Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Hydroxy-4-nitrophenyl benzenesulfonate

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Received 8 November 2006
Accepted 12 December 2006

The asymmetric unit of the title compound, $C_{12}H_9NO_6S$, contains two independent molecules. There are significant differences between the torsion angles and dihedral angles of the two molecules. An intramolecular O—H···O hydrogen bond is formed between the hydroxy group and the nitro O atom in both molecules.

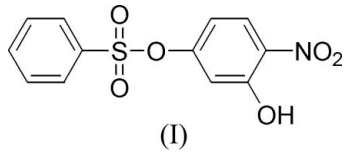
Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.122
Data-to-parameter ratio = 16.4

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

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 synthesic route to some phenolic esters. In this paper, the structure of the title compound, (I), is reported.



(I)

The molecular structure of (I) is illustrated in Fig. 1. There are two independent molecules in the asymmetric unit. In the first molecule, the two aromatic rings form a dihedral angle of 72.8 (2)°, and the C7—S1—O2—C3 torsion angle is −65.14 (17)°. In the second molecule, the two aromatic rings form a dihedral angle of 123.0 (2)°, and the C19—S2—O8—C15 torsion angle is −57.38 (16)°.

Experimental

4-Nitroresorcinol (1 mmol) was dissolved in chloroform (30 ml). Benzenesulfonyl chloride (1 eq) and triethylamine (1 eq) were added and the reaction was stirred at room temperature for 7 h. The reaction mixture was extracted with dichloromethane and dried 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. 351–353 K. Analysis required for $C_{12}H_9NO_6S$: C 48.81; H 3.07; N 4.74%. Found: C 48.81; H 3.14; N 4.70%.

Crystal data

$C_{12}H_9NO_6S$
M_r = 295.27
Triclinic, $P\overline{1}$
a = 8.390 (2) Å
b = 8.594 (3) Å
c = 18.36 (5) Å
α = 76.888 (8)°
β = 80.400 (8)°
γ = 89.340 (9)°
V = 1271 (4) Å^3
Z = 4
D_x = 1.543 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 113 (2) K
Prism, colourless
0.26 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.930, T_{max} = 0.946
11922 measured reflections
6064 independent reflections
3572 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 0.97
6064 reflections
369 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.031 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5	0.89 (3)	1.79 (3)	2.602 (4)	151 (3)
O7—H7···O11	0.84 (3)	1.80 (3)	2.586 (2)	154 (3)

C-bound H atoms were positioned geometrically and refined as riding (C—H = 0.93 Å) with U_{iso}(H)= 1.2U_{eq}(C). Hydroxy H atoms were located in a difference density map and their atomic coordinates allowed to refine freely.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXS97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2004); software used to prepare material for publication: *CrystalStructure*.

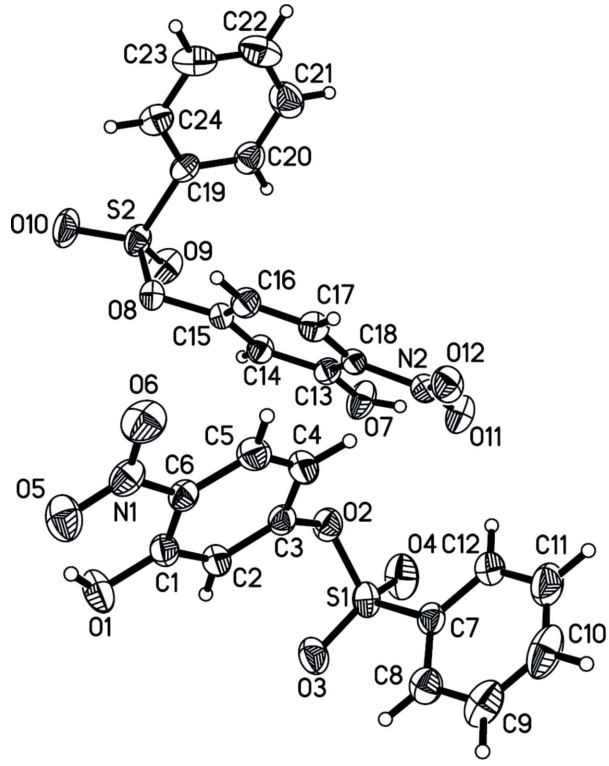


Figure 1
The asymmetric unit of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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