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m-(*p*-Tolylsulfonyloxy)aniline

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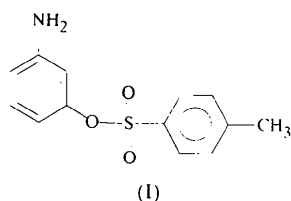
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Abstract

In the title compound, 3-aminophenyl 4-toluenesulfonate, C₁₃H₁₃NO₃S, the dihedral angle between the toluene and aniline moieties is 64.26(5)°. The crystal structure is stabilized by N—H···O intermolecular hydrogen bonds involving amino and sulfonyloxy groups.

Comment

The title compound, (I), is potentially biologically active in mimicking enzyme activity in living organisms. It is expected to show supramolecular behaviour.



The detailed X-ray structure analysis was undertaken to study the molecular conformation and intermolecular hydrogen-bonding scheme. The S—O and N—C distances, and the mean value of the C—C distances [1.381(3) Å], agree with the reported values (Allen *et al.*, 1987). The S atom is tetrahedral. The toluene and aniline moieties have a dihedral angle of 64.26(5)° between them.

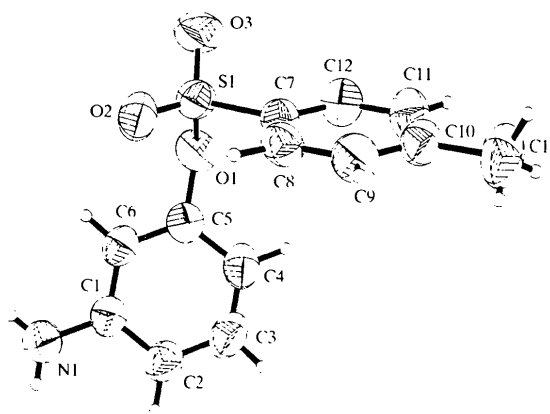


Fig. 1. SHELXTL/PC (Sheldrick, 1990) plot of the structure of (I), showing displacement ellipsoids at the 50% probability level and the atom-numbering scheme.

Experimental

To prepare the title compound, *p*-toluenesulfonyl chloride (1 equivalent) was added to a solution of 3-amino-1-hydroxybenzene (1 equivalent) and triethylamine in dry CH₂Cl₂ at 273 K. The reaction mixture was stirred for 15 min at 273 K and 45 min at room temperature. It gave the desired compound in 90% yield (Kurita, 1974). The unreacted compounds were removed by column chromatography, producing a pure sample of (I). Single crystals were grown by slow evaporation of a 1:1 dichloromethane–petroleum ether solution of the compound.

Crystal data

C₁₃H₁₃NO₃S
M_r = 263.30
Monoclinic
P2₁/c
a = 9.9807(7) Å
b = 7.5585(6) Å
c = 17.0244(12) Å
β = 90.196(7)°
V = 1284.3(2) Å³
Z = 4
D_x = 1.362 Mg m⁻³
D_n, not measured

Mo Kα radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 5.41–12.58°
μ = 0.251 mm⁻¹
T = 293(2) K
Rectangular
0.65 × 0.42 × 0.22 mm
Colourless

Data collection

Siemens P4 diffractometer
θ/2θ scans

R_{int} = 0.028
θ_{max} = 27.50°

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Absorption correction: $h = -1 \rightarrow 12$
 empirical ψ scans $k = -1 \rightarrow 9$
 (Siemens, 1994) $l = -22 \rightarrow 22$
 $T_{\min} = 0.853$, $T_{\max} = 0.939$ 3 standard reflections
 3939 measured reflections every 97 reflections
 2934 independent reflections intensity decay: $<3\%$
 1828 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2 $\Delta\rho_{\max} = 0.266 \text{ e } \text{\AA}^{-3}$
 $R[F^2 > 2\sigma(F^2)] = 0.039$ $\Delta\rho_{\min} = -0.211 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.107$ Extinction correction:
 $S = 0.901$ SHELXL93
 2934 reflections Extinction coefficient:
 216 parameters 0.019 (2)
 All H atoms refined Scattering factors from
 $w = 1/[\sigma^2(F_o^2) + (0.0603P)^2]$ International Tables for
 where $P = (F_o^2 + 2F_c^2)/3$ Crystallography (Vol. C)
 $(\Delta/\sigma)_{\max} < 0.001$

Table 1. Selected bond lengths (\AA)

S1—O2	1.4214 (15)	S1—C7	1.756 (2)
S1—O3	1.4232 (14)	O1—C5	1.431 (2)
S1—O1	1.584 (2)	N1—C1	1.365 (3)

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C8—H8 \cdots O2	0.96 (2)	2.47 (2)	2.901 (2)	107 (1)
N1—H1N1 \cdots O3 ⁱ	0.81 (2)	2.31 (2)	3.098 (3)	165 (2)
N1—H2N1 \cdots O2 ⁱⁱ	0.79 (3)	2.52 (3)	3.278 (3)	162 (2)

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

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: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXL93 and PARST (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1211). Services for accessing these data are described at the back of the journal.

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2-(4-Nitroanilino)-2-phenylethanol

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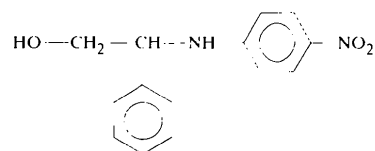
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Abstract

In the title compound, $C_{14}H_{14}N_2O_3$, the nitro group is twisted from coplanarity with the benzene ring by $3.8(3)^\circ$. The benzene ring is perpendicular to the phenyl ring. The molecules are packed around the threefold axis to form an infinite channel containing disordered solvent molecules. $C-H \cdots O$, $O-H \cdots O$ and $N-H \cdots O$ intermolecular hydrogen bonds stabilize the crystal structure.

Comment

The β -aminoalcohol sequence plays an important role in organic as well as in medicinal chemistry (Goodman & Gilman, 1980). Specifically, the β -amino alcohol subunit has been of particular value in the study of acetylcholine metabolism in intact nerve terminal preparations (Rogers *et al.*, 1989). The crystal structure determination of the title compound, (I), one of the above derivatives, was carried out in order to elucidate the molecular conformation.



(I)

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