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4-Methyl-2-phenyl-1-tosylpiperazine

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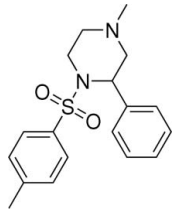
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.083; data-to-parameter ratio = 18.1.

The title compound, $C_{18}H_{22}N_2O_2S$, was obtained unintentionally in our work on the preparation of piperazine derivatives. The piperazine ring has a regular chair conformation, with the N atoms deviating by −0.586 (2) and 0.668 (2) Å from the mean plane formed by the four C atoms. This mean plane and phenyl ring make a dihedral angle of 83.03 (5)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds link the molecules into helical chains running along the *a* axis. The structure is an inversion twin.

Related literature

For related literature, see: Subba Rao & Subrahmanyam (2002).



Experimental

Crystal data

$C_{18}H_{22}N_2O_2S$
M_r = 330.44
Orthorhombic, $P2_12_12_1$
a = 10.2066 (11) Å
b = 10.6427 (11) Å
c = 15.5870 (17) Å
V = 1693.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 296 (2) K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.960
10807 measured reflections
3811 independent reflections
3449 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.04
3811 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1590 Friedel pairs
Flack parameter: 0.50 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···O1^{i}	0.96	2.57	3.453 (3)	152

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

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2229).

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supplementary materials

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4-Methyl-2-phenyl-1-tosylpiperazine

Y. Meng, W. Hu and K. Wu

Comment

Piperazine and its derivates are often are used as antielminitics, perfumes and starting materials in pharmaceutial and agro-chemical industries (Kambala V. *et al.*, 2002). In our work on the preparation of piperazine derivates we have obtained the title compound, (I).

In (I) (Fig.1), the piperazaine ring has a regular chair conformation; C2,C3,C5,C6 atoms are nearly at the same plane, atoms N1 and N4 deviate by -0.586 (2) Å and 0.668 (2) Å, respectively, from this plane. The benzene ring C7—C12 is twisted out of the plane C2/C3/C5/C6 by 83.03 (5)°. The atoms C20,S1 and the other benzene ring C14—C19 also situated in a plane which make a dihedral angle of 36.04 (6) ° with the plane C2/C3/C5/C6.

The weak intermolecular C—H···O hydrogen bonds (Table1) link the molecules into helical chains running along the a-axis (Fig. 2).

Experimental

A suspension of sodium hydroxide (4.4 g, 0.11 mol) in DMF (70 ml) was prepared. *p*-Toluenesulfonamide(8.6 g, 0.05 mol) was dissolved in DMF (40 ml) and the solution was added to the sodium hydroxide suspension.After mixing at room temperature the mixture was heated to 65-70°C for 1 h. After that, the soulution of beta-chloro-*N*-methyl-*N*-chloroethyl phenylethylamine(11.6 g,0.05 mol) in DMF(40 ml) was added gradually from a separatory funnel. The reaction mixture was stirred for 2 h and poured into water(200 ml). After 2 h, the precipitate was filtered and dried to afford 13.3 g of product (yield 80.6%). The solid product was dissolved in acetone,the solution was evaporated gradually at room temperature to afford single crystals of (I). *M*.p. 383.9–385.3 K.

Refinement

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles were refined to fit the electron density, U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

The Flack parameter of 0.50 (7) shows that the crystal is racemically twinned.

Figures

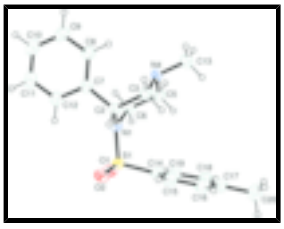

Fig. 1. The molecular structure of (I) with 30% probability displacement ellipsoids.

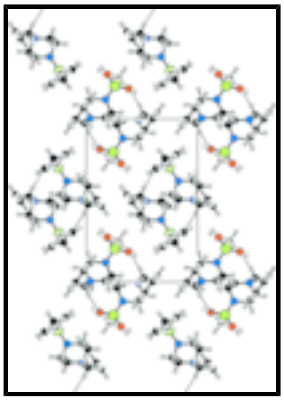

Fig. 2. Packing diagram of (I), viewed along the *a* axis, showing the hydrogen-bond as dashed lines.

4-Methyl-2-phenyl-1-tosylpiperazine

Crystal data

$C_{18}H_{22}N_2O_2S$	F_{000} = 704
M_r = 330.44	D_x = 1.296 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4930 reflections
a = 10.2066 (11) Å	θ = 2.3–28.3°
b = 10.6427 (11) Å	μ = 0.20 mm^{-1}
c = 15.5870 (17) Å	T = 296 (2) K
V = 1693.1 (3) Å^3	Prismatic, colourless
Z = 4	0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3811 independent reflections
Radiation source: fine-focus sealed tube	3449 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 296(2) K	θ_{max} = 27.5°
φ and ω scans	θ_{min} = 2.3°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.951, T_{max} = 0.960	k = −11→13
10807 measured reflections	l = −19→19

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.1655P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.083$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
3811 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}
210 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1590 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.50 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
S1	0.02151 (4)	0.24266 (4)	0.79485 (2)	0.04437 (11)
O1	0.06934 (14)	0.11953 (12)	0.81423 (9)	0.0622 (3)
O2	0.07796 (13)	0.31207 (13)	0.72578 (8)	0.0604 (4)
N1	0.04159 (13)	0.32677 (12)	0.88091 (8)	0.0388 (3)
C2	0.03934 (15)	0.46591 (14)	0.87809 (9)	0.0371 (3)
H2	0.0470	0.4909	0.8178	0.045*
C3	−0.09104 (15)	0.51519 (16)	0.91085 (10)	0.0419 (3)
H3A	−0.0876	0.6062	0.9136	0.050*
H3B	−0.1596	0.4922	0.8707	0.050*
N4	−0.12352 (13)	0.46558 (13)	0.99565 (9)	0.0432 (3)
C5	−0.12911 (19)	0.32900 (16)	0.99267 (11)	0.0495 (4)
H5A	−0.1974	0.3030	0.9532	0.059*
H5B	−0.1509	0.2967	1.0491	0.059*
C6	0.00021 (17)	0.27504 (14)	0.96419 (9)	0.0449 (4)
H6A	0.0664	0.2936	1.0070	0.054*
H6B	−0.0074	0.1844	0.9596	0.054*
C7	0.15998 (16)	0.51458 (15)	0.92445 (10)	0.0403 (3)

C8	0.15569 (18)	0.59222 (16)	0.99594 (12)	0.0499 (4)
H8	0.0752	0.6175	1.0179	0.060*
C9	0.2703 (2)	0.63240 (19)	1.03499 (13)	0.0581 (5)
H9	0.2660	0.6837	1.0832	0.070*
C10	0.38992 (19)	0.5972 (2)	1.00302 (14)	0.0613 (5)
H10	0.4666	0.6244	1.0293	0.074*
C11	0.39541 (18)	0.5215 (2)	0.93211 (14)	0.0610 (5)
H11	0.4763	0.4980	0.9099	0.073*
C12	0.28189 (17)	0.47966 (18)	0.89330 (12)	0.0505 (4)
H12	0.2873	0.4274	0.8456	0.061*
C13	−0.2484 (2)	0.5164 (2)	1.02383 (15)	0.0668 (6)
H13A	−0.2701	0.4823	1.0790	0.100*
H13B	−0.3153	0.4943	0.9833	0.100*
H13C	−0.2422	0.6062	1.0278	0.100*
C14	−0.14845 (17)	0.22811 (15)	0.77585 (9)	0.0435 (4)
C15	−0.2118 (2)	0.31740 (18)	0.72704 (12)	0.0555 (4)
H15	−0.1650	0.3822	0.7013	0.067*
C16	−0.3463 (2)	0.3089 (2)	0.71697 (14)	0.0637 (5)
H16	−0.3893	0.3685	0.6836	0.076*
C17	−0.4182 (2)	0.2146 (2)	0.75493 (12)	0.0606 (5)
C18	−0.3523 (2)	0.1267 (2)	0.80406 (13)	0.0608 (5)
H18	−0.3992	0.0628	0.8308	0.073*
C19	−0.2177 (2)	0.13219 (17)	0.81409 (11)	0.0528 (4)
H19	−0.1744	0.0716	0.8464	0.063*
C20	−0.5649 (2)	0.2056 (3)	0.74181 (18)	0.0864 (8)
H20A	−0.6046	0.1697	0.7920	0.130*
H20B	−0.5829	0.1532	0.6931	0.130*
H20C	−0.6001	0.2880	0.7321	0.130*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0496 (2)	0.0435 (2)	0.04002 (18)	0.00504 (18)	−0.00067 (16)	−0.00628 (16)
O1	0.0689 (8)	0.0465 (7)	0.0711 (8)	0.0178 (6)	−0.0130 (7)	−0.0160 (6)
O2	0.0642 (8)	0.0750 (9)	0.0421 (6)	−0.0034 (7)	0.0122 (6)	−0.0055 (6)
N1	0.0462 (8)	0.0340 (6)	0.0363 (6)	0.0001 (5)	−0.0013 (6)	0.0013 (5)
C2	0.0425 (8)	0.0339 (7)	0.0350 (7)	0.0010 (6)	0.0012 (6)	0.0039 (5)
C3	0.0409 (8)	0.0400 (8)	0.0449 (8)	0.0027 (7)	−0.0053 (7)	0.0014 (7)
N4	0.0391 (7)	0.0461 (8)	0.0444 (7)	−0.0045 (6)	0.0061 (6)	−0.0061 (6)
C5	0.0569 (10)	0.0508 (10)	0.0410 (8)	−0.0148 (8)	0.0070 (8)	0.0014 (7)
C6	0.0597 (10)	0.0376 (8)	0.0374 (7)	−0.0018 (7)	−0.0010 (7)	0.0059 (6)
C7	0.0421 (8)	0.0348 (8)	0.0441 (8)	−0.0024 (6)	0.0012 (7)	0.0090 (6)
C8	0.0470 (9)	0.0471 (9)	0.0556 (9)	−0.0071 (8)	0.0020 (8)	−0.0030 (8)
C9	0.0644 (11)	0.0503 (10)	0.0597 (11)	−0.0144 (9)	−0.0103 (9)	−0.0014 (9)
C10	0.0521 (10)	0.0598 (12)	0.0721 (12)	−0.0154 (9)	−0.0152 (9)	0.0136 (10)
C11	0.0394 (9)	0.0677 (12)	0.0758 (13)	−0.0008 (9)	0.0002 (9)	0.0149 (11)
C12	0.0447 (9)	0.0533 (10)	0.0535 (10)	0.0011 (8)	0.0031 (8)	0.0046 (8)
C13	0.0485 (10)	0.0792 (15)	0.0727 (13)	−0.0023 (10)	0.0163 (9)	−0.0186 (12)

C14	0.0540 (9)	0.0394 (8)	0.0370 (7)	0.0012 (7)	−0.0053 (6)	−0.0040 (6)
C15	0.0643 (11)	0.0507 (10)	0.0516 (10)	0.0004 (9)	−0.0101 (9)	0.0100 (8)
C16	0.0637 (12)	0.0635 (12)	0.0639 (12)	0.0108 (10)	−0.0150 (10)	0.0040 (9)
C17	0.0566 (10)	0.0706 (13)	0.0545 (10)	0.0031 (10)	−0.0052 (9)	−0.0144 (9)
C18	0.0672 (11)	0.0588 (11)	0.0564 (10)	−0.0165 (9)	−0.0040 (9)	−0.0037 (9)
C19	0.0673 (11)	0.0419 (9)	0.0492 (9)	−0.0050 (8)	−0.0097 (8)	0.0037 (7)
C20	0.0575 (12)	0.110 (2)	0.0918 (16)	0.0023 (13)	−0.0035 (12)	−0.0224 (15)

Geometric parameters (Å, °)

S1—O2	1.4271 (13)	C9—H9	0.9300
S1—O1	1.4306 (13)	C10—C11	1.369 (3)
S1—N1	1.6257 (12)	C10—H10	0.9300
S1—C14	1.7666 (18)	C11—C12	1.381 (3)
N1—C6	1.4720 (18)	C11—H11	0.9300
N1—C2	1.4816 (19)	C12—H12	0.9300
C2—C7	1.519 (2)	C13—H13A	0.9599
C2—C3	1.519 (2)	C13—H13B	0.9599
C2—H2	0.9800	C13—H13C	0.9599
C3—N4	1.462 (2)	C14—C19	1.378 (2)
C3—H3A	0.9700	C14—C15	1.378 (2)
C3—H3B	0.9700	C15—C16	1.385 (3)
N4—C13	1.452 (2)	C15—H15	0.9300
N4—C5	1.456 (2)	C16—C17	1.376 (3)
C5—C6	1.506 (3)	C16—H16	0.9300
C5—H5A	0.9700	C17—C18	1.384 (3)
C5—H5B	0.9700	C17—C20	1.514 (3)
C6—H6A	0.9700	C18—C19	1.383 (3)
C6—H6B	0.9700	C18—H18	0.9300
C7—C12	1.386 (2)	C19—H19	0.9300
C7—C8	1.388 (2)	C20—H20A	0.9599
C8—C9	1.386 (3)	C20—H20B	0.9599
C8—H8	0.9300	C20—H20C	0.9599
C9—C10	1.371 (3)		
O2—S1—O1	119.71 (9)	C10—C9—C8	120.49 (19)
O2—S1—N1	106.65 (7)	C10—C9—H9	119.8
O1—S1—N1	106.68 (7)	C8—C9—H9	119.8
O2—S1—C14	108.39 (8)	C11—C10—C9	119.41 (18)
O1—S1—C14	106.87 (8)	C11—C10—H10	120.3
N1—S1—C14	108.08 (7)	C9—C10—H10	120.3
C6—N1—C2	113.30 (12)	C10—C11—C12	120.60 (19)
C6—N1—S1	119.05 (10)	C10—C11—H11	119.7
C2—N1—S1	121.60 (10)	C12—C11—H11	119.7
N1—C2—C7	108.32 (12)	C11—C12—C7	120.89 (18)
N1—C2—C3	110.41 (13)	C11—C12—H12	119.6
C7—C2—C3	115.63 (12)	C7—C12—H12	119.6
N1—C2—H2	107.4	N4—C13—H13A	109.5
C7—C2—H2	107.4	N4—C13—H13B	109.5
C3—C2—H2	107.4	H13A—C13—H13B	109.5

N4—C3—C2	112.21 (13)	N4—C13—H13C	109.5
N4—C3—H3A	109.2	H13A—C13—H13C	109.5
C2—C3—H3A	109.2	H13B—C13—H13C	109.5
N4—C3—H3B	109.2	C19—C14—C15	120.60 (17)
C2—C3—H3B	109.2	C19—C14—S1	119.77 (13)
H3A—C3—H3B	107.9	C15—C14—S1	119.51 (14)
C13—N4—C5	110.32 (15)	C14—C15—C16	118.85 (19)
C13—N4—C3	109.75 (15)	C14—C15—H15	120.6
C5—N4—C3	109.92 (13)	C16—C15—H15	120.6
N4—C5—C6	110.84 (14)	C17—C16—C15	121.81 (19)
N4—C5—H5A	109.5	C17—C16—H16	119.1
C6—C5—H5A	109.5	C15—C16—H16	119.1
N4—C5—H5B	109.5	C16—C17—C18	118.17 (18)
C6—C5—H5B	109.5	C16—C17—C20	121.0 (2)
H5A—C5—H5B	108.1	C18—C17—C20	120.8 (2)
N1—C6—C5	111.63 (13)	C19—C18—C17	121.09 (19)
N1—C6—H6A	109.3	C19—C18—H18	119.5
C5—C6—H6A	109.3	C17—C18—H18	119.5
N1—C6—H6B	109.3	C14—C19—C18	119.48 (17)
C5—C6—H6B	109.3	C14—C19—H19	120.3
H6A—C6—H6B	108.0	C18—C19—H19	120.3
C12—C7—C8	117.97 (16)	C17—C20—H20A	109.5
C12—C7—C2	118.01 (15)	C17—C20—H20B	109.5
C8—C7—C2	124.02 (15)	H20A—C20—H20B	109.5
C9—C8—C7	120.63 (17)	C17—C20—H20C	109.5
C9—C8—H8	119.7	H20A—C20—H20C	109.5
C7—C8—H8	119.7	H20B—C20—H20C	109.5
O2—S1—N1—C6	−176.10 (12)	C2—C7—C8—C9	179.74 (16)
O1—S1—N1—C6	−47.07 (14)	C7—C8—C9—C10	0.7 (3)
C14—S1—N1—C6	67.55 (13)	C8—C9—C10—C11	−0.2 (3)
O2—S1—N1—C2	33.11 (15)	C9—C10—C11—C12	−0.6 (3)
O1—S1—N1—C2	162.13 (13)	C10—C11—C12—C7	0.9 (3)
C14—S1—N1—C2	−83.25 (14)	C8—C7—C12—C11	−0.3 (3)
C6—N1—C2—C7	77.84 (16)	C2—C7—C12—C11	179.51 (16)
S1—N1—C2—C7	−129.84 (12)	O2—S1—C14—C19	157.44 (13)
C6—N1—C2—C3	−49.71 (17)	O1—S1—C14—C19	27.15 (16)
S1—N1—C2—C3	102.62 (14)	N1—S1—C14—C19	−87.34 (15)
N1—C2—C3—N4	53.49 (16)	O2—S1—C14—C15	−26.46 (17)
C7—C2—C3—N4	−69.91 (16)	O1—S1—C14—C15	−156.75 (14)
C2—C3—N4—C13	179.64 (15)	N1—S1—C14—C15	88.76 (15)
C2—C3—N4—C5	−58.86 (17)	C19—C14—C15—C16	−0.1 (3)
C13—N4—C5—C6	−179.55 (15)	S1—C14—C15—C16	−176.18 (15)
C3—N4—C5—C6	59.28 (18)	C14—C15—C16—C17	0.6 (3)
C2—N1—C6—C5	51.48 (18)	C15—C16—C17—C18	−0.2 (3)
S1—N1—C6—C5	−101.61 (14)	C15—C16—C17—C20	−178.9 (2)
N4—C5—C6—N1	−55.84 (18)	C16—C17—C18—C19	−0.7 (3)
N1—C2—C7—C12	60.00 (18)	C20—C17—C18—C19	177.96 (19)
C3—C2—C7—C12	−175.50 (15)	C15—C14—C19—C18	−0.8 (3)
N1—C2—C7—C8	−120.20 (16)	S1—C14—C19—C18	175.28 (14)

C3—C2—C7—C8	4.3 (2)	C17—C18—C19—C14	1.2 (3)
C12—C7—C8—C9	−0.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13A···O1^{i}	0.96	2.57	3.453 (3)	152

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

Fig. 1

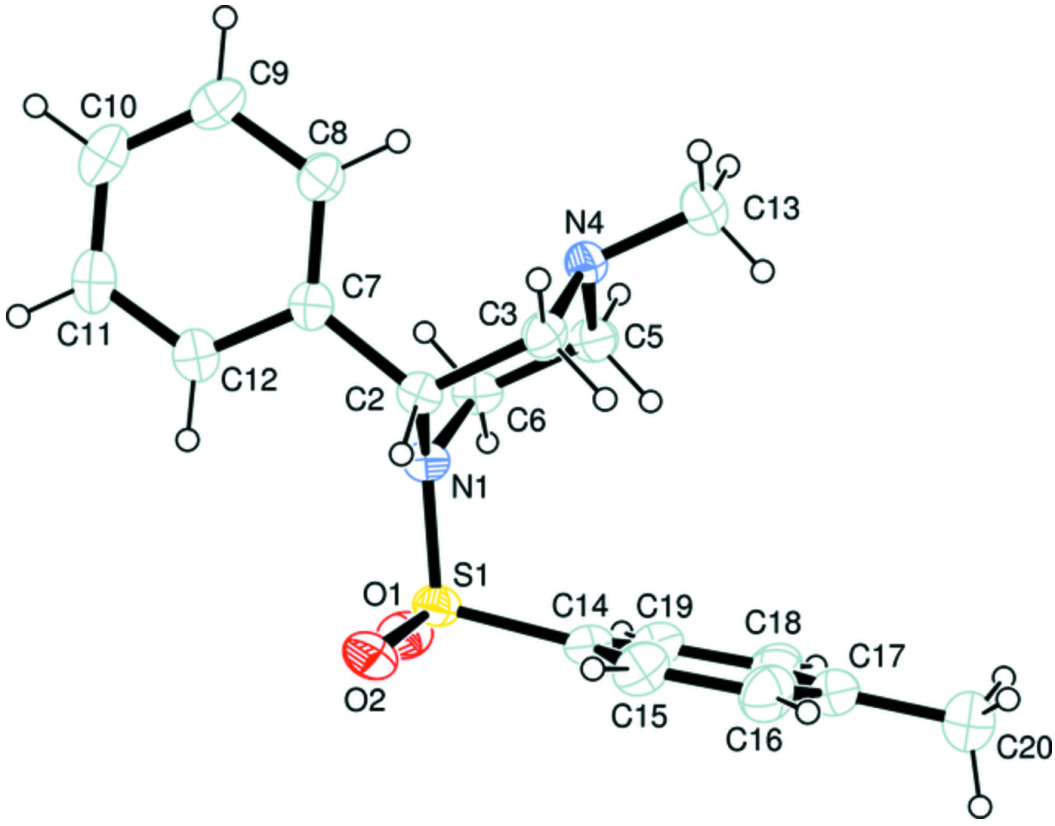
C9
C10
C8
C13
N4
C7
C3
C5
C11
C12
C2
C6
N1
S1
O1
C14
C19
C18
C17
O2
C15
C16
C20

Fig. 2