2,4,6-Trimethyl-8,9-dihydro-5*H*,7*H*-pyrido[3,2-e]-pyrazino[1,2-*b*][1,2]thiazin-5-one 11,11-dioxide

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The structure of the title compound, $C_{13}H_{15}N_3O_3S$, is the first report of a structure comprising a novel pyrido[3,2-e]-pyrazino[1,2-*b*][1,2]thiazine ring system. The partially saturated thiazine and pyrazine rings adopt screw-boat and sofa conformations, respectively. The crystal structure contains intermolecular N—H···O hydrogen bonds, short C—H···X (X = N, O) contacts and π–π interactions.

Comment

3-Acyl-4-hydroxypyrido[3,2-*e*]-1,2-thiazine 1,1-dioxides exhibit significant biological effects including analgesic, psychotropic and antimycobacterial (Malinka *et al.*, 2004). The title compound, (I), a partially rigid analogue of the above-mentioned class, was prepared. A search of the Cambridge Structural Database (2006 Release; Allen, 2002; Bruno *et al.*, 2002) did not reveal any crystal structures of compounds containing the pyrido–thiazine–pyrazine structural unit. Therefore, the X-ray crystal structure of (I) with the new pyrido[3,2-*e*]pyrazino[1,2-*b*][1,2]thiazine ring system is reported here.

(I)
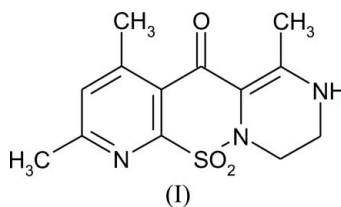
The bond lengths and angles for (I) are within expected ranges (Allen *et al.*, 1987). In the three-ring fused system the aromatic pyridine ring is planar within 0.02 (1) Å, while the partially saturated thiazine and pyrazine rings are significantly distorted from planarity with puckering amplitudes of 0.5715 (11) and 0.4665 (17) Å, respectively (Cremer & Pople, 1975). The thiazine ring exists in a screw-boat conformation with asymmetry parameter $\Delta C_2(S1,N2) = 10.73$ (15)° (Duax & Norton, 1975) and two torsion angles close to 0° [S1—C9—C10—C4 = 3.57 (18)° and N2—C3—C4—C10 = 7.45 (19)°]. The pyrazine ring adopts a sofa conformation [asymmetry parameter $\Delta C_s(C11) = 6.33$ (16)°] resulting from the conjugation of the lone pair at atom N12 with the C10, C4, O4, C3 and C11 π-electron system. The planar sp^2-hybridization of atom N12, with the sum of the bond angles of 359.8° and the N12—C11 bond length of 1.335 (2) Å significantly shorter than N12—C13 of 1.449 (2) Å, suggests conjugation in the planar part of the pyrazine ring. For comparison, in the structurally related 5*H*-2,4-dimethyl-6-phenyl-8,9-dihydro-
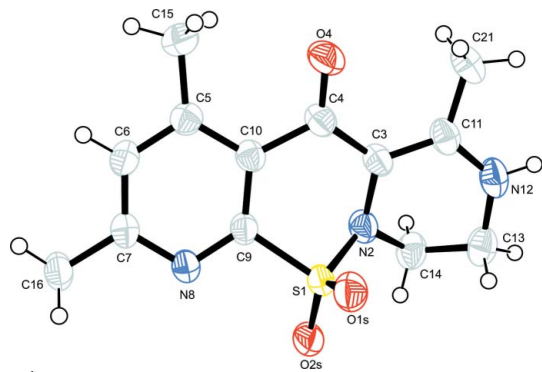
Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.
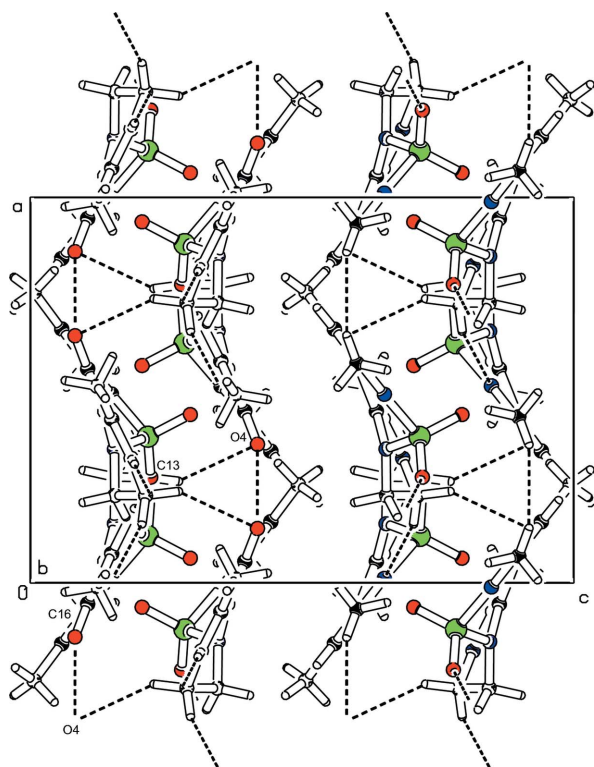

Figure 2
The molecular packing of (I). Dashed lines indicate intermolecular hydrogen bonds.

pyrido[3′,2′:5,6][1,2]thiazino[3,2-c][1,4]oxazin-5-one 11,11-dioxide (Karczmarzyk & Malinka, 2005), the thiazine and oxazine rings both adopt screw-boat conformations.

In the crystal structure, molecules are linked into chains parallel to [010] and [100] *via* intermolecular N—H···O hydrogen bonds and short intermolecular C—H···X (X = N, O) contacts, respectively (Table 1; Spek, 2003). The combination of the [010] and [100] chains generates hydrogen-bonded molecular layers in the *ab* plane (Fig. 2). Additionally, pairs of pyridine rings belonging to inversion-related molecules overlap, with a centroid-to-centroid separation of 3.7913 (11) Å; the shortest intermolecular contact [C6···C10^i = 3.590 (2) Å; symmetry code: (i) $-x, -y, -z$] is characteristic of π–π interactions.

Experimental

Compound (I) was prepared by heating 3-acetyl-4-hydroxypyrido-1,2-thiazine and ethylenediamine in dimethylformamide, according to the method of Malinka *et al.* (2006). Crystals suitable for X-ray structure analysis were grown by slow evaporation of an ethanol solution.

Crystal data

$C_{13}H_{15}N_3O_3S$	$Z = 8$
$M_r = 293.34$	$D_x = 1.494$ Mg m^{-3}
Orthorhombic, $Pbca$	Cu $K\alpha$ radiation
$a = 11.142$ (2) Å	$\mu = 2.33$ mm^{-1}
$b = 14.929$ (3) Å	$T = 293$ (2) K
$c = 15.680$ (3) Å	Prism, colourless
$V = 2608.2$ (9) Å^3	$0.25 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART APEX CCD diffractometer	27602 measured reflections
ω scans	2478 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	2437 reflections with $I > 2\sigma(I)$
$T_{min} = 0.588$, $T_{max} = 0.654$	$R_{int} = 0.017$
	$\theta_{max} = 70.1°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$+ 0.6394P]$
$wR(F^2) = 0.097$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2478 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
227 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Only H-atom coordinates refined	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.00073 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
N12—H121···O2S^i	0.84 (2)	2.13 (2)	2.9545 (18)	165 (2)
C13—H131···O4^{ii}	1.00 (2)	2.56 (2)	3.453 (2)	148.0 (17)
C13—H132···N8^i	1.00 (2)	2.53 (2)	3.479 (2)	160.8 (18)
C16—H163···O4^{iii}	0.92 (2)	2.51 (2)	3.393 (2)	160.1 (18)

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

The H atoms were located in a difference Fourier map and their coordinates were refined with isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C,N), N—H = 0.84 (2), C—H = 0.91 (2)–1.00 (2) Å].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *WinGX* (Farrugia, 1999).

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