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# Flavone-3′-sulfonamide[1]

**Engin Kendi,[a]* Süheyla Özbey,[a] Oya Bozdağ[b] and Rahmiye Ertan[b]**

[a]Hacettepe University, Department of Physics Engineering, Beytepe 06532, Ankara, Turkey, and [b]Department of Pharmaceutical Chemistry, Ankara University, Tandoğan 06100, Ankara, Turkey
Correspondence e-mail: kendi@eti.cc.hun.edu.tr

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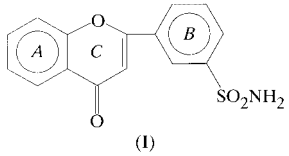
In the title molecule, $C_{15}H_{11}NO_4S$, the phenyl and benzene rings are quite planar, with maximum deviations from planarity of 0.009 (2) and 0.004 (1) Å, respectively. The $\gamma$-pyrone ring deviates from planarity and makes a dihedral angle of 8.3 (3)° with the 2-phenyl substituent. The sulfonamide group is involved in N—H···O hydrogen bonding.

## Comment

Besides the extensive biological activity of flavonoids, this class of compounds exhibits antidiabetic (Hii & Howell, 1985; Basnet *et al.*, 1993; Ragunathan & Sulochana, 1994) and aldose reductase inhibitory activity (Varma & Kinoshita, 1976; Okuda *et al.*, 1984; Aida *et al.*, 1990). Aryl sulfonamides are widely used as starting materials in the synthesis of antidiabetic sulfonylureas (AFECT, 1995). The title compound, (I), is a flavonesulfonamide and was synthesized as a starting material for antidiabetic flavonesulfonylureas. The structure was elucidated by $^1$H NMR, mass and IR spectroscopic techniques. The X-ray structure was determined in order to establish the conformation of the molecule.




(I)

All bond lengths and angles in (I) are normal. Rings *A* and *B* are quite planar: the maximum deviations are 0.009 (2) and 0.004 (1) Å from the ring planes *A* and *B*, respectively. The pyrone ring *C* is distorted ($\chi^2$ = 210.2). The angles between rings *A* and *C* and *B* and C are 1.8 (2) and 8.3 (3)°, respectively, showing that the rings *A*, *B* and *C* are coplanar.

In the generally preferred conformation of flavones, the dihedral angle between the phenyl and the $\gamma$-pyrone rings is expected to be small, as in the case of (I). This angle is 13.9 (4)° in 2-(2-ethoxycarbonyl-1,4-benzodioxan-7-yl)-4*H*-1-benzopyran-4-one (Özbey *et al.*, 1997). Another parameter of interest in flavone structures is the bond length between the benzopyrone and phenyl rings. In flavones, the increase in dihedral angle has the effect of increasing the length of this bond to the expected value for an $sp^2$—$sp^2$ single bond. In compound (I), the corresponding C2—C11 bond length is 1.478 (3) Å. In 5-hydroxyflavone, the dihedral angle is 5.2 (9)° and the C2—C11 bond is 1.465 (4) Å (Shoja, 1990). In 2′-methyl-3′-nitroflavone, these values are 139.8 (2)° and 1.491 (8) Å, respectively (Kendi *et al.*, 1996). The widening of the O1—C9—C10 angle to 121.6 (2)° and the narrowing of the C3—C4—C10 angle to 115.2 (2)° in the $\gamma$-pyrone ring may be attributed to the ring strain caused by the neighbouring C$sp^2$—C$sp^2$ atoms.

Atoms S1 and O3 of the sulfonamide group lie close to the plane of ring *B*, with deviations of 0.032 (1) and 0.045 (1) Å from the plane, respectively. Atoms O4 and N1 are displaced from the least-squares plane of the atoms in ring *B* by 0.928 (2) and −1.474 (2) Å, respectively.

The sulfonamide group in (I) is involved in intermolecular as well as intramolecular hydrogen bonding. It has been proposed by Rossi *et al.* (1980) that the hydrogen bonds involving O2 would enhance the electron-withdrawing power of the pyrone ring on the phenyl ring, thus giving C2—C11 more double-bond character and forcing coplanarity between rings *B* and *C*. The crystal packing shows that the smaller the dihedral angle between the benzopyrone and phenyl rings, the stronger the stacking interactions that occur between adjacent molecules. Details of the hydrogen bonds and short contacts in (I) are given in Table 2.

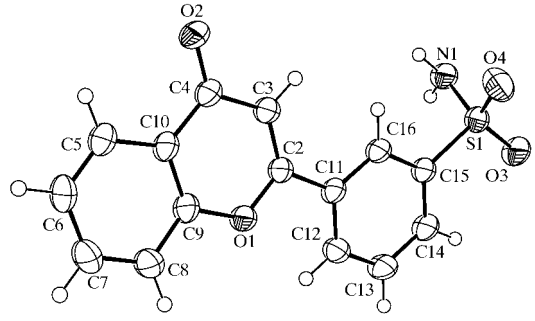



**Figure 1**
*ORTEP*II (Johnson, 1976) drawing of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H-atoms are shown as small circles of arbitrary radii.

## Experimental

Flavone (2.50 g, 0.0113 mol), obtained by the Baker–Venkataraman method, was added slowly with stirring to chlorosulfonic acid (25 ml, 0.376 mol), which had been cooled to 263 K in an ice–salt bath. The reaction mixture was stirred for 2 d at room temperature and was then poured into iced water. The crude sulfonylchloride (1.40 g, 4.37 mmol) was treated with aqueous ammonia (100 ml) and stirred for 3 h at 273 K, and flavone-3′-sulfonamide, (I), was obtained by removing the excess ammonia under reduced pressure. The crude product was crystallized from ethanol–dimethyl sulfoxide (10:1)

[1] Systematic name: 3-(4-oxo-4*H*-1-benzopyran-2-yl)benzenesulfonamide.

(yield: 1.10 g, 83.66%; m.p. 481–483 K). Spectrosopic analysis: $^1$H NMR (DMSO-$d_6$, 400 MHz, $\delta$, p.p.m.): 7.10 (*s*, 1H, 3-H), 7.52 (*ddd*, 1H, 6-H), 7.76–7.85 (*m*, 3H, 7-H, 4′-H, 6′-H), 7.98 (*d*, 1H, 8-H), 8.05 (*dd*, 1H, 5′-H), 8.28 (*d*, 1H, 5-H), 8.48 (*s*, 1H, 2′-H); mass spectroscopy (70 eV), *m*/e: 301 ($M^+$), 302 ($M$ + 1), 303 ($M$ + 2), 221, 193, 121, 120, 101, 92 (%100), 64, 63; IR (cm$^{-1}$): 1614 ($\gamma$-pyrone C=O).

*Crystal data*

$C_{15}H_{11}NO_4S$
$M_r$ = 301.32
Triclinic, $P\overline{1}$
$a$ = 8.6654 (5) Å
$b$ = 8.7898 (6) Å
$c$ = 10.0105 (6) Å
$\alpha$ = 98.183 (7)°
$\beta$ = 112.991 (6)°
$\gamma$ = 104.673 (6)°
$V$ = 653.6 (2) Å$^3$
$Z$ = 2
$D_x$ = 1.531 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta$ = 9.4–18.2°
$\mu$ = 0.263 mm$^{-1}$
$T$ = 295 K
Prismatic, colourless
0.56 × 0.40 × 0.20 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* $\psi$ scans (North *et al.*, 1968)
$T_{min}$ = 0.869, $T_{max}$ = 0.951
2834 measured reflections
2649 independent reflections
2057 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.010
$\theta_{max}$ = 26.3°
$h = -10 \rightarrow 0$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity decay: 1.5%

*Refinement*

Refinement on $F$
$R$ = 0.037
$wR$ = 0.044
$S$ = 0.90
2057 reflections
192 parameters
H atoms constrained
$w = 1/(\sigma F^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.09 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—O3 | 1.431 (2) | O1—C9 | 1.373 (3) |
| S1—O4 | 1.423 (2) | O2—C4 | 1.247 (3) |
| S1—N1 | 1.613 (2) | C2—C3 | 1.346 (3) |
| S1—C15 | 1.776 (2) | C2—C11 | 1.478 (3) |
| O1—C2 | 1.348 (3) | | |
| O3—S1—O4 | 120.1 (1) | C3—C4—C10 | 115.2 (2) |
| O3—S1—N1 | 107.3 (9) | O1—C9—C10 | 121.6 (2) |
| O3—S1—C15 | 107.2 (1) | | |
| O3—S1—C15—C16 | 176.82 (15) | O1—C2—C11—C16 | −173.58 (16) |
| O4—S1—C15—C14 | −135.03 (16) | C13—C14—C15—S1 | −179.03 (15) |
| N1—S1—C15—C14 | 110.72 (16) | | |

**Table 2**
Hydrogen-bond and short-contact geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···O3$^{i}$ | 0.95 | 2.67 | 3.442 (3) | 139 |
| C6—H6···N1$^{i}$ | 0.95 | 2.78 | 3.526 (4) | 137 |
| C8—H8···O4$^{ii}$ | 0.95 | 2.61 | 3.417 (3) | 144 |
| C12—H12···O4$^{ii}$ | 0.95 | 2.71 | 3.643 (3) | 168 |
| C12—H12···O4$^{iii}$ | 0.95 | 2.89 | 3.391 (2) | 115 |
| N1—H1N···O2$^{iv}$ | 0.95 | 1.99 | 2.928 (3) | 172 |
| N1—H2N···O2$^{v}$ | 0.94 | 2.05 | 2.976 (3) | 169 |

Symmetry codes: (i) $x-1, 1+y, z$; (ii) $x, 1+y, z$; (iii) $1-x, 1-y, 1-z$; (iv) $1+x, y, z$; (v) $1-x, 1-y, 2-z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *MolEN* and *PARST* (Nardelli, 1995); program(s) used to refine structure: *MolEN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1229). Services for accessing these data are described at the back of the journal.

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