

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

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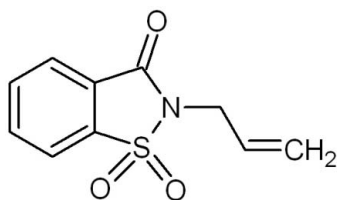
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.118; data-to-parameter ratio = 17.2.

In the title compound,  $\text{C}_{10}\text{H}_9\text{NO}_3\text{S}$ , the benzisothiazole group is almost planar (with a maximum deviation of 1.61 Å). The crystal structure is stabilized by weak intermolecular C—H $\cdots$ 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.* (2007a,b,c).



### Experimental

#### Crystal data

$\text{C}_{10}\text{H}_9\text{NO}_3\text{S}$   
 $M_r = 223.24$   
 Triclinic,  $P\bar{1}$   
 $a = 7.2169$  (8) Å  
 $b = 7.8347$  (7) Å  
 $c = 10.3849$  (12) Å  
 $\alpha = 105.530$  (3)°  
 $\beta = 91.586$  (3)°

$\gamma = 112.047$  (3)°  
 $V = 518.95$  (10) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.30$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.37 \times 0.26 \times 0.18$  mm

#### Data collection

Bruker APEXII CCD area-detector  
 diffractometer  
 Absorption correction: none  
 5460 measured reflections

2342 independent reflections  
 1728 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

#### 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  
 $\Delta\rho_{\text{max}} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O1}^i$	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*.

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

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**supplementary materials**

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

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### 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-HT1a 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.*, 2007a; Siddiqui, Ahmad, Siddiqui *et al.*, 2007b; Siddiqui, Ahmad, Siddiqui *et al.*, 2007c). Each molecule is linked to its adjacent one through C—H $\cdots$ O contacts forming a chain of molecules along *b* (Figure 2).

### 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.

### Refinement

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

### Figures

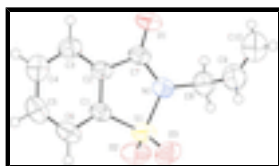


Fig. 1. The molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

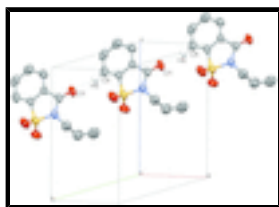


Fig. 2. Perspective view of the crystal packing showing hydrogen-bonded interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

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

### Crystal data

$C_{10}H_9NO_3S$	$Z = 2$
$M_r = 223.24$	$F_{000} = 232$
Triclinic, $P\bar{1}$	$D_x = 1.429 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 7.2169 (8) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 7.8347 (7) \text{ \AA}$	Cell parameters from 2362 reflections
$c = 10.3849 (12) \text{ \AA}$	$\theta = 3.1\text{--}27.3^\circ$
$\alpha = 105.530 (3)^\circ$	$\mu = 0.30 \text{ mm}^{-1}$
$\beta = 91.586 (3)^\circ$	$T = 296 \text{ K}$
$\gamma = 112.047 (3)^\circ$	Needles, colourless
$V = 518.95 (10) \text{ \AA}^3$	$0.37 \times 0.26 \times 0.18 \text{ mm}$

### Data collection

Bruker APEXII CCD area-detector diffractometer	1728 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.022$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 296 \text{ K}$	$\theta_{\text{min}} = 2.9^\circ$
$\varphi$ and $\omega$ scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -10 \rightarrow 6$
5460 measured reflections	$l = -11 \rightarrow 13$
2342 independent reflections	

### 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
$wR(F^2) = 0.118$	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1101P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2342 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
136 parameters	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
	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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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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, °)*

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 ( $\text{\AA}$ ,  $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C6—H6 $\cdots$ O1 <sup>i</sup>	0.93	2.36	3.216 (3)	153

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

Fig. 1

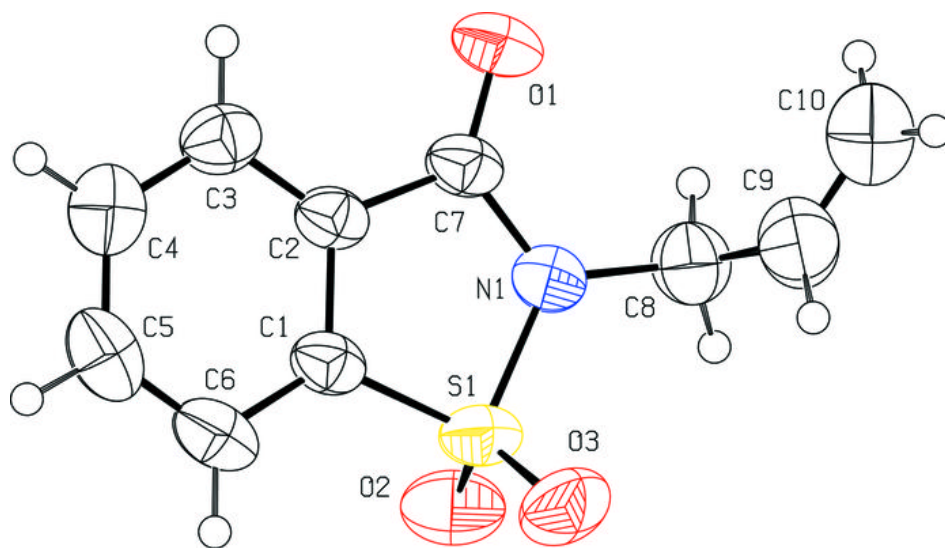


Fig. 2

