organic papers

4-Bromo-2-formyl-6-methoxyphenyl 4-methylbenzenesulfonate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.083
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{15}H_{13}BrO_5S$, the *o*-vanillin group makes a dihedral angle of 41.54 (9)° with the benzene ring. The crystal packing is stabilized by weak intermolecular C—H···Br interactions, leading to chains of molecules.

Comment

Schiff base ligands have received a good deal of attention in biology and chemistry (Kahwa *et al.*, 1986 and Klayman *et al.*, 1979). Many Schiff base derivatives have been synthesized and employed in the development of protein and enzyme mimics (Santos *et al.*, 2001). As part of our interest in the coordination properties of Schiff bases functioning as ligands, we have investigated the title compound, (I) (Fig. 1), which is used as a precursor in the preparation of Schiff bases.
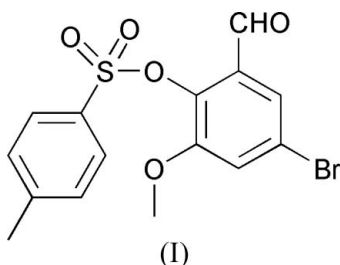
The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The *o*-vanillin group (atoms C8–C14/O3/
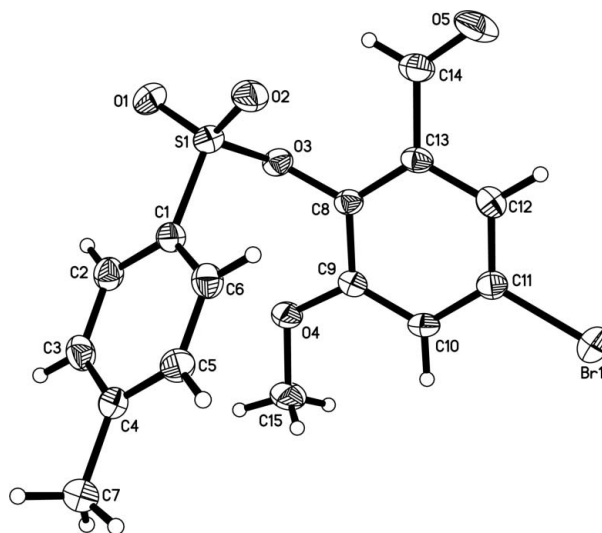
Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

O4) is essentially planar, with an r.m.s. deviation for the fitted atoms of 0.029 Å. This group makes a dihedral angle of 41.54 (9)° with the C1–C6 benzene ring.

The crystal packing is stabilized by weak intermolecular C—H···Br interactions (Table 1) which link adjacent molecules into chains running along the *b*-axis direction (Fig. 2).

Experimental

An anhydrous benzene solution (100 ml) of 5-bromo-2-hydroxy-3-methoxybenzaldehyde (2.31 g, 10 mmol) was added dropwise to a solution (50 ml) of 4-methylbenzene-1-sulfonyl chloride (1.91 g, 10 mmol) and pyridine (0.79 g, 10 mmol) in benzene, over a period of 30 min. The mixture was then refluxed for 24 h under a nitrogen atmosphere. The solvent was removed and the mixture poured into ice–water (100 ml). The resulting white precipitate was isolated, recrystallized from acetonitrile and then dried in a vacuum to give the pure compound in 58% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{15}H_{13}BrO_5S$	$V = 758.9$ (6) Å^3
$M_r = 385.22$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.686$ Mg m^{-3}
$a = 7.752$ (3) Å	Mo $K\alpha$ radiation
$b = 9.025$ (4) Å	$\mu = 2.87$ mm^{-1}
$c = 11.638$ (5) Å	$T = 294$ (2) K
$\alpha = 71.559$ (7)°	Block, colorless
$\beta = 87.746$ (7)°	$0.24 \times 0.20 \times 0.14$ mm
$\gamma = 79.326$ (7)°	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3898 measured reflections
	2662 independent reflections
ω scans	1991 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.017$
$T_{min} = 0.485$, $T_{max} = 0.669$	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.033P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$+ 0.3049P]$
$wR(F^2) = 0.083$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2662 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
201 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···Br1^i	0.93	2.96	3.755 (4)	144

Symmetry code: (i) $x, y + 1, z$.
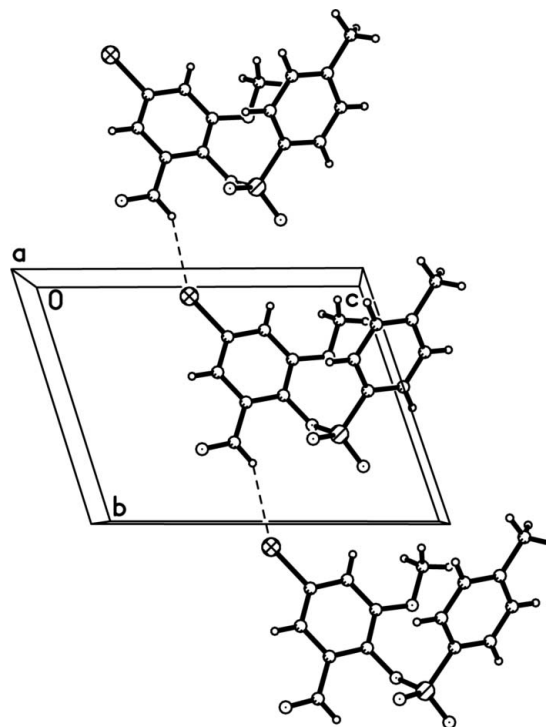
Figure 2
A packing diagram for (I), with C—H···Br interactions shown as dashed lines.

The H atoms were included in calculated positions (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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