Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Hydroxy-4-nitrophenyl 4-toluenesulfonate

Xiujie Ji,[a] Chunbao Li[a]* and Chao Liu[b]*

[a]Department of Chemistry, Tianjin University, Tianjin 300072, People's Republic of China, and [b]School of Materials Science and Engineering, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: lichunbao@tju.edu.cn, liuchao@tju.edu.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.136
Data-to-parameter ratio = 14.8

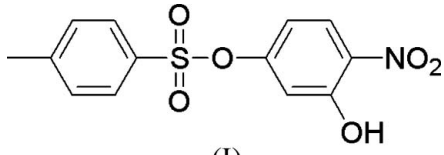
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 January 2006
Accepted 19 January 2006

In the molecule of the title compound, $C_{13}H_{11}NO_6S$, an intramolecular O—H···O hydrogen bond is formed between the hydroxy group and the nitro O atom. The two aromatic rings form a dihedral angle of 48.8 (2)°.

Comment

Phenolic esters are useful intermediates in organic synthesis (Trollsås *et al.*, 1996; Svensson *et al.*, 1998; Atkinson *et al.*, 2005; Hu *et al.*, 2001). We have developed a new method for the synthesis of some phenolic esters. In this paper, the structure of the title compound, (I), is reported.



(I)

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles (Table 1) in (I) are within normal ranges (Allen *et al.*, 1987).

There is an intramolecular O—H···O hydrogen bond (Table 2). The dihedral angle between the two aromatic rings is 48.8 (2)°.

Experimental

2-Nitrohydroquinone (155 mg, 1.0 mmol) was dissolved in chloroform (30 ml). To this solution, 4-toluenesulfonyl chloride (191 mg, 1.0 mmol) and triethylamine (101 mg, 1.0 mmol) were added and the reaction mixture was stirred at room temperature for 7 h. The 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 ethanol (yield 31 mg, 10%; m.p. 342-344 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3197, 2974, 1622, 1534, 1270. Analysis required for $C_{13}H_{11}NO_6S$: C 50.48, H 3.58, N 4.53%; found: C 50.50, H 3.64, N 4.48%.

Crystal data

$C_{13}H_{11}NO_6S$
M_r = 309.30
Orthorhombic, *Pbca*
a = 6.2331 (9) Å
b = 16.000 (2) Å
c = 27.741 (4) Å
V = 2766.6 (7) Å^3
Z = 8
D_x = 1.485 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3444 reflections
θ = 2.6–23.8°
μ = 0.26 mm^{-1}
T = 294 (2) K
Block, colourless
0.26 × 0.24 × 0.12 mm

Data collection

Bruker 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.935, T_{max} = 0.969
14390 measured reflections
2830 independent reflections
1547 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.4°
$h = -7 \rightarrow 7$
$k = -19 \rightarrow 19$
$l = -34 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.136
S = 1.00
2830 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 0.8662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.4142 (19)	O4—C12	1.346 (3)
S1—O1	1.4195 (19)	O5—N1	1.234 (3)
S1—O3	1.6160 (18)	O6—N1	1.224 (4)
S1—C1	1.740 (2)	N1—C11	1.453 (4)
O3—C8	1.402 (3)		
O2—S1—O1	120.55 (12)	O6—N1—O5	122.5 (3)
O2—S1—O3	102.84 (11)	O6—N1—C11	118.5 (3)
O1—S1—O3	107.73 (11)	O5—N1—C11	119.0 (3)
O2—S1—C1	111.42 (12)	C4—C7—H7*A*	109.5
O1—S1—C1	109.92 (12)	O4—C12—C13	117.0 (2)
O3—S1—C1	102.52 (10)	O4—C12—C11	124.7 (3)
C8—O3—S1	118.20 (14)		
C1—S1—O3—C8	56.56 (18)	N1—C11—C12—O4	−0.5 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O5	0.82	1.88	2.581 (3)	143

Most of the reflections were weak, probably due to the crystal quality. The H atoms were positioned geometrically, with O—H = 0.82 Å, and C—H = 0.93 (CH) or 0.96 Å (CH_3), and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C,O), or 1.2U_{eq}(C) for CH.

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*.

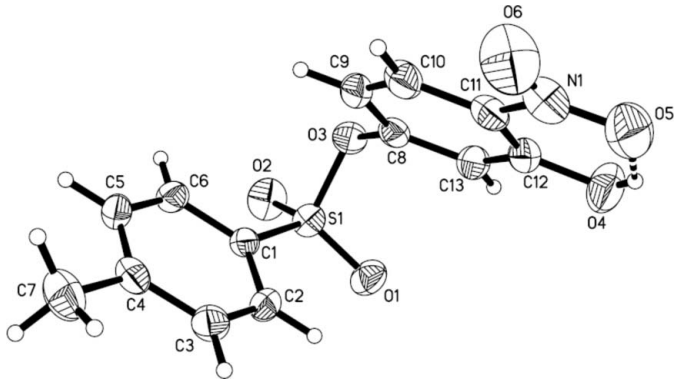


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

The authors thank the TJU Young Teachers Foundation (grant No. W50501) for financial support.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Atkinson, P. J., Bromidge, S. M., Duxon, M. S., Gaster, L. M., Hadley, M. S., Hammond, B., Johnson, C. N., Middlemiss, D. N., North, S. E., Price, G. W., Rami, H. K., Riley, G. J., Scott, C. M., Shaw, T. E., Starr, K. R., Stemp, G., Thewlis, K. M., Thomas, D. R., Thompson, M., Vong, A. K. K. & Watson, J. M. (2005). *Bioorg. Med. Chem. Lett.* **15**, 737–741.
Bruker (1997). *SMART* (Version 5.10), *SAINT* (Version 5.10) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Hu, B., Ellingboe, J., Gunawan, I., Han, S., Largis, E., Li, Z., Malamas, M., Mulvey, R., Oliphant, A., Sum, F.-W., Tillett, J. & Wong, V. (2001). *Bioorg. Med. Chem. Lett.* **11**, 757–760.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Svensson, M., Helgee, B., Skarp, K. & Andersson, G. (1998). *J. Mater. Chem.* **8**, 353–362.
Trollsås, M., Orrenius, C., Sahlén, F., Gedde, U. W., Norin, T., Hult, A., Hermann, D., Rudquist, P., Komitov, L., Lagerwall, S. T. & Lindström, J. (1996). *J. Am. Chem. Soc.* **118**, 8542–8548.