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

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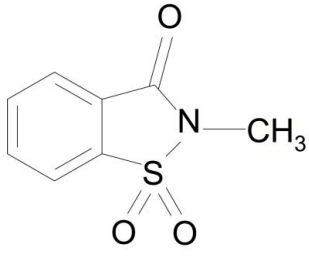
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 13.8.

All atoms of the title molecule, $C_8H_7NO_3S$, except the two oxide O atoms and two H atoms of the methyl group, lie on a crystallographic mirror plane. The crystal structure is stabilized by weak inter- and intramolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Hu *et al.* (2004); Kap-Sun & Nicholas (1998); Liang *et al.* (2006); Masashi *et al.* (1999); Nagasawa *et al.* (1995); Siddiqui *et al.* (2006, 2007*a*,*b*,*c*); Siddiqui, Ahmad, Khan & Siddiqui (2007); Siddiqui, Ahmad, Khan, Siddiqui & Ahmad (2007); Siddiqui, Ahmad, Khan, Siddiqui & Parvez (2007).



Experimental

Crystal data

$C_8H_7NO_3S$
M_r = 197.21
Monoclinic, $P2_1/m$
a = 7.463 (7) Å
b = 6.761 (6) Å
c = 8.748 (8) Å
β = 103.78 (3)°
V = 428.7 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 173 (2) K
0.12 × 0.08 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.960, T_{max} = 0.976
1724 measured reflections
1045 independent reflections
889 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.106
S = 1.03
1045 reflections
76 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1	0.96	2.49	2.869 (4)	104
C2—H2···O1^{i}	0.95	2.29	3.227 (4)	169
C8—H8B···O2^{ii}	0.96	2.49	3.358 (3)	151

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *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, 2008); 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: LH2597).

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supplementary materials

Acta Cryst. (2008). E**64**, o724 [**doi:10.1107/S1600536808004637**]

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

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Comment

Benzisothiazolone-1,1-dioxide is part of a class of heterocycles which has been investigated in pharmaceutical research (Kap-Sun & Nicholas, 1998). 1,2-benzisothiazole-3-one 1,1-dioxide (saccharin) has been widely incorporated into a variety of biologically active compounds. It has been identified as an important molecular component in various classes of 5-HTla antagonists, analgesics and human mast cell tryptase inhibitors (Liang *et al.*, 2006). In particular, N-substituted derivatives, e.g with *N*-hydroxy and *N*-alkyl substituents, have shown important biological activites (Nagasawa *et al.*, 1995). Among *N*-alkyl derivatives, various synthetic routes have been reported for the synthesis of the title compound involving ionic liquids and free radical mechanisms (Hu *et al.*, 2004; Masashi *et al.*, 1999). In continuation of our research on the synthesis of 1,2-benzothiazine 1,1-dioxide derivatives, we have in addtion, embarked on the synthesis of benzisothiazole derivatives (Siddiqui *et al.*, 2006; Siddiqui *et al.*, 2007*a*,b,c; Siddiqui, Ahmad, Khan & Siddiqui, 2007; Siddiqui, Ahmad, Khan, Siddiqui & Ahmad, 2007; Siddiqui, Ahmad, Khan, Siddiqui & Parvez, 2007). Herein, we report the synthesis and crystal structure of the title compound, (I).

With the exception atoms O2 and H8B, all atoms of the molecule of (I) (Fig. 1) lie on a crystallographic mirror plane. The benzisothiazole moiety is exactly planar. The molecular dimensions are in accord with the corresponding dimensions reported in similar structures (Siddiqui *et al.*, 2007*a*-c; Siddiqui, Ahmad, Khan, Siddiqui & Parvez, 2007). The structure is stabilized by one intramolecular and two intermolecular interactions of the type C—H···O (details are in Table).

Experimental

Saccharin (2.0 g, 11.0 mmol.) was added to a solution of sodium hydroxide (0.875 g, 22.0 mmol.) in distilled water (25 ml) under constant stirring to give a transparent solution. A solution of dimethylsulfate (2.08 ml, 22.0 mmol.) in methanol (10.0 ml) was then added dropwise over 2 minutes. Precipitates started appearing within 5 minutes and stirring was continued for 20 min. at room temperature. The precipitates were filtered, washed with cold water and dried (343 K) to get 1.75 g of (I) (8.9 mmol. 81%). Recrystallization Solvent: $CHCl_3$. The solution was subjected to slow evaporation at 313 K to obtain colourless crystals.

Refinement

H-atoms bonded were included in the refinements at geometrically idealized positions with aromatic and methyl C—H distances 0.95 and 0.96 Å, respectively, 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

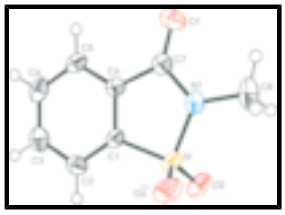

Fig. 1. *ORTEPII* (Johnson, 1976) drawing of (I) with displacement ellipsoids plotted at 50% probability level. Symmetry code: (iii) $x, -y + 1/2, z$.

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

Crystal data

$C_8H_7NO_3S$	$F_{000} = 204$
$M_r = 197.21$	$D_x = 1.528$ Mg m^{-3}
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yb	Cell parameters from 1724 reflections
$a = 7.463$ (7) Å	$\theta = 3.2$–27.4°
$b = 6.761$ (6) Å	$\mu = 0.35$ mm^{-1}
$c = 8.748$ (8) Å	$T = 173$ (2) K
$\beta = 103.78$ (3)°	Prism, colorless
$V = 428.7$ (7) Å^3	0.12 × 0.08 × 0.07 mm
$Z = 2$	

Data collection

Nonius KappaCCD diffractometer	1045 independent reflections
Radiation source: fine-focus sealed tube	889 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
$T = 173$(2) K	$\theta_{max} = 27.4^\circ$
ω and φ scans	$\theta_{min} = 3.2^\circ$
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	$h = -9 \rightarrow 9$
$T_{min} = 0.960$, $T_{max} = 0.976$	$k = -8 \rightarrow 8$
1724 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.2966P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$

1045 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
76 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.68037 (10)	0.2500	0.26611 (7)	0.0314 (2)
O1	0.2425 (3)	0.2500	0.4038 (3)	0.0416 (5)
O2	0.7324 (2)	0.0698 (2)	0.20266 (16)	0.0445 (4)
N1	0.4534 (3)	0.2500	0.2520 (3)	0.0314 (5)
C1	0.7314 (4)	0.2500	0.4722 (3)	0.0254 (5)
C2	0.9043 (4)	0.2500	0.5750 (3)	0.0337 (6)
H2	1.0141	0.2500	0.5381	0.040*
C3	0.9090 (4)	0.2500	0.7337 (3)	0.0403 (7)
H3	1.0249	0.2500	0.8079	0.048*
C4	0.7486 (4)	0.2500	0.7873 (3)	0.0379 (7)
H4	0.7566	0.2500	0.8974	0.045*
C5	0.5767 (4)	0.2500	0.6832 (3)	0.0308 (6)
H5	0.4670	0.2500	0.7203	0.037*
C6	0.5690 (3)	0.2500	0.5235 (3)	0.0251 (5)
C7	0.4012 (4)	0.2500	0.3931 (3)	0.0289 (6)
C8	0.3218 (5)	0.2500	0.0986 (3)	0.0455 (8)
H8A	0.1982	0.2500	0.1130	0.055*
H8B	0.3405	0.1341	0.0410	0.055*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0415 (4)	0.0319 (4)	0.0237 (3)	0.000	0.0133 (3)	0.000
O1	0.0284 (10)	0.0449 (13)	0.0521 (13)	0.000	0.0107 (9)	0.000
O2	0.0587 (10)	0.0435 (9)	0.0369 (8)	0.0058 (7)	0.0223 (7)	−0.0101 (7)
N1	0.0355 (12)	0.0301 (12)	0.0266 (11)	0.000	0.0031 (9)	0.000
C1	0.0319 (13)	0.0222 (12)	0.0237 (12)	0.000	0.0099 (10)	0.000
C2	0.0276 (13)	0.0367 (16)	0.0371 (14)	0.000	0.0085 (11)	0.000
C3	0.0402 (16)	0.0417 (17)	0.0340 (15)	0.000	−0.0010 (12)	0.000
C4	0.0553 (18)	0.0343 (16)	0.0238 (13)	0.000	0.0087 (12)	0.000
C5	0.0392 (15)	0.0257 (13)	0.0324 (14)	0.000	0.0181 (12)	0.000
C6	0.0280 (12)	0.0189 (12)	0.0297 (13)	0.000	0.0093 (10)	0.000
C7	0.0330 (14)	0.0221 (13)	0.0320 (13)	0.000	0.0086 (11)	0.000

C8	0.0551 (19)	0.0452 (19)	0.0284 (15)	0.000	−0.0053 (13)	0.000

Geometric parameters (Å, °)

S1—O2^{i}	1.430 (2)	C2—H2	0.9500
S1—O2	1.430 (2)	C3—C4	1.386 (4)
S1—N1	1.668 (3)	C3—H3	0.9500
S1—C1	1.752 (3)	C4—C5	1.385 (4)
O1—C7	1.211 (3)	C4—H4	0.9500
N1—C7	1.380 (4)	C5—C6	1.384 (4)
N1—C8	1.462 (4)	C5—H5	0.9500
C1—C2	1.386 (4)	C6—C7	1.479 (4)
C1—C6	1.389 (4)	C8—H8A	0.9600
C2—C3	1.380 (4)	C8—H8B	0.9600
O2^{i}—S1—O2	116.79 (14)	C4—C3—H3	119.2
O2^{i}—S1—N1	109.63 (8)	C5—C4—C3	121.1 (3)
O2—S1—N1	109.63 (8)	C5—C4—H4	119.5
O2^{i}—S1—C1	112.76 (8)	C3—C4—H4	119.5
O2—S1—C1	112.76 (8)	C6—C5—C4	118.2 (2)
N1—S1—C1	92.54 (12)	C6—C5—H5	120.9
C7—N1—C8	123.3 (2)	C4—C5—H5	120.9
C7—N1—S1	115.6 (2)	C5—C6—C1	119.7 (2)
C8—N1—S1	121.1 (2)	C5—C6—C7	127.0 (2)
C2—C1—C6	122.7 (2)	C1—C6—C7	113.2 (2)
C2—C1—S1	127.5 (2)	O1—C7—N1	124.0 (3)
C6—C1—S1	109.9 (2)	O1—C7—C6	127.2 (3)
C3—C2—C1	116.7 (3)	N1—C7—C6	108.8 (2)
C3—C2—H2	121.6	N1—C8—H8A	109.6
C1—C2—H2	121.6	N1—C8—H8B	109.4
C2—C3—C4	121.6 (3)	H8A—C8—H8B	109.5
C2—C3—H3	119.2		
O2^{i}—S1—N1—C7	−115.28 (8)	C3—C4—C5—C6	0.000 (1)
O2—S1—N1—C7	115.28 (8)	C4—C5—C6—C1	0.0
C1—S1—N1—C7	0.0	C4—C5—C6—C7	180.0
O2^{i}—S1—N1—C8	64.72 (8)	C2—C1—C6—C5	0.0
O2—S1—N1—C8	−64.72 (8)	S1—C1—C6—C5	180.0
C1—S1—N1—C8	180.0	C2—C1—C6—C7	180.0
O2^{i}—S1—C1—C2	−67.46 (9)	S1—C1—C6—C7	0.0
O2—S1—C1—C2	67.46 (9)	C8—N1—C7—O1	0.0
N1—S1—C1—C2	180.0	S1—N1—C7—O1	180.0
O2^{i}—S1—C1—C6	112.54 (9)	C8—N1—C7—C6	180.0
O2—S1—C1—C6	−112.54 (9)	S1—N1—C7—C6	0.0
N1—S1—C1—C6	0.0	C5—C6—C7—O1	0.0
C6—C1—C2—C3	0.0	C1—C6—C7—O1	180.0
S1—C1—C2—C3	180.0	C5—C6—C7—N1	180.0
C1—C2—C3—C4	0.000 (1)	C1—C6—C7—N1	0.0
C2—C3—C4—C5	0.000 (1)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···O1	0.96	2.49	2.869 (4)	104
C2—H2···O1^{ii}	0.95	2.29	3.227 (4)	169
C8—H8B···O2^{iii}	0.96	2.49	3.358 (3)	151

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

Fig. 1