

N-Acetylsaccharin

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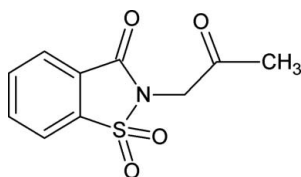
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.110; data-to-parameter ratio = 16.3.

In the title compound [systematic name: 2-(2-oxopropyl)-1,2-benzothiazol-3(2H)-one 1,1-dioxide], $\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$, the benzothiazole unit is essentially planar [maximum deviation = 0.0490 (9) Å for the S atom] and the oxopropyl group is inclined at an angle 75.61 (8)° with respect to its mean plane. In the crystal, molecules are held together by weak intermolecular C—H...O non-classical hydrogen bonds, resulting in centrosymmetric dimeric units, forming 14-membered ring systems which may be described as $R_2^2(14)$ ring motifs. Moreover, molecules lying about inversion centers show π - π interactions, with centroid-centroid separations between the benzene rings of 3.676 (2) Å.

Related literature

For the crystal structure of a benzothiazine, see: Ahmad *et al.* (2008). For the biological activity of saccharine derivatives, see: Kapui *et al.* (2003); Singh *et al.* (2007); Vaccarino *et al.* (2007). For graph-set notation of ring motifs, see: Bernstein *et al.* (1994).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$	$V = 1048.5$ (8) Å ³
$M_r = 239.24$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.475$ (3) Å	$\mu = 0.31$ mm ⁻¹
$b = 8.975$ (4) Å	$T = 200$ K
$c = 15.923$ (7) Å	$0.12 \times 0.12 \times 0.06$ mm
$\beta = 101.028$ (18)°	

Data collection

Nonius KappaCCD diffractometer	3984 measured reflections
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	2382 independent reflections
$T_{\min} = 0.964$, $T_{\max} = 0.982$	2106 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	146 parameters
$wR(F^2) = 0.110$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.30$ e Å ⁻³
2382 reflections	$\Delta\rho_{\text{min}} = -0.41$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C10}-\text{H10B}\cdots\text{O2}^i$	0.98	2.57	3.324 (3)	134

Symmetry code: (i) $-x, -y, -z + 1$.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997); data reduction: SCALEPACK (Otwinowski & Minor, 1997); 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); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2191).

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supplementary materials

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N-Acetonylsaccharin

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Comment

Saccharine derivatives are extensively reported in the literature for their diverse range of biological activities like cyclooxygenase-2 (COX-2) inhibitors (Singh *et al.* 2007), analgesic (Vaccarino *et al.* 2007), human leucocyte elastase (HLE) inhibitors (Kapui *et al.* 2003). In continuation to our project to explore potentially biologically active derivatives of benzothiazines (Ahmad *et al.* 2008), we herein report the crystal structure of the title compound, (I), in this paper.

The structure of the title compound is depicted in Figure 1. The benzothiazol moiety (C1—C7/N1/S1) is essentially planar with maximum deviation observed for S1 (0.0490 (9) Å) and the oxopropyl group (C8/C9/C10/O4) forms an angle 75.61 (8)° with the mean-plane of the former. The molecular dimensions in (I) agree with the corresponding molecular dimensions reported for a closely related compound (Ahmad *et al.* 2008). In the crystal structure, the molecules of (I) are held together by rather weak intermolecular C—H...O type non-classical hydrogen bonds resulting in dimeric units about inversion centers, forming fourteen membered ring systems which may be described in terms of graph set notation (Bernstein *et al.* 1994) as $R_2^2(14)$ ring motif; details are given in Table 1 and Figure 2. The molecules lying about inversion centers show π - π interactions with the separation between the centroids of the benzene rings (C1—C6) which are related by the symmetry operation: 1-x, 1-y, 1-z, is 3.676 (2) Å (Spek, 2009); with perpendicular distance being 3.354 Å and the slippage of 1.504 Å.

Experimental

Sodium saccharin (73.2 mmole, 15.0 g) and chloroacetone (87.8 mmole, 7.0 ml) were added in a round bottom flask containing 30 ml of anhydrous DMF. The mixture was stirred under inert atmosphere for one hour at 393 K. The contents of the flask were poured in ice cold water. Brownish ppts. formed were filtered and washed with excess of water. Crystals suitable for XRD were grown in chloroform. Yield: 15.2 g, 87%; m.p. 386–387 K.

Refinement

Though all the H atoms could be distinguished in the difference Fourier map the H-atoms were included at geometrically idealized positions and refined in riding-model approximation with the following constraints: C—H distances were set to 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The final difference map was free of any chemically significant features.

Figures

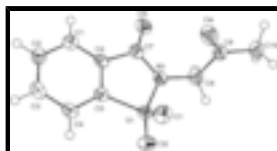


Fig. 1. ORTEP-3 (Farrugia, 1997) drawing of (I) with displacement ellipsoids plotted at 50% probability level.

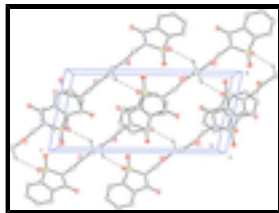


Fig. 2. Unit cell of (I) showing dimers of molecules formed by C—H...O interactions; H-atoms not involved in H-bonding interactions have been excluded.

2-(2-oxopropyl)-1,2-benzothiazol-3(2H)-one 1,1-dioxide

Crystal data

$C_{10}H_9NO_4S$	$F_{000} = 496$
$M_r = 239.24$	$D_x = 1.516 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 3984 reflections
$a = 7.475 (3) \text{ \AA}$	$\theta = 3.4\text{--}27.5^\circ$
$b = 8.975 (4) \text{ \AA}$	$\mu = 0.31 \text{ mm}^{-1}$
$c = 15.923 (7) \text{ \AA}$	$T = 200 \text{ K}$
$\beta = 101.028 (18)^\circ$	Block, colorless
$V = 1048.5 (8) \text{ \AA}^3$	$0.12 \times 0.12 \times 0.06 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	2382 independent reflections
Radiation source: fine-focus sealed tube	2106 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
$T = 200 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ω and φ scans	$\theta_{\text{min}} = 3.4^\circ$
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.964$, $T_{\text{max}} = 0.982$	$k = -8 \rightarrow 11$
3984 measured reflections	$l = -20 \rightarrow 20$

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
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.8524P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2382 reflections	$(\Delta/\sigma)_{\text{max}} = 0.005$
146 parameters	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.26215 (6)	0.14487 (5)	0.47133 (3)	0.02833 (15)
O1	0.09274 (19)	0.22262 (17)	0.44859 (10)	0.0401 (4)
O2	0.2659 (2)	-0.00422 (16)	0.44012 (10)	0.0406 (4)
O3	0.5536 (2)	0.25086 (18)	0.68078 (9)	0.0397 (4)
O4	0.1159 (2)	0.30470 (18)	0.66321 (11)	0.0504 (4)
N1	0.3345 (2)	0.14693 (19)	0.57704 (10)	0.0319 (4)
C1	0.7056 (3)	0.3839 (2)	0.53131 (12)	0.0306 (4)
H1	0.7806	0.4139	0.5837	0.037*
C2	0.7458 (3)	0.4271 (2)	0.45301 (13)	0.0335 (4)
H2	0.8482	0.4894	0.4520	0.040*
C3	0.6389 (3)	0.3809 (2)	0.37630 (13)	0.0355 (4)
H3	0.6702	0.4113	0.3238	0.043*
C4	0.4873 (3)	0.2911 (2)	0.37500 (12)	0.0317 (4)
H4	0.4148	0.2579	0.3227	0.038*
C5	0.4466 (2)	0.2519 (2)	0.45337 (12)	0.0267 (4)
C6	0.5534 (2)	0.29595 (19)	0.53062 (11)	0.0263 (4)
C7	0.4875 (3)	0.2339 (2)	0.60579 (12)	0.0295 (4)
C8	0.2358 (3)	0.0693 (2)	0.63437 (13)	0.0350 (4)
H8A	0.3243	0.0170	0.6788	0.042*
H8B	0.1557	-0.0068	0.6014	0.042*
C9	0.1209 (3)	0.1729 (2)	0.67757 (13)	0.0346 (4)
C10	0.0182 (3)	0.1008 (3)	0.73859 (14)	0.0500 (6)
H10A	0.0730	0.0039	0.7564	0.060*
H10B	-0.1092	0.0867	0.7104	0.060*
H10C	0.0235	0.1646	0.7890	0.060*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0268 (2)	0.0309 (2)	0.0263 (2)	0.00255 (17)	0.00270 (17)	-0.00209 (17)
O1	0.0271 (7)	0.0465 (8)	0.0446 (9)	0.0070 (6)	0.0015 (6)	0.0000 (7)

supplementary materials

O2	0.0477 (9)	0.0311 (7)	0.0415 (8)	-0.0004 (6)	0.0049 (7)	-0.0067 (6)
O3	0.0419 (8)	0.0536 (9)	0.0221 (7)	-0.0003 (7)	0.0024 (6)	0.0009 (6)
O4	0.0612 (11)	0.0388 (8)	0.0582 (11)	0.0046 (8)	0.0288 (9)	0.0006 (7)
N1	0.0303 (8)	0.0411 (9)	0.0252 (8)	-0.0019 (7)	0.0077 (6)	-0.0004 (6)
C1	0.0278 (9)	0.0337 (9)	0.0289 (9)	0.0050 (7)	0.0021 (7)	-0.0009 (7)
C2	0.0286 (9)	0.0344 (10)	0.0393 (11)	0.0029 (8)	0.0105 (8)	0.0018 (8)
C3	0.0367 (10)	0.0430 (11)	0.0291 (10)	0.0065 (8)	0.0124 (8)	0.0051 (8)
C4	0.0334 (9)	0.0382 (10)	0.0230 (9)	0.0071 (8)	0.0043 (7)	-0.0016 (7)
C5	0.0251 (8)	0.0287 (8)	0.0259 (9)	0.0051 (7)	0.0040 (7)	-0.0006 (7)
C6	0.0278 (9)	0.0280 (8)	0.0229 (8)	0.0061 (7)	0.0043 (7)	-0.0008 (7)
C7	0.0290 (9)	0.0345 (9)	0.0249 (9)	0.0052 (7)	0.0045 (7)	0.0005 (7)
C8	0.0389 (10)	0.0345 (10)	0.0330 (10)	-0.0006 (8)	0.0103 (8)	0.0053 (8)
C9	0.0322 (10)	0.0435 (11)	0.0288 (10)	-0.0055 (9)	0.0075 (8)	0.0006 (8)
C10	0.0500 (13)	0.0703 (16)	0.0330 (12)	-0.0204 (12)	0.0159 (10)	-0.0010 (11)

Geometric parameters (Å, °)

S1—O2	1.4295 (15)	C3—C4	1.388 (3)
S1—O1	1.4305 (15)	C3—H3	0.9500
S1—N1	1.6667 (18)	C4—C5	1.385 (3)
S1—C5	1.7485 (19)	C4—H4	0.9500
O3—C7	1.211 (2)	C5—C6	1.389 (3)
O4—C9	1.204 (3)	C6—C7	1.487 (3)
N1—C7	1.388 (3)	C8—C9	1.517 (3)
N1—C8	1.456 (2)	C8—H8A	0.9900
C1—C6	1.383 (3)	C8—H8B	0.9900
C1—C2	1.392 (3)	C9—C10	1.496 (3)
C1—H1	0.9500	C10—H10A	0.9800
C2—C3	1.389 (3)	C10—H10B	0.9800
C2—H2	0.9500	C10—H10C	0.9800
O2—S1—O1	116.35 (9)	C6—C5—S1	110.42 (14)
O2—S1—N1	109.71 (9)	C1—C6—C5	120.11 (17)
O1—S1—N1	110.53 (9)	C1—C6—C7	127.31 (17)
O2—S1—C5	112.90 (9)	C5—C6—C7	112.55 (16)
O1—S1—C5	112.22 (9)	O3—C7—N1	123.44 (18)
N1—S1—C5	92.60 (8)	O3—C7—C6	127.64 (18)
C7—N1—C8	123.13 (17)	N1—C7—C6	108.91 (16)
C7—N1—S1	115.29 (13)	N1—C8—C9	112.95 (16)
C8—N1—S1	121.49 (14)	N1—C8—H8A	109.0
C6—C1—C2	118.03 (18)	C9—C8—H8A	109.0
C6—C1—H1	121.0	N1—C8—H8B	109.0
C2—C1—H1	121.0	C9—C8—H8B	109.0
C3—C2—C1	121.19 (19)	H8A—C8—H8B	107.8
C3—C2—H2	119.4	O4—C9—C10	123.2 (2)
C1—C2—H2	119.4	O4—C9—C8	121.05 (18)
C4—C3—C2	121.16 (18)	C10—C9—C8	115.78 (19)
C4—C3—H3	119.4	C9—C10—H10A	109.5
C2—C3—H3	119.4	C9—C10—H10B	109.5
C5—C4—C3	116.97 (18)	H10A—C10—H10B	109.5

C5—C4—H4	121.5	C9—C10—H10C	109.5
C3—C4—H4	121.5	H10A—C10—H10C	109.5
C4—C5—C6	122.52 (18)	H10B—C10—H10C	109.5
C4—C5—S1	127.07 (15)		
O2—S1—N1—C7	120.12 (15)	C2—C1—C6—C7	178.29 (17)
O1—S1—N1—C7	-110.26 (15)	C4—C5—C6—C1	0.9 (3)
C5—S1—N1—C7	4.66 (15)	S1—C5—C6—C1	-179.02 (13)
O2—S1—N1—C8	-63.36 (17)	C4—C5—C6—C7	-177.06 (16)
O1—S1—N1—C8	66.26 (17)	S1—C5—C6—C7	3.04 (19)
C5—S1—N1—C8	-178.83 (15)	C8—N1—C7—O3	1.2 (3)
C6—C1—C2—C3	-1.4 (3)	S1—N1—C7—O3	177.61 (15)
C1—C2—C3—C4	0.6 (3)	C8—N1—C7—C6	179.96 (16)
C2—C3—C4—C5	0.9 (3)	S1—N1—C7—C6	-3.59 (19)
C3—C4—C5—C6	-1.7 (3)	C1—C6—C7—O3	1.1 (3)
C3—C4—C5—S1	178.21 (14)	C5—C6—C7—O3	178.91 (18)
O2—S1—C5—C4	63.10 (19)	C1—C6—C7—N1	-177.59 (17)
O1—S1—C5—C4	-70.77 (19)	C5—C6—C7—N1	0.2 (2)
N1—S1—C5—C4	175.78 (17)	C7—N1—C8—C9	74.6 (2)
O2—S1—C5—C6	-117.01 (13)	S1—N1—C8—C9	-101.65 (19)
O1—S1—C5—C6	109.12 (14)	N1—C8—C9—O4	0.4 (3)
N1—S1—C5—C6	-4.32 (14)	N1—C8—C9—C10	-178.98 (18)
C2—C1—C6—C5	0.7 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10B...O2 ⁱ	0.98	2.57	3.324 (3)	134

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

Fig. 1

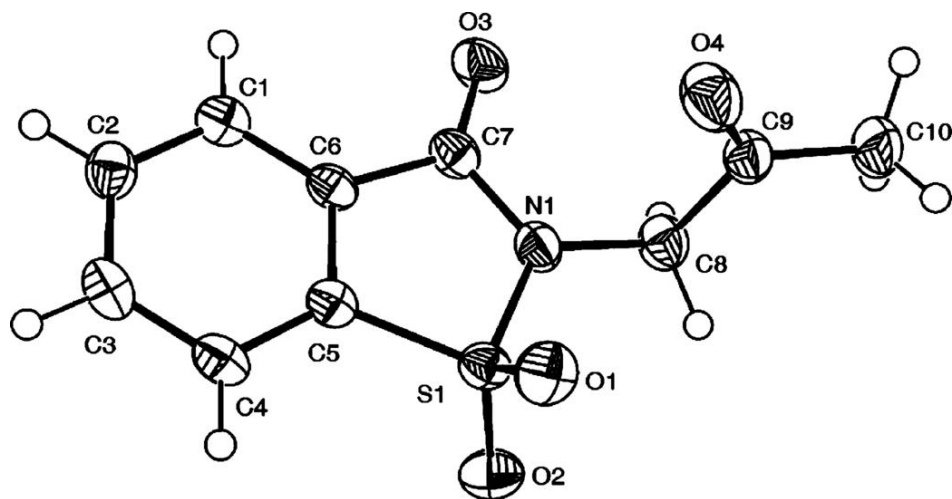


Fig. 2

