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N-(2-Bromo-1-phenylethyl)-4-methylbenzenesulfonamide

Yue Meng, Guang-Xiang Zhong and Wei-Xiao Hu*

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310032, People's Republic of China
Correspondence e-mail: huyang@mail.hz.zj.cn

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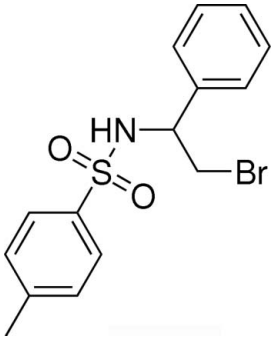
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 17.0.

The title compound, $C_{15}H_{16}BrNO_2S$, was obtained unintentionally in our work on the preparation of vicinal haloamine derivatives. The molecule lies across a crystallographic mirror plane with the NH group disordered across this mirror plane. The dihedral angle between the benzene and phenyl rings is 25.10 (17)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into chains running along the b axis. In addition, C—H···Br hydrogen bonds are observed.

Related literature

For related literature, see: Thakur *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{16}BrNO_2S$
M_r = 354.26
Monoclinic, $P2_1/m$
a = 7.8866 (13) Å
b = 9.5474 (16) Å
c = 10.2881 (16) Å
β = 97.280 (2)°
V = 768.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.81 mm^{-1}
T = 293 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.50, T_{max} = 0.56
4943 measured reflections
1873 independent reflections
1211 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 0.99
1873 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.87	2.14	3.010 (5)	175
C11—H11···Br1^{ii}	0.93	2.90	3.603 (4)	134

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2421).

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supplementary materials

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N-(2-Bromo-1-phenylethyl)-4-methylbenzenesulfonamide

Y. Meng, G.-X. Zhong and W.-X. Hu

Comment

Vicinal haloamine derivates are versatile synthetic intermediates for the synthesis of functional materials and biologically active compounds (Thakur *et al.*, 2003). In our work on the preparation of haloamine derivates we have obtained the title compound, (I).

The molecular structure of (I) is illustrated in Fig.1. The molecule of (I) possesses mirror symmetry, with atoms Br1, S1, C1, C4, C5, C6, C7, C8, C11, H5A and H11 lying on the crystallographic mirror plane. Atom N1 is disordered across the mirror plane. The dihedral angle between the benzene and phenyl rings is 25.10 (17)°.

As shown in Fig.2, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into a chain running along the *b* axis. In addition, C—H···Br hydrogen bonds are observed.

Experimental

Bromine (21.5 g, 0.134 mol) was added to *p*-toluenesulfonamide (11.5 g, 0.067 mol) and cooled to 273 K in an ice bath. Sodium hydroxide hydroxide (20%, 5.4 g, 0.135 mol) in water (21.6 ml) was then added dropwise from a separatory funnel. The lower layer containing the reaction product was dissolved in chloroform (70 ml). To the chloroform solution, styrene (13.6 g, 0.131 mol) was added gradually from a separatory funnel. After the addition, the reaction mixture was heated to 318 K for 1 h. The mixture was cooled in an ice bath, then the precipitate was filtered off and washed with ice-cold absolute ethanol (yield 14.8 g, 0.042 mol). Another semi-solid product (3.6 g, 0.01 mol) was obtained by the evaporation of the chloroform solution and it was washed with cold absolute ethanol. The solid product was dissolved in acetone, the solution was evaporated gradually at room temperature to afford single crystals of (I) (m.p. 442–444 K).

Refinement

Atoms Br1, S1, C1, C4, C5, C6, C7, C8, C11, H5A and H11 lie on the mirror plane. Atom N1 is disordered across the mirror plane. H atoms were placed in calculated positions (N—H = 0.87 Å and C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2–1.5 U_{eq}(C).

Figures

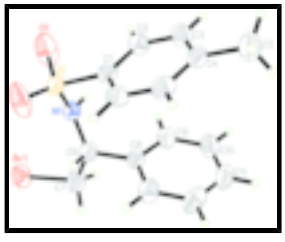

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids. Atoms Br1, S1, C1, C4, C5, C6, C7, C8, C11, H5A and H11 lie on the mirror plane. Atom N1 is disordered across the mirror plane. Unlabelled atoms are related to labelled atoms by the symmetry operation (x, 1/2 − y, z). Only one disorder component is shown.

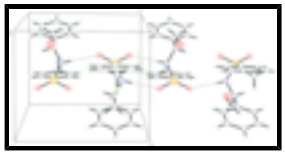

Fig. 2. Packing diagram of (I), showing hydrogen bonds as dashed lines. Only one disorder component is shown.

N-(2-Bromo-1-phenylethyl)-4-methylbenzenesulfonamide

Crystal data

$C_{15}H_{16}BrNO_2S$	F_{000} = 360
M_r = 354.26	D_x = 1.531 Mg m^{-3}
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yb	Cell parameters from 1415 reflections
a = 7.8866 (13) Å	θ = 2.6–23.3º
b = 9.5474 (16) Å	μ = 2.81 mm^{-1}
c = 10.2881 (16) Å	T = 293 (2) K
β = 97.280 (2)º	Block, colourless
V = 768.4 (2) Å^3	0.25 × 0.20 × 0.20 mm
Z = 2	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1873 independent reflections
Radiation source: fine-focus sealed tube	1211 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 293(2) K	θ_{max} = 28.4º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 10$
T_{min} = 0.50, T_{max} = 0.56	$k = -12 \rightarrow 10$
4943 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.098	$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.4403P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.001
1873 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
110 parameters	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}	Occ. (<1)
Br1	0.83529 (7)	0.2500	0.77727 (4)	0.0852 (2)	
S1	0.39919 (13)	0.2500	0.45965 (10)	0.0614 (3)	
O1	0.3345 (5)	0.1259 (2)	0.5024 (2)	0.1211 (11)	
N1	0.5905 (5)	0.1825 (4)	0.5114 (4)	0.0517 (10)	0.50
H1N	0.6121	0.0939	0.5025	0.047 (14)*	0.50
C1	0.3791 (5)	0.2500	0.2874 (3)	0.0509 (9)	
C2	0.3740 (4)	0.1250 (3)	0.2209 (3)	0.0626 (7)	
H2	0.3767	0.0405	0.2662	0.075*	
C3	0.3647 (4)	0.1264 (4)	0.0858 (3)	0.0696 (8)	
H3	0.3601	0.0419	0.0406	0.084*	
C4	0.3621 (5)	0.2500	0.0166 (4)	0.0640 (11)	
C5	0.3536 (7)	0.2500	−0.1320 (4)	0.0944 (17)	
H5A	0.4675	0.2500	−0.1557	0.142*	
H5B	0.2941	0.3321	−0.1670	0.142*	0.50
H5C	0.2941	0.1679	−0.1670	0.142*	0.50
C6	0.7406 (5)	0.2500	0.4975 (4)	0.0647 (11)	
H6	0.7030	0.3453	0.5021	0.078*	0.50
C7	0.8864 (6)	0.2500	0.5997 (4)	0.0940 (17)	
H7A	0.9556	0.3319	0.5875	0.113*	0.50
H7B	0.9556	0.1681	0.5875	0.113*	0.50
C8	0.8036 (5)	0.2500	0.3641 (4)	0.0576 (10)	
C9	0.8369 (4)	0.1261 (4)	0.3045 (3)	0.0771 (9)	
H9	0.8145	0.0414	0.3436	0.093*	
C10	0.9037 (4)	0.1267 (5)	0.1866 (3)	0.0924 (11)	
H10	0.9256	0.0424	0.1466	0.111*	
C11	0.9375 (6)	0.2500	0.1287 (4)	0.0878 (17)	
H11	0.9836	0.2500	0.0498	0.105*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1040 (4)	0.0977 (4)	0.0524 (3)	0.000	0.0044 (2)	0.000

S1	0.0596 (6)	0.0699 (7)	0.0578 (5)	0.000	0.0197 (5)	0.000
O1	0.243 (3)	0.0521 (14)	0.0787 (15)	−0.0046 (18)	0.0605 (18)	0.0099 (12)
N1	0.064 (3)	0.038 (2)	0.055 (2)	0.003 (2)	0.014 (2)	0.0030 (18)
C1	0.047 (2)	0.048 (2)	0.058 (2)	0.000	0.0100 (16)	0.000
C2	0.0691 (18)	0.0509 (17)	0.0686 (17)	0.0065 (14)	0.0116 (14)	0.0017 (14)
C3	0.0711 (19)	0.068 (2)	0.0700 (18)	0.0048 (16)	0.0116 (15)	−0.0138 (16)
C4	0.049 (2)	0.085 (3)	0.059 (2)	0.000	0.0116 (18)	0.000
C5	0.086 (4)	0.134 (5)	0.065 (3)	0.000	0.014 (2)	0.000
C6	0.059 (3)	0.080 (3)	0.056 (2)	0.000	0.0103 (19)	0.000
C7	0.079 (3)	0.151 (5)	0.052 (2)	0.000	0.010 (2)	0.000
C8	0.050 (2)	0.072 (3)	0.051 (2)	0.000	0.0069 (16)	0.000
C9	0.086 (2)	0.078 (2)	0.0699 (19)	−0.0067 (18)	0.0212 (17)	−0.0076 (17)
C10	0.085 (2)	0.118 (3)	0.078 (2)	0.000 (2)	0.0223 (18)	−0.033 (2)
C11	0.058 (3)	0.155 (6)	0.051 (2)	0.000	0.011 (2)	0.000

Geometric parameters (Å, °)

Br1—C7	1.920 (4)	C5—H5A	0.96
S1—O1^{i}	1.384 (2)	C5—H5B	0.96
S1—O1	1.384 (2)	C5—H5C	0.96
S1—N1^{i}	1.665 (4)	C6—N1^{i}	1.371 (5)
S1—N1	1.665 (4)	C6—C7	1.457 (6)
S1—C1	1.759 (4)	C6—C8	1.518 (5)
N1—N1^{i}	1.289 (8)	C6—H6	0.96
N1—C6	1.371 (5)	C7—H7A	0.97
N1—H1N	0.87	C7—H7B	0.97
C1—C2	1.374 (3)	C8—C9^{i}	1.373 (4)
C1—C2^{i}	1.374 (3)	C8—C9	1.373 (4)
C2—C3	1.382 (4)	C9—C10	1.383 (4)
C2—H2	0.93	C9—H9	0.93
C3—C4	1.377 (4)	C10—C11	1.361 (5)
C3—H3	0.93	C10—H10	0.93
C4—C3^{i}	1.377 (4)	C11—C10^{i}	1.361 (5)
C4—C5	1.522 (6)	C11—H11	0.93
O1^{i}—S1—O1	117.9 (2)	C4—C5—H5C	109.5
O1^{i}—S1—N1^{i}	85.6 (2)	H5A—C5—H5C	109.5
O1—S1—N1^{i}	125.8 (2)	H5B—C5—H5C	109.5
O1^{i}—S1—N1	125.8 (2)	N1—C6—N1^{i}	56.1 (4)
O1—S1—N1	85.6 (2)	N1—C6—C7	122.5 (3)
O1^{i}—S1—C1	109.29 (12)	N1^{i}—C6—C7	122.5 (3)
O1—S1—C1	109.29 (12)	N1—C6—C8	118.7 (3)
N1^{i}—S1—C1	106.52 (18)	N1^{i}—C6—C8	118.7 (3)
N1—S1—C1	106.52 (18)	C7—C6—C8	109.5 (3)
N1^{i}—N1—C6	61.97 (19)	N1—C6—H6	99.5
N1^{i}—N1—S1	67.23 (15)	C7—C6—H6	100.6
C6—N1—S1	123.0 (3)	C8—C6—H6	100.6

N1^{i}—N1—H1N	166.5	C6—C7—Br1	116.4 (3)
C6—N1—H1N	105.3	C6—C7—H7A	108.2
S1—N1—H1N	121.6	Br1—C7—H7A	108.2
C2—C1—C2^{i}	120.6 (3)	C6—C7—H7B	108.2
C2—C1—S1	119.67 (17)	Br1—C7—H7B	108.2
C2^{i}—C1—S1	119.67 (17)	H7A—C7—H7B	107.3
C1—C2—C3	119.1 (3)	C9^{i}—C8—C9	119.0 (4)
C1—C2—H2	120.4	C9^{i}—C8—C6	120.44 (19)
C3—C2—H2	120.4	C9—C8—C6	120.44 (19)
C4—C3—C2	121.6 (3)	C8—C9—C10	120.2 (4)
C4—C3—H3	119.2	C8—C9—H9	119.9
C2—C3—H3	119.2	C10—C9—H9	119.9
C3^{i}—C4—C3	117.9 (4)	C11—C10—C9	120.4 (4)
C3^{i}—C4—C5	121.03 (19)	C11—C10—H10	119.8
C3—C4—C5	121.03 (19)	C9—C10—H10	119.8
C4—C5—H5A	109.3	C10^{i}—C11—C10	119.8 (4)
C4—C5—H5B	109.5	C10^{i}—C11—H11	120.1
H5A—C5—H5B	109.5	C10—C11—H11	120.1
O1^{i}—S1—N1—N1^{i}	−32.58 (15)	C2—C3—C4—C5	179.4 (3)
O1—S1—N1—N1^{i}	−154.03 (12)	S1—N1—C6—N1^{i}	29.8 (4)
C1—S1—N1—N1^{i}	97.15 (9)	N1^{i}—N1—C6—C7	109.9 (3)
O1^{i}—S1—N1—C6	−61.0 (4)	S1—N1—C6—C7	139.6 (3)
O1—S1—N1—C6	177.6 (3)	N1^{i}—N1—C6—C8	−107.0 (3)
N1^{i}—S1—N1—C6	−28.4 (4)	S1—N1—C6—C8	−77.2 (3)
C1—S1—N1—C6	68.8 (3)	N1—C6—C7—Br1	−33.9 (3)
O1^{i}—S1—C1—C2	−156.1 (3)	N1^{i}—C6—C7—Br1	33.9 (3)
O1—S1—C1—C2	−25.8 (4)	C8—C6—C7—Br1	180.0
N1^{i}—S1—C1—C2	112.8 (3)	N1—C6—C8—C9^{i}	124.2 (3)
N1—S1—C1—C2	65.2 (3)	N1^{i}—C6—C8—C9^{i}	59.4 (5)
O1^{i}—S1—C1—C2^{i}	25.8 (4)	C7—C6—C8—C9^{i}	−88.2 (3)
O1—S1—C1—C2^{i}	156.1 (3)	N1—C6—C8—C9	−59.4 (5)
N1^{i}—S1—C1—C2^{i}	−65.2 (3)	N1^{i}—C6—C8—C9	−124.2 (3)
N1—S1—C1—C2^{i}	−112.8 (3)	C7—C6—C8—C9	88.2 (3)
C2^{i}—C1—C2—C3	0.4 (6)	C9^{i}—C8—C9—C10	0.3 (6)
S1—C1—C2—C3	−177.6 (2)	C6—C8—C9—C10	−176.3 (3)
C1—C2—C3—C4	0.6 (5)	C8—C9—C10—C11	0.2 (6)
C2—C3—C4—C3^{i}	−1.6 (6)	C9—C10—C11—C10^{i}	−0.7 (7)

Symmetry codes: (i) *x*, −*y*+1/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1^{ii}	0.87	2.14	3.010 (5)	175
C11—H11···Br1^{iii}	0.93	2.90	3.603 (4)	134

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

Fig. 1

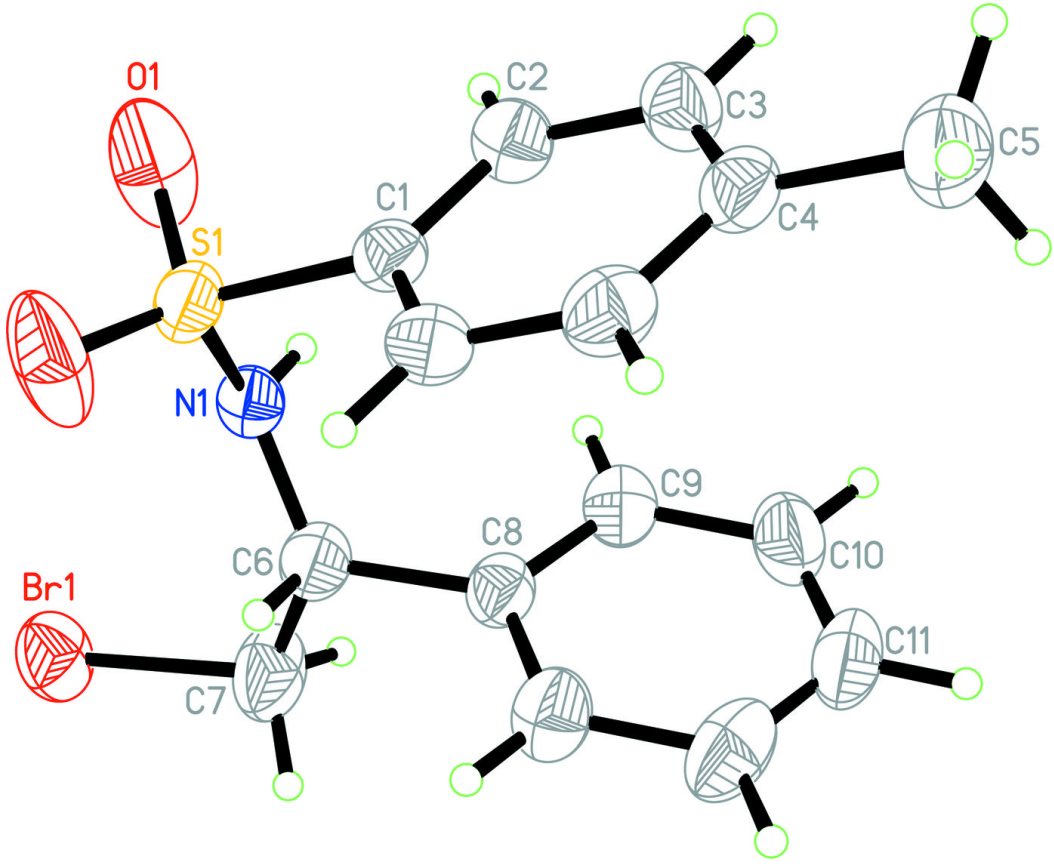
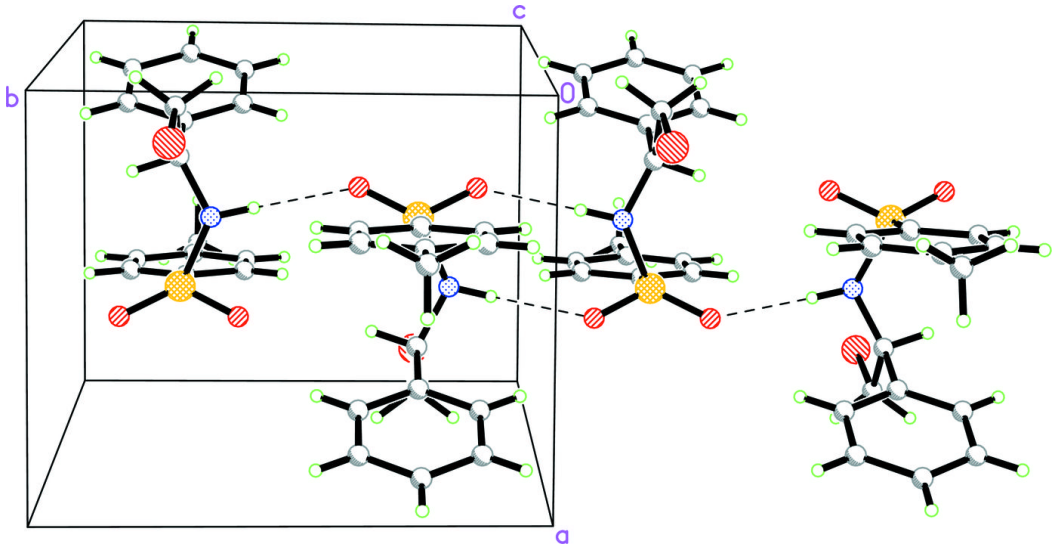
Fig. 2