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(–)-*N*-[(3*S*)-3-(4-Fluorophenyl-4-methyl)-hexanoyl]bornane-10,2-sultam

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.135
Data-to-parameter ratio = 19.6

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

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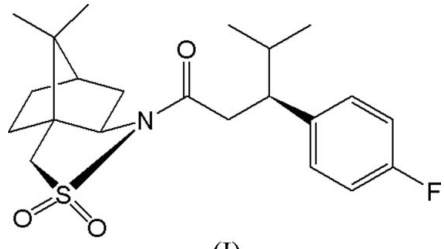
In the title compound, $C_{22}H_{30}FNO_3S$, molecules are linked *via* C—H···O interactions.

Comment

The readily available enantiomers of bornane-10,2-sultam serve as efficient, versatile and practical chiral auxiliaries (Oppolzer, 1990), and we have focused our attention on this field. In this paper, we present the X-ray crystallographic analysis of the title compound, (I).



(I)

In (I), the six-membered ring of sultam shows a boat form (Fig. 1). The C7/C6/C5/C4 and C4/C9/C8/C7 planes form a dihedral angle of 92.6 (3)°. The C4/C3/C7 plane forms dihedral angles to the aforementioned planes of 123.6 (1) and 125.8 (4)°, respectively. The molecules are linked *via* C—H···O interactions (Table 2).

Experimental

For the preapration of compound (I), *N*-[3-(4-fluorophenyl)propenoyl]bornane-10,2-sultam (2.908 g, 8.0 mmol) was reacted with *i*-PrMgCl (16.0 mmol) in anhydrous THF (60 ml) at 193 K (yield 2.184 g, 67%); $[\alpha]_D^{31}$ = −84.2°(c 1.05, $CHCl_3$) (Huang *et al.*, 1999). Crystals appropriate for X-ray data collection were obtained by slow evaporation of a dichloromethane solution at 293 K.

Crystal data

$C_{22}H_{30}FNO_3S$
M_r = 407.53
Orthorhombic, $P2_12_12_1$
a = 11.3245 (11) Å
b = 12.7667 (12) Å
c = 14.6079 (14) Å
V = 2112.0 (3) Å^3
Z = 4
D_x = 1.282 Mg m^{-3}
Mo *Kα* radiation
μ = 0.18 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
20597 measured reflections
5040 independent reflections
4546 reflections with $I > 2\sigma(I)$
R_{int} = 0.088
θ_{max} = 28.2°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 0.1599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.052$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.135$	$\Delta\rho_{max} = 0.46$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
5040 reflections	Absolute structure: Flack (1983), 1860 Friedel pairs
257 parameters	Flack parameter: 0.00 (8)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C9—N1	1.475 (2)	C22—F1	1.360 (3)
C10—S1	1.789 (2)	N1—S1	1.6878 (16)
C11—O3	1.199 (3)	O1—S1	1.425 (2)
C11—N1	1.394 (3)	O2—S1	1.427 (2)
C4—C10—S1	106.61 (14)	N1—S1—C10	95.63 (10)
C4—C5—C6—C7	−4.3 (2)	C12—C13—C17—C18	−56.1 (2)
C7—C8—C9—C4	−9.9 (2)	C4—C9—N1—S1	18.55 (18)
C10—C4—C9—N1	−30.1 (2)	C9—N1—S1—C10	−1.72 (16)
C9—C4—C10—S1	28.4 (2)	C4—C10—S1—N1	−15.94 (19)
C11—C12—C13—C14	166.99 (18)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*B*···N1	0.96	2.58	3.226 (3)	125
C12—H12*A*···O2	0.97	2.43	3.158 (3)	131
C10—H10*A*···O3^{i}	0.97	2.36	3.236 (3)	150
C8—H8*A*···O2^{ii}	0.97	2.60	3.458 (3)	148
C6—H6*A*···O2^{ii}	0.97	2.56	3.477 (3)	157

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

All H atoms were constrained to an ideal geometry with C—H distances of 0.93–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). The absolute configuration of (I) based on the Flack (1983) parameter is consistent with the known absolute configuration of (−)-2,10-sultam (Boiadjiev & Lightner, 2001).

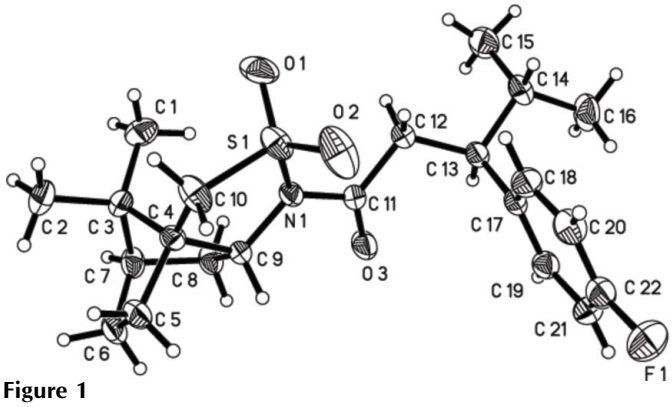


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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