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5-(*p*-Tolylsulfonyl)-3-oxa-5-azatricyclo[5.2.1.0^{4,8}]-decane

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.132
Data-to-parameter ratio = 15.3

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

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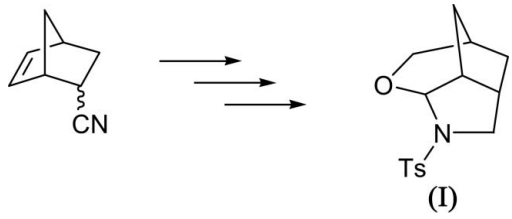
In the title compound, $C_{15}H_{19}NO_3S$, the tricyclodecane part of the molecule can be described in terms of three rings. The C_4N ring is in an envelope conformation, as is the C_5 ring. The C_5O ring adopts a boat conformation. There are no inter- or intramolecular hydrogen-bonding interactions.

Comment

Nitrogen heterocycles, especially pyrrolidine derivatives, are widespread among both natural products and medicinally important synthetic compounds (Dogan & Garner, 2000). Bicyclo derivatives of these compounds, such as those based on the 3-azabicyclo[3.3.0]octane framework, are, however, quite rare. They are only known in a few synthetic analogues, such as prostocyclin (Malleron *et al.*,1995) (PGI2), antidiabetic gliclazide (Bergmeier *et al.*, 1999) and some antibacterial quinolonecarboxylic acid derivatives (Ogata *et al.*, 1991). Besides their physiological activity (Franzky *et al.*, 2000), bicyclopyrrolidine derivatives also serve as chiral auxiliaries (Martens & Wallbaum, 1993) in asymmetric transformations. There is also considerable interest in the development of new methods for preparing cage-like oxaheterocycles (Marchand *et al.*, 2001) and rigid amine-containing heterocycles (Becker *et al.*, 1997) (azacycles). Our studies of the synthesis of the 3-azabicyclo[3.3.0]octane framework resulted in the formation of a new tricycloaminoether, the title compound, (I). We report here the X-ray crystal structure of this interesting compound.



(I)

The tricyclodecane part of (I) can be described in terms of three rings, *A* (N/C8/C9/C13/C14), *B* (C9–C13) and *C* (O3/C11–C15). Ring *A* is in an envelope conformation, with the flap atom, C9, displaced by 0.503 (3) Å from the plane of the other four atoms. Ring *B* is also in an envelope conformation, with the flap atom, C12, lying 0.715 (3) Å from the plane of the other four atoms. Ring *C* adopts a boat conformation, in which atoms C12 and O3 are displaced from the plane through the other four atoms by 0.866 (3) and 0.515 (2) Å, respectively.

No inter- or intramolecular hydrogen-bonding interactions are present in the crystal structure of (I).

Experimental

5-(Toluene-4-sulfonyl)-3-oxa-5-azatricyclo[5.2.1.0^{4,8}]decan-2-one (100 mg, 0.326 mmol) was dissolved in tetrahydrofuran (THF; 1 ml) and added to a round-bottomed flask equipped with a magnetic stirrer, containing a 1 *M* solution of $LiAlH_4$ (10 mg, 0.261 mmol) in THF (0.2 ml) at 273 K. The reaction mixture was then stirred at room temperature for 2 h (monitored by thin-layer chromatography). A 1 N HCl solution (5 ml) and diethyl ether (5 ml) were then added and the layers were separated. The aqueous layer was extracted with diethyl ether (2 × 5 ml) and the combined organic layers were dried over Na_2SO_4 and then concentrated under reduced pressure. The crude product was purified by flash column chromatography (silica gel; hexane–EtOAc 1:5 *v*/*v*) to give 55.6 mg (58%) of the title compound, which was recrystallized from EtOH for X-ray analysis. The elemental analysis and IR and NMR spectroscopic data of (I) have already been published (Kaniskan & Dogan, 2003).

Crystal data

$C_{15}H_{19}NO_3S$
M_r = 293.39
Monoclinic, $P2_1/n$
a = 6.0285 (12) Å
b = 16.4319 (11) Å
c = 14.7046 (14) Å
β = 99.258 (3)°
V = 1437.7 (3) Å^3
Z = 4
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 100 (2) K
Block, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ-scan (*MolEN*; Fair, 1990)
T_{min} = 0.946, T_{max} = 0.966
3026 measured reflections
2928 independent reflections
1739 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.3°
3 standard reflections
frequency: 120 min
intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.02
2776 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.2195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and U_{eq}(H) = 1.2U_{eq}(C).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *CAD-4 EXPRESS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

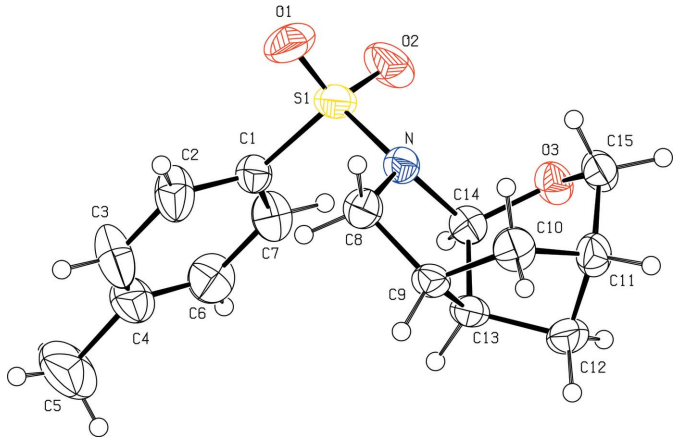


Figure 1
A plot of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

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