Pearson, W. H., Lian, B. W. & Bergmeier, S. C. (1996). *Comprehensive Heterocyclic Chemistry* II, Vol. IA, edited by A. Padwa, pp. 1–60. New York: Pergamon Press.

Sheldrick, G. M. (1996a). *SADABS. Program for Empirical Absorption Correction of Area Detector Data.* University of Göttingen, Germany.

Sheldrick, G. M. (1996b). *SHELXTL Reference Manual.* Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1996a). *SAINT Software Reference Manual.* Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1996b). *SMART Software Reference Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sriraghavan, K. & Ramakrishnan, V. T. (1998). Unpublished work.

Tanner, D. (1994). *Angew. Chem. Int. Ed. Eng.* **33**, 599–619.

N-(3-Hydroxyphenyl)-p-toluenesulfonamide

SHYAMAPROSAD GOSWAMI,[a] AJIT KUMAR MAHAPATRA,[b] GUR DAYAL NIGAM,[c] KANDASAMY CHINNAKALI[d]† AND HOONG-KUN FUN[d]

[a]*Department of Chemistry, Bengal Engineering College, Botanical Garden, Howrah 711 103, India,* [b]*Department of Chemistry, Indian Institute of Technology, Kharagpur 721 302, West Bengal, India,* [c]*Department of Physics and Meteorology, Indian Institute of Technology, Kharagpur 721 302, West Bengal, India, and* [d]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my*

Abstract

In the title compound, $C_{13}H_{13}NO_3S$, the dihedral angle between the toluene and phenol moieties is 69.48 (5)°. The molecules form columns linked by N—H···O and O—H···O hydrogen bonds.

Comment

Molecular recognition is central to the understanding of biological activity and enzymatic systems (Goswami & Ghosh, 1997). The title compound, (I), contains a potentially active sulfonamide moiety. Sulfonamides have been extensively used against many common grampositive bacterial infections like pneumonia, meningitis, dysentery and urinary-tract infection (Kar, 1993). Hydrogen bonding is expected to play an important role

---

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.

in governing the crystal structure of the title compound (Aoyama *et al.*, 1995). The X-ray structure determination was carried out in order to elucidate the molecular conformation and hydrogen bonding.

(I)
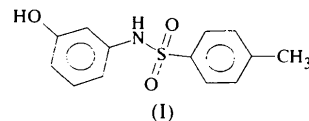
Bond lengths agree with other reported values (Allen *et al.*, 1987). The sum of the bond angles around the N1 atom [349 (1)°] indicates that it is in a pyramidal configuration. The dihedral angle between the toluene and phenol moieties is 69.48 (5)°. Chains of molecules along **b** are linked by O—H···O hydrogen bonds, and glide-related chains are interlinked by N—H···O hydrogen bonds (see Table 2 for details). These two chains form a column-like structure along the **b** axis, with the phenol groups occupying a plane and the toluene groups projecting outwards from it on either side. The toluene groups of neighbouring columns are arranged close to each other, but are not stacked to allow any π–π interactions.
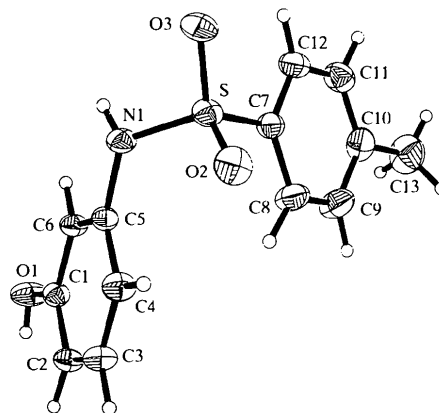

Fig. 1. The structure and atom-numbering scheme of the title compound, shown with 50% probability displacement ellipsoids.

Experimental

For the preparation of the title compound, *p*-toluenesulfonyl chloride (875 mg, 4.5 mmol) was added to a solution of *m*-aminophenol (500 mg, 4.5 mmol) and dry pyridine (0.5 ml) in dry CH_2Cl_2 solvent at 283 K. The reaction mixture was stirred for 15 min at this temperature and for a further 1 h at room temperature. After the usual work-up, the desired compound was obtained in 80% yield (Kurita, 1974). Single crystals were grown by slow evaporation of a 4:1 dichloromethane–methanol solution of the compound.

Crystal data

$C_{13}H_{13}NO_3S$	Mo $K\alpha$ radiation
$M_r = 263.30$	$\lambda = 0.71073$ Å

Monoclinic
$C2/c$
$a = 23.9096\,(7)$ Å
$b = 9.5192\,(3)$ Å
$c = 11.9629\,(3)$ Å
$\beta = 112.136\,(1)^\circ$
$V = 2522.07\,(13)$ Å^3
$Z = 8$
$D_x = 1.387$ Mg m^{-3}
D_m not measured

Cell parameters from 6375
reflections
$\theta = 1.5\text{–}33.0^\circ$
$\mu = 0.256$ mm^{-1}
$T = 293\,(2)$ K
Block
$0.52 \times 0.36 \times 0.30$ mm
Colourless

Data collection

Siemens SMART CCD area-
detector diffractometer
ω scans
Absorption correction:
empirical (*SADABS*;
Sheldrick, 1996*a*)
$T_{min} = 0.871$, $T_{max} = 0.955$
8060 measured reflections

2898 independent reflections
2474 reflections with
$I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.5^\circ$
$h = -30 \rightarrow 28$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.102$
$S = 1.058$
2897 reflections
215 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2$
$+ 1.4298P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.218$ e Å^{-3}
$\Delta\rho_{min} = -0.280$ e Å^{-3}
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S—O2	1.4259 (13)	S—C7	1.760 (2)
S—O3	1.4347 (12)	O1—C1	1.371 (2)
S—N1	1.6214 (13)	N1—C5	1.432 (2)
O2—S—O3	119.55 (8)	O3—S—C7	107.62 (8)
O2—S—N1	108.65 (8)	N1—S—C7	107.96 (7)
O3—S—N1	104.59 (7)	C5—N1—S	123.62 (11)
O2—S—C7	107.97 (8)		
C4—C5—N1—S	52.0 (2)	N1—S—C7—C8	−74.3 (2)
C5—N1—S—C7	64.19 (14)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots$A	D—H	H$\cdots$A	D$\cdots$A	D—H$\cdots$A
O1—H1O1$\cdots$O3^i	0.79 (3)	1.95 (3)	2.727 (2)	171 (2)
N1—H1N1$\cdots$O1^{ii}	0.85 (2)	2.14 (2)	2.983 (2)	174 (2)

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

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was -35°. Coverage of the unique set is over 99% complete. Crystal decay, monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*b*). Cell refinement: *SAINT* (Siemens, 1996*a*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996*b*). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Aoyama, Y., Imai, Y., Endo, K. & Kobayashi, K. (1995). *Tetrahedron*, 51, 343–352.
Goswami, S. P. & Ghosh, K. (1997). *Tetrahedron Lett.* 38, 4503–4506.
Kar, A. (1993). In *Medicinal Chemistry*. New Delhi: Wiley Eastern.
Kurita, K. (1974). *Chem. Ind. (London)*, p. 345.
Nardelli, M. (1995). *J. Appl. Cryst.* 28, 659.
Sheldrick, G. M. (1996*a*). *SADABS. Program for Empirical Absorption Correction of Area Detector Data.* University of Göttingen, Germany.
Sheldrick, G. M. (1996*b*). *SHELXTL Reference Manual.* Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*a*). *SAINT Software Reference Manual.* Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*b*). *SMART Software Reference Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Cyclodecanol

Edward J. Valente,[a] Diwakar M. Pawar[b] and Eric A. Noe[b]

[a]*Department of Chemistry, Mississippi College, Clinton, MS 39058-4065, USA, and* [b]*Department of Chemistry, Jackson State University, Jackson, MS 39217-0510, USA. E-mail: valente@mc.edu*

Abstract

Cyclodecanol crystals, $C_{10}H_{20}O$, grown by slow sublimation, occur in the monoclinic system in space group