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4-(Tosylamino)benzoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.119
Data-to-parameter ratio = 13.8

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

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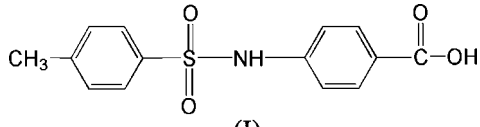
In the molecule of the title compound, $C_{14}H_{13}NO_4S$, the dihedral angle between the two benzene rings is 34.7°. The C—N—S—C torsion angle in the central part of the molecule is 65.67°. The molecular packing involves centrosymmetrically related carboxyl groups connected by O—H···O hydrogen bonds forming dimers, whereas intermolecular N—H···O═S hydrogen bonds connect molecules into a chain along the [011] direction.

Comment

Derivatives of *p*-$(CO_2H)C_6H_4NHSO_2C_6H_4$-*p*-Me have been used as starting materials for the preparation of a variety of sulfonamide drugs, such as an inhibitor of HIV infection (Kazmierski *et al.*, 2004) and an antihypertensive drug (Beate *et al.*, 1998). In addition, they have also been employed in the preparation of gene probe labelling (Skrzipczyk *et al.*, 1994).



(I)

The molecular structure of the title compound, (I), is illustrated in Fig. 1 and selected bond distances and angles are given in Table 1. The dihedral angle between the two benzene rings of the benzoic acid moiety and the tolysulfonamide moiety is 34.7°. The C8—N1—S1—C5 torsion angle in the central part of the molecule is 65.67°.

In the crystal structure, adjacent molecules are linked *via* N—H···O and O—H···O hydrogen bonds (Table 2 and Fig. 2).

Experimental

The title compound was prepared according to the method of Weisblat *et al.* (1953). Ethyl *p*-aminobenzoate (165.1 g, 1.0 mol) was dissolved in 2,4-lutidine (450 ml, 4.0 mol). *p*-Toluenesulfonyl chloride (200 g, 1.05 mol) was added to the vigorously stirred solution at such a rate that the temperature of the solution did not exceed 353 K. After all the *p*-toluenesulfonyl chloride had been added, the temperature was maintained at 348–353 K for an additional 45 min. The hot solution was poured slowly into water (1.5 l) with vigorous stirring and the mixture was cooled to room temperature. The mixture was then filtered and the solid washed well with water. The wet solid was added to sodium hydroxide (100 g) in water (1.5 l) and the solution was brought to the boil and kept boiling for 15 min. The hot solution was clarified and then added slowly to a vigorously stirred solution of acetic acid (152 ml) in water (400 ml). The mixture

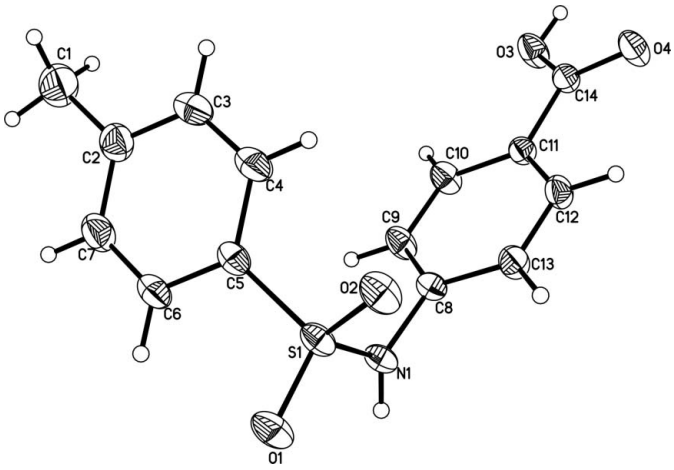


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

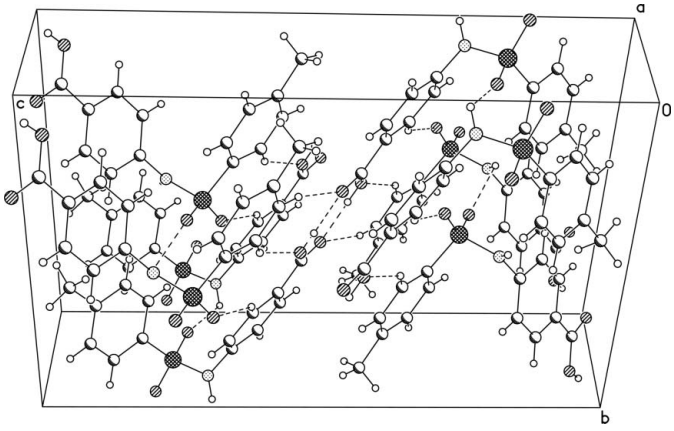


Figure 2
The crystal packing of (I). Intermolecular N—H···O and O—H···O hydrogen bonds are shown as dashed lines.

was cooled, filtered, washed well with water and dried [yield 286 g, 98%; m.p. 503–505 K (decomposition)]. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetone solution.

Crystal data

$C_{14}H_{13}NO_4S$
M_r = 291.31
Monoclinic, $P2_1/c$
a = 5.1626 (10) Å
b = 13.178 (3) Å
c = 20.003 (4) Å
β = 92.11 (3)°
V = 1360.0 (5) Å^3
Z = 4
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 (2) K
Bar, white
0.45 × 0.14 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.896, T_{max} = 0.971
10798 measured reflections
2491 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.02
2491 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.1493P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.4340 (19)	S1—C5	1.7574 (18)
N1—S1	1.6490 (15)	O4—C14	1.2502 (19)
S1—O1	1.4287 (13)	C11—C12	1.385 (2)
O1—S1—O2	120.33 (8)	O4—C14—C11	119.24 (14)
C3—C2—C1	121.4 (2)	O3—C14—C11	117.19 (14)
C12—C11—C14	119.47 (14)		
C8—N1—S1—O1	−178.53 (12)	O2—S1—C5—C4	28.83 (17)
C1—C2—C3—C4	−178.5 (2)	C12—C11—C14—O4	−3.3 (2)
O1—S1—C5—C4	161.90 (15)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.55	2.9882 (19)	113
O3—H3B···O4^{ii}	0.82	1.80	2.6109 (18)	171

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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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References

Beate, G. *et al.* (1998). WO patent No. 9855481.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Kazmierski, W. M., Aquino, C. J., Bifulco, N., Boros, E. E., Chauder, B. A., Chong, P. Y., Duan, M., Deanda, F. Jr, Koble, C. S., Mclean, E. W., Peckham, J. P., Perkins, A. C., Thompson, J. B. & Vanderwall. D. (2004). WO patent No. 2004054974.
Rigaku (2004). *RAPID AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Skrzipczyk, H. J., Uhlmann, E. & Mayer, A. (1994). EP Patent No. 602 524.
Weisblat, D. I., Magerlein, B. J., Hanze, A. R., Myers, D. R. & Rolfson, S. T. (1953). *J. Am. Chem. Soc.* **75**, 3625–3629.