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7-(*p*-Tolylsulfonyl)-7-azabicyclo[4.1.0]heptane

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The title compound, $C_{13}H_{17}NO_2S$, is an aziridination product of cyclohexene using chloramine-T as a substrate. The aziridine ring is axially bound to the cyclohexane ring, which has a disordered half-chair-like conformation.

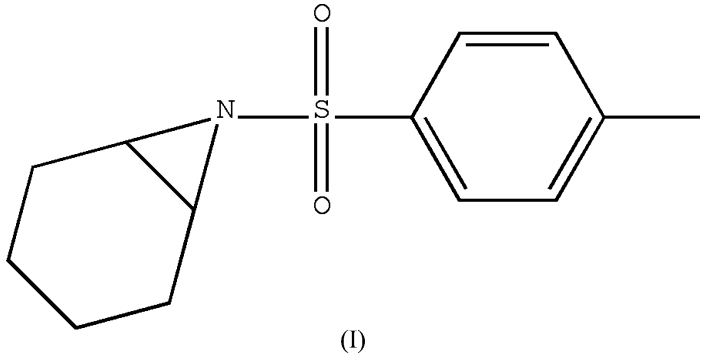
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.147
Data-to-parameter ratio = 15.8

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

Comment

The aziridination of olefins using chloramine-T as a nitrogen source (Jeong *et al.*, 1998) is a practical method (Thakur & Sudalai, 2003) widely used in the synthesis of aziridines. We have recently prepared the title compound, (I), by this method and report its crystal structure here.



(I)

The molecular structure of (I) is shown in Fig. 1. The benzene and aziridine rings are inclined at 38.5 (1)°. The aziridine ring is fused in an axial fashion to the cyclohexane ring. The four C atoms of the cyclohexane ring which are not fused to the aziridine ring are disordered over two sites with relative occupancies of 0.57 and 0.43.

Experimental

Anhydrous chloramine-T (12.5 g) and *N*-bromosuccinamide (2.0 g) were dissolved in anhydrous acetonitrile (250 ml) and then newly distilled cyclohexene (4.0 g) was slowly added. The resulting mixture was stirred at room temperature for 12 h (monitored by thin-layer chromatography). The reaction mixture was then diluted with EtOAc (50 ml) and washed successively with water and brine. The organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether and EtOAc in the proportion of 6:1 as eluent to afford colourless crystals of (I) (yield 64%). These crystals melted at 328 K.

Crystal data

$C_{13}H_{17}NO_2S$
M_r = 251.34
Monoclinic, $P2_1/c$
a = 8.1431 (10) Å
b = 15.0711 (16) Å
c = 10.8067 (11) Å
β = 93.266 (7)°
V = 1324.1 (3) Å^3
Z = 4
D_x = 1.261 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 294 (2) K
Block, colourless
0.14 × 0.12 × 0.10 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.959, T_{max} = 0.982
11462 measured reflections
3151 independent reflections
2095 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
S = 1.11
3151 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.1615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4290 (18)	S1—C7	1.756 (2)
S1—O2	1.4315 (18)	N1—C2	1.483 (3)
S1—N1	1.654 (2)	N1—C1	1.495 (3)
O1—S1—O2	118.23 (12)		

Aziridine atoms H1 and H2 were located in a difference map and refined freely with isotropic displacement parameters. Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

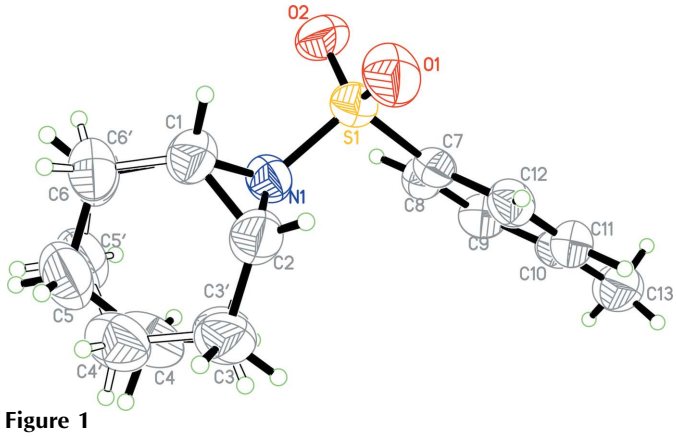


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2004); software used to prepare material for publication: *CrystalStructure*.

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