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2,3-Dihydro-1λ^6,2-benzothiazine-1,1,4-trione

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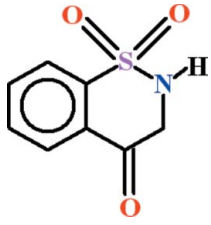
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 16.7.

In the title compound, $C_8H_7NO_3S$, the benzene ring is oriented at a dihedral angle of 69.25 (7)° to the S and O atoms of the sulfonyl group. The heterocyclic ring approximates to an envelope, with the N atom in the flap position. In the crystal, molecules are linked by N—H···O$_c$ (c = carbonyl) hydrogen bonds, forming $C(5)$ chains along [001]. Two $R_2^2(10)$ loops arise from pairs of C—H···O hydrogen bonds and a weak aromatic π–π stacking interaction [centroid–centorid separation = 3.8404 (11) Å] also occurs.

Related literature

For chemical background and related structures, see: Siddiqui *et al.* (2007, 2008). For graph-set notation, see: Bernstein *et al.* (1995). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_8H_7NO_3S$
M_r = 197.21
Monoclinic, $P2_1/c$
a = 8.4950 (4) Å
b = 13.7560 (5) Å
c = 7.6677 (3) Å
β = 113.214 (1)°
V = 823.48 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 296 K
0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.915, T_{max} = 0.938
7644 measured reflections
2017 independent reflections
1692 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.04
2017 reflections
121 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.80 (2)	2.34 (2)	3.028 (2)	144 (2)
C2—H2···O2^{ii}	0.93	2.58	3.443 (2)	154
C8—H8A···O2^{iii}	0.97	2.48	3.273 (2)	139

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan. The authors also acknowledge the technical support provided by Syed Muhammad Hussain Rizvi of Bana International, Karachi, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6713).

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supplementary materials

Acta Cryst. (2012). E**68**, o1306 [doi:10.1107/S1600536812013827]

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Comment

The title compound (I), (Fig. 1) has been synthesized as a pre-cursor. The crystal structures of 2-methyl-2*H*-1,2-benzothiazin-4(3*H*)-one 1,1-dioxide (Siddiqui *et al.*, 2007) has been published which is related to (I).

The dihedral angle between the benzene ring and S1/O1/O2 is 69.25 (7)°. The heterocyclic ring C (C1/C6—C8/N1/S1) is twisted with puckering parameters (Cremer & Pople, 1975) Q = 0.5149 (15) Å, θ = 62.25 (19)° and π = 43.7 (2)°. The molecules are linked in the form of C(5) chains (Bernstein *et al.*, 1995) along the *c*-axis due to H-bondings between amide and carbonyl O-atoms (Table 1, Fig. 2). The neighbouring polyneric chains are interlinked due to C—H···O bonds with two $R_2^2(10)$ ring motifs (Table 1, Fig. 2), where CH are of benzene and methylene groups and the same O-atom is of sulfonyl group. There exist π–π interaction between the centroids of the benzene rings at a distance of 3.8404 (11)°.

Experimental

A mixture of (0.5 g, 1.96 mmol) 4-hydroxy-3-carbomethoxy-2*H*-1,2- benzothiazine 1,1-dioxide (Siddiqui *et al.*, 2008) and anhydrous lithium iodide (1.3 g, 9.79 mmol) was subjected to reflux in dimethyl sulphoxide (15 ml) for five h. The dark brown reaction mixture was then poured into crushed ice (100 g). The formed dark yellow precipitates were filtered, washed with cold water (3×25 ml) and dried to get (0.36 g, 1.8 mmol, 92%) of the crude product. Recrystallization of the crude product in ethyl acetate yielded light yellow needles of (I) (m.p. 415–417 K).

IR (KBr) (ν_{max}, cm^{-1}): 3269 (NH), 3078 (Ar. CH), 1683 (C=O), 1577 (NH, def.), 1419 (CH, def.) 1330, 1176 (SO_2).

Refinement

The coordinates of H-atom of amide were refined. The H-atoms of aryl and methylene groups were positioned geometrically at C—H = 0.93 and C—H = 0.97 Å, respectively and included in the refinement as riding with U_{iso}(H) = xU_{eq}(C, N), where x = 1.2 for all H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

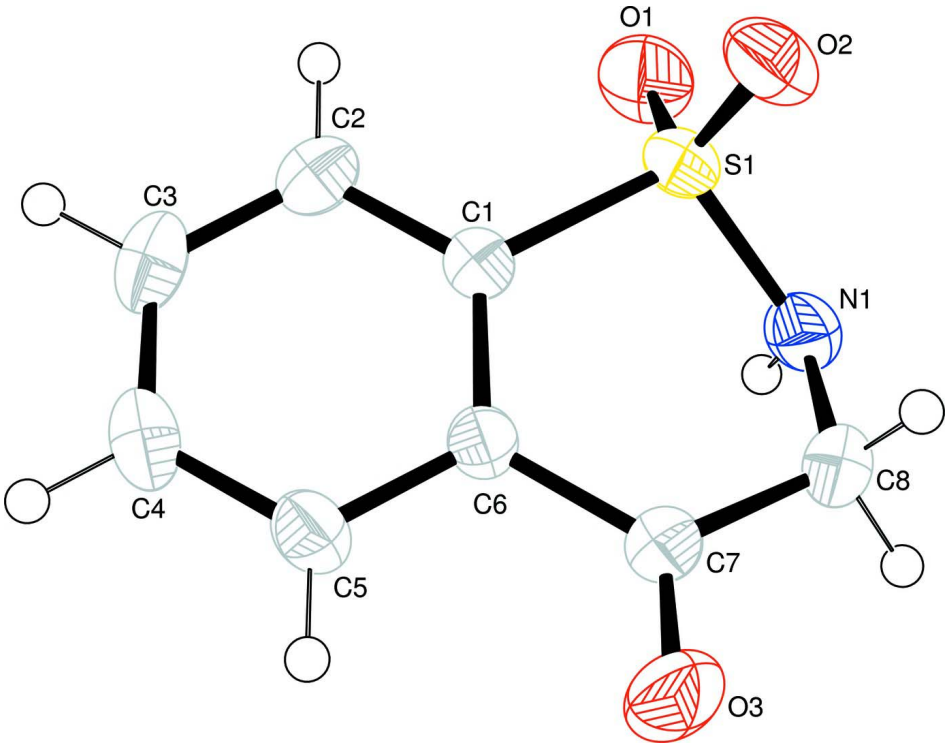

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level.

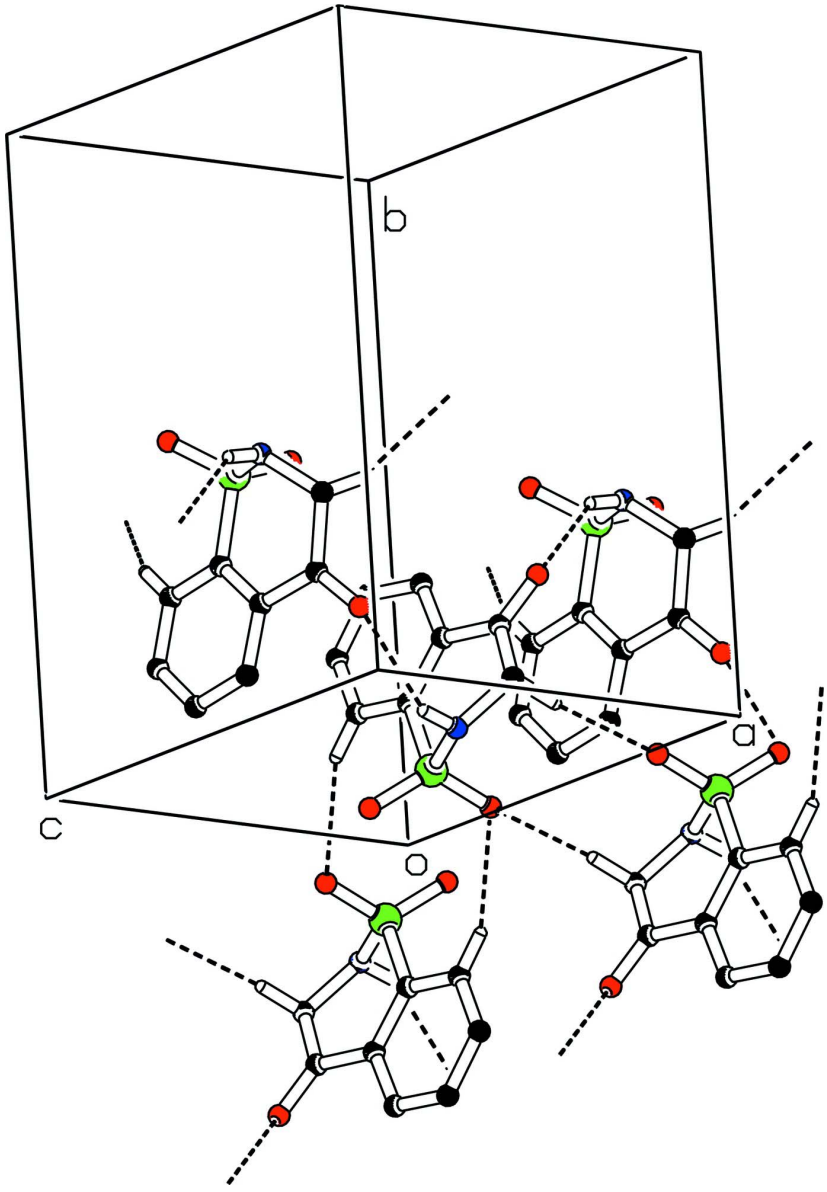

Figure 2

Partial packing diagram for (I) showing C(5) chains extending along [001]. The chains are interlinked with $R_2^2(10)$ rings due to C—H···O bondings. The H-atoms not involved in H-bondings are omitted for clarity.

2,3-Dihydro-1λ^6,2-benzothiazine-1,1,4-trione

Crystal data

$C_8H_7NO_3S$
M_r = 197.21
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.4950 (4) Å
b = 13.7560 (5) Å
c = 7.6677 (3) Å
β = 113.214 (1)°
V = 823.48 (6) Å^3
Z = 4
F(000) = 408
D_x = 1.591 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1692 reflections
θ = 2.6–28.3°
μ = 0.36 mm^{-1}

T = 296 K
Needle, light yellow

0.35 × 0.15 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.915, T_{max} = 0.938

7644 measured reflections
2017 independent reflections
1692 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.3°, θ_{min} = 2.6°
h = −11→8
k = −16→18
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.04
2017 reflections
121 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.3408P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.28018 (5)	0.04479 (3)	0.18815 (6)	0.0352 (2)
O1	0.21934 (19)	−0.00868 (10)	0.3084 (2)	0.0539 (5)
O2	0.25497 (18)	0.00747 (10)	0.00536 (19)	0.0499 (5)
O3	0.55335 (18)	0.29145 (11)	0.1183 (2)	0.0532 (5)
N1	0.4843 (2)	0.06118 (11)	0.3034 (2)	0.0405 (5)
C1	0.1985 (2)	0.16427 (11)	0.1625 (2)	0.0297 (4)
C2	0.0353 (2)	0.18011 (14)	0.1537 (3)	0.0392 (5)
C3	−0.0234 (2)	0.27484 (15)	0.1446 (3)	0.0447 (6)
C4	0.0788 (2)	0.35176 (14)	0.1427 (3)	0.0430 (6)
C5	0.2406 (2)	0.33587 (13)	0.1476 (3)	0.0363 (5)
C6	0.30357 (19)	0.24184 (11)	0.1576 (2)	0.0285 (4)
C7	0.4782 (2)	0.22672 (12)	0.1609 (2)	0.0331 (5)
C8	0.5621 (2)	0.12808 (13)	0.2121 (3)	0.0429 (6)
H1	0.502 (3)	0.0766 (17)	0.410 (3)	0.0486*

H2	−0.03448	0.12804	0.15398	0.0471*
H3	−0.13293	0.28633	0.13970	0.0537*
H4	0.03854	0.41499	0.13799	0.0516*
H5	0.30824	0.38842	0.14422	0.0436*
H8A	0.56037	0.09765	0.09725	0.0514*
H8B	0.68115	0.13745	0.29619	0.0514*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0387 (3)	0.0236 (2)	0.0436 (3)	−0.0023 (2)	0.0165 (2)	−0.0012 (2)
O1	0.0633 (10)	0.0355 (7)	0.0697 (10)	−0.0056 (6)	0.0335 (8)	0.0110 (6)
O2	0.0576 (9)	0.0383 (7)	0.0513 (8)	−0.0023 (6)	0.0188 (7)	−0.0148 (6)
O3	0.0408 (8)	0.0479 (8)	0.0783 (10)	−0.0039 (6)	0.0314 (7)	0.0131 (7)
N1	0.0384 (8)	0.0292 (8)	0.0486 (8)	0.0044 (6)	0.0116 (7)	0.0025 (6)
C1	0.0308 (8)	0.0278 (8)	0.0307 (7)	0.0003 (6)	0.0123 (6)	−0.0008 (6)
C2	0.0305 (9)	0.0446 (10)	0.0442 (9)	−0.0037 (7)	0.0164 (7)	0.0001 (8)
C3	0.0320 (9)	0.0579 (12)	0.0475 (10)	0.0120 (8)	0.0192 (8)	0.0034 (9)
C4	0.0462 (11)	0.0384 (10)	0.0452 (10)	0.0154 (8)	0.0189 (8)	0.0017 (8)
C5	0.0425 (10)	0.0273 (8)	0.0409 (9)	0.0022 (7)	0.0184 (7)	0.0013 (7)
C6	0.0295 (8)	0.0273 (8)	0.0299 (7)	0.0012 (6)	0.0129 (6)	0.0006 (6)
C7	0.0309 (8)	0.0327 (9)	0.0372 (8)	−0.0018 (7)	0.0152 (7)	−0.0016 (7)
C8	0.0321 (9)	0.0371 (10)	0.0625 (12)	0.0031 (7)	0.0219 (8)	−0.0036 (8)

Geometric parameters (Å, º)

S1—O1	1.4262 (16)	C4—C5	1.378 (3)
S1—O2	1.4273 (14)	C5—C6	1.390 (2)
S1—N1	1.6219 (18)	C6—C7	1.488 (2)
S1—C1	1.7646 (16)	C7—C8	1.511 (2)
O3—C7	1.213 (2)	C2—H2	0.9300
N1—C8	1.462 (3)	C3—H3	0.9300
N1—H1	0.80 (2)	C4—H4	0.9300
C1—C2	1.379 (3)	C5—H5	0.9300
C1—C6	1.401 (2)	C8—H8A	0.9700
C2—C3	1.387 (3)	C8—H8B	0.9700
C3—C4	1.372 (3)		
O1—S1—O2	119.80 (9)	C1—C6—C7	122.30 (14)
O1—S1—N1	107.55 (9)	O3—C7—C6	121.31 (16)
O1—S1—C1	109.00 (9)	O3—C7—C8	119.00 (17)
O2—S1—N1	107.42 (9)	C6—C7—C8	119.66 (15)
O2—S1—C1	108.99 (8)	N1—C8—C7	115.70 (16)
N1—S1—C1	102.72 (8)	C1—C2—H2	121.00
S1—N1—C8	114.49 (12)	C3—C2—H2	120.00
C8—N1—H1	112.6 (18)	C2—C3—H3	120.00
S1—N1—H1	108.9 (19)	C4—C3—H3	120.00
C2—C1—C6	121.15 (15)	C3—C4—H4	120.00
S1—C1—C6	119.06 (13)	C5—C4—H4	120.00
S1—C1—C2	119.76 (13)	C4—C5—H5	120.00

C1—C2—C3	119.05 (17)	C6—C5—H5	120.00
C2—C3—C4	120.56 (18)	N1—C8—H8A	108.00
C3—C4—C5	120.39 (18)	N1—C8—H8B	108.00
C4—C5—C6	120.48 (17)	C7—C8—H8A	108.00
C5—C6—C7	119.36 (15)	C7—C8—H8B	108.00
C1—C6—C5	118.34 (16)	H8A—C8—H8B	107.00
O1—S1—N1—C8	−170.75 (13)	C2—C1—C6—C7	−178.08 (15)
O2—S1—N1—C8	59.04 (14)	S1—C1—C6—C5	−176.60 (13)
C1—S1—N1—C8	−55.82 (14)	C1—C2—C3—C4	0.6 (3)
O1—S1—C1—C2	−36.22 (16)	C2—C3—C4—C5	0.8 (3)
O2—S1—C1—C2	96.19 (16)	C3—C4—C5—C6	−1.0 (3)
N1—S1—C1—C2	−150.09 (14)	C4—C5—C6—C1	0.0 (3)
O1—S1—C1—C6	141.74 (12)	C4—C5—C6—C7	179.44 (17)
O2—S1—C1—C6	−85.85 (14)	C1—C6—C7—C8	−13.2 (2)
N1—S1—C1—C6	27.87 (14)	C5—C6—C7—O3	−14.6 (2)
S1—N1—C8—C7	53.31 (19)	C1—C6—C7—O3	164.83 (15)
S1—C1—C2—C3	176.30 (15)	C5—C6—C7—C8	167.41 (16)
C6—C1—C2—C3	−1.6 (3)	O3—C7—C8—N1	166.31 (15)
S1—C1—C6—C7	3.99 (19)	C6—C7—C8—N1	−15.6 (2)
C2—C1—C6—C5	1.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.80 (2)	2.34 (2)	3.028 (2)	144 (2)
C2—H2···O2^{ii}	0.93	2.58	3.443 (2)	154
C8—H8*A*···O2^{iii}	0.97	2.48	3.273 (2)	139

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*, −*y*, −*z*; (iii) −*x*+1, −*y*, −*z*.