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

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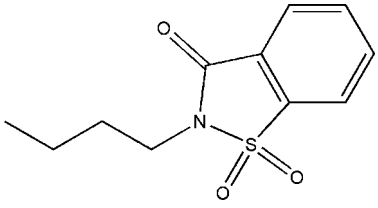
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.110; data-to-parameter ratio = 14.1.

The crystal packing of the title compound, $C_{11}H_{13}NO_3S$, exhibits weak intermolecular C—H···O hydrogen bonding, which links molecules related by translation along the b axis into chains, and π–π interactions [centroid–centroid distance of 3.778 (2) Å between benzene rings].

Related literature

For similar crystal structures, see: Feeder & Jones (1994, 1996); Glidewell *et al.* (2000). For related literature, see: Xiong (2004); Rice & Pettit (1954).



Experimental

Crystal data

$C_{11}H_{13}NO_3S$
M_r = 239.28
Triclinic, $P\overline{1}$
a = 7.3130 (15) Å
b = 7.7219 (15) Å
c = 11.416 (2) Å
α = 102.76 (3)°
β = 94.23 (3)°
γ = 109.75 (3)°
V = 584.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 153 (2) K
0.30 × 0.24 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.924, T_{max} = 0.953
4589 measured reflections
2061 independent reflections
1712 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.109
S = 1.08
2061 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O3^{ii}	0.95	2.35	3.279 (2)	165

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2391).

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supporting information

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S1. Comment

The title compound, (I), also called THIAZONE, is a new skin penetration enhancer. The tests of penetration enhancing behaviors to berberine, ciclopirox olamino and cypermethrin show that penetration enhancing effect ofTHIAZONE is 2.99 times higher than that of AZONE. THIAZONE is widely applied in pharmaceutic industry, cosmetic and health care industry, agriculture and forest industry, and many others (Xiong, 2004). Herewith we report the crystal structure of (I).

In (I) (Fig. 1), all bond lengths and angles within the saccharin group are similar to those observed in the series of N-saccharin acids (Feeder & Jones, 1996), N-saccharin peracids (Feeder & Jones, 1994) and saccharin (Glidewell *et al.*, 2000).

In the crystal, the relatively short distance between the centroids of benzene rings from neighbouring molecules (Table 1) suggests an existence of $\pi\cdots\pi$ interactions. The crystal packing exhibits also exhibits weak intermolecular C—H···O hydrogen bonds (Table 2), which link the molecules related by translation along *b* axis into chains.

S2. Experimental

The title compound has been synthesized following the known procedure (Rice & Pettit, 1954). Saccharin sodium 2.65 g (0.011 mol) was dissolved in 20 ml of dried DMF. To the solution,1-butyl bromide 1.37 g (0.01 mol) was added. The mixture was stirred for half an hour at room temperature and then the mixture was heated with strring for 2 h at 100° C. The mixture was poured into water, and 2.50 g of the product were obtained (yield 95.7%). Single crystals suitable for X-ray measurement were obtained by recrystallization from dichloromethane at room temperature.

S3. Refinement

All H atoms were placed in idealized positions and constrained to ride on their parent atoms,with C—H distances of 0.95 (aromatic), 0.98 (CH_3) and 0.99 Å (CH_2),and with U_{iso}(H) values set at 1.5 U_{eq}(C)(for CH_3) or 1.2 U_{eq}(C)(for CH_2, aromatic CH).

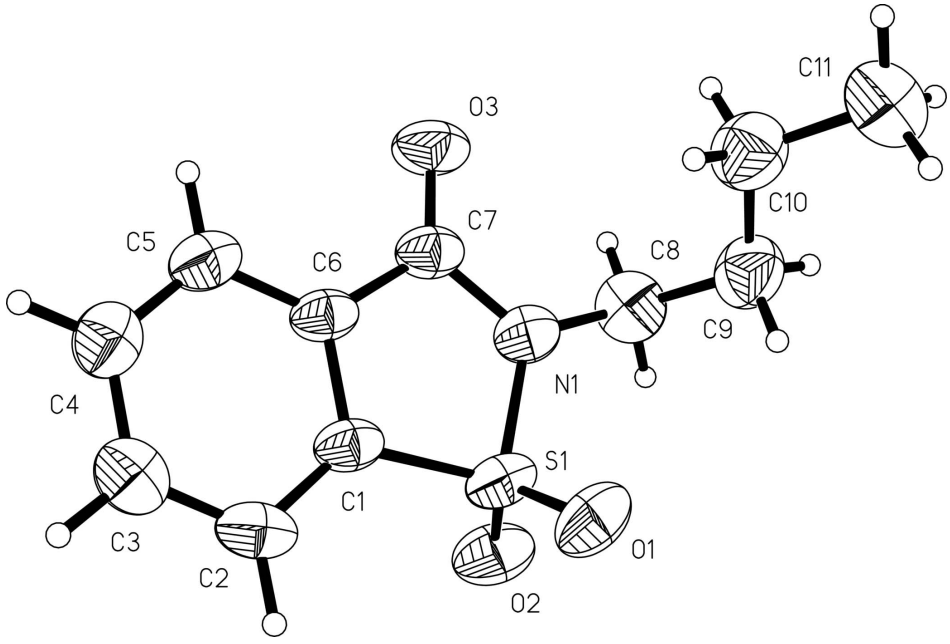

Figure 1

The molecular structure of (I) showing the atomic numbering and displacement ellipsoids drawn at the 40% probability level.

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

Crystal data

$C_{11}H_{13}NO_3S$
$M_r = 239.28$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3130\ (15)$ Å
$b = 7.7219\ (15)$ Å
$c = 11.416\ (2)$ Å
$\alpha = 102.76\ (3)°$
$\beta = 94.23\ (3)°$
$\gamma = 109.75\ (3)°$
$V = 584.0\ (2)$ Å^3
$Z = 2$
$F(000) = 252$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1494 reflections
$\theta = 2.6–26.4°$
$\mu = 0.27$ mm^{-1}
$T = 153$ K
Block, colourless
0.30 × 0.24 × 0.18 mm

Data collection

Rigaku R-Axis Rapid IP area-detector diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω Oscillation scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.924$, $T_{max} = 0.953$
4589 measured reflections
2061 independent reflections
1712 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -8\rightarrow8$
$k = -9\rightarrow9$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.109$
$S = 1.08$
2061 reflections
146 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.1349P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.129 (10)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.42409 (7)	0.81664 (7)	0.72568 (5)	0.0606 (2)
O1	0.3065 (2)	0.7267 (2)	0.80490 (15)	0.0795 (5)
O2	0.6279 (2)	0.8432 (2)	0.74251 (17)	0.0828 (5)
O3	0.3009 (2)	1.1828 (2)	0.60625 (15)	0.0757 (5)
N1	0.4016 (2)	1.0256 (2)	0.73053 (15)	0.0597 (4)
C1	0.3124 (3)	0.7195 (3)	0.57339 (18)	0.0521 (5)
C2	0.2711 (3)	0.5368 (3)	0.5017 (2)	0.0624 (5)
H2B	0.3056	0.4455	0.5327	0.075*
C3	0.1780 (3)	0.4928 (3)	0.3836 (2)	0.0685 (6)
H3A	0.1489	0.3691	0.3317	0.082*
C4	0.1262 (3)	0.6256 (3)	0.3393 (2)	0.0675 (6)
H4A	0.0605	0.5910	0.2580	0.081*
C5	0.1682 (3)	0.8068 (3)	0.41115 (19)	0.0603 (5)
H5A	0.1332	0.8977	0.3800	0.072*
C6	0.2625 (3)	0.8541 (3)	0.52964 (18)	0.0506 (4)
C7	0.3201 (3)	1.0391 (3)	0.62121 (19)	0.0555 (5)
C8	0.4943 (3)	1.1887 (3)	0.8376 (2)	0.0770 (7)
H8A	0.5302	1.3073	0.8103	0.092*
H8B	0.6178	1.1805	0.8731	0.092*
C9	0.3700 (4)	1.2038 (4)	0.9357 (2)	0.0828 (7)
H9A	0.3341	1.0851	0.9628	0.099*
H9B	0.4509	1.3101	1.0061	0.099*
C10	0.1870 (4)	1.2355 (4)	0.9009 (3)	0.0896 (8)

H10A	0.0964	1.1205	0.8392	0.108*
H10B	0.2196	1.3434	0.8629	0.108*
C11	0.0818 (5)	1.2781 (4)	1.0081 (3)	0.1019 (9)
H11A	−0.0369	1.2987	0.9794	0.153*
H11B	0.1698	1.3930	1.0690	0.153*
H11C	0.0449	1.1701	1.0446	0.153*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0564 (3)	0.0666 (4)	0.0714 (4)	0.0257 (3)	0.0102 (2)	0.0377 (3)
O1	0.0874 (11)	0.0866 (11)	0.0758 (10)	0.0256 (9)	0.0202 (8)	0.0509 (9)
O2	0.0591 (9)	0.0988 (12)	0.1038 (12)	0.0374 (8)	0.0022 (8)	0.0426 (10)
O3	0.0906 (11)	0.0589 (9)	0.0971 (12)	0.0388 (8)	0.0214 (9)	0.0387 (8)
N1	0.0593 (10)	0.0567 (10)	0.0665 (10)	0.0210 (8)	0.0087 (8)	0.0232 (8)
C1	0.0454 (10)	0.0560 (11)	0.0688 (12)	0.0231 (8)	0.0181 (8)	0.0338 (9)
C2	0.0565 (11)	0.0555 (11)	0.0927 (16)	0.0284 (9)	0.0287 (11)	0.0358 (11)
C3	0.0599 (13)	0.0654 (13)	0.0783 (15)	0.0195 (10)	0.0227 (11)	0.0166 (11)
C4	0.0589 (12)	0.0796 (15)	0.0638 (13)	0.0218 (11)	0.0142 (10)	0.0228 (11)
C5	0.0533 (11)	0.0721 (13)	0.0695 (13)	0.0275 (10)	0.0156 (9)	0.0368 (11)
C6	0.0439 (9)	0.0530 (10)	0.0669 (11)	0.0209 (8)	0.0164 (8)	0.0319 (9)
C7	0.0512 (11)	0.0544 (11)	0.0736 (13)	0.0235 (9)	0.0196 (9)	0.0322 (9)
C8	0.0637 (14)	0.0725 (15)	0.0824 (16)	0.0144 (11)	0.0050 (12)	0.0142 (12)
C9	0.0883 (17)	0.0833 (16)	0.0685 (14)	0.0276 (14)	−0.0029 (12)	0.0141 (12)
C10	0.0831 (17)	0.0983 (19)	0.0853 (17)	0.0308 (15)	0.0070 (14)	0.0255 (14)
C11	0.104 (2)	0.095 (2)	0.107 (2)	0.0410 (17)	0.0282 (18)	0.0150 (16)

Geometric parameters (Å, º)

S1—O1	1.4243 (15)	C5—C6	1.383 (3)
S1—O2	1.4265 (16)	C5—H5A	0.9500
S1—N1	1.6661 (17)	C6—C7	1.476 (3)
S1—C1	1.747 (2)	C8—C9	1.503 (3)
O3—C7	1.210 (2)	C8—H8A	0.9900
N1—C7	1.383 (3)	C8—H8B	0.9900
N1—C8	1.470 (3)	C9—C10	1.481 (4)
C1—C2	1.384 (3)	C9—H9A	0.9900
C1—C6	1.386 (2)	C9—H9B	0.9900
C2—C3	1.381 (3)	C10—C11	1.527 (4)
C2—H2B	0.9500	C10—H10A	0.9900
C3—C4	1.383 (3)	C10—H10B	0.9900
C3—H3A	0.9500	C11—H11A	0.9800
C4—C5	1.375 (3)	C11—H11B	0.9800
C4—H4A	0.9500	C11—H11C	0.9800
Cg1···Cg1^{i}	3.778 (2)		
O1—S1—O2	117.55 (10)	O3—C7—N1	123.7 (2)

O1—S1—N1	109.67 (10)	O3—C7—C6	126.81 (19)
O2—S1—N1	109.17 (10)	N1—C7—C6	109.45 (16)
O1—S1—C1	111.98 (10)	N1—C8—C9	115.25 (19)
O2—S1—C1	112.60 (10)	N1—C8—H8A	108.5
N1—S1—C1	93.09 (9)	C9—C8—H8A	108.5
C7—N1—C8	123.88 (18)	N1—C8—H8B	108.5
C7—N1—S1	114.58 (14)	C9—C8—H8B	108.5
C8—N1—S1	120.69 (15)	H8A—C8—H8B	107.5
C2—C1—C6	121.99 (19)	C10—C9—C8	115.6 (2)
C2—C1—S1	128.18 (16)	C10—C9—H9A	108.4
C6—C1—S1	109.81 (15)	C8—C9—H9A	108.4
C3—C2—C1	117.30 (19)	C10—C9—H9B	108.4
C3—C2—H2B	121.3	C8—C9—H9B	108.4
C1—C2—H2B	121.3	H9A—C9—H9B	107.4
C2—C3—C4	121.1 (2)	C9—C10—C11	113.4 (2)
C2—C3—H3A	119.4	C9—C10—H10A	108.9
C4—C3—H3A	119.4	C11—C10—H10A	108.9
C5—C4—C3	121.1 (2)	C9—C10—H10B	108.9
C5—C4—H4A	119.4	C11—C10—H10B	108.9
C3—C4—H4A	119.4	H10A—C10—H10B	107.7
C4—C5—C6	118.64 (19)	C10—C11—H11A	109.5
C4—C5—H5A	120.7	C10—C11—H11B	109.5
C6—C5—H5A	120.7	H11A—C11—H11B	109.5
C5—C6—C1	119.82 (19)	C10—C11—H11C	109.5
C5—C6—C7	127.23 (17)	H11A—C11—H11C	109.5
C1—C6—C7	112.96 (17)	H11B—C11—H11C	109.5

O1—S1—N1—C7	−117.32 (15)	C4—C5—C6—C7	−179.82 (17)
O2—S1—N1—C7	112.58 (16)	C2—C1—C6—C5	0.2 (3)
C1—S1—N1—C7	−2.65 (15)	S1—C1—C6—C5	−178.69 (14)
O1—S1—N1—C8	72.84 (17)	C2—C1—C6—C7	179.99 (16)
O2—S1—N1—C8	−57.26 (18)	S1—C1—C6—C7	1.14 (19)
C1—S1—N1—C8	−172.49 (16)	C8—N1—C7—O3	−7.0 (3)
O1—S1—C1—C2	−65.33 (19)	S1—N1—C7—O3	−176.51 (15)
O2—S1—C1—C2	69.76 (19)	C8—N1—C7—C6	173.10 (17)
N1—S1—C1—C2	−177.99 (17)	S1—N1—C7—C6	3.63 (19)
O1—S1—C1—C6	113.43 (14)	C5—C6—C7—O3	−3.0 (3)
O2—S1—C1—C6	−111.48 (14)	C1—C6—C7—O3	177.15 (18)
N1—S1—C1—C6	0.77 (14)	C5—C6—C7—N1	176.82 (17)
C6—C1—C2—C3	0.2 (3)	C1—C6—C7—N1	−3.0 (2)
S1—C1—C2—C3	178.82 (14)	C7—N1—C8—C9	101.9 (2)
C1—C2—C3—C4	−0.7 (3)	S1—N1—C8—C9	−89.2 (2)
C2—C3—C4—C5	0.9 (3)	N1—C8—C9—C10	−63.7 (3)
C3—C4—C5—C6	−0.5 (3)	C8—C9—C10—C11	−171.7 (2)
C4—C5—C6—C1	0.0 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O3^{ii}	0.95	2.35	3.279 (2)	165

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