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2-Methyl-2*H*-1,2-benzothiazin-4(3*H*)-one 1,1-dioxide

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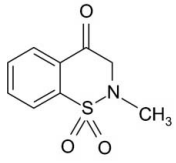
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.124; data-to-parameter ratio = 16.2.

In the title compound, $C_9H_9NO_3S$, the thiazine ring adopts a half-chair conformation. The structure is stabilized by an extensive hydrogen-bonded network involving two intramolecular and three intermolecular interactions.

Related literature

For related literature, see: Bernstein *et al.* (1994); Kwon & Park (1996); Lombardino & Wiseman (1972); Croce *et al.* (1992); Consonni *et al.* (1990); Siddiqui, Ahmad, Khan & Siddiqui (2007); Siddiqui, Ahmad, Khan, Siddiqui & Ahmad (2007); Siddiqui *et al.* (2007*a*,*b*); Siddiqui, Ahmad, Siddiqui, Khan & Parvez (2007); Cremer & Pople (1975); Siddiqui *et al.* (2006).



Experimental

Crystal data

$C_9H_9NO_3S$
M_r = 211.23
Orthorhombic, $P2_12_12_1$
a = 6.778 (5) Å
b = 8.634 (6) Å
c = 15.704 (10) Å
V = 919.0 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 173 (2) K
0.12 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.962, T_{max} = 0.974
6478 measured reflections
2075 independent reflections
1692 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.125
S = 1.09
2075 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (1983), 848 Friedel pairs
Flack parameter: −0.14 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O3^{i}	0.95	2.55	3.467 (5)	164
C8—H8A···O3^{ii}	0.99	2.42	3.240 (5)	140
C9—H9A···O3^{iii}	0.98	2.40	3.226 (5)	141
C8—H8A···O2	0.99	2.58	2.933 (5)	101
C9—H9B···O3	0.98	2.49	2.865 (5)	102

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2541).

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supplementary materials

Acta Cryst. (2007). E**63**, o4585 [**doi:10.1107/S1600536807054736**]

2-Methyl-2*H*-1,2-benzothiazin-4(3*H*)-one 1,1-dioxide

W. A. Siddiqui, S. Ahmad, H. L. Siddiqui, M. I. Tariq and M. Parvez

Comment

The term 'oxicam' describes a relatively new enolic acid class of 4-hydroxy-1,2-benzothiazine carboxamides with anti-inflammatory and analgesic properties as tested by Writhing Syndrome (Kwon & Park, 1996). The first member of this class, piroxicam (Lombardino & Wiseman, 1972), was introduced in the United States in 1982 and it gained immediate acceptance and remained among the top 50 prescription drugs for several years. Continuing our investigations in this area (Siddiqui *et al.*, 2006; Siddiqui, Ahmad, Khan, & Siddiqui, 2007; Siddiqui, Ahmad, Khan, Siddiqui & Ahmad, 2007; Siddiqui *et al.*, 2007*a*,b; Siddiqui, Ahmad, Siddiqui, Khan & Parvez, 2007) we report herein the structure of the title compound, (I).

The heterocyclic thiazine ring in (I) (Fig. 1) adopts a half-chair conformation wherein N1 is displaced by −0.685 (4) Å from the plane defined by the remaining atoms in the ring, with puckering parameters (Cremer & Pople, 1975): Q = 0.526 (1) Å, θ = 50.1 (3)° and φ = 54.6 (5)°. The structure is stabilized by two intramolecular hydrogen bonds C8—H8A···O2 and C9—H9B···O3 that result in graph set patterns S(5) and S(6), respectively (Bernstein *et al.*, 1994); details of hydrogen bonding geometry have been provided in a Table. It is interesting to note that O3 is involved in three rather weak intermolecular interactions of the type C—H···O (Fig. 2).

Experimental

The title compound was synthesized as reported earlier (Roberto *et al.*, 1990; Piero *et al.*, 1992) and was recrystallized from a solution of MeOH at 313 K to obtain colorless crystals.

Refinement

An absolute strucrute was established by the Flack (1983) method using 848 Friedel pairs; Flack parameter, *x*, was 1.15 (15) for the inverted structure which was therefore, rejected to be the one present in the crystal. H-atoms bonded to C-atoms were included in the refinements at geometrically idealized positions with C—$H_{aromatic}$ type = 0.95, CH_2 type = 0.99 and CH_3 type = 0.98 Å and U_{iso} = 1.2 times U_{eq} of the atoms to which they were bonded. The final difference map was free of any chemically significant features.

Figures

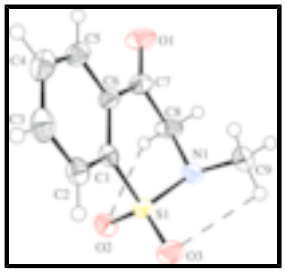

Fig. 1. The molecular structure with displacement ellipsoids plotted at 50% probability level; intramolecular interactions have been indicated by broken lines.

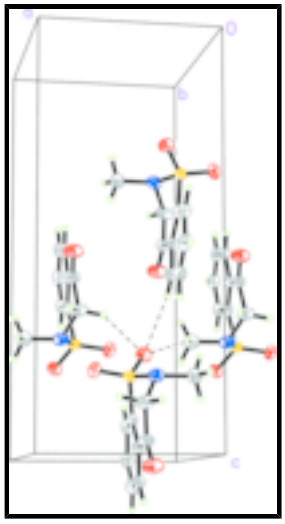

Fig. 2. Part of the crystal structure, showing intermolecular interactions of the type C—H···O; intramolecular interactions have been ignored for clarity.

2-Methyl-2*H*-1,2-benzothiazin-4(3*H*)-one 1,1-dioxide

Crystal data

$C_9H_9NO_3S$	F_{000} = 440
M_r = 211.23	D_x = 1.527 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2075 reflections
a = 6.778 (5) Å	θ = 2.6–27.4º
b = 8.634 (6) Å	μ = 0.33 mm^{-1}
c = 15.704 (10) Å	T = 173 (2) K
V = 919.0 (11) Å^3	Prism, colorless
Z = 4	0.12 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer	2075 independent reflections
Radiation source: fine-focus sealed tube	1692 reflections with $I > 2\ \sigma(I)$
Monochromator: graphite	R_{int} = 0.045
T = 173(2) K	θ_{max} = 27.4º
ω and φ scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	h = −8→8
T_{min} = 0.962, T_{max} = 0.974	k = −11→11
6478 measured reflections	l = −20→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.93P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.125$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.09$	$\Delta\rho_{max} = 0.70$ e Å^{-3}
2075 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}
128 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 848 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.14 (15)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.38859 (14)	0.53500 (10)	0.80195 (6)	0.0257 (2)
O1	0.3254 (5)	0.2461 (3)	1.02285 (17)	0.0434 (8)
O2	0.5943 (4)	0.5091 (3)	0.78864 (16)	0.0334 (6)
O3	0.2839 (4)	0.6302 (3)	0.74272 (17)	0.0349 (7)
N1	0.2779 (5)	0.3677 (3)	0.8062 (2)	0.0316 (7)
C1	0.3568 (5)	0.6086 (4)	0.9056 (2)	0.0242 (8)
C2	0.3501 (6)	0.7667 (4)	0.9181 (2)	0.0303 (9)
H2	0.3551	0.8351	0.8708	0.036*
C3	0.3359 (6)	0.8258 (5)	1.0006 (3)	0.0356 (10)
H3	0.3313	0.9345	1.0097	0.043*
C4	0.3285 (6)	0.7255 (5)	1.0688 (3)	0.0369 (10)
H4	0.3226	0.7658	1.1250	0.044*
C5	0.3296 (5)	0.5657 (4)	1.0563 (2)	0.0306 (9)
H5	0.3203	0.4979	1.1038	0.037*
C6	0.3441 (5)	0.5051 (4)	0.9746 (2)	0.0247 (8)
C7	0.3406 (6)	0.3334 (4)	0.9631 (2)	0.0287 (8)
C8	0.3629 (7)	0.2676 (4)	0.8739 (2)	0.0344 (9)
H8A	0.5049	0.2517	0.8620	0.041*
H8B	0.2977	0.1651	0.8717	0.041*
C9	0.0596 (6)	0.3734 (6)	0.8058 (3)	0.0479 (11)
H9A	0.0069	0.2681	0.8008	0.057*
H9B	0.0145	0.4357	0.7574	0.057*
H9C	0.0127	0.4202	0.8589	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0278 (4)	0.0258 (4)	0.0235 (4)	−0.0017 (4)	0.0001 (4)	0.0021 (4)
O1	0.064 (2)	0.0348 (15)	0.0317 (15)	0.0023 (14)	0.0011 (14)	0.0103 (13)
O2	0.0234 (13)	0.0411 (15)	0.0358 (14)	−0.0007 (12)	0.0056 (12)	−0.0005 (11)
O3	0.0352 (16)	0.0381 (15)	0.0313 (14)	0.0037 (12)	−0.0035 (12)	0.0094 (12)
N1	0.0404 (19)	0.0290 (15)	0.0254 (16)	−0.0083 (13)	0.0017 (16)	−0.0002 (15)
C1	0.0183 (19)	0.0256 (17)	0.0288 (18)	−0.0021 (15)	0.0017 (15)	−0.0007 (14)
C2	0.025 (2)	0.029 (2)	0.037 (2)	−0.0005 (16)	−0.0019 (17)	−0.0006 (16)
C3	0.024 (2)	0.034 (2)	0.049 (3)	0.0015 (16)	−0.0018 (18)	−0.0111 (19)
C4	0.027 (2)	0.047 (3)	0.036 (2)	0.0051 (18)	−0.0013 (17)	−0.0170 (19)
C5	0.028 (2)	0.040 (2)	0.0242 (18)	0.0044 (16)	0.0009 (15)	−0.0044 (15)
C6	0.0243 (19)	0.0281 (19)	0.0218 (16)	0.0013 (13)	−0.0004 (15)	−0.0021 (13)
C7	0.033 (2)	0.0288 (19)	0.0246 (18)	0.0029 (16)	0.0000 (16)	0.0040 (15)
C8	0.055 (3)	0.0204 (17)	0.0274 (18)	0.0010 (19)	0.0067 (19)	0.0033 (14)
C9	0.040 (3)	0.067 (3)	0.037 (2)	−0.027 (2)	−0.003 (2)	0.005 (2)

Geometric parameters (Å, °)

S1—O2	1.428 (3)	C3—H3	0.9500
S1—O3	1.430 (3)	C4—C5	1.393 (6)
S1—N1	1.629 (3)	C4—H4	0.9500
S1—C1	1.760 (4)	C5—C6	1.390 (5)
O1—C7	1.207 (4)	C5—H5	0.9500
N1—C9	1.481 (5)	C6—C7	1.494 (5)
N1—C8	1.487 (5)	C7—C8	1.519 (5)
C1—C2	1.380 (5)	C8—H8A	0.9900
C1—C6	1.407 (5)	C8—H8B	0.9900
C2—C3	1.395 (5)	C9—H9A	0.9800
C2—H2	0.9500	C9—H9B	0.9800
C3—C4	1.379 (6)	C9—H9C	0.9800
O2—S1—O3	118.66 (16)	C6—C5—C4	120.2 (4)
O2—S1—N1	108.48 (17)	C6—C5—H5	119.9
O3—S1—N1	107.92 (18)	C4—C5—H5	119.9
O2—S1—C1	108.16 (16)	C5—C6—C1	118.5 (3)
O3—S1—C1	109.45 (17)	C5—C6—C7	118.9 (3)
N1—S1—C1	103.05 (16)	C1—C6—C7	122.6 (3)
C9—N1—C8	114.2 (3)	O1—C7—C6	121.8 (3)
C9—N1—S1	115.5 (3)	O1—C7—C8	119.4 (3)
C8—N1—S1	111.5 (3)	C6—C7—C8	118.7 (3)
C2—C1—C6	121.1 (3)	N1—C8—C7	113.8 (3)
C2—C1—S1	119.6 (3)	N1—C8—H8A	108.8
C6—C1—S1	119.3 (3)	C7—C8—H8A	108.8
C1—C2—C3	119.8 (4)	N1—C8—H8B	108.8
C1—C2—H2	120.1	C7—C8—H8B	108.8
C3—C2—H2	120.1	H8A—C8—H8B	107.7

C4—C3—C2	119.6 (4)	N1—C9—H9A	109.5
C4—C3—H3	120.2	N1—C9—H9B	109.5
C2—C3—H3	120.2	H9A—C9—H9B	109.5
C3—C4—C5	120.9 (4)	N1—C9—H9C	109.5
C3—C4—H4	119.6	H9A—C9—H9C	109.5
C5—C4—H4	119.6	H9B—C9—H9C	109.5
O2—S1—N1—C9	−170.5 (3)	C3—C4—C5—C6	2.0 (6)
O3—S1—N1—C9	−40.7 (3)	C4—C5—C6—C1	−0.3 (5)
C1—S1—N1—C9	75.0 (3)	C4—C5—C6—C7	−178.7 (3)
O2—S1—N1—C8	57.0 (3)	C2—C1—C6—C5	−1.6 (5)
O3—S1—N1—C8	−173.3 (2)	S1—C1—C6—C5	176.6 (3)
C1—S1—N1—C8	−57.5 (3)	C2—C1—C6—C7	176.8 (3)
O2—S1—C1—C2	93.8 (3)	S1—C1—C6—C7	−5.0 (5)
O3—S1—C1—C2	−36.9 (3)	C5—C6—C7—O1	0.0 (6)
N1—S1—C1—C2	−151.5 (3)	C1—C6—C7—O1	−178.3 (4)
O2—S1—C1—C6	−84.5 (3)	C5—C6—C7—C8	−178.2 (3)
O3—S1—C1—C6	144.9 (3)	C1—C6—C7—C8	3.4 (6)
N1—S1—C1—C6	30.3 (3)	C9—N1—C8—C7	−70.9 (5)
C6—C1—C2—C3	1.7 (6)	S1—N1—C8—C7	62.3 (4)
S1—C1—C2—C3	−176.5 (3)	O1—C7—C8—N1	149.6 (4)
C1—C2—C3—C4	−0.1 (6)	C6—C7—C8—N1	−32.2 (5)
C2—C3—C4—C5	−1.8 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O3^{i}	0.95	2.55	3.467 (5)	164
C8—H8A···O3^{ii}	0.99	2.42	3.240 (5)	140
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Symmetry codes: (i) −*x*+1/2, −*y*+1, *z*+1/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*, *y*−1/2, −*z*+3/2.

Fig. 1

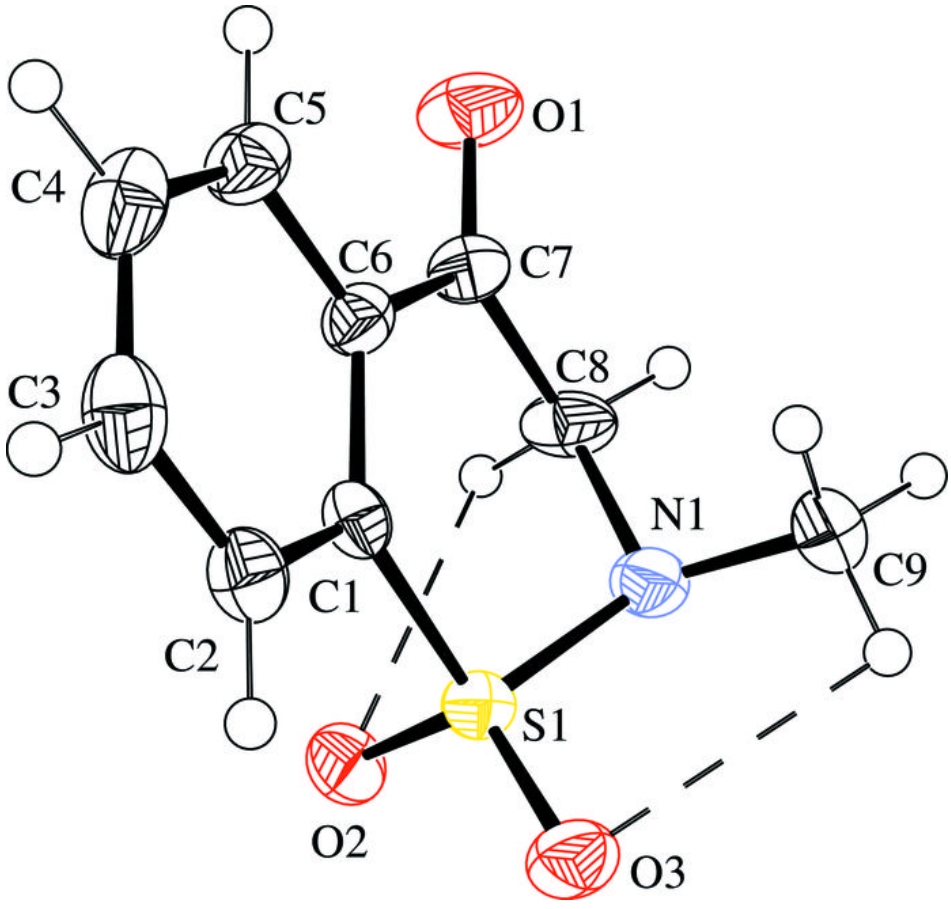

Fig. 2