Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Ethyl 3-oxo-1,2-benzisothiazoline-2-acetate 1,1-dioxide

Liang-Zhong Xu,* Guo-Dong Si, Zai-Feng Li, Shuang-Hua Yang and Kai Li

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China

Correspondence e-mail: qknhs@163169.net

Received 23 March 2005
Accepted 7 April 2005
Online 16 April 2005

In the title compound, $C_{11}H_{11}NO_5S$, the saccharin and ethyl acetate moieties are nearly orthogonal. There are some weak intermolecular hydrogen-bond interactions which stabilize the crystal structure.

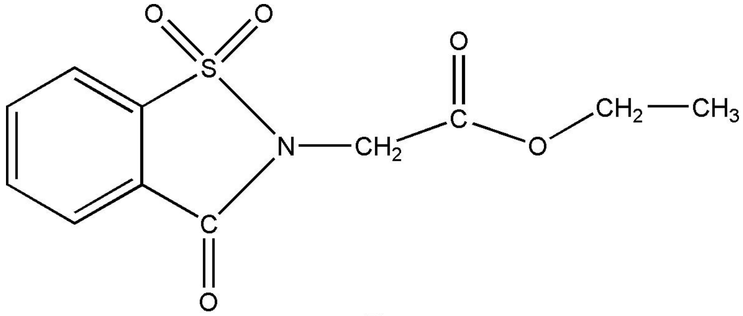
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.075
Data-to-parameter ratio = 17.9

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

Comment

Saccharin was the earliest synthetic sweetener, and was applied widely in drug synthesis. Many medicines were made starting from saccharin, including the *anti*-inflammatory and antirheumatic drug meloxicam (Xu *et al.*, 1999), and the title compound, (I), was an important intermediate in the preparation of these medicines. Here we report the crystal structure of (I).



(I)

All bond lengths and angles (Table 1) within the saccharin group of (I) are similar to those found for a series of *N*-saccharin acids (Feeder & Jones, 1996) and *N*-saccharin peracids (Feeder & Jones, 1994) as well as those of *N*-(2-nitrophenylthio)saccharin (Glidewell *et al.*, 2000). The saccharin and ethyl acetate moieties are each planar, the largest deviations from the least-squares planes being 0.046 (2) and 0.055 (2) Å for C7 and C11, respectively; these planes are nearly orthogonal to each other, forming a dihedral angle of 82.92 (4)°. The packing is stabilized by weak intermolecular C—H···O hydrogen-bond interactions (Table 2).

Experimental

The title compound was prepared by reaction of sodium saccharin (4.17 g, 0.02 mol) and ethyl chloroacetate (2.17 ml, 0.02 mol) in refluxing *N*,*N*-dimethylformamide (5 ml) for 5 h. The solid product obtained (4.88 g, yield 83.6%) was recrystallized from ethyl acetate at room temperature to give colourless crystals suitable for X-ray measurements.

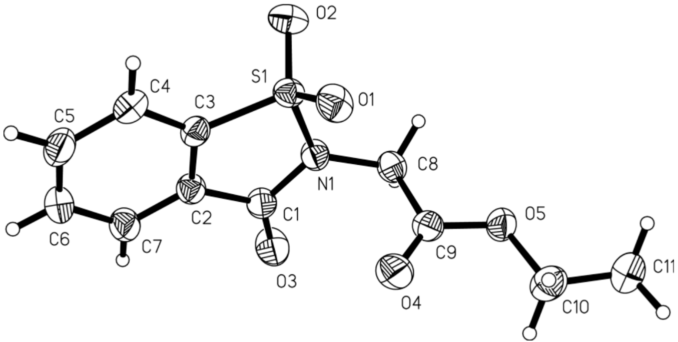


Figure 1
View of the title compound, with displacement ellipsoids drawn at the 40% probability level.

Crystal data

$C_{11}H_{11}NO_5S$
$M_r = 269.27$
Orthorhombic, $P2_12_12_1$
$a = 4.9278\ (6)$ Å
$b = 12.6217\ (14)$ Å
$c = 19.601\ (2)$ Å
$V = 1219.1\ (2)$ Å^3
$Z = 4$
$D_x = 1.467$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2794 reflections
$\theta = 2.6$–$24.3°$
$\mu = 0.28$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
$0.20 \times 0.18 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.934$, $T_{max} = 0.962$
8240 measured reflections
2928 independent reflections
2523 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 28.0°$
$h = -5 \rightarrow 6$
$k = -14 \rightarrow 16$
$l = -25 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.075$
$S = 1.02$
2928 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Flack (1983), 1194 Friedel pairs
Flack parameter = −0.10 (6)

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4233 (11)	N1—C1	1.389 (2)
S1—O2	1.4234 (12)	N1—C8	1.456 (2)
S1—N1	1.6692 (13)	O3—C1	1.2053 (18)
S1—C3	1.7567 (16)		
O1—S1—O2	116.97 (7)	N1—S1—C3	92.39 (7)
O1—S1—N1	109.53 (7)	C1—N1—C8	122.29 (13)
O2—S1—N1	110.13 (7)	C1—N1—S1	115.27 (11)
O1—S1—C3	113.74 (8)	C8—N1—S1	120.21 (10)
O2—S1—C3	111.34 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.93	2.58	3.487 (2)	166.2
C8—H8A···O1^{ii}	0.97	2.58	3.140 (2)	116.8
C8—H8B···O4^{ii}	0.97	2.38	3.253 (2)	149.1

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $1+x, y, z$.

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ for the aryl and methylene H atoms and 1.5 $U_{eq}(C)$ for the methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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*.

References

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Feeder, N. & Jones, W. (1994). *Acta Cryst.* C**50**, 1118–1122.
Feeder, N. & Jones, W. (1996). *Acta Cryst.* C**52**, 2323–2326.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Glidewell, C., Low, J. N. & Wardell, J. L. (2000). *Acta Cryst.* C**56**, 1462–1464.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xu, Y. J., Liu, F. & Yang, Z. M. (1999). *Hecheng Huaxue,* **7**, 117–119.