*) at room temperature. Crystals were obtained by slow evaporation and dried under high vacuum.

Crystal data

$C_{17}H_{15}N_3O_5S\cdot C_2H_6OS$
M_r = 451.51
Triclinic, $P\overline{1}$
a = 8.4973 (2) Å
b = 10.1959 (4) Å
c = 12.0545 (4) Å
α = 92.132 (2)°
β = 101.540 (2)°
γ = 95.550 (2)°
V = 1016.75 (6) Å^3
Z = 2
D_x = 1.475 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 120 (2) K
Plate, colourless
0.54 × 0.42 × 0.11 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.853, T_{max} = 0.967
20897 measured reflections
4663 independent reflections
3397 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 1.04
4663 reflections
286 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.2807P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4	0.91 (2)	1.75 (2)	2.5771 (18)	150 (2)
O3—H3···O4^{i}	0.91 (2)	2.48 (2)	2.9028 (19)	108.4 (17)
N2—H2···O5	0.87 (2)	1.84 (2)	2.583 (2)	142.8 (18)
N2—H2···N1	0.87 (2)	2.27 (2)	2.720 (2)	112.0 (16)
N3—H3*A*···O6	0.905 (17)	2.043 (18)	2.911 (2)	160 (2)
N3—H3*B*···O6^{ii}	0.896 (17)	2.064 (18)	2.941 (2)	166 (2)

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x-1, -y+1, -z+1$.

H atoms bound to C atoms were placed geometrically and refined using a riding model, with C—H = 0.95 Å, U_{iso}(H) = 1.2 U_{eq}(C) for aryl H, or C—H = 0.98 Å, U_{iso}(H) = 1.5 U_{eq}(C) for methyl H. The methyl groups were allowed to rotate about their local threefold axes. H atoms bound to N and O atoms were located in difference Fourier maps and their coordinates were refined freely with U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

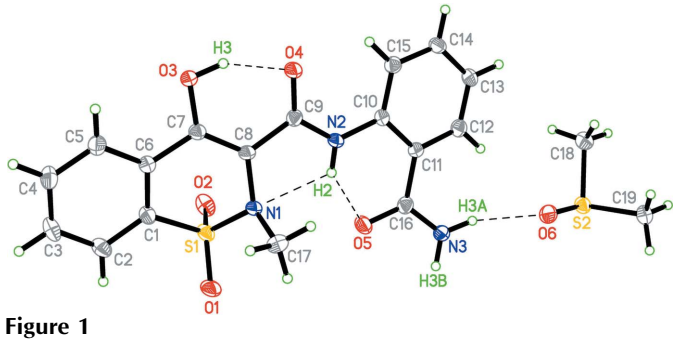


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. Dashed lines denote hydrogen bonds.

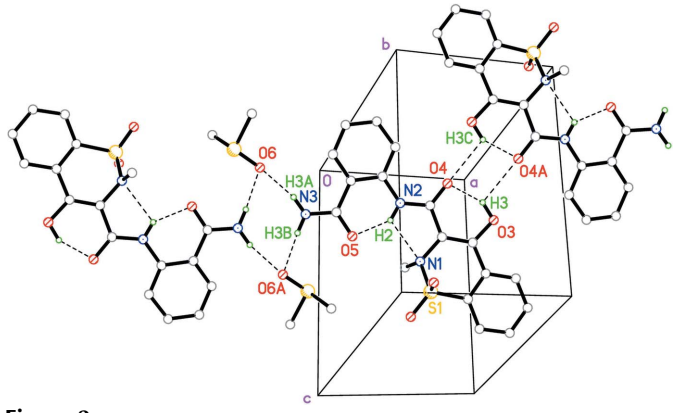


Figure 2
Projection approximately on to the plane of one hydrogen-bonded chain in (I). H atoms not involved in hydrogen bonding have been omitted. Dashed lines denote hydrogen bonds. (Symmetry operators to generate molecules containing O4A and O6A, respectively: $1-x, 2-y, 1-z$; $-x-1, 1-y, 1-z$.)

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