Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(3-Methylanilino)pyridine-3-sulfonamide

Gu-Ping Tang[a]* and Jian-Ming Gu[b]

[a]Institute of Chemical Biology and Pharmaceutical Chemistry, Zhejiang University, Hangzhou, Zhejiang 310028, People's Republic of China, and [b]Center of Analysis and Measurement, Zhejiang University, Hangzhou, Zhejiang 310028, People's Republic of China

Correspondence e-mail: tangguping@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.070
wR factor = 0.180
Data-to-parameter ratio = 12.6

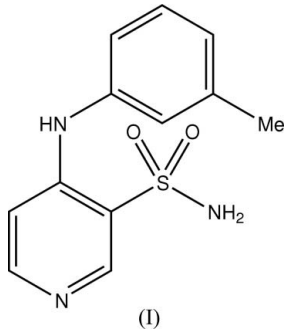
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{12}H_{13}N_3O_2S$, the dihedral angle between the pyridine and benzene rings is 62.1 (1)°. Molecules are linked *via* N—H···N and N—H···O hydrogen bonds, forming a ribbon motif along the *a* axis.

Received 24 August 2005
Accepted 31 August 2005
Online 7 September 2005

Comment

The title compound, (I), is an important intermediate in the preparation of torasemide, which belongs to a group of medications known as loop diuretics (Rollinger *et al.*, 2002; Wouters *et al.*, 2000). The structure of (I) (Fig. 1) exhibits an elaborate hydrogen-bonding network involving N—H···O dimers and two other hydrogen-bonding motifs (Fig. 2). Selected geometric parameters are listed in Table 1, and the hydrogen-bonding geometry in Table 2. Atom N1 acts as a hydrogen-bond donor to atom O1^{ii} [symmetry code: (ii) $-x$, $-y$, $-z$], so generating a centrosymmetric $R_2^2(8)$ graph-set (Etter, 1990) dimer. Intramolecular hydrogen-bond association from N3—H301 to O2 forms an $S(6)$ graph-set motif. Another hydrogen-bond interaction, N1—H101···N2^{i} [symmetry code: (i) $1 + x$, y, z], links molecules into a hydrogen-bonded ribbon motif along the *a* axis.



(I)

Experimental

Compound (I) was supplied by Linhai Dongdong Chemical Factory. Crystals of (I) suitable for X-ray diffraction were grown from an acetone solution by slow evaporation.

Crystal data

$C_{12}H_{13}N_3O_2S$
M_r = 263.31
Triclinic, $P\overline{1}$
a = 6.714 (3) Å
b = 8.630 (4) Å
c = 11.403 (4) Å
α = 98.640 (11)°
β = 102.57 (2)°
γ = 102.911 (12)°
V = 614.7 (4) Å^3
Z = 2
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5401 reflections
θ = 2.5–27.5°
μ = 0.26 mm^{-1}
T = 296 (1) K
Block, colorless
0.30 × 0.25 × 0.12 mm

© 2005 International Union of Crystallography
Printed in Great Britain – all rights reserved

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.921, T_{max} = 0.969
4455 measured reflections
2766 independent reflections
2055 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.085
θ_{max} = 27.5°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.180
S = 1.00
2055 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 4F_o^2/[0.004F_o^2 + 4\sigma(F_o^2) + 0.5]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.416 (2)	S1—C1	1.752 (3)
S1—O2	1.436 (2)	N3—C5	1.356 (4)
S1—N1	1.589 (3)	N3—C6	1.416 (3)
O1—S1—O2	119.25 (14)		
O2—S1—C1—C5	−36.3 (3)	C5—N3—C6—C11	62.2 (5)
N1—S1—C1—C5	78.4 (3)	C6—N3—C5—C4	2.6 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H101···N2^{i}	0.88	2.12	2.890 (4)	147
N1—H102···O1^{ii}	0.86	2.17	2.985 (3)	157
N3—H301···O2	0.86	2.10	2.824 (3)	142
N3—H301···O2^{iii}	0.86	2.47	3.124 (4)	134

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

H atoms bonded to N atoms were located in difference Fourier maps and included in the refinement based on the as-found N—H bond lengths, but their U_{iso} paramenters were refined and fixed in the final stage. All other H atoms were placed in calculated positions with C—H = 0.96–0.98 Å and included in the refinement as riding, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

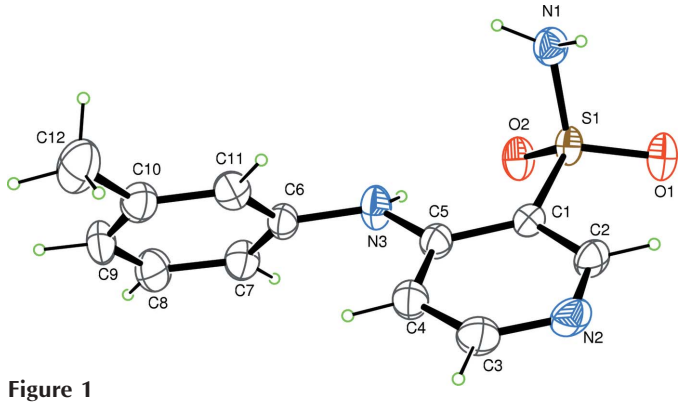


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level.

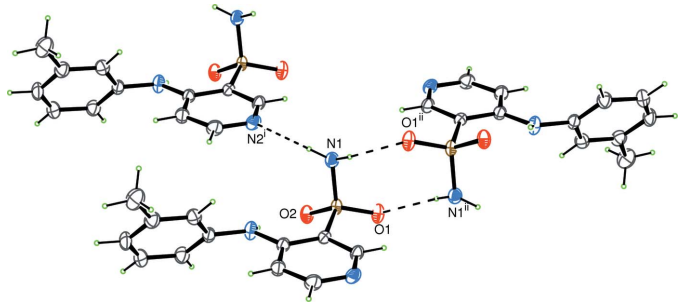


Figure 2
A partial packing diagram for (I), showing the hydrogen-bonded (dashed lines) motif. [Symmetry codes: (i) $1+x, y, z$; (ii) $-x, -y, -z$.]

References

Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.60. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Rollinger, J. M., Gstrein, E. M. & Burger, A. (2002). *Eur. J. Pharm. Biopharm.* **53**, 75–86.
Wouters, J., Michaux, C., Durant, F., Dogne, J. M., Delarge, J. & Masereel, B. (2000). *Eur. J. Med. Chem.* **35**, 923–929.