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4-Hydroxy-3-nitrophenyl benzenesulfonate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.096
Data-to-parameter ratio = 11.6

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

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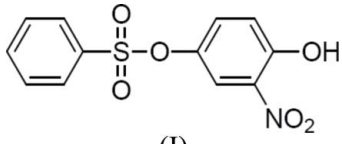
In the title compound, $C_{12}H_9NO_6S$, the two aromatic rings form a dihedral angle of 50.0 (2)°. An intermolecular O—H···O hydrogen bond is formed between the hydroxy group and the sulfonyl O atom of an adjacent molecule.

Comment

Phenolic esters are useful intermediates in organic synthesis (Trollsas *et al.*, 1996; Svensson *et al.*, 1998; Atkinson *et al.*, 2005; Hu *et al.*, 2001). We have developed a new synthetic route to some phenolic esters. In this paper, the structure of the title compound, (I), is reported. The molecular structure of (I) is illustrated in Fig. 1. The two aromatic rings form a dihedral angle of 50.0 (2)°. The torsion angle C7—S1—O1—C1 is 74.30 (13)°.



(I)

Experimental

2-Nitrohydroquinone (1 mmol) was dissolved in chloroform (30 ml). Benzenesulfonyl chloride (1 mmol) and triethylamine (1 mmol) were then added and the reaction was stirred at room temperature for 7 h. The reaction mixture was extracted with dichloromethane and dried

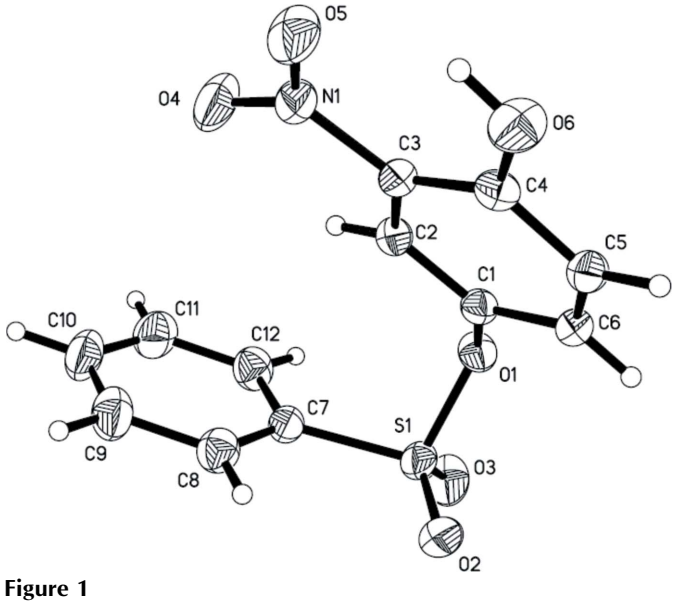


Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

with anhydrous sodium sulfate. After concentration, the residue was separated by flash column chromatography and purified by recrystallization from ethyl acetate (yield 8%; m.p. 342–344 K). IR (KBr, ν, cm^{-1}): 3310, 1625, 1532, 1243. Analysis required for $C_{12}H_9NO_6S$: C 48.81; H 3.07; N 4.74%; found: C 48.83; H 3.15; N 4.73%.

Crystal data

$C_{12}H_9NO_6S$	$Z = 2$
$M_r = 295.27$	$D_x = 1.574$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.1372$ (11) Å	Cell parameters from 1923 reflections
$b = 7.7339$ (13) Å	$\theta = 2.6$–$28.0°$
$c = 13.152$ (2) Å	$\mu = 0.29$ mm^{-1}
$\alpha = 87.833$ (2)°	$T = 293$ (2) K
$\beta = 89.884$ (2)°	Block, colourless
$\gamma = 87.135$ (2)°	0.24 × 0.22 × 0.18 mm
$V = 623.03$ (18) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2192 independent reflections
	1853 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.017$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -5 \rightarrow 7$
$T_{min} = 0.934$, $T_{max} = 0.950$	$k = -9 \rightarrow 9$
3245 measured reflections	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.1779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.035$	
$wR(F^2) = 0.096$	
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
2192 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
184 parameters	$\Delta\rho_{min} = -0.44$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O5	0.825 (10)	1.922 (19)	2.617 (2)	141 (3)
O6—H6···O3^{i}	0.825 (10)	2.35 (2)	2.914 (2)	126 (2)

Symmetry code: (i) $x - 1, y - 1, z$.

All C-bound H atoms were positioned geometrically [C—H = 0.93 Å] and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. The hydroxy H atom was located in a difference Fourier map and refined with O—H restrained to 0.825 (10) Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

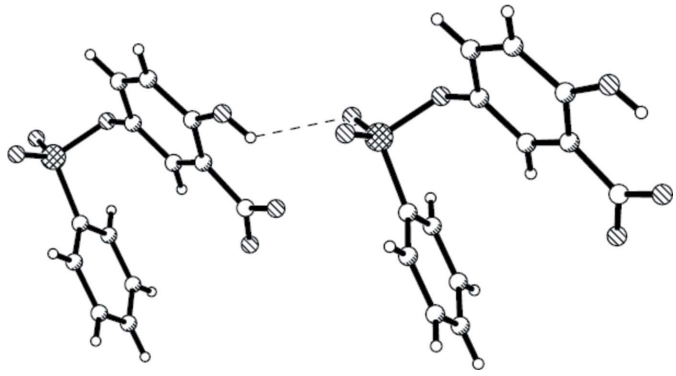


Figure 2
The intermolecular O—H···O hydrogen bond (dashed line) in (I).

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

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