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2-(4-Chlorobenzyl)-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.152
Data-to-parameter ratio = 12.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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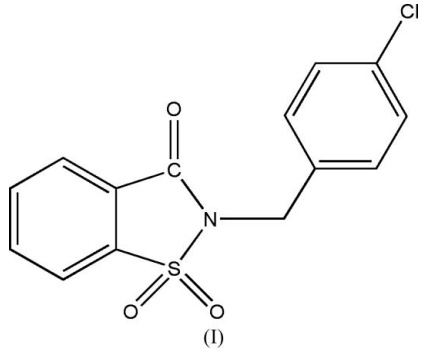
In the title compound, $C_{14}H_{10}ClNO_3S$, the mean planes of benzisothiazole moiety and the 4-chlorobenzene ring make a dihedral angle of 66.2 (5)°. Weak intermolecular C—H···O hydrogen bonds link molecules into linear chains extending along the *b* axis.

Comment

Saccharin, one of the earliest synthetic sweeteners, has been widely applied in drug synthesis. Many pharmaceuticals have been made starting from saccharin, for example, the anti-inflammatory and antirheumatic drug Meloxicam (Xu *et al.*, 1999). The 4-chlorobenzyl group is an important intermediate in organic synthesis. We report here the crystal structure of the title compound, (I).



(I)

In (I) (Fig. 1), all bond lengths and angles (Table 1) 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). The mean plane of the benzisothiazole (C1–C7/S1/

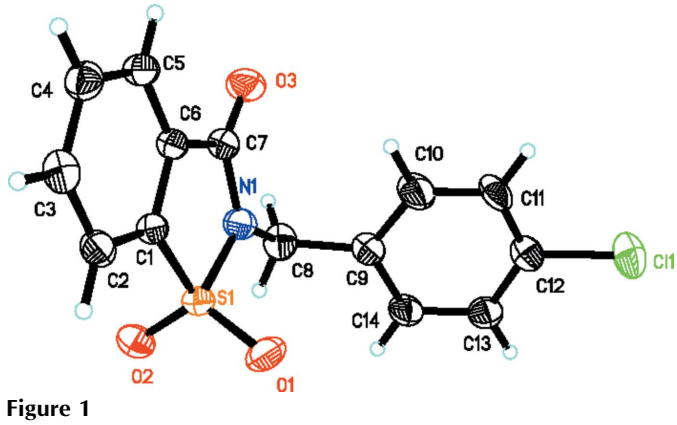


Figure 1
View of (I), with displacement ellipsoids drawn at the 40% probability level.

N1) moiety and the benzene (C9–C14) ring make a dihedral angle of 66.2 (5)°. Weak intermolecular C—H···O hydrogen bonds (Table 2) link molecules into linear chains extending along the *b* axis.

Experimental

The title compound was prepared by reaction of sodium saccharinate (4.17 g, 0.02 mol) and 4-chlorobenzyl chloride (2.50 g, 0.02 mol) in *N*,*N*-dimethylformamide (5 ml) under reflux for 5 h; 4.67 g product was obtained (yield 76.0%). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethyl acetate at room temperature.

Crystal data

$C_{14}H_{10}ClNO_3S$
M_r = 307.74
Triclinic, $P\overline{1}$
a = 7.2657 (15) Å
b = 7.4743 (15) Å
c = 13.317 (3) Å
α = 97.50 (3)°
β = 102.82 (3)°
γ = 111.25 (3)°
V = 639.3 (3) Å^3
Z = 2
D_x = 1.599 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5601 reflections
θ = 1.6–27.5°
μ = 0.47 mm^{-1}
T = 293 (2) K
Plate, colourless
0.53 × 0.47 × 0.11 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.753, T_{max} = 0.953
2851 measured reflections
2201 independent reflections
2065 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -8 \rightarrow 8$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.152
S = 1.11
2201 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2 + 0.4878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C12	1.735 (3)	S1—C1	1.748 (3)
S1—O2	1.418 (2)	N1—C7	1.376 (4)
S1—O1	1.421 (2)	N1—C8	1.463 (4)
S1—N1	1.666 (2)		
O2—S1—O1	117.21 (14)	N1—C8—C9	113.3 (2)
C7—N1—S1	115.0 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O3^{i}	0.93	2.39	3.300 (4)	165

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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