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3-Benzylamino-1,2-benzisothiazole 1,1-dioxide

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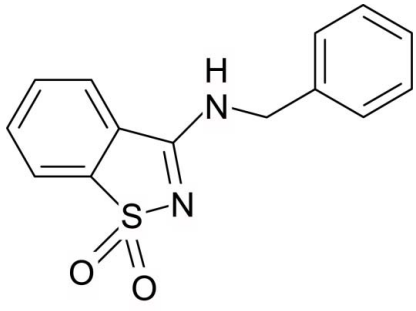
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.092; data-to-parameter ratio = 15.9.

The structure of the title compound, $C_{14}H_{12}N_2O_2S$, contains an essentially planar benzisothiazole ring system which is inclined at 76.52 (5)° with respect to the phenyl ring. The molecules are linked into chains along the b axis by N—H···O hydrogen bonds.

Related literature

For related literature, see: Brigas *et al.* (2001); Cowan *et al.* (2000); Delclaux *et al.* (1996); Groutas *et al.* (1993); Janoff (1985); Kapui *et al.* (2003); Lee *et al.* (1981); Llewellyn-Jones *et al.* (1996); Piccioni *et al.* (1992); Siddiqui *et al.* (2006, 2007); Varga *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{12}N_2O_2S$
M_r = 272.32
Monoclinic, $P2_1/c$
a = 7.061 (3) Å
b = 7.052 (2) Å
c = 24.959 (11) Å
β = 93.997 (18)°
V = 1239.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 173 (2) K
0.10 × 0.09 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.975, T_{max} = 0.980
4760 measured reflections
2790 independent reflections
2268 reflections with $(I) > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.092
S = 1.04
2790 reflections
175 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.85 (2)	2.12 (2)	2.958 (2)	166 (2)

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

Data collection: *COLLECT* (Hooft, 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: WN2201).

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supplementary materials

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3-Benzylamino-1,2-benzisothiazole 1,1-dioxide

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Comment

The interest in obtaining new derivatives of saccharin has not diminished in recent times. Saccharin derivatives are considered to be the most potent orally active human leucocyte elastase (HLE) inhibitors (Kapui *et al.*, 2003). HLE belongs to the chymotrypsin family of serine proteinases that aids in the migration of neutrophils from blood to various tissues such as the airways in response to chemotactic factors. It is capable of degrading a variety of proteins, including different types of collagens and structural matrix proteins (Delclaux *et al.*, 1996). In a number of pulmonary pathophysiological states relative insufficiency of endogenous elastase inhibitors may result in severe conditions, such as pulmonary emphysema, adult respiratory distress syndrome (ARDS), chronic bronchitis, chronic obstructive pulmonary disease (COPD), pulmonary hypertension and other inflammatory diseases (Janoff, 1985; Lee *et al.*, 1981; Llewellyn-Jones *et al.*, 1996; Piccioni *et al.*, 1992; Cowan *et al.*, 2000). The inhibitors of HLE may provide a way for alleviating these diseases whereas, saccharin derivatives are well recognized to be such agents which are orally active (Groutas *et al.*, 1993; Varga *et al.*, 2003).

In continuation of our investigation of the chemistry of saccharin and its derivatives (Siddiqui *et al.*, 2006, 2007), we have synthesized the title compound, 3-benzylamino-1,2-benzisothiazole 1,1-dioxide, to utilize it as a precursor for the synthesis of new saccharin derivatives. In this paper, its structure is described. The structures of two closely related compounds have been reported (Brigas *et al.*, 2001).

In the structure (Fig. 1) the benzisothiazole ring system is essentially planar; the maximum deviation of any atom from the mean plane through S1/N2/C1—C7 being 0.022 (1) Å for atom C7. The phenyl ring (C9—C14) is inclined at 76.52 (5)° with respect to the benzisothiazole ring system. The molecules are linked *via* N—H···O hydrogen bonds, resulting in chains along the *b* axis (Fig. 2).

Experimental

A mixture of saccharin (1.0 g, 5.46 mmol) and benzylamine (5 ml, in excess) was heated to reflux on an oil-bath (4 hrs), cooled to room temperature and kept overnight in a freezer. The solvent was evaporated under reduced pressure and the yellow paste obtained was washed with petroleum ether (4 *x* 25 ml) to obtain the bright yellow title product (1.17 g, 78%) which was recrystallized from a mixture of MeOH:AcOEt (1:1) by slow evaporation at room temperature to obtain light yellow crystals. m.p 482–483 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 H atom bonded to N1 was refined freely, with U_{iso}(H) = 1.2U_{eq}(N); N—H = 0.85 (2) Å. The final difference map was free of any chemically significant features.

Figures

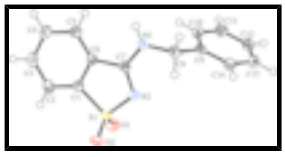

Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound, with displacement ellipsoids drawn at the 50% probability level.

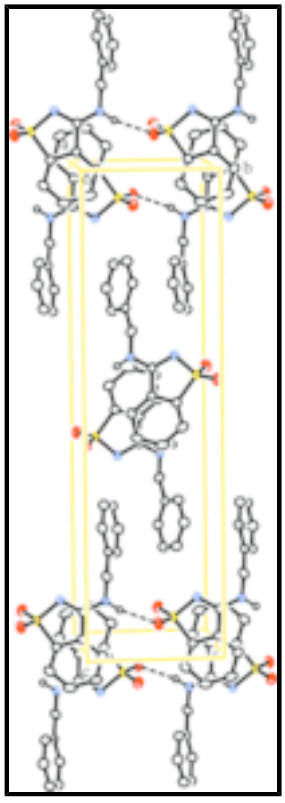

Fig. 2. Unit cell packing of the title compound, showing the N—H···O hydrogen bonds as dashed lines. Only those H atoms involved in hydrogen bonding are shown.

(I)

Crystal data

$C_{14}H_{12}N_2O_2S$	F_{000} = 568
M_r = 272.32	D_x = 1.459 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 482-483 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.061 (3) Å	Cell parameters from 4760 reflections
b = 7.052 (2) Å	θ = 4.1–27.5º
c = 24.959 (11) Å	μ = 0.26 mm^{-1}
β = 93.997 (18)º	T = 173 (2) K
V = 1239.8 (8) Å^3	Prism, colorless
Z = 4	0.10 × 0.09 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer	2790 independent reflections
Radiation source: fine-focus sealed tube	2268 reflections with $(I) > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 173(2)$ K	$\theta_{max} = 27.5°$
ω and φ scans	$\theta_{min} = 4.1°$
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	$h = -9\rightarrow9$
$T_{min} = 0.975$, $T_{max} = 0.980$	$k = -8\rightarrow9$
4760 measured reflections	$l = -32\rightarrow32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.5721P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2790 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
175 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. IR (Neat, ν_{max}, cm^{-1}): NH 3318 (*s*), C=N 1621, SO_2 1280 and 1149; ^{1}H-NMR (300 MHz, acetone-d_6) δ: 4.82 (s, 2H, CH_2), 7.31–7.47 (m, 3H), 7.48–7.50 (m, 2H), 7.79–7.95 (m, 2H), 7.96–7.97 (d, J = 7.5, 1H), 8.10–8.18 (d, J = 7.5, 1H), 8.80 (s, 1H, NH); ^{13}C-NMR δ: 206.2, 160.6, 144.2, 137.9, 134.1, 133.6, 129.4, 128.9, 128.8, 128.5, 123.1, 122.0, 47.4 LRMS (ES^+): m/*z*: 273 [*M* + H]+ (17.2%), 336 $[M + Na + MeCN]^+$ (59.3%), 567 $[M + Na]^+$ (100.0%), 839 $[3M + Na]^+$ (25.9%).

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	0.77714 (6)	0.34415 (5)	0.073954 (16)	0.02266 (12)
O1	0.60219 (17)	0.43540 (16)	0.08534 (5)	0.0310 (3)
O2	0.93690 (17)	0.47104 (15)	0.07037 (5)	0.0295 (3)
N1	0.8855 (2)	−0.14795 (19)	0.11674 (5)	0.0239 (3)

H1N	0.891 (3)	−0.250 (3)	0.0983 (7)	0.029*
N2	0.83151 (19)	0.17576 (17)	0.11673 (5)	0.0226 (3)
C1	0.7469 (2)	0.1995 (2)	0.01616 (6)	0.0211 (3)
C2	0.6989 (2)	0.2474 (2)	−0.03662 (6)	0.0272 (4)
H2	0.6778	0.3754	−0.0473	0.033*
C3	0.6828 (2)	0.0990 (3)	−0.07339 (7)	0.0315 (4)
H3	0.6488	0.1258	−0.1101	0.038*
C4	0.7157 (2)	−0.0880 (3)	−0.05744 (7)	0.0304 (4)
H4	0.7036	−0.1864	−0.0834	0.036*
C5	0.7659 (2)	−0.1331 (2)	−0.00401 (7)	0.0258 (3)
H5	0.7896	−0.2605	0.0068	0.031*
C6	0.7803 (2)	0.0139 (2)	0.03276 (6)	0.0197 (3)
C7	0.8337 (2)	0.0111 (2)	0.09166 (6)	0.0200 (3)
C8	0.9706 (2)	−0.1511 (2)	0.17166 (6)	0.0254 (3)
H8A	1.0398	−0.0305	0.1785	0.031*
H8B	1.0650	−0.2549	0.1748	0.031*
C9	0.8327 (2)	−0.1775 (2)	0.21492 (6)	0.0225 (3)
C10	0.6538 (2)	−0.2560 (2)	0.20472 (7)	0.0272 (4)
H10	0.6122	−0.2921	0.1692	0.033*
C11	0.5347 (3)	−0.2822 (2)	0.24633 (7)	0.0311 (4)
H11	0.4117	−0.3346	0.2390	0.037*
C12	0.5951 (3)	−0.2324 (2)	0.29831 (7)	0.0316 (4)
H12	0.5136	−0.2498	0.3266	0.038*
C13	0.7746 (3)	−0.1569 (2)	0.30897 (7)	0.0314 (4)
H13	0.8169	−0.1235	0.3447	0.038*
C14	0.8930 (2)	−0.1300 (2)	0.26747 (7)	0.0266 (4)
H14	1.0163	−0.0787	0.2750	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0269 (2)	0.01635 (19)	0.0247 (2)	0.00192 (15)	0.00152 (15)	0.00038 (15)
O1	0.0328 (7)	0.0267 (6)	0.0337 (7)	0.0102 (5)	0.0051 (5)	−0.0007 (5)
O2	0.0341 (7)	0.0181 (5)	0.0360 (7)	−0.0048 (5)	0.0018 (5)	0.0015 (5)
N1	0.0319 (7)	0.0170 (6)	0.0232 (7)	0.0008 (6)	0.0050 (6)	0.0006 (5)
N2	0.0289 (7)	0.0178 (6)	0.0209 (7)	0.0017 (5)	0.0003 (5)	−0.0001 (5)
C1	0.0185 (7)	0.0230 (7)	0.0220 (8)	−0.0001 (6)	0.0029 (6)	0.0006 (6)
C2	0.0230 (8)	0.0323 (9)	0.0263 (9)	0.0025 (7)	0.0017 (6)	0.0070 (7)
C3	0.0249 (8)	0.0498 (11)	0.0198 (8)	−0.0021 (8)	0.0020 (6)	0.0025 (8)
C4	0.0276 (9)	0.0411 (10)	0.0229 (8)	−0.0065 (8)	0.0045 (7)	−0.0080 (7)
C5	0.0260 (8)	0.0253 (8)	0.0269 (8)	−0.0046 (7)	0.0064 (6)	−0.0022 (7)
C6	0.0173 (7)	0.0216 (7)	0.0206 (8)	−0.0027 (6)	0.0042 (6)	0.0002 (6)
C7	0.0191 (7)	0.0191 (7)	0.0222 (8)	−0.0014 (6)	0.0052 (6)	0.0004 (6)
C8	0.0264 (8)	0.0242 (8)	0.0255 (8)	0.0023 (6)	0.0001 (6)	0.0055 (6)
C9	0.0277 (8)	0.0159 (7)	0.0238 (8)	0.0028 (6)	0.0018 (6)	0.0031 (6)
C10	0.0316 (9)	0.0273 (8)	0.0224 (8)	−0.0026 (7)	−0.0002 (7)	−0.0025 (7)
C11	0.0299 (9)	0.0298 (8)	0.0338 (9)	−0.0057 (7)	0.0046 (7)	−0.0018 (8)
C12	0.0385 (10)	0.0308 (9)	0.0264 (9)	0.0001 (8)	0.0084 (7)	0.0029 (7)

C13	0.0417 (10)	0.0299 (9)	0.0222 (8)	0.0020 (8)	−0.0011 (7)	0.0008 (7)
C14	0.0285 (9)	0.0237 (8)	0.0267 (9)	−0.0016 (7)	−0.0045 (6)	0.0015 (7)

Geometric parameters (Å, °)

S1—O1	1.4387 (13)	C5—H5	0.9500
S1—O2	1.4475 (12)	C6—C7	1.492 (2)
S1—N2	1.6250 (13)	C8—C9	1.515 (2)
S1—C1	1.7673 (16)	C8—H8A	0.9900
N1—C7	1.324 (2)	C8—H8B	0.9900
N1—C8	1.458 (2)	C9—C10	1.386 (2)
N1—H1N	0.854 (19)	C9—C14	1.391 (2)
N2—C7	1.3198 (19)	C10—C11	1.393 (2)
C1—C2	1.379 (2)	C10—H10	0.9500
C1—C6	1.388 (2)	C11—C12	1.383 (2)
C2—C3	1.391 (3)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.383 (3)
C3—C4	1.392 (3)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.389 (2)
C4—C5	1.393 (2)	C13—H13	0.9500
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.384 (2)		
O1—S1—O2	114.79 (7)	N2—C7—N1	122.22 (14)
O1—S1—N2	111.33 (7)	N2—C7—C6	116.43 (13)
O2—S1—N2	110.06 (7)	N1—C7—C6	121.32 (13)
O1—S1—C1	111.28 (7)	N1—C8—C9	115.40 (14)
O2—S1—C1	110.88 (7)	N1—C8—H8A	108.4
N2—S1—C1	97.13 (7)	C9—C8—H8A	108.4
C7—N1—C8	122.67 (14)	N1—C8—H8B	108.4
C7—N1—H1N	118.5 (12)	C9—C8—H8B	108.4
C8—N1—H1N	117.7 (12)	H8A—C8—H8B	107.5
C7—N2—S1	109.96 (11)	C10—C9—C14	118.96 (15)
C2—C1—C6	122.76 (15)	C10—C9—C8	122.75 (15)
C2—C1—S1	130.25 (13)	C14—C9—C8	118.20 (15)
C6—C1—S1	106.99 (11)	C9—C10—C11	120.38 (15)
C1—C2—C3	116.71 (16)	C9—C10—H10	119.8
C1—C2—H2	121.6	C11—C10—H10	119.8
C3—C2—H2	121.6	C12—C11—C10	120.18 (16)
C2—C3—C4	121.30 (16)	C12—C11—H11	119.9
C2—C3—H3	119.4	C10—C11—H11	119.9
C4—C3—H3	119.4	C13—C12—C11	119.79 (16)
C3—C4—C5	121.08 (16)	C13—C12—H12	120.1
C3—C4—H4	119.5	C11—C12—H12	120.1
C5—C4—H4	119.5	C12—C13—C14	120.02 (16)
C6—C5—C4	117.79 (15)	C12—C13—H13	120.0
C6—C5—H5	121.1	C14—C13—H13	120.0
C4—C5—H5	121.1	C13—C14—C9	120.65 (16)
C5—C6—C1	120.36 (14)	C13—C14—H14	119.7
C5—C6—C7	130.19 (14)	C9—C14—H14	119.7

C1—C6—C7	109.43 (13)		
O1—S1—N2—C7	117.29 (12)	S1—N2—C7—N1	175.66 (12)
O2—S1—N2—C7	−114.26 (12)	S1—N2—C7—C6	−2.39 (17)
C1—S1—N2—C7	1.09 (12)	C8—N1—C7—N2	−9.3 (2)
O1—S1—C1—C2	63.92 (16)	C8—N1—C7—C6	168.69 (14)
O2—S1—C1—C2	−65.15 (16)	C5—C6—C7—N2	−178.74 (16)
N2—S1—C1—C2	−179.84 (15)	C1—C6—C7—N2	2.80 (19)
O1—S1—C1—C6	−115.74 (11)	C5—C6—C7—N1	3.2 (2)
O2—S1—C1—C6	115.19 (11)	C1—C6—C7—N1	−175.27 (14)
N2—S1—C1—C6	0.50 (12)	C7—N1—C8—C9	92.95 (18)
C6—C1—C2—C3	0.7 (2)	N1—C8—C9—C10	21.1 (2)
S1—C1—C2—C3	−178.95 (13)	N1—C8—C9—C14	−162.43 (14)
C1—C2—C3—C4	−0.6 (2)	C14—C9—C10—C11	1.6 (2)
C2—C3—C4—C5	0.0 (3)	C8—C9—C10—C11	178.07 (15)
C3—C4—C5—C6	0.6 (2)	C9—C10—C11—C12	−0.8 (3)
C4—C5—C6—C1	−0.6 (2)	C10—C11—C12—C13	−0.3 (3)
C4—C5—C6—C7	−178.89 (15)	C11—C12—C13—C14	0.5 (3)
C2—C1—C6—C5	−0.1 (2)	C12—C13—C14—C9	0.3 (3)
S1—C1—C6—C5	179.61 (12)	C10—C9—C14—C13	−1.4 (2)
C2—C1—C6—C7	178.56 (14)	C8—C9—C14—C13	−177.96 (15)
S1—C1—C6—C7	−1.75 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2^{i}	0.85 (2)	2.12 (2)	2.958 (2)	166 (2)

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

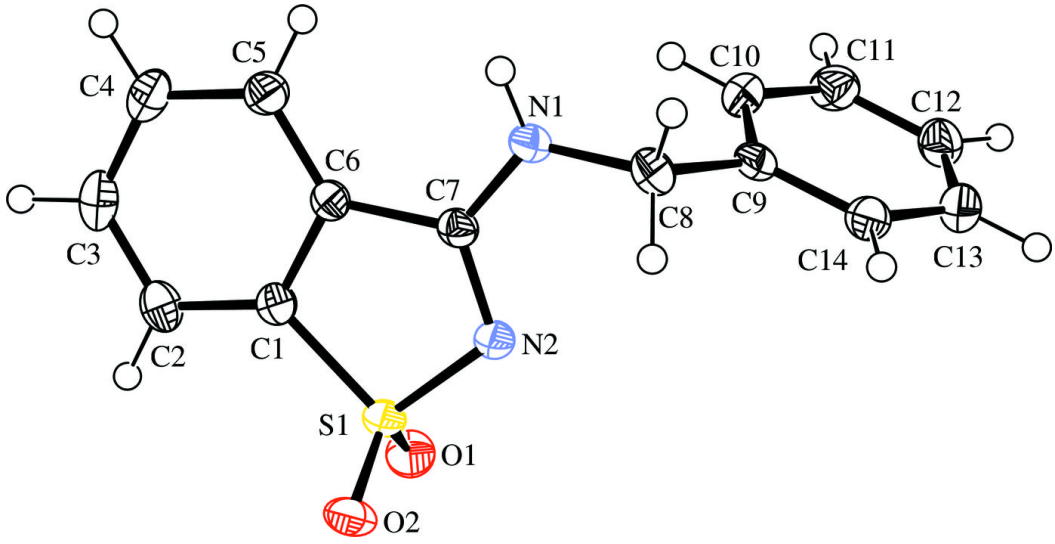

Fig. 2