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

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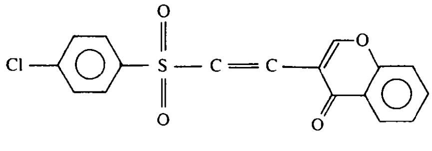
## Abstract

The molecule of the title compound, $C_{17}H_{11}ClO_4S$, contains three nearly planar segments (the benzopyranone group, the chlorophenyl ring and the ethene group with its immediate substituents). The sulfonyl plane is inclined at 60.3° to the plane of the ethene moiety and at 51.5° to the plane of the benzopyranone group.

## Comment

The crystal structure of the title compound, (I), has been determined as part of a series of studies of the stereochemistry of sulfones containing ethene and benzopyran groups. These compounds are effective fungicides (Hawthorne, 1960).



(I)

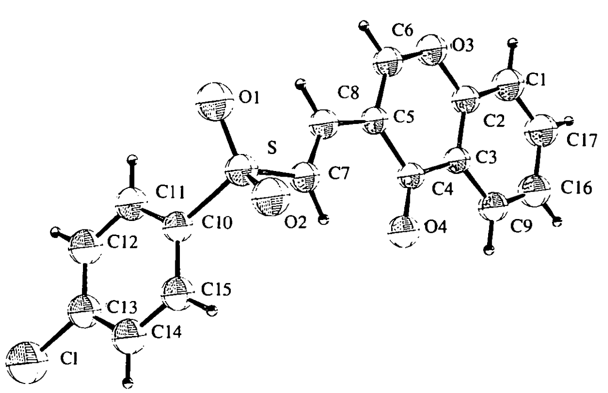


Fig. 1. View of the title compound with ellipsoids plotted at the 50% probability level.

## Experimental

The title compound was synthesized by Mukundam and S. R. Naidu at the Department of Chemistry, Sri Venkateswara University, Tirupati, India. To a solution of 1.74 g (0.01 mol) of 4-oxo-4*H*-1-benzopyran-3-carboxaldehyde and 2.35 g (0.01 mol) of 4-chlorophenylsulfonylacetic acid in 10 ml of glacial acetic acid was added 0.2 ml of benzylamine; the resulting solution was refluxed for 3 h. The resultant reaction mixture was warmed to yield 1.5 g (43%) of the title compound, which was recrystallized from glacial acetic acid, affording yellow crystals (m.p. 503–504 K).

*Crystal data*

$C_{17}H_{11}ClO_4S$
$M_r = 346.79$
Monoclinic
$P2_1/c$
$a = 17.5076\,(1)$ Å
$b = 6.6859\,(1)$ Å
$c = 13.0147\,(1)$ Å
$\beta = 94.48\,(1)^\circ$
$V = 1518.7$ Å$^3$
$Z = 4$
$D_x = 1.52$ Mg m$^{-3}$
$D_m = 1.51$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9\text{–}15^\circ$
$\mu = 0.39$ mm$^{-1}$
$T = 23\,(1)$ K
Needle
1.0 × 0.3 × 0.1 mm
Yellow

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$ scans
Absorption correction: none
2982 measured reflections
2919 independent reflections
1781 observed reflections [$I > 3\sigma(I)$]

$\theta_{\max} = 36.5^\circ$
$h = 0 \rightarrow 20$
$k = -7 \rightarrow 0$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 100 reflections intensity decay: <5%

*Refinement*

Refinement on $F$
$R = 0.033$
$wR = 0.050$
$S = 1.48$
1781 reflections
252 parameters
Only H-atom $U$'s refined

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.22$
$\Delta\rho_{\max} = 2.10$ e Å$^{-3}$
$\Delta\rho_{\min} = 0.06$ e Å$^{-3}$
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| Cl | 0.52300 (5) | 0.6936 (2) | 0.83849 (7) | 6.32 (2) |
| S | 0.70190 (4) | 1.1920 (1) | 1.18289 (6) | 3.35 (1) |
| O1 | 0.6845 (1) | 1.3993 (3) | 1.1669 (2) | 4.60 (5) |
| O2 | 0.6888 (1) | 1.1028 (4) | 1.2808 (1) | 4.38 (5) |
| O3 | 1.0348 (1) | 1.5052 (3) | 1.1153 (1) | 3.47 (4) |
| O4 | 0.9431 (1) | 0.9494 (3) | 1.1435 (2) | 3.63 (4) |
| C1 | 1.1581 (2) | 1.3750 (5) | 1.1023 (2) | 4.07 (7) |
| C2 | 1.0812 (2) | 1.3391 (4) | 1.1125 (2) | 2.97 (6) |
| C3 | 1.0518 (1) | 1.1484 (4) | 1.1201 (2) | 2.69 (5) |
| C4 | 0.9701 (2) | 1.1169 (4) | 1.1329 (2) | 2.66 (5) |
| C5 | 0.9247 (2) | 1.3000 (4) | 1.1331 (2) | 2.71 (5) |

| | | | | |
|---|---|---|---|---|
| C6 | 0.9601 (2) | 1.4783 (4) | 1.1251 (2) | 3.14 (6) |
| C7 | 0.7979 (2) | 1.1513 (4) | 1.1616 (2) | 3.12 (6) |
| C8 | 0.8430 (2) | 1.3044 (4) | 1.1448 (2) | 3.05 (6) |
| C9 | 1.1029 (2) | 0.9874 (4) | 1.1165 (2) | 3.32 (6) |
| C10 | 0.6511 (1) | 1.0519 (5) | 1.0851 (2) | 3.08 (6) |
| C11 | 0.6332 (2) | 1.1369 (5) | 0.9891 (2) | 3.79 (7) |
| C12 | 0.5939 (2) | 1.0260 (5) | 0.9129 (2) | 4.29 (7) |
| C13 | 0.5731 (2) | 0.8330 (5) | 0.9338 (2) | 3.96 (7) |
| C14 | 0.5912 (2) | 0.7459 (5) | 1.0283 (2) | 4.16 (7) |
| C15 | 0.6310 (2) | 0.8569 (5) | 1.1050 (2) | 3.68 (7) |
| C16 | 1.1789 (2) | 1.0203 (5) | 1.1060 (2) | 4.01 (7) |
| C17 | 1.2065 (2) | 1.2145 (5) | 1.0989 (2) | 4.31 (7) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Cl—C13 | 1.733 (2) | C4—C5 | 1.459 (3) |
| S—O1 | 1.430 (2) | C5—C6 | 1.351 (3) |
| S—O2 | 1.442 (2) | C5—C8 | 1.451 (3) |
| S—C7 | 1.746 (2) | C7—C8 | 1.322 (3) |
| S—C10 | 1.764 (2) | C9—C16 | 1.365 (3) |
| O3—C2 | 1.378 (2) | C10—C11 | 1.385 (3) |
| O3—C6 | 1.337 (3) | C10—C15 | 1.380 (3) |
| O4—C4 | 1.228 (2) | C11—C12 | 1.378 (3) |
| C1—C2 | 1.384 (3) | C12—C13 | 1.374 (4) |
| C1—C17 | 1.370 (4) | C13—C14 | 1.375 (3) |
| C2—C3 | 1.382 (3) | C14—C15 | 1.387 (3) |
| C3—C4 | 1.469 (3) | C16—C17 | 1.391 (4) |
| C3—C9 | 1.403 (3) | | |
| O1—S—O2 | 118.8 (1) | C4—C5—C8 | 123.9 (2) |
| O1—S—C7 | 109.0 (1) | C6—C5—C8 | 116.8 (2) |
| O1—S—C10 | 108.7 (1) | O3—C6—C5 | 125.7 (2) |
| O2—S—C7 | 107.3 (1) | S—C7—C8 | 120.0 (2) |
| O2—S—C10 | 108.2 (1) | C5—C8—C7 | 127.5 (2) |
| C7—S—C10 | 103.9 (1) | C3—C9—C16 | 120.5 (2) |
| C2—O3—C6 | 118.5 (2) | S—C10—C11 | 119.9 (2) |
| C2—C1—C17 | 118.4 (2) | S—C10—C15 | 119.2 (2) |
| O3—C2—C1 | 116.3 (2) | C11—C10—C15 | 120.9 (2) |
| O3—C2—C3 | 121.2 (2) | C10—C11—C12 | 119.5 (3) |
| C1—C2—C3 | 122.5 (2) | C11—C12—C13 | 119.2 (2) |
| C2—C3—C4 | 120.8 (2) | Cl—C13—C12 | 119.4 (2) |
| C2—C3—C9 | 117.6 (2) | Cl—C13—C14 | 118.7 (2) |
| C4—C3—C9 | 121.6 (2) | C12—C13—C14 | 121.9 (2) |
| O4—C4—C3 | 122.0 (2) | C13—C14—C15 | 119.0 (3) |
| O4—C4—C5 | 123.4 (2) | C10—C15—C14 | 119.4 (2) |
| C3—C4—C5 | 114.5 (2) | C9—C16—C17 | 120.2 (2) |
| C4—C5—C6 | 119.2 (2) | C1—C17—C16 | 120.6 (2) |
| O3—C2—C3—C4 | −0.9 (4) | C2—C3—C4—O4 | −177.4 (2) |
| C1—C2—C3—C4 | 178.7 (2) | | |

The structure was solved using direct methods as incorporated in *MULTAN*80 (Main *et al.*, 1980). The remaining atoms were located in succeeding difference Fourier syntheses. The structure was refined by full-matrix least squares where the function minimized was $\Sigma w(|F_o|-|F_c|)^2$. The standard deviation on intensities, $\sigma(F_o^2)$, is defined as follows: $\sigma(F_o^2) = [S^2(C + R^2B)(pF_o^2)^2]/\mathrm{Lp}^2$, where $S$ is the scan rate, $C$ is the total integrated peak count, $R$ is the ratio of scan time to background counting time, $B$ is the total background count, Lp is the Lorentz–polarization factor and the parameter $p$ is a factor introduced to down-weight intense reflections; in this case, $p$ was set to 0.040. Anomalous-dispersion effects were included in $F_c$; the values for $f'$ and $f''$ were those of Cromer (1974). The largest parameter shift was 0.01 times its e.s.d.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1186). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Hawthorne, M. P. (1960). *J. Am. Chem. Soc.* **82**, 1886–1888.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

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# 5-(2,4-Dihydroxyphenyl)tetrazole Sesquihydrate

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## Abstract

The structure of the title compound [4-(5-tetrazolyl)-1,3-benzenediol sesquihydrate, $C_7H_6N_4O_2.3/2H_2O$] which crystallizes with two tetrazolylbenzenediol molecules per asymmetric unit together with three water molecules, has been determined by single-crystal X-ray diffraction at room temperature. The two molecules are linked through a hydrogen-bonded network to water molecules, forming layers extending along the *bc* face of the unit cell.

## Comment

The structure of the title compound, (I), has been determined as part of our studies of conventional liquid crystals and metalomesogens which contain heterocyclic rings (Gallardo & Merlo, 1993; Gallardo, Müller, Taylor, Bartulín & Martinez, 1993; Gallardo & Favarin, 1993; Gallardo & Begnini, 1995). The phenyl ring and the tetrazole ring of each molecule in the asymmetric unit are coplanar, which indicates an extension of the delocalized $\pi$-bonding system across the rings. A least-squares-plane calculation involving the atoms of both rings plus the atoms O1 and O2 in molecules *A* and *B* showed that the maximum deviation from planarity was less than 0.05 Å at O1 and O2′.

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