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2-Chloromethyl-1,2-benzisothiazole-1,1,3(2*H*)-trione

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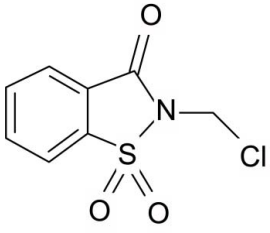
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.102; data-to-parameter ratio = 17.0.

The molecule of the title compound, $C_8H_6ClNO_3S$, contains a planar benzisothiazole ring system. The crystal structure is stabilized by C—H···O intermolecular interactions, with C···O distances of 3.222 (3) and 3.288 (3) Å.

Related literature

For related literature, see: Groutas *et al.* (1993, 1996); Güzel & Salman (2006); Siddiqui *et al.* (2007); Subramanyam *et al.* (1994).



Experimental

Crystal data

$C_8H_6ClNO_3S$
M_r = 231.65
Monoclinic, $P2_1/n$
a = 9.208 (5) Å
b = 8.643 (4) Å
c = 12.458 (6) Å
β = 107.46 (2)°
V = 945.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 173 (2) K
0.16 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.910, T_{max} = 0.965
4086 measured reflections
2161 independent reflections
1671 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.02
2161 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.95	2.52	3.222 (3)	131
C8—H8B···O3^{ii}	0.99	2.33	3.288 (3)	163

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

Data collection: *COLLECT* (Nonius, 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: GG2045).

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supplementary materials

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2-Chloromethyl-1,2-benzisothiazole-1,1,3(2*H*)-trione

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

Comment

Saccharin derivatives have been reported as important biological agents in the treatment of a wide range of diseases for many years (Subramanyam *et al.*, 1994; Groutas *et al.*, 1996; Güzel & Salman, 2006). Chloromethylsaccharin being one of the saccharin derivatives available has already been tested for its activity as human leucocyte elastase (HLE) inhibitor (Groutas *et al.*, 1993). In our recent work we have reported the title compound (I) as an important intermediate in the synthesis of 1,2-benzothiazine 1,1-dioxide derivatives belonging to the oxicam class of non-steroidal anti-inflammatory drugs (NSAIDs) (Siddiqui *et al.*, 2007). Herein, we report the structure of the title compound (I).

In the structure (Fig. 1) the benzisothiazole ring system is essentially planar; the maximum deviation of any atom from the mean plane through S1/O1/N1/C1–C7 being 0.029 (1) Å for S1 and C7 atoms. The molecules are linked *via* weak C—H···O type hydrogen bonds, thus stabilizing the crystal structure (Fig. 2).

Experimental

Crystals of the title compound (I) were grown by slow evaporation from a solution of $CHCl_3$ at 313 K.

Refinement

Carbon-bound H atoms were included in the refinement at geometrically idealized positions, with C—H = 0.95 and 0.99 Å and U_{iso}(H) = 1.2U_{eq}(carrier atom). The final difference map was free of any chemically significant features.

Figures

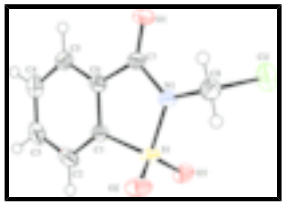

Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound (I), with displacement ellipsoids drawn at the 50% probability level.

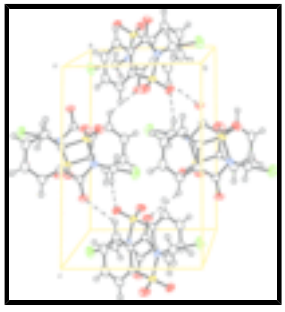

Fig. 2. Unit cell packing of the title compound (I), showing the C—H···O interactions as dashed lines.

2-Chloromethyl-1,2-benzisothiazole-1,1,3(2*H*)-trione

Crystal data

$C_8H_6ClNO_3S$	F_{000} = 472
M_r = 231.65	D_x = 1.627 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4086 reflections
a = 9.208 (5) Å	θ = 3.3–27.5º
b = 8.643 (4) Å	μ = 0.60 mm^{-1}
c = 12.458 (6) Å	T = 173 (2) K
β = 107.46 (2)º	Prism, colourless
V = 945.8 (8) Å^3	0.16 × 0.10 × 0.06 mm
Z = 4	

Data collection

Nonius KappaCCD area-detector diffractometer	2161 independent reflections
Radiation source: fine-focus sealed tube	1671 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 173(2) K	θ_{max} = 27.5º
ω and φ scans	θ_{min} = 3.3º
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	h = −11→11
T_{min} = 0.910, T_{max} = 0.965	k = −11→11
4086 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.102	$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.6083P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2161 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
127 parameters	$\Delta\rho_{min}$ = −0.40 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}
Cl1	0.13760 (9)	0.96958 (7)	−0.09257 (6)	0.0450 (2)
S1	0.21025 (6)	0.61766 (6)	0.09596 (4)	0.02421 (16)
O1	0.17085 (19)	0.58867 (19)	−0.21198 (12)	0.0307 (4)
O2	0.07883 (19)	0.5821 (2)	0.13012 (13)	0.0349 (4)
O3	0.31276 (19)	0.73035 (19)	0.16087 (12)	0.0311 (4)
N1	0.1551 (2)	0.6681 (2)	−0.03988 (14)	0.0243 (4)
C1	0.3062 (3)	0.4538 (2)	0.07062 (17)	0.0236 (5)
C2	0.3863 (3)	0.3462 (3)	0.14818 (19)	0.0320 (6)
H2	0.3917	0.3532	0.2254	0.038*
C3	0.4585 (3)	0.2278 (3)	0.1091 (2)	0.0359 (6)
H3	0.5138	0.1517	0.1602	0.043*
C4	0.4512 (3)	0.2185 (3)	−0.0042 (2)	0.0345 (6)
H4	0.5030	0.1372	−0.0286	0.041*
C5	0.3699 (3)	0.3257 (3)	−0.08149 (19)	0.0280 (5)
H5	0.3646	0.3186	−0.1587	0.034*
C6	0.2963 (3)	0.4440 (2)	−0.04326 (17)	0.0226 (5)
C7	0.2032 (3)	0.5687 (3)	−0.11165 (17)	0.0234 (5)
C8	0.0460 (3)	0.7888 (3)	−0.08138 (19)	0.0296 (5)
H8A	−0.0169	0.8017	−0.0300	0.036*
H8B	−0.0224	0.7593	−0.1563	0.036*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0490 (5)	0.0296 (3)	0.0581 (4)	0.0029 (3)	0.0184 (3)	0.0067 (3)
S1	0.0253 (3)	0.0299 (3)	0.0174 (3)	−0.0027 (2)	0.0063 (2)	−0.0028 (2)
O1	0.0385 (10)	0.0359 (9)	0.0182 (7)	0.0017 (8)	0.0092 (7)	0.0021 (7)
O2	0.0295 (10)	0.0503 (10)	0.0292 (8)	−0.0062 (8)	0.0153 (7)	−0.0047 (8)
O3	0.0318 (10)	0.0349 (9)	0.0238 (8)	−0.0067 (8)	0.0042 (7)	−0.0086 (7)
N1	0.0269 (11)	0.0276 (9)	0.0174 (8)	0.0026 (8)	0.0053 (7)	−0.0009 (7)
C1	0.0247 (12)	0.0234 (11)	0.0228 (10)	−0.0038 (9)	0.0073 (9)	0.0001 (9)
C2	0.0333 (14)	0.0353 (13)	0.0257 (11)	−0.0050 (11)	0.0059 (10)	0.0079 (10)
C3	0.0353 (15)	0.0259 (11)	0.0430 (14)	−0.0004 (11)	0.0064 (11)	0.0102 (11)
C4	0.0319 (14)	0.0232 (11)	0.0514 (15)	−0.0001 (11)	0.0171 (12)	0.0008 (11)
C5	0.0291 (13)	0.0273 (11)	0.0299 (11)	−0.0041 (10)	0.0122 (10)	−0.0033 (9)
C6	0.0208 (12)	0.0234 (10)	0.0236 (10)	−0.0048 (9)	0.0066 (9)	0.0001 (9)
C7	0.0250 (12)	0.0256 (10)	0.0203 (10)	−0.0030 (9)	0.0081 (9)	−0.0004 (8)

C8	0.0272 (13)	0.0307 (12)	0.0281 (11)	0.0039 (10)	0.0038 (10)	−0.0004 (10)

Geometric parameters (Å, °)

Cl1—C8	1.801 (3)	C2—H2	0.9500
S1—O3	1.427 (2)	C3—C4	1.394 (4)
S1—O2	1.431 (2)	C3—H3	0.9500
S1—N1	1.672 (2)	C4—C5	1.384 (3)
S1—C1	1.748 (2)	C4—H4	0.9500
O1—C7	1.207 (3)	C5—C6	1.387 (3)
N1—C7	1.404 (3)	C5—H5	0.9500
N1—C8	1.433 (3)	C6—C7	1.478 (3)
C1—C2	1.384 (3)	C8—H8A	0.9900
C1—C6	1.397 (3)	C8—H8B	0.9900
C2—C3	1.386 (4)		
O3—S1—O2	116.90 (10)	C5—C4—C3	121.2 (2)
O3—S1—N1	110.12 (10)	C5—C4—H4	119.4
O2—S1—N1	109.25 (10)	C3—C4—H4	119.4
O3—S1—C1	111.79 (11)	C4—C5—C6	118.2 (2)
O2—S1—C1	113.48 (11)	C4—C5—H5	120.9
N1—S1—C1	92.56 (10)	C6—C5—H5	120.9
C7—N1—C8	122.46 (18)	C5—C6—C1	120.2 (2)
C7—N1—S1	115.29 (15)	C5—C6—C7	126.72 (19)
C8—N1—S1	121.46 (15)	C1—C6—C7	113.05 (19)
C2—C1—C6	121.8 (2)	O1—C7—N1	123.0 (2)
C2—C1—S1	127.66 (18)	O1—C7—C6	128.4 (2)
C6—C1—S1	110.49 (16)	N1—C7—C6	108.55 (17)
C3—C2—C1	117.5 (2)	N1—C8—Cl1	111.45 (17)
C3—C2—H2	121.2	N1—C8—H8A	109.3
C1—C2—H2	121.2	Cl1—C8—H8A	109.3
C2—C3—C4	121.0 (2)	N1—C8—H8B	109.3
C2—C3—H3	119.5	Cl1—C8—H8B	109.3
C4—C3—H3	119.5	H8A—C8—H8B	108.0
O3—S1—N1—C7	114.39 (17)	C4—C5—C6—C1	0.6 (3)
O2—S1—N1—C7	−115.93 (17)	C4—C5—C6—C7	−179.5 (2)
C1—S1—N1—C7	0.05 (18)	C2—C1—C6—C5	−1.1 (3)
O3—S1—N1—C8	−75.6 (2)	S1—C1—C6—C5	177.41 (18)
O2—S1—N1—C8	54.1 (2)	C2—C1—C6—C7	178.9 (2)
C1—S1—N1—C8	170.05 (18)	S1—C1—C6—C7	−2.5 (2)
O3—S1—C1—C2	67.0 (2)	C8—N1—C7—O1	9.2 (3)
O2—S1—C1—C2	−67.8 (2)	S1—N1—C7—O1	179.07 (18)
N1—S1—C1—C2	179.9 (2)	C8—N1—C7—C6	−171.3 (2)
O3—S1—C1—C6	−111.42 (17)	S1—N1—C7—C6	−1.4 (2)
O2—S1—C1—C6	113.75 (17)	C5—C6—C7—O1	2.1 (4)
N1—S1—C1—C6	1.45 (18)	C1—C6—C7—O1	−178.0 (2)
C6—C1—C2—C3	0.6 (3)	C5—C6—C7—N1	−177.4 (2)
S1—C1—C2—C3	−177.65 (19)	C1—C6—C7—N1	2.5 (3)
C1—C2—C3—C4	0.4 (4)	C7—N1—C8—Cl1	−93.0 (2)
C2—C3—C4—C5	−1.0 (4)	S1—N1—C8—Cl1	97.72 (18)

C3—C4—C5—C6	0.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.95	2.52	3.222 (3)	131
C8—H8B···O3^{ii}	0.99	2.33	3.288 (3)	163

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*−1/2; (ii) *x*−1/2, −*y*+3/2, *z*−1/2.

Fig. 1

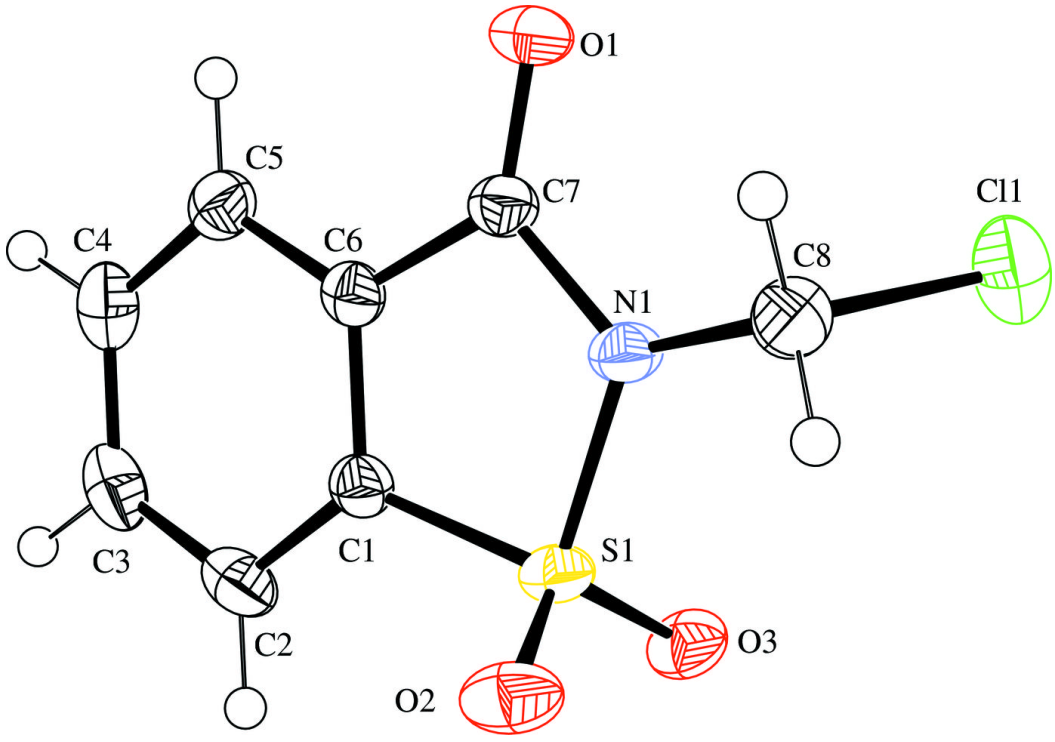

Fig. 2