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o-Sulfamoylbenzoic acid

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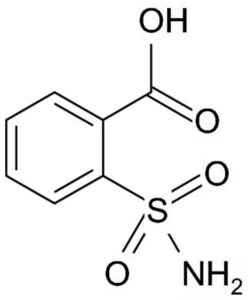
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 15.4.

The title compound, $C_7H_7NO_4S$, contains an extensive hydrogen-bonded network involving three intermolecular and three intramolecular interactions that stabilize the structure.

Related literature

For related literature, see: Bernstein *et al.* (1994); Culf *et al.* (1997); Eatedal *et al.* (2002); Groutas *et al.* (1993); González-Álvarez *et al.* (2003); Siddiqui *et al.* (2006); Siddiqui, Ahmad, Khan & Siddiqui (2007); Siddiqui, Ahmad, Khan, Siddiqui & Ahmad (2007).



Experimental

Crystal data

$C_7H_7NO_4S$
M_r = 201.20
Monoclinic, $C2/c$
a = 12.200 (4) Å
b = 15.824 (6) Å
c = 9.012 (4) Å
β = 100.45 (2)°
V = 1710.9 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 173 (2) K
0.20 × 0.18 × 0.14 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.932, T_{max} = 0.952
3769 measured reflections
1952 independent reflections
1718 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.04
1952 reflections
127 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 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
O1—H1···O2^{i}	0.84 (2)	1.90 (2)	2.731 (2)	169 (2)
N1—H1A···O2	0.86 (2)	2.24 (2)	2.867 (2)	130 (2)
N1—H1A···O4^{ii}	0.86 (2)	2.21 (2)	2.987 (2)	150 (2)
N1—H1B···O3^{iii}	0.88 (2)	2.11 (2)	2.976 (2)	168 (2)
C2—H2···O3	0.95	2.36	2.805 (2)	108
C5—H5···O1	0.95	2.37	2.709 (2)	100

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

Data collection: *COLLECT* (Nonius, 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, 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: LH2510).

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supplementary materials

Acta Cryst. (2007). E**63**, o4117 [**doi:10.1107/S1600536807045710**]

o-Sulfamoylbenzoic acid

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

Comment

Saccharin is an old synthetic compound. Its derivatives have always been of interest due to their diverse applications (González-Álvarez *et al.*, 2003; Culf *et al.*, 1997). The open ring benzene sulfonamide derivatives of saccharin have shown cyclooxygenase-2 inhibitory action and act as analgesic and anti-inflammatory agents (Eatedal *et al.*, 2002). The title compound is one of the very first derivatives of saccharin that was prepared by an indirect route involving hydroxymethylsaccharin (Groutas *et al.*, 1993). In continuation of our research program to synthesize new biologically important 1,2-benzothiazine 1,1-dioxide molecules (Siddiqui *et al.*, 2006; Siddiqui, Ahmad, Khan & Siddiqui, 2007; Siddiqui, Ahmad, Khan, Siddiqui & Ahmad, 2007), we embarked on derivatization of saccharin as well. Herein, we report the crystal structure of the title compound that has been synthesized by a straight forward method.

The structure of the title compound (Fig. 1) contains an extensive hydrogen bonded net work (Fig. 2). The acid and the amino hydrogen atoms are involved in three intermolecular H-bonds of the types O—H···O and N—H···O, respectively. The acid group, in its synplanar conformation, forms cyclic dimers, typical of the carboxylic acids, about inversion centers. The eight membered ring thus formed exhibits an $R_2^2(8)$-type motif (Bernstein *et al.*, 1994). One of the amino H-atoms, H1A is hydrogen bonded to O4 of a symmetry related molecule (details are in Table) thus resulting in chains of molecules. The second H-atom of the amino group, H1B, on the other hand, is hydrogen bonded to O3 of an adjacent molecule which is oriented in such a manner that its amino H1B is hydrogen bonded to the reference molecule, thus forming an eight membered ring $S1^{iii}/O3^{iii}/H1B/N1/S1/O3/H1B^{iii}/N1^{iii}$ (details are in Table). In terms of graph-set presentation (Bernstein *et al.*, 1994), this ring also exhibits an $R_2^2(8)$ hydrogen bonding pattern. The structure is further stabilized by three intramolecular interactions of the type C—H···O and N—H···O (details are in Table).

Experimental

Experimental: A mixture of saccharin (0.997 g, 5.5 mmol.) and sodium hydroxide (0.5 g, 12.5 mmol.) in distilled water (25 ml) was heated to reflux (1 hr), cooled to room temperature and acidified to pH = 4 (15% HCl). The reaction mixture was subjected to cooling in freezer overnight and white flakes-like product was filtered, washed with cold water and dried to yield 1.06 g of the title compound (5.3 mmol. 97%) which was recrystallized from a solution of MeOH and $CHCl_3$ (1:1) at 313 K to obtain colorless crystals.

Refinement

H-atoms bonded to C-atoms were included in the refinements at geometrically idealized positions with C—H = 0.95 and 0.99 Å and U_{iso} = 1.2 times U_{eq} of the atoms to which they were bonded. The H-atoms bonded to N1 and O1 were allowed to refine with U_{iso} = 1.2 times U_{eq} of the parent atoms. The final difference map was free of any chemically significant features.

Figures

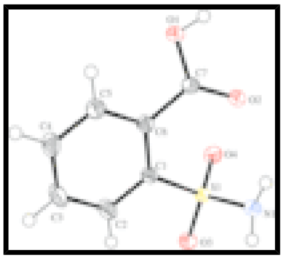

Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the title compound with displacement ellipsoids plotted at 50% probability level.

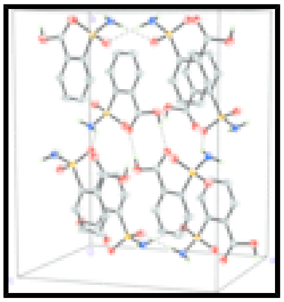

Fig. 2. Unit cell packing of the title compound showing intramolecular hydrogen bonds; only the H-atoms involved in H-bonds have been shown.

o-Sulfamoylbenzoic acid

Crystal data

$C_7H_7NO_4S$

$M_r = 201.20$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 12.200\ (4)$ Å

$b = 15.824\ (6)$ Å

$c = 9.012\ (4)$ Å

$\beta = 100.45\ (2)°$

$V = 1710.9\ (11)$ Å^3

$Z = 8$

$F_{000} = 832$

$D_x = 1.562$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3769 reflections

$\theta = 4.2–27.5°$

$\mu = 0.36$ mm^{-1}

$T = 173\ (2)$ K

Prism, colourless

0.20 × 0.18 × 0.14 mm

Data collection

Nonius KappaCCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173(2)$ K

ω and φ scans

Absorption correction: multi-scan (SORTAV; Blessing, 1997)

$T_{min} = 0.932$, $T_{max} = 0.952$

3769 measured reflections

1952 independent reflections

1718 reflections with $I > 2\sigma(I)$

$R_{int} = 0.024$

$\theta_{max} = 27.5°$

$\theta_{min} = 4.2°$

$h = -15 \rightarrow 15$

$k = -19 \rightarrow 20$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 1.65P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1952 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
127 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. IR (Neat, ν_{max}, cm^{-1}): NH 3321 (br), CO 1715, SO_2 1270 and 1157; ^{1}H-NMR (400 MHz, DMSO-d$_6$) δ: 7.18 (s, 2H, NH_2), 7.62–7.75 (m, 3H, aromatic), 7.95 (m, 1H, aromatic); ^{13}C-NMR δ: 169.0, 141.1, 132.1, 131.8, 130.9, 129.3, 127.3. LRMS (FAB$^+$): m/*z*: 202 $[M]^+$ (53.5%), 403 $[2M]^+$ (18.3%).

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	0.32466 (3)	0.08825 (2)	0.29800 (4)	0.01783 (13)
O1	−0.03197 (9)	0.10966 (8)	0.06197 (17)	0.0358 (3)
H1	−0.0561 (18)	0.0621 (14)	0.031 (3)	0.043*
O2	0.12390 (9)	0.03527 (7)	0.07062 (14)	0.0262 (3)
O3	0.42650 (9)	0.11678 (7)	0.39115 (13)	0.0253 (3)
O4	0.25219 (9)	0.03529 (7)	0.36559 (13)	0.0241 (3)
N1	0.36169 (11)	0.03865 (8)	0.16031 (16)	0.0220 (3)
H1A	0.3086 (16)	0.0220 (12)	0.090 (2)	0.026*
H1B	0.4203 (16)	0.0615 (12)	0.131 (2)	0.026*
C1	0.24768 (12)	0.18088 (9)	0.23164 (17)	0.0186 (3)
C2	0.29921 (14)	0.25731 (10)	0.2747 (2)	0.0284 (4)
H2	0.3740	0.2578	0.3277	0.034*
C3	0.24279 (15)	0.33333 (10)	0.2413 (2)	0.0328 (4)
H3	0.2792	0.3854	0.2704	0.039*
C4	0.13346 (14)	0.33287 (10)	0.1656 (2)	0.0293 (4)
H4	0.0940	0.3845	0.1444	0.035*
C5	0.08186 (13)	0.25648 (10)	0.12075 (18)	0.0243 (3)
H5	0.0070	0.2567	0.0681	0.029*

C6	0.13656 (12)	0.17981 (9)	0.15061 (16)	0.0186 (3)
C7	0.07735 (12)	0.10081 (9)	0.09228 (17)	0.0195 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0154 (2)	0.0169 (2)	0.0196 (2)	0.00077 (13)	−0.00111 (14)	−0.00002 (13)
O1	0.0156 (6)	0.0250 (6)	0.0617 (9)	0.0008 (5)	−0.0062 (5)	−0.0151 (6)
O2	0.0168 (5)	0.0228 (6)	0.0371 (7)	−0.0011 (4)	0.0003 (5)	−0.0100 (5)
O3	0.0184 (5)	0.0264 (6)	0.0271 (6)	0.0022 (4)	−0.0064 (4)	−0.0048 (5)
O4	0.0226 (5)	0.0226 (6)	0.0271 (6)	0.0013 (4)	0.0047 (4)	0.0059 (4)
N1	0.0161 (6)	0.0244 (7)	0.0248 (7)	−0.0027 (5)	0.0018 (5)	−0.0059 (5)
C1	0.0167 (7)	0.0175 (7)	0.0202 (7)	−0.0015 (5)	−0.0002 (6)	−0.0002 (6)
C2	0.0250 (8)	0.0204 (8)	0.0351 (9)	−0.0030 (6)	−0.0072 (7)	−0.0007 (7)
C3	0.0352 (9)	0.0166 (8)	0.0414 (10)	−0.0048 (7)	−0.0070 (8)	−0.0009 (7)
C4	0.0348 (9)	0.0170 (7)	0.0325 (9)	0.0040 (7)	−0.0031 (7)	0.0027 (7)
C5	0.0236 (8)	0.0220 (8)	0.0244 (8)	0.0024 (6)	−0.0031 (6)	0.0014 (6)
C6	0.0185 (7)	0.0178 (7)	0.0181 (7)	−0.0006 (6)	0.0000 (6)	0.0000 (5)
C7	0.0172 (7)	0.0210 (7)	0.0187 (7)	0.0004 (6)	−0.0007 (6)	−0.0020 (6)

Geometric parameters (Å, °)

S1—O4	1.4319 (12)	C1—C6	1.419 (2)
S1—O3	1.4402 (11)	C2—C3	1.391 (2)
S1—N1	1.6010 (15)	C2—H2	0.9500
S1—C1	1.7844 (16)	C3—C4	1.384 (2)
O1—C7	1.3194 (18)	C3—H3	0.9500
O1—H1	0.84 (2)	C4—C5	1.389 (2)
O2—C7	1.2152 (18)	C4—H4	0.9500
N1—H1A	0.860 (19)	C5—C6	1.387 (2)
N1—H1B	0.88 (2)	C5—H5	0.9500
C1—C2	1.386 (2)	C6—C7	1.491 (2)
O4—S1—O3	118.00 (7)	C4—C3—C2	119.80 (15)
O4—S1—N1	109.00 (8)	C4—C3—H3	120.1
O3—S1—N1	105.70 (7)	C2—C3—H3	120.1
O4—S1—C1	107.17 (7)	C3—C4—C5	119.58 (14)
O3—S1—C1	106.49 (7)	C3—C4—H4	120.2
N1—S1—C1	110.39 (8)	C5—C4—H4	120.2
C7—O1—H1	104.7 (15)	C6—C5—C4	121.87 (14)
S1—N1—H1A	116.0 (13)	C6—C5—H5	119.1
S1—N1—H1B	112.8 (12)	C4—C5—H5	119.1
H1A—N1—H1B	116.7 (18)	C5—C6—C1	118.08 (13)
C2—C1—C6	119.82 (14)	C5—C6—C7	118.66 (13)
C2—C1—S1	116.01 (11)	C1—C6—C7	123.24 (13)
C6—C1—S1	123.95 (11)	O2—C7—O1	122.80 (14)
C1—C2—C3	120.82 (15)	O2—C7—C6	124.16 (13)
C1—C2—H2	119.6	O1—C7—C6	113.01 (13)
C3—C2—H2	119.6		

O4—S1—C1—C2	129.47 (14)	C4—C5—C6—C1	1.1 (2)
O3—S1—C1—C2	2.32 (15)	C4—C5—C6—C7	−177.08 (16)
N1—S1—C1—C2	−111.95 (14)	C2—C1—C6—C5	−1.8 (2)
O4—S1—C1—C6	−45.14 (15)	S1—C1—C6—C5	172.67 (12)
O3—S1—C1—C6	−172.29 (13)	C2—C1—C6—C7	176.32 (15)
N1—S1—C1—C6	73.44 (15)	S1—C1—C6—C7	−9.3 (2)
C6—C1—C2—C3	0.9 (3)	C5—C6—C7—O2	157.01 (16)
S1—C1—C2—C3	−173.97 (15)	C1—C6—C7—O2	−21.1 (2)
C1—C2—C3—C4	0.7 (3)	C5—C6—C7—O1	−21.2 (2)
C2—C3—C4—C5	−1.4 (3)	C1—C6—C7—O1	160.75 (15)
C3—C4—C5—C6	0.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84 (2)	1.90 (2)	2.731 (2)	169 (2)
N1—H1A···O2	0.86 (2)	2.24 (2)	2.867 (2)	130 (2)
N1—H1A···O4^{ii}	0.86 (2)	2.21 (2)	2.987 (2)	150 (2)
N1—H1B···O3^{iii}	0.88 (2)	2.11 (2)	2.976 (2)	168 (2)
C2—H2···O3	0.95	2.36	2.805 (2)	108
C5—H5···O1	0.95	2.37	2.709 (2)	100

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

Fig. 1

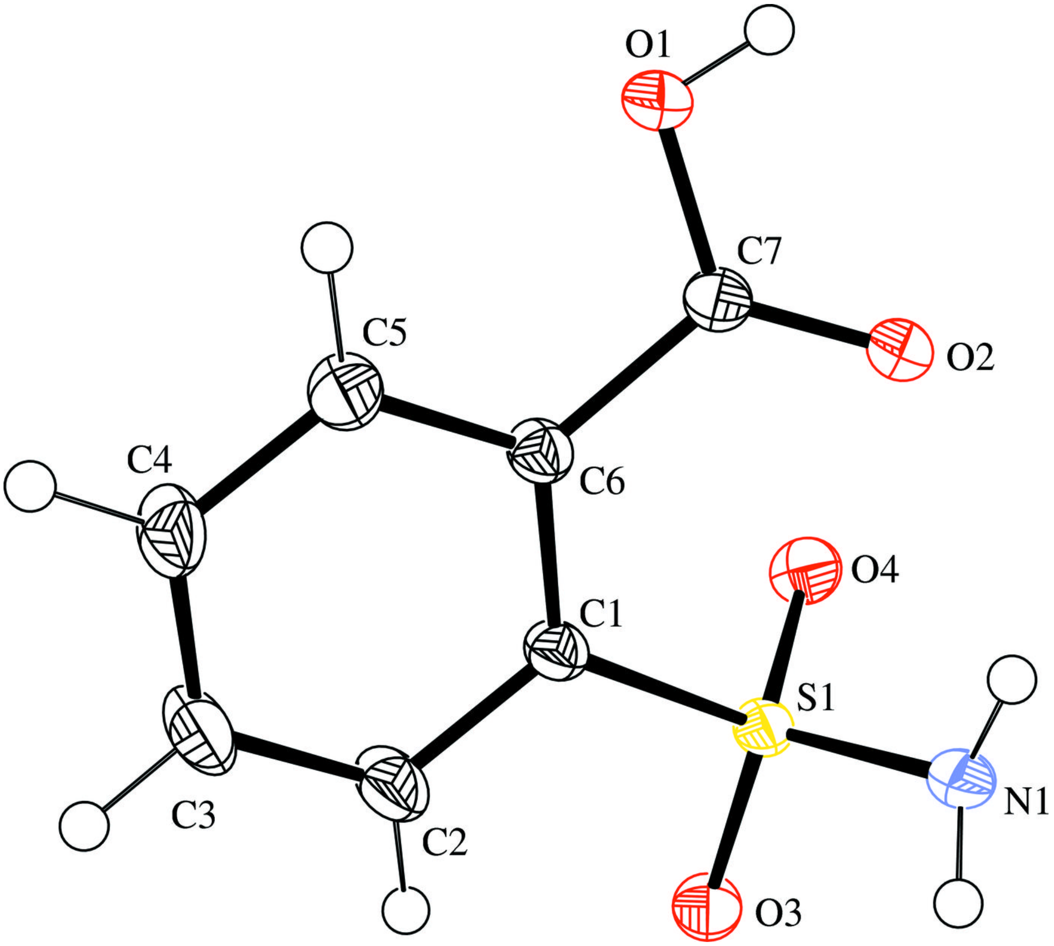

Fig. 2