Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-(3,4-Dimethylphenyl)methanesulfonamide

B. Thimme Gowda,[a]* Sabine Foro[b] and Hartmut Fuess[b]

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

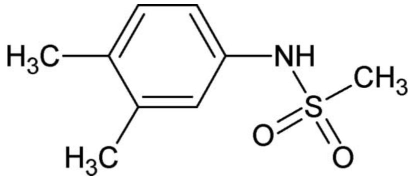
Received 23 May 2007; accepted 28 May 2007

Key indicators: single-crystal X-ray study; *T* = 299 K; mean σ(C–C) = 0.012 Å; *R* factor = 0.082; *wR* factor = 0.266; data-to-parameter ratio = 13.9.

In the structure of the title compound, $C_9H_{13}NO_2S$, the conformation of the N—H bond is *anti* to the *meta*-methyl group, similar to the *anti* conformation observed in *N*-(3-methylphenyl)methanesulfonamide. The amide H atoms lies on one side of the plane of the benzene ring, while the methanesulfonyl group is on the opposite side of the plane, similar to what is observed in other methanesulfonanilides. It is thus available to a receptor molecule during its biological activity. The molecules are packed into chains through N—H···O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2007*a*,*b*,*c*,*d*,*e*,*f*,*g*,*h*,*i*); Jayalakshmi & Gowda (2004); Klug (1968); Spek (2003).



Experimental

Crystal data

$C_9H_{13}NO_2S$
M_r = 199.26
Monoclinic, $P2_1$
a = 8.258 (9) Å
b = 5.847 (9) Å
c = 10.414 (9) Å
β = 97.70 (8)°
V = 498.3 (10) Å^3
Z = 2
Cu *Kα* radiation
μ = 2.64 mm^{-1}
T = 299 (2) K
0.22 × 0.13 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.565, T_{max} = 0.767
1809 measured reflections
1712 independent reflections
1375 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
3 standard reflections
frequency: 120 min
intensity decay: 1.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.082
$wR(F^2)$ = 0.266
S = 1.12
1712 reflections
123 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Absolute structure: Flack (1983), with 734 Friedel pairs
Flack parameter: −0.05 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5N···O3^i	0.85 (4)	2.23 (5)	3.055 (10)	164 (9)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

BTG gratefully thanks the Alexander von Humboldt Foundation, Bonn, Germany, for extensions of his research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2021).

References

Enraf–Nonius (1996). *CAD-4-PC* Software. Version 2.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gowda, B. T., Foro, S. & Fuess, H. (2007*a*). *Acta Cryst.* E**63**, o2337.
Gowda, B. T., Foro, S. & Fuess, H. (2007*b*). *Acta Cryst.* E**63**, o2338.
Gowda, B. T., Foro, S. & Fuess, H. (2007*c*). *Acta Cryst.* E**63**, o2339.
Gowda, B. T., Foro, S. & Fuess, H. (2007*d*). *Acta Cryst.* E**63**, o2340.
Gowda, B. T., Foro, S. & Fuess, H. (2007*e*). *Acta Cryst.* E**63**, o2569.
Gowda, B. T., Foro, S. & Fuess, H. (2007*f*). *Acta Cryst.* E**63**, o2570.
Gowda, B. T., Foro, S. & Fuess, H. (2007*g*). *Acta Cryst.* E**63**, o2597.
Gowda, B. T., Foro, S. & Fuess, H. (2007*h*). *Acta Cryst.* E**63**, o3014.
Gowda, B. T., Foro, S. & Fuess, H. (2007*i*). *Acta Cryst.* E**63**, o3015.
Jayalakshmi, K. L. & Gowda, B. T. (2004). *Z. Naturforsch. Teil A*, **59**, 491–500.
Klug, H. P. (1968). *Acta Cryst.* B**24**, 792–802.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1987). *REDU4*. Stoe & Cie GmbH, Darmstadt, Germany.

doi:10.1107/S1600536807026086

supplementary materials

Acta Cryst. (2007). E**63**, o3085 [**doi:10.1107/S1600536807026086**]

N-(3,4-Dimethylphenyl)methanesulfonamide

B. T. Gowda, S. Foro and H. Fuess

Comment

The structural studies of sulfonanilides are of interest as their biological activity is thought to be due to the amide hydrogen portion of the molecules as it can align itself in relation to a receptor site. In the present work, the structure of *N*-(3,4-dimethylphenyl)-methanesulfonamide (34DMPMSA) has been determined to explore the substituent effects on the solid state structures of sulfonanilides (Gowda *et al.*, 2007*a-i*). The structure of 34DMPMSA (Fig. 1) is similar to those of *N*-(phenyl)- methanesulfonamide (PMSA) (Klug, 1968), *N*-(2-methylphenyl)- methanesulfonamide (2MPMSA)(Gowda *et al.*, 2007*d*), *N*-(3-methylphenyl)-methanesulfonamide (3MPMSA)(Gowda *et al.*, 2007*b*), *N*-(2,3-dimethylphenyl)-methanesulfonamide (23DMPMSA) (Gowda *et al.*, 2007*h*), *N*-(2,4-dimethylphenyl)- methanesulfonamide (24DMPMSA) (Gowda *et al.*, 2007*i*) and other alkyl sulfonanilides (Gowda *et al.*, 2007*a,c,e-g*). The conformation of the N—H bond in 34DMPMSA is *anti* to the *meta* methyl group similar to the *anti* conformation observed in *N*-(3-methylphenyl)-methanesulfonamide (3MPMSA)(Gowda *et al.*, 2007*b*). The substitution of an additional methyl group at the *para* position in 3MPMSA to produce 34DMPMSA changes the space group from orthorhombic Pccn to monoclinic $P2_1$, in contrast to the change over from orthorhombic Pccn to orthorhombic $P2_12_12_1$ space group on an additional methyl group substitution at the *ortho* position in 3MPMSA to produce *N*-(2,3-dimethylphenyl)-methanesulfonamide (23DMPMSA)(Gowda *et al.*, 2007*h*). The bond parameters in 34DMPMSA are similar to those in PMSA, 3MPMSA, 23DMPMSA, 24DMPMSA and other alkyl sulpfonanilides, except for some difference in the torsional angles, C1S2N5C6, S2N5C6C7, S2N5C6C11, O3S2N5C6 and O4S2N5C6. 62.2 (2)°, 75.5 (2)°, −106.6 (2)°, −54.4 (2)°, 177.7 (2)° (PMSA); −64.5 (2)°, 117.1 (2)°, −65.3 (3)°, 51.3 (2)°, 179.1 (2)° (2MPMSA); 57.9 (3)°, 68.1 (4)°, −114.3 (3)°, −57.7 (3)°, 174.7 (3)° (3MPMSA); 71.4 (3)°, 70.1 (4)°, −110.8 (3)°, −44.9 (3)°, −172.6 (3)° (23DMPMSA); −62.9 (3)°, −67.8 (4)°, 113.5 (3)°, 53.1 (3)°, −178.4 (3)° (24DMPMSA); −59.7 (8)°, −58.0 (10)°, 126.2 (7)°, 56.2 (8)°, −175.5 (6)° (34DMPMSA), respectively. The data included for PMSA are the values determined under the present conditions as the literature values were determined by Klug 1968. The N—H hydrogen sits alone on one side of the plane of the phenyl group, while the whole methanesulfonyl group is on the opposite side of the plane, similar to that in other alkyl sulfonanilides. The amide hydrogen is thus available to a receptor molecule during its biological activity. The molecules in 34DMPMSA are packed into chains in the direction of *b* axis (Fig. 2) through N—H···O hydrogen bonds (Fig. 3 and Table 1).

Experimental

The title compound was prepared according to the literature method (Jayalakshmi & Gowda, 2004). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Jayalakshmi & Gowda, 2004). Single crystals of the title compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studied at room temperature.

Refinement

The H atom of the NH group was located in a diffrerence map and its position refined. The carbon-bound H atoms were positioned with idealized geometry and refined using a riding model with C—H = 0.93 Å (CH aromatic) or 0.96 Å (CH_3). Isotropic displacement parameters for all H atoms were set equal to 1.2 U_{eq} (parent atom).

Figures

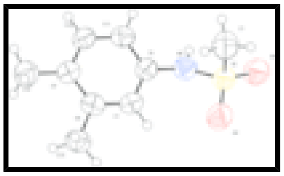


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

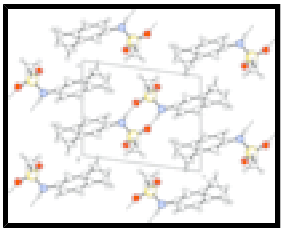


Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

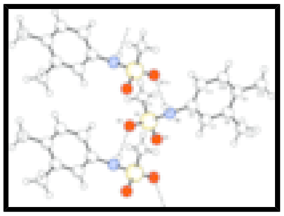


Fig. 3. Hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

N-(3,4-Dimethylphenyl)methanesulfonamide

Crystal data

$C_9H_{13}NO_2S$

M_r = 199.26

Monoclinic, $P2_1$

Hall symbol: P 2yb

a = 8.258 (9) Å

b = 5.847 (9) Å

c = 10.414 (9) Å

β = 97.70 (8)°

V = 498.3 (10) $Å^3$

Z = 2

F_{000} = 212

D_x = 1.328 Mg m^{-3}

Cu $K\alpha$ radiation

λ = 1.54180 Å

Cell parameters from 20 reflections

θ = 5.4–23.6°

μ = 2.64 mm^{-1}

T = 299 (2) K

Prism, red

0.22 × 0.13 × 0.10 mm

Data collection

Enraf–Nonius CAD-4

R_{int} = 0.034

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299(2)$ K

ω scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.565$, $T_{\max} = 0.767$

1809 measured reflections

1712 independent reflections

1375 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 67.0°$

$\theta_{\min} = 4.3°$

$h = -9 \rightarrow 9$

$k = -6 \rightarrow 6$

$l = 0 \rightarrow 12$

3 standard reflections

every 120 min

intensity decay: 1.1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.082$

$wR(F^2) = 0.266$

$S = 1.12$

1712 reflections

123 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1519P)^2 + 0.6985P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.45$ e Å^{-3}

$\Delta\rho_{\min} = -0.55$ e Å^{-3}

Extinction correction: none

Absolute structure: Flack (1983), with 734 Friedel pairs

Flack parameter: −0.05 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.1485 (14)	0.223 (2)	0.4455 (12)	0.088 (3)
H1A	0.0856	0.1951	0.5151	0.106*
H1B	0.0761	0.2465	0.3665	0.106*
H1C	0.2146	0.3569	0.4647	0.106*

C6	0.3492 (9)	0.1408 (14)	0.2002 (7)	0.0535 (18)
C7	0.2552 (8)	0.0009 (17)	0.1121 (7)	0.0552 (16)
H7	0.2202	−0.1403	0.1388	0.066*
C8	0.2127 (9)	0.0697 (15)	−0.0162 (8)	0.0571 (19)
C9	0.2713 (9)	0.2767 (14)	−0.0574 (8)	0.0543 (18)
C10	0.3647 (10)	0.4145 (14)	0.0320 (8)	0.060 (2)
H10	0.4032	0.5537	0.0052	0.071*
C11	0.4020 (10)	0.3495 (14)	0.1602 (9)	0.061 (2)
H11	0.4623	0.4463	0.2192	0.073*
C12	0.2361 (12)	0.3477 (17)	−0.1972 (9)	0.074 (3)
H12A	0.2840	0.4949	−0.2081	0.089*
H12B	0.1201	0.3562	−0.2222	0.089*
H12C	0.2819	0.2375	−0.2504	0.089*
C13	0.1065 (12)	−0.0813 (17)	−0.1082 (10)	0.074 (2)
H13A	0.1548	−0.0992	−0.1865	0.089*
H13B	0.0003	−0.0132	−0.1281	0.089*
H13C	0.0963	−0.2283	−0.0691	0.089*
N5	0.4010 (8)	0.0667 (12)	0.3283 (7)	0.0608 (18)
H5N	0.478 (9)	0.149 (15)	0.367 (8)	0.073*
O3	0.3720 (8)	−0.0573 (12)	0.5494 (6)	0.0723 (18)
O4	0.1756 (9)	−0.1921 (13)	0.3693 (7)	0.084 (2)
S2	0.2748 (2)	−0.0132 (3)	0.42799 (18)	0.0600 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.094 (7)	0.091 (8)	0.082 (7)	0.029 (6)	0.020 (6)	0.012 (6)
C6	0.048 (4)	0.058 (4)	0.054 (4)	0.002 (3)	0.007 (3)	0.003 (3)
C7	0.055 (4)	0.052 (4)	0.060 (4)	0.000 (4)	0.012 (3)	−0.001 (4)
C8	0.047 (4)	0.065 (5)	0.060 (4)	0.002 (3)	0.007 (3)	−0.009 (3)
C9	0.050 (4)	0.058 (5)	0.057 (5)	0.001 (3)	0.011 (3)	−0.001 (4)
C10	0.062 (4)	0.056 (4)	0.061 (5)	−0.005 (3)	0.008 (4)	0.002 (3)
C11	0.063 (5)	0.055 (5)	0.062 (5)	−0.006 (4)	0.001 (4)	−0.007 (4)
C12	0.075 (6)	0.081 (7)	0.063 (6)	0.001 (5)	0.003 (5)	0.001 (5)
C13	0.080 (5)	0.071 (6)	0.070 (6)	−0.009 (5)	0.001 (4)	−0.014 (4)
N5	0.059 (4)	0.069 (4)	0.053 (4)	−0.002 (3)	0.005 (3)	0.004 (3)
O3	0.079 (3)	0.084 (5)	0.053 (3)	0.013 (3)	0.005 (3)	0.013 (3)
O4	0.087 (5)	0.089 (5)	0.078 (5)	−0.023 (4)	0.018 (4)	0.001 (4)
S2	0.0616 (10)	0.0653 (11)	0.0528 (10)	0.0061 (10)	0.0063 (7)	0.0059 (10)

Geometric parameters (Å, °)

C1—S2	1.756 (11)	C10—C11	1.383 (12)
C1—H1A	0.9600	C10—H10	0.9300
C1—H1B	0.9600	C11—H11	0.9300
C1—H1C	0.9600	C12—H12A	0.9600
C6—C11	1.379 (12)	C12—H12B	0.9600
C6—C7	1.386 (11)	C12—H12C	0.9600
C6—N5	1.413 (10)	C13—H13A	0.9600

C7—C8	1.395 (12)	C13—H13B	0.9600
C7—H7	0.9300	C13—H13C	0.9600
C8—C9	1.392 (12)	N5—S2	1.635 (7)
C8—C13	1.497 (11)	N5—H5N	0.85 (4)
C9—C10	1.385 (11)	O3—S2	1.427 (6)
C9—C12	1.504 (11)	O4—S2	1.416 (7)

S2—C1—H1A	109.5	C10—C11—H11	120.1
S2—C1—H1B	109.5	C9—C12—H12A	109.5
H1A—C1—H1B	109.5	C9—C12—H12B	109.5
S2—C1—H1C	109.5	H12A—C12—H12B	109.5
H1A—C1—H1C	109.5	C9—C12—H12C	109.5
H1B—C1—H1C	109.5	H12A—C12—H12C	109.5
C11—C6—C7	119.6 (8)	H12B—C12—H12C	109.5
C11—C6—N5	119.2 (7)	C8—C13—H13A	109.5
C7—C6—N5	121.0 (7)	C8—C13—H13B	109.5
C6—C7—C8	120.7 (9)	H13A—C13—H13B	109.5
C6—C7—H7	119.6	C8—C13—H13C	109.5
C8—C7—H7	119.6	H13A—C13—H13C	109.5
C9—C8—C7	119.5 (8)	H13B—C13—H13C	109.5
C9—C8—C13	120.9 (8)	C6—N5—S2	123.3 (6)
C7—C8—C13	119.6 (9)	C6—N5—H5N	113 (7)
C10—C9—C8	119.0 (8)	S2—N5—H5N	111 (7)
C10—C9—C12	120.5 (8)	O4—S2—O3	118.2 (4)
C8—C9—C12	120.5 (8)	O4—S2—N5	108.5 (4)
C11—C10—C9	121.4 (8)	O3—S2—N5	106.5 (4)
C11—C10—H10	119.3	O4—S2—C1	108.0 (5)
C9—C10—H10	119.3	O3—S2—C1	108.7 (5)
C6—C11—C10	119.8 (8)	N5—S2—C1	106.2 (5)
C6—C11—H11	120.1		

C11—C6—C7—C8	−0.4 (11)	C12—C9—C10—C11	−178.3 (8)
N5—C6—C7—C8	−176.2 (7)	C7—C6—C11—C10	−1.9 (12)
C6—C7—C8—C9	2.8 (11)	N5—C6—C11—C10	174.0 (8)
C6—C7—C8—C13	−178.0 (7)	C9—C10—C11—C6	1.7 (13)
C7—C8—C9—C10	−2.9 (11)	C11—C6—N5—S2	126.2 (7)
C13—C8—C9—C10	177.9 (7)	C7—C6—N5—S2	−58.0 (10)
C7—C8—C9—C12	176.1 (8)	C6—N5—S2—O4	56.2 (8)
C13—C8—C9—C12	−3.1 (12)	C6—N5—S2—O3	−175.5 (6)
C8—C9—C10—C11	0.7 (12)	C6—N5—S2—C1	−59.7 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5N···O3$^{\mathrm{i}}$	0.85 (4)	2.23 (5)	3.055 (10)	164 (9)

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

Fig. 1

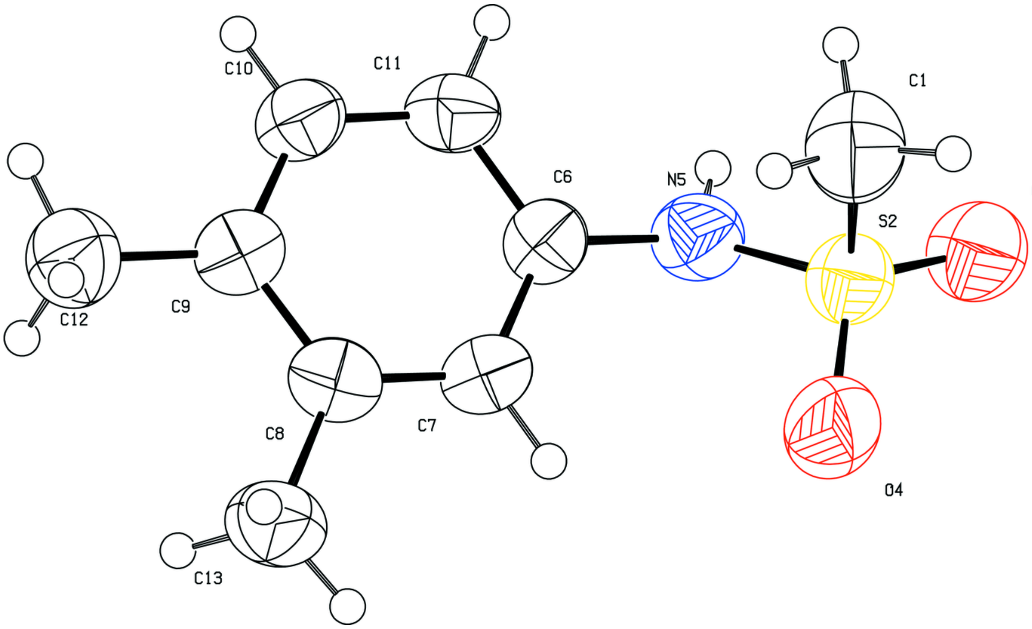


Fig. 2

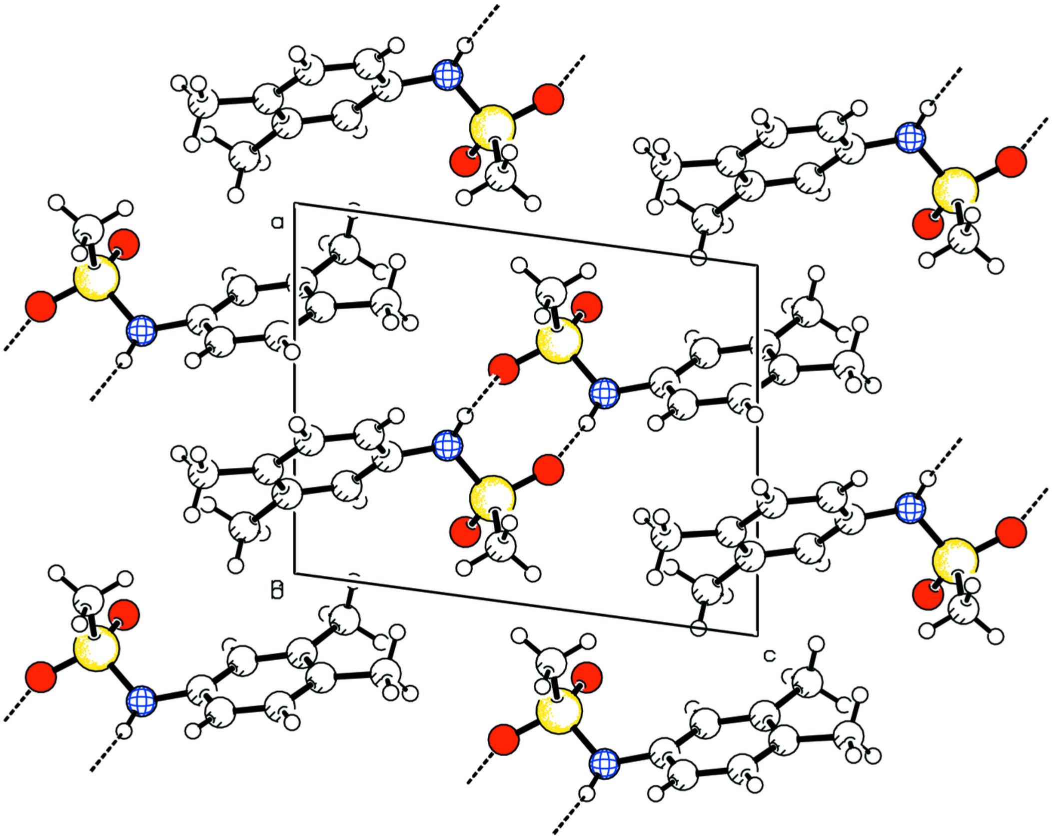


Fig. 3