N-Cinnamoylsaccharin

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The title compound [systematic name: 2-cinnamoyl-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide], $C_{16}H_{11}NO_4S$, contains both saccharin and cinnamoyl groups. The molecule is approximately planar in the solid state, and adjacent molecules are connected by C—H⋯O and C—H⋯π(phenyl) interactions. In the C—H⋯π interaction, the C⋯*CgA* distance is 3.916 (4) Å (*CgA* is the non-fused benzene ring centroid) and the C—H⋯π angle is 156 (2)°. A feature of the molecular geometry is the narrow C—S—N angle of 92.51 (9)° in the five-membered ring. This angle relieves strain from the ring and makes it possible for the whole saccharin group to become quite planar.

Comment

Derivatives of saccharin are known for their biological activity (Strupczewski *et al.*, 1995). The saccharyl system has also been used as a cheap and effective leaving group in important chemical transformations, such as the derivatization of phenols prior to their conversion into arenes by transfer hydrogenolysis (Brigas & Johnstone, 1990). Cinnamic acid derivatives show antibacterial, antifungal (Takeichi, 1962) and antioxidant activities (Natella *et al.*, 1999). Cinnamic acids with substitutents on the phenyl ring showing antibacterial and antimicrobial activities have also been reported (Ramanan & Rao, 1987). Taking into account these important features of the saccharin and cinnamoyl groups, we have undertaken the X-ray diffraction study of the title compound,

(I)
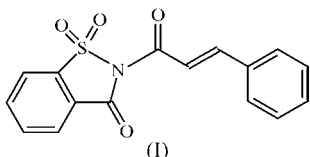
(I), in order to understand the molecular features which stabilize its observed conformation.

A view of (I) with the atom-labelling scheme is shown in Fig. 1. The saccharin group (C10/C11–C16/N1/S1/O4) is planar to within 0.0526 (2) Å, and the two sulfone O atoms (O2 and O3) lie approximately 1.23 Å above and below this plane. The crystal structure of (I) can be described as being built from essentially planar fragments, *viz.* a three-atom bridge (C7/C8/C9) linking the aromatic ring (C1–C6) with the saccharin group. The dihedral angle between the saccharin group and the aromatic ring (C1–C6) is 13.3 (2)°. Two intramolecular C—H⋯O hydrogen-bond-like contacts (Fig. 1 and Table 2) may contribute to the stability of this conformation. The small C12—S1—N1 angle of 92.51 (9)° in the five-membered ring, similar to the value of 92.7 (1)° found in saccharin itself (Bart, 1968), results from a compromise with ring strain, and makes it possible for the whole saccharin group to become quite planar.

There are two intermolecular C—H⋯O contacts in the structure of (I) (Table 2). The supramolecular aggregation is
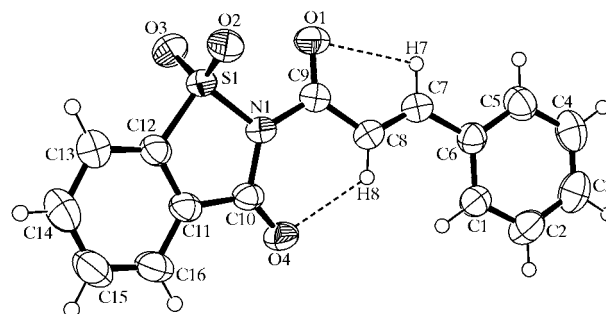

Figure 1
A view of the molecule of (I), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. Dashed lines indicate the intramolecular hydrogen bonds.
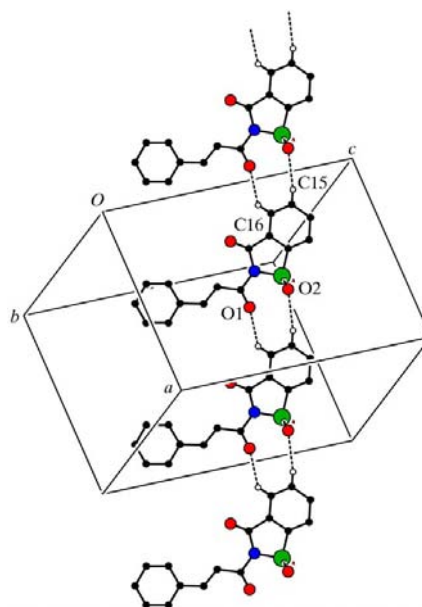

Figure 2
A view showing part of the chain of molecules which are linked by C—H⋯O hydrogen bonds (dashed lines).
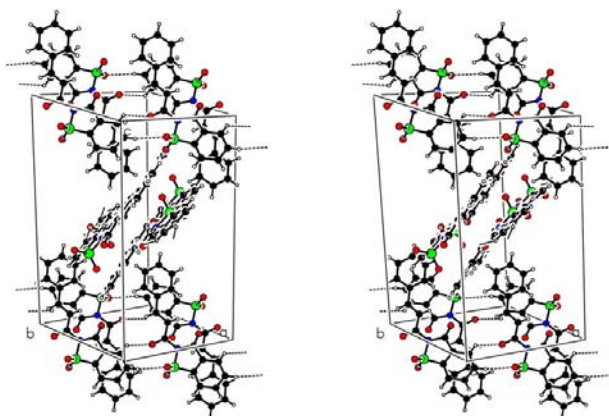
Figure 3
A stereoview of part of the crystal structure of (I). Dashed lines indicate the intermolecular hydrogen bonds.

determined by two almost equal C—H···O hydrogen bonds. In these two interactions, aromatic atom C16 in the molecule at (x, y, z) acts as a hydrogen-bond donor to carbonyl atom O1 in the molecule at $(x - \frac{1}{2}, y - \frac{1}{2}, z)$ and aromatic atom C15 in the molecule at (x, y, z) acts as a hydrogen-bond donor to sulfoxide atom O2 in the molecule at $(x - \frac{1}{2}, y - \frac{1}{2}, z)$. Because of these interactions, molecules of (I) are arranged so that C—H···O hydrogen bonds form an $S(5)S(6)[R_2^2(9)]S(5)S(6)$ motif (Bernstein *et al.*, 1995) (Fig. 2).

In the extended structure of (I), there is a weak intermolecular C—H···π interaction involving the C14—H14 group and the centroid (CgA) of ring A (atoms C1–C6) of the molecule at $(x, -y, \frac{1}{2} + z)$; see Table 2 for details. This varied set of hydrogen-bonding and π···ring interactions contributes to the stabilization of the crystal structure (Fig. 3).

Experimental

A mixture of sodium saccharinate (0.1 mol) and cinnamoyl chloride (0.1 mol) in tetrahydrofuran (100 ml) was stirred under reflux for 5 h. The precipitated NaCl was filtered off. After filtration and evaporation of the solvent, the residual solid was recrystallized from tetrahydrofuran as colourless crystals of (I) (m.p. 497–501 K, 65% yield).

Crystal data

$C_{16}H_{11}NO_4S$	$D_x = 1.425$ Mg m^{-3}
$M_r = 313.32$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3237
$a = 13.714$ (3) Å	reflections
$b = 10.456$ (4) Å	$\theta = 2.5$–$30.0°$
$c = 20.781$ (3) Å	$\mu = 0.24$ mm^{-1}
$\beta = 101.365$ (5)°	$T = 293$ (2) K
$V = 2921.4$ (14) Å^3	Prism, colourless
$Z = 8$	$0.75 \times 0.65 \times 0.50$ mm

Data collection

Rigaku AFC-7S diffractometer	$R_{int} = 0.063$
$\omega/2\theta$ scans	$\theta_{max} = 30.0°$
Absorption correction: spherical	$h = -19 \rightarrow 19$
(Dwiggins, 1975)	$k = -14 \rightarrow 14$
$T_{min} = 0.818$, $T_{max} = 0.866$	$l = -29 \rightarrow 29$
3054 measured reflections	3 standard reflections
2939 independent reflections	every 150 reflections
2325 reflections with $I > 2\sigma(I)$	intensity decay: 0.6%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.088P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$+ 1.695P]$
$wR(F^2) = 0.138$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2939 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXL97*
	(Sheldrick, 1997)
	Extinction coefficient: 0.0034 (7)

Table 1
Selected geometric parameters (Å, °).

C9—O1	1.209 (3)	C12—S1	1.742 (2)
C9—N1	1.428 (3)	N1—S1	1.705 (2)
C10—O4	1.200 (2)	O2—S1	1.426 (2)
C10—N1	1.422 (2)	O3—S1	1.420 (2)
C8—C7—C6	126.1 (2)	C10—N1—C9	130.2 (2)
C7—C8—C9	120.2 (2)	C10—N1—S1	113.8 (1)
O1—C9—N1	116.8 (2)	C9—N1—S1	115.9 (1)
O1—C9—C8	125.5 (2)	O3—S1—O2	119.0 (1)
N1—C9—C8	117.6 (2)	O3—S1—N1	110.7 (1)
O4—C10—N1	125.1 (2)	O2—S1—N1	109.79 (9)
O4—C10—C11	126.5 (2)	O3—S1—C12	110.4 (1)
N1—C10—C11	108.4 (2)	O2—S1—C12	111.2 (1)
C11—C12—S1	111.2 (2)	N1—S1—C12	92.51 (9)
C13—C12—S1	125.8 (2)		
C5—C6—C7—C8	−171.2 (2)	O4—C10—N1—C9	−5.2 (4)
C6—C7—C8—C9	179.9 (2)	C8—C9—N1—C10	−8.3 (3)
C7—C8—C9—N1	178.5 (2)	O1—C9—N1—S1	−8.2 (3)

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

CgA is the centroid of ring C1–C6.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1	0.95 (3)	2.49 (3)	2.832 (3)	101 (2)
C8—H8···O4	0.91 (3)	2.26 (3)	2.901 (3)	127 (2)
C15—H15···O2^i	0.92 (3)	2.57 (3)	3.478 (3)	169 (2)
C16—H16···O1^i	0.94 (2)	2.56 (3)	3.277 (3)	133 (2)
C14—H14···CgA^{ii}	0.91 (3)	3.07 (3)	3.916 (4)	156 (2)

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

All H atoms were located in a difference Fourier map and their positional and isotropic displacement parameters were refined. The C—H bond lengths are in the range 0.91 (3)–1.00 (3) Å and the U_{iso}(H) values are in the range 0.049 (6)–0.085 (9) Å^2.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2000); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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