

*SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1180). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 3-[(2-Phenylsulfonyl)ethenyl]-4H-1-benzopyran-4-one

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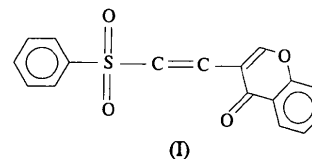
## Abstract

In the title compound, C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>S, the bond distances reflect electron delocalization in the O4—C10=C9—C8=C7 chain. The molecule contains three nearly planar segments, namely, the benzopyranone group, the

phenyl ring and the ethylene group. The sulfonyl plane is inclined at an angle of 84.6(2)° to the plane of the ethylene group, including its immediate substituents, and at an angle of 14.4(1)° to the benzopyranone group. In the structure of the related compound 3-[2-(4-chlorophenylsulfonyl)ethenyl]-4H-1-benzopyran-4-one [Krishnaiah, Narayana Raju, Lu, Chen & Narasinga Rao (1995). *Acta Cryst.* **C51**, 2429–2430], the corresponding angles are 60.3(2) and 51.5(2)°, respectively.

## Comment

Sulfones are compounds in which the S atom is bonded to two C atoms and two terminal O atoms in a tetrahedral arrangement (Truce, Klingler & Brand, 1984). Sulfones have shown activity as antibacterial and antifungal agents. Dapsone has proven effective against leprosy, while diasone is highly effective against streptococcal and pneumococcal infections (Kharasch, Stampa & Nudenberg, 1953). The antifungal activity of some unsaturated sulfones has been found to be dependent upon substituent and stereochemical effects. The title compound, 3-[2-(phenylsulfonyl)ethenyl]-4H-1-benzopyran-4-one, (I), has been observed to display antifungal activity against *Curvularia lunata* and *Furarium oxysporum* (Mukundam, 1990).



The crystal and molecular structure of (I) (Fig. 1) has been determined in order to study its stereochemistry and is part of a series of compounds derived from these antifungal agents having different substituents at the 6 position of the 4H-1-benzopyran-4-one ring. Our aim is to observe the influences of these changes on the conformation of the ethenylsulfone moiety.

The title molecule contains three nearly planar segments, namely, the benzopyranone group, the phenyl

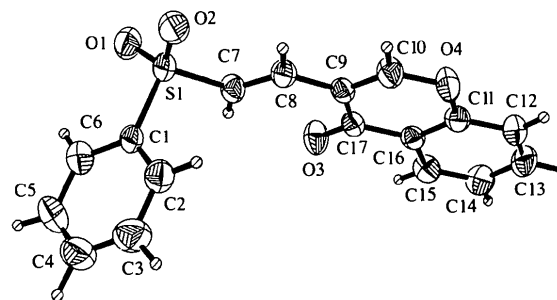


Fig. 1. An ORTEP plot (Johnson, 1965) of (I) with ellipsoids at the 50% probability level.

ring and the ethylene group. The sulfonyl plane is inclined at an angle of 84.6(2)° to the plane of the ethylene group, including its immediate substituents. The benzopyranone group is inclined at an angle of 14.4(1)° to the sulfonyl plane. In the structure of the related compound 3-[2-(4-chlorophenylsulfonyl)ethenyl]-4*H*-1-benzopyran-4-one (Krishnaiah, Narayana Raju, Lu, Chen & Narasinga Rao, 1995), where chlorine is attached to the phenyl ring, the corresponding angles are 60.3(2) and 51.5(2)°, respectively.

## Experimental

A mixture of 1.74 g (0.01 mol) of 4-oxo-4*H*-1-benzopyran-3-carboxaldehyde, 2.0 g (0.01 mol) of phenylsulfonylacetic acid, 10 ml of glacial acetic acid and a catalytic amount of benzylamine (0.2 ml) was heated under reflux for 2 h. The reaction mixture was then treated with 50 ml of dry ether and allowed to stand overnight. The compound which separated was collected and recrystallized from glacial acetic acid to yield 1.1 g (35%) of (I) as light-yellow flakes (m.p. 481–482 K).

### Crystal data

C<sub>17</sub>H<sub>12</sub>O<sub>4</sub>S  
*M<sub>r</sub>* = 312.3  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 12.062 (5) Å  
*b* = 11.652 (4) Å  
*c* = 10.304 (4) Å  
 $\beta$  = 94.13 (3)°  
*V* = 1444.4 (12) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.436 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 10–25°  
 $\mu$  = 0.24 mm<sup>-1</sup>  
*T* = 293 K  
 Thin plate  
 0.50 × 0.30 × 0.20 mm  
 Colorless

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction: none  
 5511 measured reflections  
 2836 independent reflections  
 1999 observed reflections  
 $[F > 4\sigma(F)]$

*R*<sub>int</sub> = 0.0318  
 $\theta_{\max}$  = 26°  
 $h = 0 \rightarrow 14$   
 $k = -14 \rightarrow 14$   
 $l = -12 \rightarrow 12$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: <3%

### Refinement

Refinement on *F*  
*R* = 0.039  
*wR* = 0.042  
*S* = 1.78  
 1999 reflections  
 247 parameters  
 H-atom parameters refined  
 $w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.19 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from Cromer & Waber (1974)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
S(1)	0.7561 (1)	0.3129 (1)	0.0868 (1)	0.039 (1)
O(1)	0.7406 (1)	0.3819 (2)	−0.0290 (2)	0.053 (1)
O(2)	0.7751 (1)	0.1919 (1)	0.0726 (2)	0.052 (1)
O(3)	0.4708 (1)	0.4400 (1)	0.3290 (2)	0.051 (1)
O(4)	0.4072 (1)	0.1161 (1)	0.4565 (2)	0.047 (1)
C(1)	0.8704 (2)	0.3717 (2)	0.1817 (2)	0.037 (1)
C(2)	0.9033 (2)	0.3224 (2)	0.3003 (2)	0.048 (1)
C(3)	0.9931 (3)	0.3676 (3)	0.3736 (3)	0.064 (1)
C(4)	1.0490 (3)	0.4601 (3)	0.3288 (3)	0.068 (1)
C(5)	1.0159 (3)	0.5088 (3)	0.2123 (3)	0.071 (1)
C(6)	0.9249 (2)	0.4658 (3)	0.1370 (3)	0.055 (1)
C(7)	0.6447 (2)	0.3324 (2)	0.1833 (2)	0.038 (1)
C(8)	0.5984 (2)	0.2421 (2)	0.2356 (2)	0.037 (1)
C(9)	0.5107 (2)	0.2401 (2)	0.3251 (2)	0.034 (1)
C(10)	0.4830 (2)	0.1367 (2)	0.3708 (2)	0.042 (1)
C(11)	0.3531 (2)	0.2090 (2)	0.5061 (2)	0.036 (1)
C(12)	0.2768 (2)	0.1834 (2)	0.5967 (2)	0.045 (1)
C(13)	0.2219 (2)	0.2727 (2)	0.6500 (3)	0.051 (1)
C(14)	0.2424 (2)	0.3856 (3)	0.6147 (3)	0.051 (1)
C(15)	0.3172 (2)	0.4097 (2)	0.5244 (2)	0.043 (1)
C(16)	0.3745 (2)	0.3201 (2)	0.4678 (2)	0.034 (1)
C(17)	0.4542 (2)	0.3431 (2)	0.3691 (2)	0.035 (1)

Table 2. Selected geometric parameters (Å, °)

S(1)—O(1)	1.439 (2)	S(1)—O(2)	1.438 (2)
S(1)—C(1)	1.770 (2)	S(1)—C(7)	1.742 (2)
O(3)—C(17)	1.224 (3)	O(4)—C(10)	1.338 (3)
O(4)—C(11)	1.381 (3)	C(1)—C(2)	1.382 (3)
C(1)—C(6)	1.375 (4)	C(2)—C(3)	1.380 (4)
C(3)—C(4)	1.369 (5)	C(4)—C(5)	1.361 (5)
C(5)—C(6)	1.390 (4)	C(7)—C(8)	1.325 (3)
C(8)—C(9)	1.454 (3)	C(9)—C(10)	1.345 (3)
C(9)—C(17)	1.467 (3)	C(11)—C(12)	1.390 (3)
C(11)—C(16)	1.382 (3)	C(12)—C(13)	1.369 (4)
C(13)—C(14)	1.392 (4)	C(14)—C(15)	1.371 (4)
C(15)—C(16)	1.403 (3)	C(16)—C(17)	1.473 (3)
O(1)—S(1)—O(2)	118.4 (1)	O(1)—S(1)—C(1)	107.0 (1)
O(2)—S(1)—C(1)	108.2 (2)	O(1)—S(1)—C(7)	109.9 (1)
O(2)—S(1)—C(7)	108.8 (1)	C(1)—S(1)—C(7)	103.7 (1)
C(10)—O(4)—C(11)	117.9 (2)	S(1)—C(1)—C(2)	119.4 (2)
S(1)—C(1)—C(6)	119.7 (2)	C(2)—C(1)—C(6)	120.9 (2)
C(1)—C(2)—C(3)	119.3 (3)	C(2)—C(3)—C(4)	120.2 (3)
C(3)—C(4)—C(5)	120.3 (3)	C(4)—C(5)—C(6)	120.8 (3)
C(1)—C(6)—C(5)	118.5 (3)	S(1)—C(7)—C(8)	119.6 (2)
C(7)—C(8)—C(9)	128.2 (2)	C(8)—C(9)—C(10)	116.5 (2)
C(8)—C(9)—C(17)	124.0 (2)	C(10)—C(9)—C(17)	119.5 (2)
O(4)—C(10)—C(9)	126.2 (2)	O(4)—C(11)—C(12)	115.7 (2)
O(4)—C(11)—C(16)	121.6 (2)	C(12)—C(11)—C(16)	122.6 (2)
C(11)—C(12)—C(13)	118.0 (2)	C(12)—C(13)—C(14)	120.9 (3)
C(13)—C(14)—C(15)	120.6 (3)	C(14)—C(15)—C(16)	119.9 (2)
C(11)—C(16)—C(15)	118.0 (2)	C(11)—C(16)—C(17)	120.8 (2)
C(15)—C(16)—C(17)	121.2 (2)	O(3)—C(17)—C(9)	124.0 (2)
O(3)—C(17)—C(16)	122.1 (2)	C(9)—C(17)—C(16)	113.9 (2)

C—H distances vary between 0.91 (3) and 0.98 (3) Å. The structure was solved by direct methods and refined by full-matrix least squares minimizing  $\sum w(F_o - F_c)^2$ .

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1226). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A New Precursor of 1,3,4-Thiadiazole-sulfonamides: 5-*tert*-Butyloxycarbonylamino-1,3,4-thiadiazole-2-sulfonyl Chloride

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### Abstract

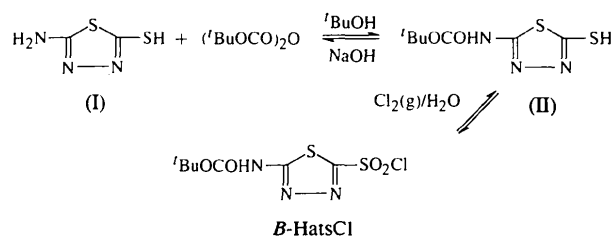
The title compound, *tert*-butyl 2-chlorosulfonyl-1,3,4-thiadiazole-5-carbamate, C<sub>7</sub>H<sub>10</sub>ClN<sub>3</sub>O<sub>4</sub>S<sub>2</sub>, is a 1,3,4-thiadiazole derivative related to sulfonamides. Bond lengths and angles indicate a strong interaction between the sulfonyl group and the thiadiazole ring. The sulfonyl moiety presents a distorted arrangement around the S atom.

### Comment

Since Davenport (1945) reported that thiophene-2-sulfonamide is 40 times more active as a carbonic anhydrase (CA) inhibitor than the sulfonamides, many

heterocyclic sulfonamides have been prepared. Two important facts connecting chemical structure with CA inhibitory action have been established. Firstly, five-membered derivatives are more effective than six-membered ones, and secondly, the presence of nitrogen and sulfur within the ring lead to the most potent CA inhibitors. We have obtained and determined the crystal structures of several 1,3,4-thiadiazolesulfonamides following the method proposed by Young, Wood, Eichler, Vaughan & Anderson (1956) with minor modifications (Alzuet, Ferrer & Borrás, 1991a; Pedregosa, Alzuet, Borrás, Fustero, García-Granda & Díaz, 1993).

In this work, as part of our program of systematic studies on unsubstituted sulfonamides and their metal complexes (Alzuet, Ferrer & Borrás, 1991b; Pedregosa, Casanova, Alzuet, Borrás, García-Granda & Gutierrez-Rodriguez, 1995), we have obtained and determined the crystal structure of 5-*tert*-butyloxycarbonylamino-1,3,4-thiadiazole-2-sulfonyl chloride (*B*-HatsCl) (*B* = butyloxycarbonyl), which is an intermediate in the synthesis of 5-*tert*-butyloxycarbonylamino-1,3,4-thiadiazole-2-sulfonamide (*B*-H<sub>2</sub>ats) and 5-amino-1,3,4-thiadiazole-2-sulfonamide (Hats), in order to establish structural relations with sulfonamides. The presence of an S—Cl bond instead of the S—N bond in the structure may, however, provide an insight into the reactivity of the intermediate and coordination properties of these compounds.



The endocyclic bond length C(2)—N(2) [1.294 (4) Å] clearly indicates a double bond. The longer bond distances [C(1)—N(1) 1.317 (4) and C(1)—N(3) 1.354 (4) Å] can be explained by a slight delocalization of electron density through the N(1), C(1) and

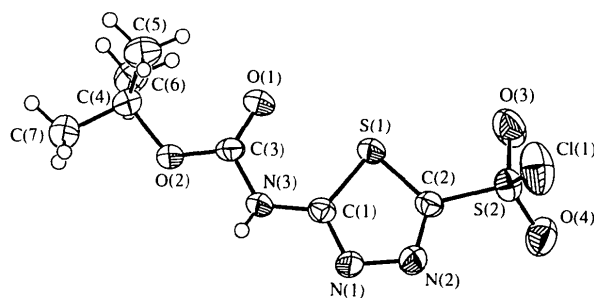


Fig. 1. The molecular structure of *B*-HatsCl showing the atom-numbering scheme and 50% probability displacement ellipsoids.