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# 1-(1-Naphthylsulfonyl)-1a,2,6,6a-tetrahydro-1*H*,4*H*-1,3-dioxepino[5,6-*b*]azirine

**Biserka Prugovečki,[a] Marina Marinković,[b] Mladen Vinković[b]‡ and Miljenko Dumić[b]***

[a]Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Kralja Zvonimira 8, HR-10000 Zagreb, Croatia, and [b]PLIVA d.d., Research and Development, Prilaz baruna Filipovića 29, HR-10000 Zagreb, Croatia

‡ Present address: Astex Therapeutics, 436 Cambridge Science Park, Cambridge CB4 0QA, England.

Correspondence e-mail: biserka@chem.pmf.hr

**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.051
*wR* factor = 0.128
Data-to-parameter ratio = 21.7

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



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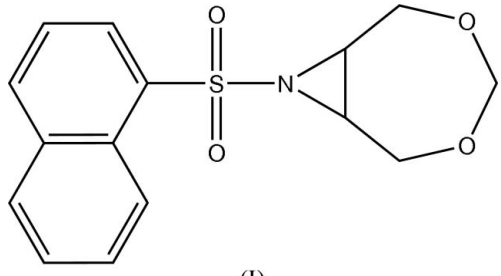
The solid-state conformation of the title compound, $C_{15}H_{15}NO_4S$, has been established by X-ray crystallography. The orientation of the sulfonyl moiety is defined by the C—S—N—C torsion angle of −108.24 (16)°. The angle between the naphthyl ring and the direction of the S—N bond is 71.24 (18)°. The dioxepinoaziridine moiety system a boat–chair (BC) conformation. The crystal packing is defined by optimized π–π interactions between parallel naphthyl groups.

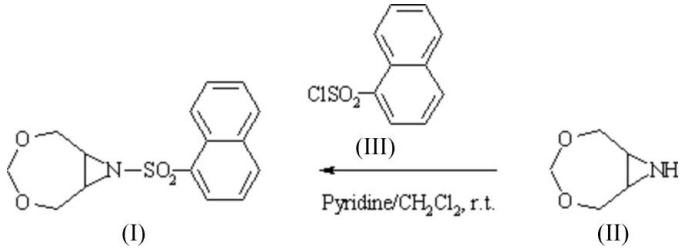
## Comment

Following on from our studies of the design and synthesis of *N*-sulfonyl-1a,2,6,6a-tetrahydro-1*H*,4*H*-[1,3]dioxepino[5,6-*b*]-aziridines as potent antihyperglycaemics (Dumić *et al.*, 1993, 1995; Filić *et al.*, 1996; Vinković *et al.*, 1993) and the conformational behavior of their different analogs bearing sulfanyl-, sulfenyl-, sulfonyl-, carbonyl- and methylene groups (Orešić *et al.*, 2001; Prugovečki *et al.*, 2005), we have now studied the structure of the title compound, (I), in which the bulky planar naphthyl group is attached at the sulfonyl group (Fig. 1).




(I)

Compound (I) was prepared in 62% yield by reaction of the previously prepared dioxepinoaziridine (II) (Dumić *et al.*, 1993) with 1-naphthalenesulfonyl chloride, (III), in the presence of pyridine in methylene chloride at 273 K.




(I) (II)

The orientation of the sulfonyl moiety in relation to the aziridine ring is defined by the C7—S1—N1—C6*A* torsion angle of −108.24 (16)° and adopts the more stable (*A*) (Orešić

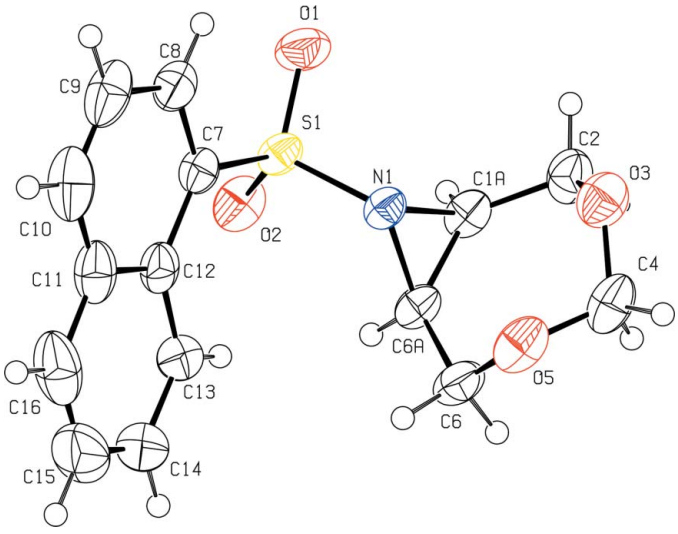



**Figure 1**
View of the molecule of (I), with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

*et al.*, 2001) of the two possible orientations (*A* and *B*; Fig. 2). The dioxepinoaziridine group adopts a boat–chair (BC) conformation. The substituent on the aziridine N atom is in a *trans* position in relation to the dioxepane ring. The aziridine N atom is $sp^3$-hybridized. There are no hydrogen bonds in the crystal packing of (I). $\pi$–$\pi$ interactions between naphthyl groups, with an average distance between atoms in two naphthyl rings of 3.697 Å, stabilize the crystal structure (Fig. 3).

## Experimental

A dry three-necked flask was charged with dioxepinoaziridine (1.9 mmol, 216.0 mg), 1-naphthalenesulfonyl chloride (2.3 mmol, 520.0 mg), pyridine (3.2 mmol, 0.26 ml) and methylene chloride (8 ml). The resulting mixture was stirred at 273 K for 1 h. Upon addition of further methylene chloride (20 ml), the mixture was worked up with aqueous NaOH solution (vol. ratio 1:1) (2·10 ml). The organic layer was separated, washed with water (10 ml), neutralized with diluted HCl up to pH 6, washed once more with water (10 ml) and dried ($Na_2SO_4$). Evaporation of methylene chloride under reduced pressure yielded crude, TLC-pure (I) (360.0 mg, 62.1%), which was recrystallized from ethyl acetate; m.p. 441–442 K. Long thick prismatic crystals suitable for structure determination were obtained by crystallization from methylene chloride. Analysis, $C_{15}H_{15}NO_4S$ requires: C 59.00, H 4.95, N 4.59, S 10.50%; found: C 59.02, H 4.97, N 4.56, S 10.51%. IR (KBr, nmax, $cm^{-1}$): 2960, 2900, 1600, 1510, 1445, 1320, 1180, 1135. MS (FAB): 306 $(M+H)^+$.

*Crystal data*

$C_{15}H_{15}NO_4S$
$M_r$ = 305.34
Monoclinic, $P2_1/c$
$a$ = 10.2540 (7) Å
$b$ = 17.0933 (9) Å
$c$ = 8.0635 (11) Å
$\beta$ = 92.560 (6)°
$V$ = 1411.9 (2) Å$^3$
$Z$ = 4
$D_x$ = 1.436 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 32 reflections
$\theta$ = 12.3–17.8°
$\mu$ = 0.25 mm$^{-1}$
$T$ = 295 (2) K
Prism, colorless
0.4 × 0.3 × 0.2 mm

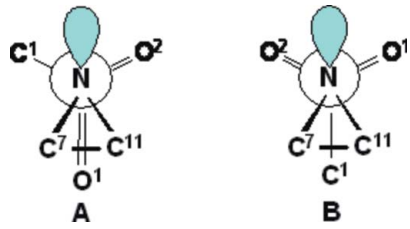



**Figure 2**
Two possible conformations, *A* and *B*, that could be adopted by the sulfonyl moiety of (I).

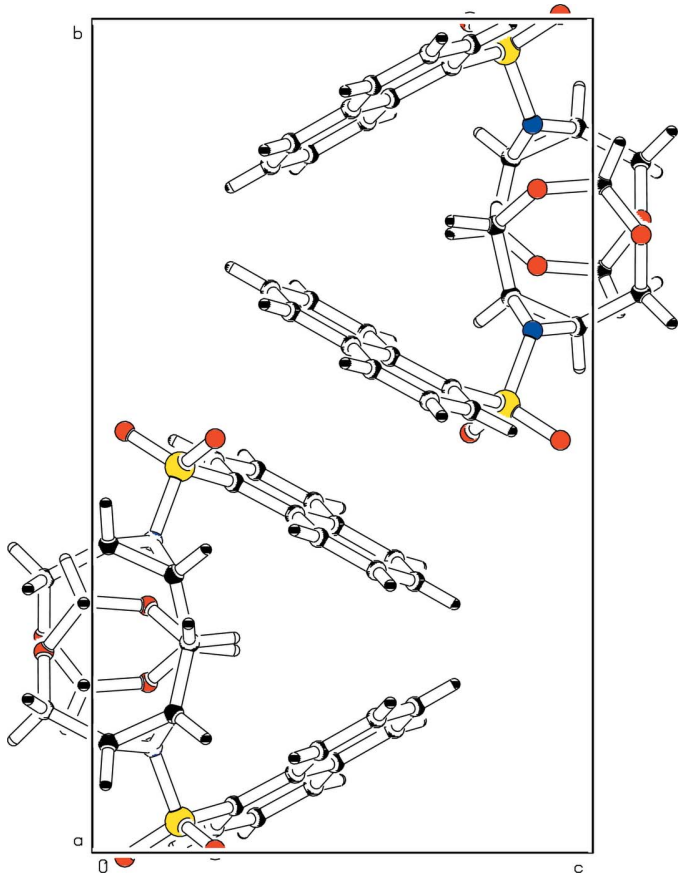



**Figure 3**
Packing of the molecules in the unit cell.

*Data collection*

Philips PW1100 diffractometer updated by Stoe
$\omega$–2$\theta$ scans
4327 measured reflections
4125 independent reflections
1880 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060
$\theta_{max}$ = 30.0°
$h = 0 \rightarrow 14$
$k = -24 \rightarrow 0$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: 3.2%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
$S$ = 0.95
4125 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and treated as riding [$U_{iso}$(H) = 1.2$U_{eq}$(C)].

Data collection: *STADI4* (Stoe & Cie, 1995); cell refinement: *STADI4*; data reduction: *X-RED* (Stoe & Cie, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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