

**(1,3-Benzodioxol-5-ylmethyl)ammonium
2-methoxy-5-[(1*E*)-3-oxo-3-phenylprop-1-en-
1-yl]benzenesulfonate monohydrate****Luiz Everson da Silva,^a
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Key indicatorsSingle-crystal X-ray study
T = 299 K
Mean $\sigma(C-C)$ = 0.005 Å
R factor = 0.045
wR factor = 0.128
Data-to-parameter ratio = 12.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

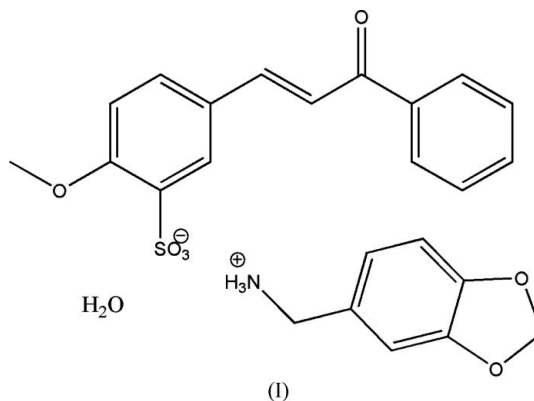
The reaction of a chalcone derivative and an arylmethylamine yields the title organic salt, $C_8H_{10}NO_2^+ \cdot C_{16}H_{13}O_5S^- \cdot H_2O$. The 1,3-dioxolane ring exhibits an envelope conformation. The C—C—C torsion angle of 16.7 (7)° between the 2-methoxy-5-vinylbenzenesulfonate unit and the phenyl ring indicates the non-planarity of the system. The polar heads of the anion and cation are connected by two bifurcated N—H···O hydrogen bonds. The water molecule completes hydrophilic interactions by N—H···O hydrogen bonds with the cation and O—H···O bonds with the anion along the *a* axis.

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Comment

Chalcones, 1,3-diaryl-2-propen-1-ones, are natural and synthetic compounds belonging to the flavonoid family. Chalcones possess a broad spectrum of biological activities, including antibacterial, antihelmintic, amoebicidal, anti-ulcer, antiviral, insecticidal, antiprotzoal, anticancer, cytotoxic and immunosuppressive activities (Dimmock *et al.*, 1999). We have investigated a series of novel synthetic chalcone derivatives for their trypanocidal and leishmanicidal properties (Lunardi *et al.*, 2003). Much to our surprise, the reaction of the chalcone derivatives 2-methoxy-5-[(*E*)-3-oxo-3-phenylprop-1-enyl]benzenesulfonic acid with the arylmethylamine (benzo[*d*][1,3]-dioxol-6-yl)methanamine leads to the formation of the title compound, (I), which has been characterized by X-ray structure analysis; the results are shown here (Fig. 1).



The 1,3-dioxolane ring exhibits an envelope conformation with C24 in the flap position. The N1—C17—C18—C19 torsion angle between the NH₃ group and the benzene ring is 91.6 (4)°. The 2-methoxy-5-vinylbenzenesulfonate unit is nearly planar with a deviation from the mean plane of 0.098 (3) Å for C7 and −0.186 (4) Å for C8. The C7—C8—

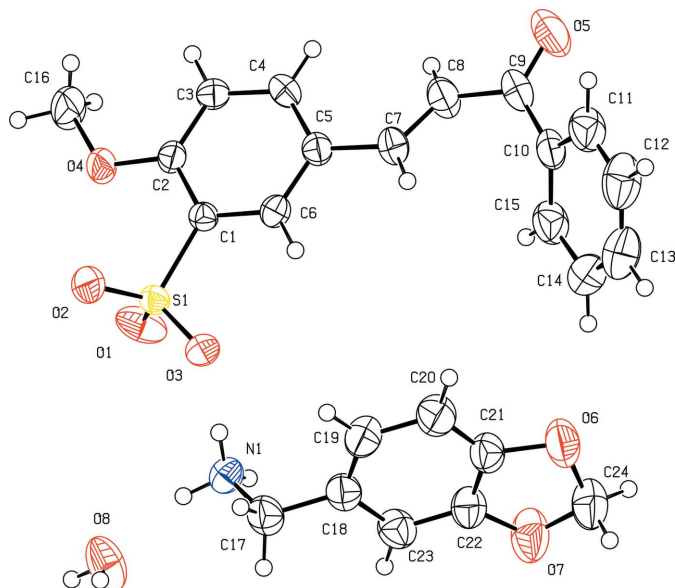


Figure 1
The molecular structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

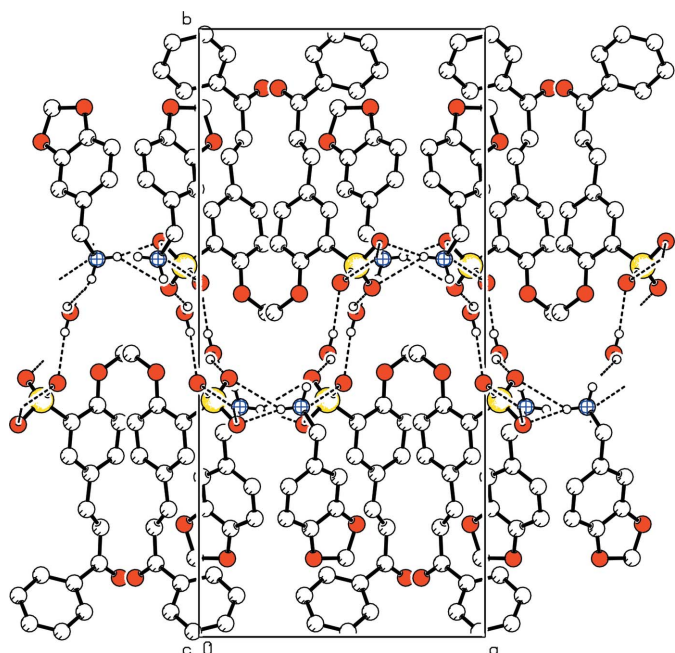


Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines; cation and anion polar heads form a hydrophilic channel running along *a*. H atoms not involved in the interactions shown have been omitted.

C9—C10 torsion angle of 16.7 (7)° between this group and the phenyl ring indicate the non-planarity of the system.

The crystal packing is dominated by hydrogen bonds between the polar cation and anion heads and water molecules that connect them into a hydrophilic chain along the *a* axis (Table 1 and Fig. 2). The compound is achiral, but to optimize crystal packing and requirements of polar interactions the non-centrosymmetric space group *Pca*₂₁ seems to be more appropriate.

Experimental

4-Methoxychalcone (6.63 g, 27.87 mmol) was reacted with chlorosulfonic acid (19.48 g, 167.2 mmol) at room temperature for five days. The red solution was poured on to crushed ice with stirring to give the sulfonic acid derivative as a yellow solid. The sulfonic acid derivative (0.50 g, 1.48 mmol) was treated with piperonylamine (0.33 g, 2.19 mmol) in methanol at 273 K. The mixture was reacted at room temperature for 1 h. The precipitate was collected by filtration and washed with ice–methanol. Recrystallization from hot ethanol yielded 0.338 g (47%) of (I). Single crystals suitable for X-ray diffraction study were obtained by recrystallization from a hot ethanol–water (95:5) solution of (I).

Crystal data

$C_8H_{10}NO_2^+ \cdot C_{16}H_{13}O_5S^- \cdot H_2O$
 $M_r = 487.51$
 Orthorhombic, *Pca*₂₁
 $a = 11.631$ (1) Å
 $b = 24.686$ (1) Å
 $c = 8.198$ (1) Å
 $V = 2353.8$ (4) Å³

$Z = 4$
 $D_x = 1.376$ Mg m⁻³
 Cu $K\alpha$ radiation
 $\mu = 1.66$ mm⁻¹
 $T = 299$ (2) K
 Prism, colourless
 0.60 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{min} = 0.688$, $T_{max} = 0.854$
 4302 measured reflections

3968 independent reflections
 3553 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.045$
 $\theta_{max} = 66.7^\circ$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.128$
 $S = 1.04$
 3968 reflections
 324 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2 + 0.2858P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.014$
 $\Delta\rho_{max} = 0.31$ e Å⁻³
 $\Delta\rho_{min} = -0.34$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0046 (4)
 Absolute structure: Flack (1983),
 1725 Friedel pairs
 Flack parameter: -0.01 (2)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H11N...O3	0.84 (2)	2.18 (2)	2.947 (4)	153 (4)
N1—H11N...O1	0.84 (2)	2.48 (3)	3.166 (4)	140 (3)
N1—H12N...O8	0.88 (2)	1.88 (2)	2.723 (4)	160 (4)
N1—H13N...O3 ⁱ	0.84 (2)	2.25 (2)	3.021 (4)	153 (3)
N1—H13N...O2 ⁱ	0.84 (2)	2.34 (3)	3.055 (4)	144 (3)
O8—H81O...O2 ⁱⁱ	0.84 (2)	2.17 (3)	2.968 (4)	159 (4)
O8—H82O...O1 ⁱⁱⁱ	0.85 (2)	2.07 (3)	2.837 (4)	151 (5)

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

The OH and NH H atoms were located in a difference map and refined with restrained geometry: N—H distances were restrained to 0.86 (2) Å, O—H distances to 0.85 (2) Å; the H—H distance was restrained to 1.365 (2) Å, thus leading to an H—O—H angle of 107° (Nardelli, 1999). The CH atoms were positioned with idealized geometry and refined using a riding model, with C—H = 0.93 Å

(aromatic), 0.96 Å (methyl) and 0.97 Å (methylene). All H atoms were refined with isotropic displacement parameters (set to 1.2 or 1.5 times U_{eq} of the parent atom).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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