Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Prop-2-enyl)-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide

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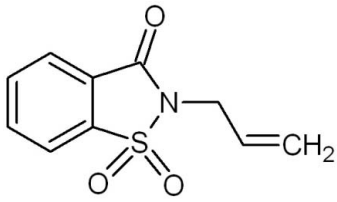
Received 29 April 2009; accepted 30 April 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.118; data-to-parameter ratio = 17.2.

In the title compound, $C_{10}H_9NO_3S$, the benzisothiazole group is almost planar (with a maximum deviation of 1.61 Å). The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds, forming a chain of molecules along b.

Related literature

For the synthesis of benzothiazine and benzisothiazol derivatives, see: Zia-ur-Rehman, Anwar & Ahmad (2006); Zia-ur-Rehman, Anwar, Ahmad & Siddiqui (2006); Siddiqui *et al.* (2007) Zia-ur-Rehman *et al.* (2009). For the biological activity of benzisothiazols, see: Kapui *et al.* (2003); Liang *et al.* (2006). For related structures, see: Siddiqui, Ahmad, Siddiqui *et al.* (2007*a*,*b*,*c*).



Experimental

Crystal data

$C_{10}H_9NO_3S$
M_r = 223.24
Triclinic, $P\overline{1}$
a = 7.2169 (8) Å
b = 7.8347 (7) Å
c = 10.3849 (12) Å
α = 105.530 (3)°
β = 91.586 (3)°
γ = 112.047 (3)°
V = 518.95 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 K
0.37 × 0.26 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
5460 measured reflections
2342 independent reflections
1728 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.118
S = 1.06
2342 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.93	2.36	3.216 (3)	153

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON*.

The authors are grateful to the Higher Education Commission of Pakistan for financial support to purchase the diffractometer. MNA acknowledges the Higher Education Commission, Pakistan, for providing a PhD Scholarship under PIN 042-120607-PS2–183.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2942).

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supporting information

Acta Cryst. (2009). E**65**, o1236 [doi:10.1107/S1600536809016328]

2-(Prop-2-enyl)-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide

Muhammad Nadeem Arshad, Hafiz Mubashar-ur-Rehman, Muhammad Zia-ur-Rehman, Islam Ullah Khan and Muhammad Shafiq

S1. Comment

Besides being used as a sweetener, saccharin and its various derivatives are well known for their different type of biological activities *e.g.*, it has been identified as an important molecular component in various classes of 5-HTla antagonists, analgesics and human mast cell tryptase inhibitors (Kapui *et al.*, 2003; Liang *et al.*, 2006). *N*-alkyl derivatives of saccharin have been successfully transformed to non-steroidal anti-inflammatory drugs *e.g.*, piroxicam and meloxicam.

As part of a research program synthesizing various bioactive benzothiazines (Zia-ur-Rehman *et al.*, 2009; Siddiqui *et al.*, 2007), we have in addition, worked on the synthesis of benzisothiazole derivatives. We herein report the crystal structure of the title compound (Scheme and figure 1). The benzisothiazole moiety is exactly planar. The molecular dimensions are in accord with the corresponding dimensions reported in similar structures (Siddiqui, Ahmad, Siddiqui *et al.*, 2007*a*; Siddiqui, Ahmad, Siddiqui *et al.*, 2007*b*; Siddiqui, Ahmad, Siddiqui *et al.*, 2007*c*). Each molecule is linked to its adjacent one through C—H···O contacts forming a chain of molecules along *b* (Figure 2).

S2. Experimental

A mixture of 2,3-dihydro-1,2-benzisothiazol-3-one-1,1-dioxide (1.83 g, 10.0 mmoles), dimethyl formamide (5.0 ml) and allyl bromide (1.20 g, 10.0 mmoles) was stirred for a period of one hour at 90°C. Contents were cooled to room temperature; poured over crushed ice to get white coloured precipitates which were filtered, washed and dried. Crystallization of the white precipitate in methanol afforded suitable crystals for X-ray studies.

S3. Refinement

H atoms were placed in geometric positions (C—H distance = 0.93 to 0.96 Å) using a riding model with U_{iso}(H) = 1.2 U_{eq}(C).

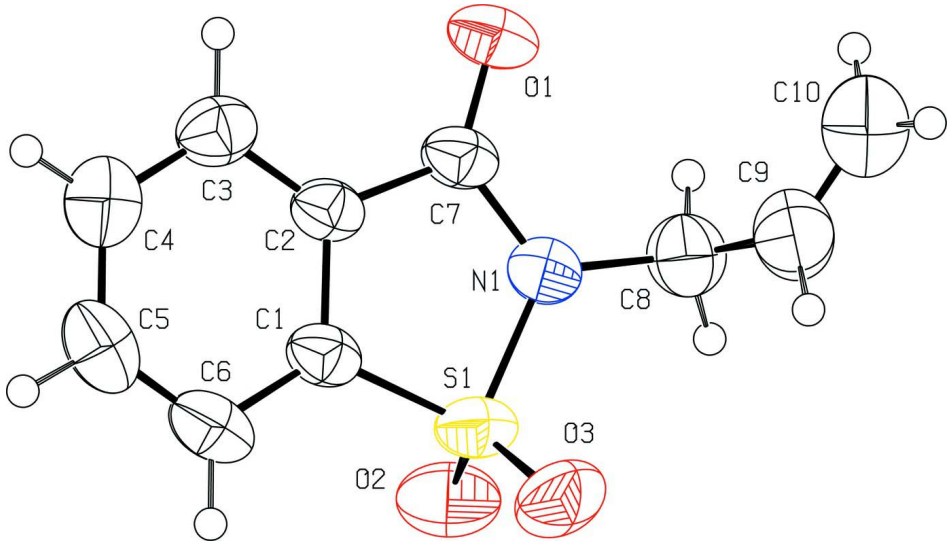

Figure 1

The molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

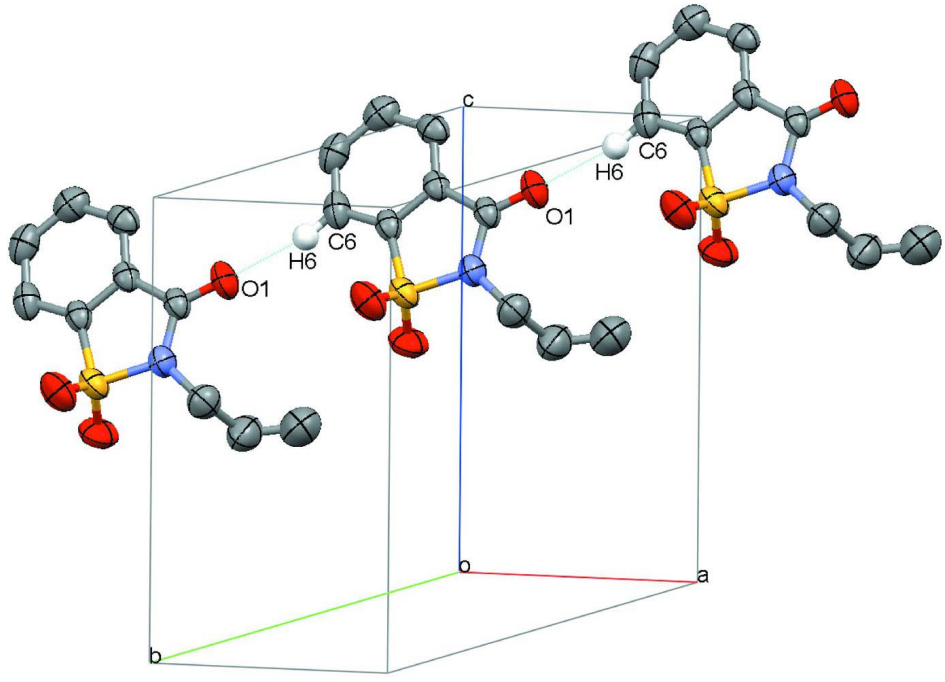

Figure 2

Perspective view of the crystal packing showing hydrogen-bonded interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

2-(Prop-2-enyl)-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide

Crystal data

$C_{10}H_9NO_3S$
$M_r = 223.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.2169\ (8)$ Å
$b = 7.8347\ (7)$ Å

c = 10.3849 (12) Å
α = 105.530 (3)°
β = 91.586 (3)°
γ = 112.047 (3)°
V = 518.95 (10) Å^3
Z = 2
F(000) = 232
D_x = 1.429 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2362 reflections
θ = 3.1–27.3°
μ = 0.30 mm^{-1}
T = 296 K
Needles, colourless
0.37 × 0.26 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
5460 measured reflections
2342 independent reflections

1728 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −9→9
k = −10→6
l = −11→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.118
S = 1.06
2342 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1101P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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	0.39784 (8)	0.35030 (7)	0.26041 (5)	0.0536 (2)
O1	0.2640 (2)	0.6659 (2)	0.09710 (17)	0.0646 (4)
O2	0.6073 (2)	0.3953 (2)	0.29080 (18)	0.0759 (5)
O3	0.2677 (3)	0.2648 (2)	0.34679 (16)	0.0726 (5)
N1	0.3652 (3)	0.5452 (2)	0.25013 (17)	0.0515 (4)
C1	0.3079 (3)	0.2289 (3)	0.0897 (2)	0.0450 (4)
C2	0.2602 (3)	0.3453 (3)	0.0270 (2)	0.0431 (4)
C3	0.1889 (3)	0.2789 (3)	−0.1090 (2)	0.0533 (5)
H3	0.1557	0.3555	−0.1521	0.064*
C4	0.1684 (3)	0.0951 (3)	−0.1791 (2)	0.0635 (6)

H4	0.1223	0.0479	−0.2713	0.076*
C5	0.2147 (3)	−0.0205 (3)	−0.1155 (3)	0.0645 (6)
H5	0.1983	−0.1442	−0.1656	0.077*
C6	0.2844 (3)	0.0433 (3)	0.0204 (2)	0.0572 (6)
H6	0.3145	−0.0347	0.0636	0.069*
C7	0.2931 (3)	0.5357 (3)	0.1224 (2)	0.0463 (5)
C8	0.4052 (4)	0.7114 (3)	0.3697 (2)	0.0642 (6)
H8A	0.4591	0.8293	0.3443	0.077*
H8B	0.5060	0.7163	0.4359	0.077*
C9	0.2176 (5)	0.6993 (4)	0.4312 (3)	0.0823 (8)
H9	0.1606	0.5989	0.4683	0.099*
C10	0.1299 (5)	0.8116 (5)	0.4373 (3)	0.0945 (9)
H10A	0.1814	0.9141	0.4015	0.113*
H10B	0.0134	0.7926	0.4777	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0584 (3)	0.0538 (3)	0.0598 (4)	0.0253 (2)	0.0088 (2)	0.0306 (3)
O1	0.0772 (10)	0.0506 (8)	0.0818 (11)	0.0342 (7)	0.0108 (8)	0.0319 (8)
O2	0.0616 (10)	0.0850 (11)	0.0897 (12)	0.0328 (8)	−0.0054 (9)	0.0362 (10)
O3	0.0904 (12)	0.0726 (10)	0.0647 (10)	0.0278 (9)	0.0214 (9)	0.0418 (9)
N1	0.0612 (10)	0.0455 (9)	0.0537 (10)	0.0248 (8)	0.0095 (8)	0.0187 (8)
C1	0.0420 (10)	0.0435 (9)	0.0588 (12)	0.0201 (8)	0.0149 (9)	0.0249 (9)
C2	0.0391 (10)	0.0428 (9)	0.0554 (11)	0.0178 (8)	0.0139 (8)	0.0243 (9)
C3	0.0468 (11)	0.0607 (12)	0.0572 (13)	0.0207 (9)	0.0085 (9)	0.0262 (10)
C4	0.0540 (13)	0.0652 (14)	0.0616 (14)	0.0180 (11)	0.0098 (11)	0.0117 (11)
C5	0.0568 (13)	0.0470 (12)	0.0819 (17)	0.0195 (10)	0.0182 (12)	0.0080 (11)
C6	0.0514 (12)	0.0468 (11)	0.0845 (17)	0.0249 (9)	0.0194 (11)	0.0285 (11)
C7	0.0452 (10)	0.0429 (10)	0.0600 (12)	0.0198 (8)	0.0134 (9)	0.0259 (9)
C8	0.0702 (15)	0.0559 (12)	0.0604 (14)	0.0234 (11)	0.0031 (11)	0.0108 (11)
C9	0.111 (2)	0.0722 (16)	0.0749 (18)	0.0451 (16)	0.0323 (16)	0.0258 (14)
C10	0.109 (2)	0.098 (2)	0.0787 (19)	0.0495 (19)	0.0176 (17)	0.0177 (17)

Geometric parameters (Å, º)

S1—O2	1.4220 (16)	C4—C5	1.379 (3)
S1—O3	1.4253 (15)	C4—H4	0.9300
S1—N1	1.6596 (16)	C5—C6	1.374 (3)
S1—C1	1.743 (2)	C5—H5	0.9300
O1—C7	1.206 (2)	C6—H6	0.9300
N1—C7	1.385 (3)	C8—C9	1.495 (3)
N1—C8	1.467 (3)	C8—H8A	0.9700
C1—C6	1.382 (3)	C8—H8B	0.9700
C1—C2	1.384 (2)	C9—C10	1.253 (4)
C2—C3	1.376 (3)	C9—H9	0.9300
C2—C7	1.481 (3)	C10—H10A	0.9300
C3—C4	1.378 (3)	C10—H10B	0.9300

C3—H3	0.9300		
O2—S1—O3	117.16 (10)	C6—C5—C4	121.4 (2)
O2—S1—N1	109.80 (9)	C6—C5—H5	119.3
O3—S1—N1	109.80 (9)	C4—C5—H5	119.3
O2—S1—C1	111.86 (10)	C5—C6—C1	116.9 (2)
O3—S1—C1	112.76 (9)	C5—C6—H6	121.5
N1—S1—C1	92.73 (8)	C1—C6—H6	121.5
C7—N1—C8	123.33 (17)	O1—C7—N1	123.46 (19)
C7—N1—S1	115.04 (13)	O1—C7—C2	127.23 (19)
C8—N1—S1	121.60 (14)	N1—C7—C2	109.31 (15)
C6—C1—C2	122.1 (2)	N1—C8—C9	111.41 (19)
C6—C1—S1	127.33 (16)	N1—C8—H8A	109.3
C2—C1—S1	110.60 (14)	C9—C8—H8A	109.3
C3—C2—C1	120.34 (18)	N1—C8—H8B	109.3
C3—C2—C7	127.38 (17)	C9—C8—H8B	109.3
C1—C2—C7	112.27 (17)	H8A—C8—H8B	108.0
C2—C3—C4	117.8 (2)	C10—C9—C8	126.1 (3)
C2—C3—H3	121.1	C10—C9—H9	116.9
C4—C3—H3	121.1	C8—C9—H9	116.9
C3—C4—C5	121.5 (2)	C9—C10—H10A	120.0
C3—C4—H4	119.3	C9—C10—H10B	120.0
C5—C4—H4	119.3	H10A—C10—H10B	120.0
O2—S1—N1—C7	112.66 (16)	C7—C2—C3—C4	−179.51 (17)
O3—S1—N1—C7	−117.16 (15)	C2—C3—C4—C5	0.9 (3)
C1—S1—N1—C7	−1.76 (15)	C3—C4—C5—C6	−0.4 (3)
O2—S1—N1—C8	−69.00 (18)	C4—C5—C6—C1	−0.6 (3)
O3—S1—N1—C8	61.18 (18)	C2—C1—C6—C5	1.2 (3)
C1—S1—N1—C8	176.58 (16)	S1—C1—C6—C5	−178.53 (15)
O2—S1—C1—C6	69.07 (19)	C8—N1—C7—O1	2.9 (3)
O3—S1—C1—C6	−65.5 (2)	S1—N1—C7—O1	−178.81 (15)
N1—S1—C1—C6	−178.31 (17)	C8—N1—C7—C2	−177.24 (16)
O2—S1—C1—C2	−110.65 (14)	S1—N1—C7—C2	1.1 (2)
O3—S1—C1—C2	114.77 (14)	C3—C2—C7—O1	−0.5 (3)
N1—S1—C1—C2	1.96 (14)	C1—C2—C7—O1	−179.66 (19)
C6—C1—C2—C3	−0.7 (3)	C3—C2—C7—N1	179.65 (18)
S1—C1—C2—C3	179.07 (14)	C1—C2—C7—N1	0.5 (2)
C6—C1—C2—C7	178.58 (16)	C7—N1—C8—C9	84.2 (3)
S1—C1—C2—C7	−1.68 (19)	S1—N1—C8—C9	−94.0 (2)
C1—C2—C3—C4	−0.4 (3)	N1—C8—C9—C10	−114.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.36	3.216 (3)	153

Symmetry code: (i) *x*, *y*−1, *z*.