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(1*R*,2*R*,2′*S*)-2-(4-Methylphenylsulfonamido)-1-(pyrrolidine-2′-carboxamido)cyclohexane

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.101
Data-to-parameter ratio = 14.5

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

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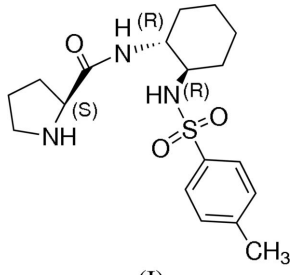
The title compound, $C_{18}H_{27}N_3O_3S$, is an organocatalyst prepared from (1*R*,2*R*)-cyclohexane-1,2-diamine. There are bifurcated N—H···N/O intramolecular hydrogen bonds and N—H···O and C—H···O intermolecular interactions.

Comment

Asymmetric organocatalytic reactions have received much attention recently. The design and synthesis of a bifunctional organocatalyst is challenging field. L-proline and derivatives have been used as effective catalysts in various kinds of asymmetric reactions, such as the Aldol, Mannich and Michael reactions (Notz *et al.*, 2004). The title compound, (I), can be used as a novel organocatalyst and applied in asymmetric transformations. In this paper, we present an X-ray crystallographic analysis of (I).



(I)

The cyclohexane ring adopts a chair form, and the pyrrolidine ring shows conformational disorder (Fig. 1). In the molecule, a bifurcated hydrogen bond (N2—H2···N3/O2) is observed. There are also intermolecular N—H···O and C—H···O interactions (Fig. 2 and Table 2).

Experimental

To a solution of (*R*,*R*)-1,2-cyclohexanediamine (30 mmol) in dichloromethane (40 ml) was dropped slowly a solution of *p*-toluene sulfonyl chloride (10 mmol) in dichloromethane (30 ml) in an ice bath. The mixture was stirred for 20 h at room temperature. The resulting mixture was filtered and was purified by column chromatography on silica gel, with diethyl ether/methanol as eluant. The isolated single-substituted product (6 mmol) was added to a solution of Boc-protected L-proline (10 mmol) in dichloromethane (50 ml) at room temperature. After 12 h, the mixture solution was filtered, trifluoroacetic acid (60 mmol) was added, and the mixture was stirred for 1 h at room temperature. The resulting solution was washed with Na_2CO_3 solution, extracted with dichloromethane and dried, then filtered and purified to afford compound (I) by column chromatography on silica gel, eluting with diethyl ether/methanol (yield 65%).

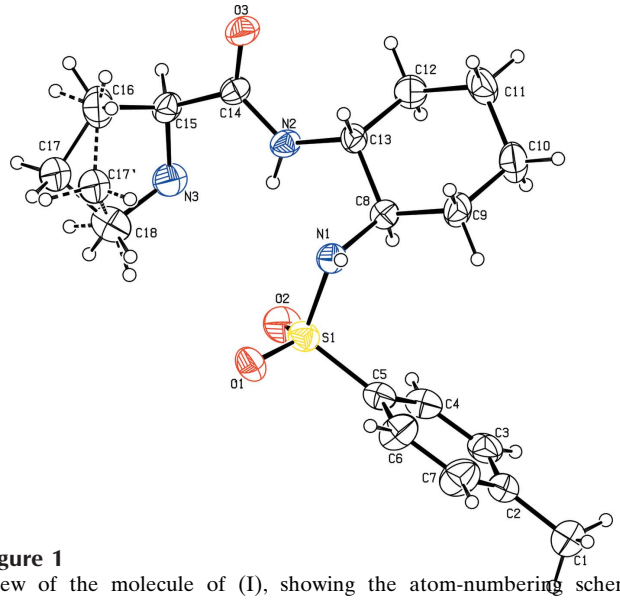


Figure 1
View of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

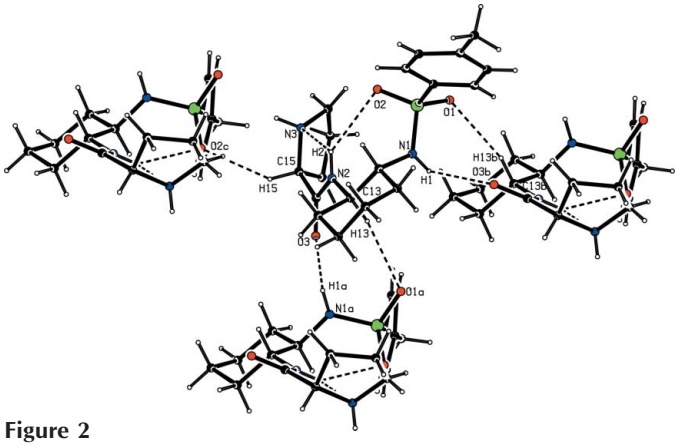


Figure 2
View of the intermolecular and intramolecular hydrogen bonds (dashed lines). Symmetry codes: (*a*) $\frac{1}{2}+x, \frac{3}{2}-y, -z$; (*b*) $x-\frac{1}{2}, \frac{3}{2}-y, -z$; (*c*) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.

Crystals suitable for X-ray diffraction were grown from a dichloromethane-hexane solution at 298 K.

Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz): 1.09–1.27(*m*, 4H), 1.57–2.13 (*m*, 8H), 2.38 (*s*, 3H), 3.02–3.08 (*m*, 1H), 3.55–3.62 (*m*, 1H), 3.70–3.73 (*m*, 1H), 7.24 (*d*, *J* = 8.4 Hz, 2H), 7.71(*d*, *J* = 8.0 Hz, 2H), 7.81(*d*, *J* = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 21.4, 24.5, 26.2, 30.6, 32.3, 33.3, 47.1, 51.8, 58.6, 60.3, 126.6, 129.4, 139.3, 142.7, 176.4; MS (ESI): cald for *M*Na$^+$ 388, found 388.

Crystal data

$C_{18}H_{27}N_3O_3S$
M_r = 365.49
Orthorhombic, $P2_12_12_1$
a = 9.8855 (7) Å
b = 9.8856 (7) Å
c = 20.3477 (14) Å
V = 1988.5 (2) Å^3
Z = 4
D_x = 1.221 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1748 reflections
θ = 2.3–19.6°
μ = 0.18 mm^{-1}
T = 292 (2) K
Block, colorless
0.24 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
10678 measured reflections
3506 independent reflections
2489 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 25.0°
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -24 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.101
S = 0.95
3506 reflections
242 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1482 Friedel pairs
Flack parameter: −0.08 (10)

Table 1
Selected geometric parameters (Å, °).

C5—S1	1.755 (3)	C15—N3	1.459 (4)
C8—N1	1.482 (4)	N1—S1	1.611 (3)
C13—N2	1.448 (4)	O1—S1	1.430 (2)
C14—O3	1.234 (3)	O2—S1	1.429 (2)
C14—N2	1.328 (4)		
N1—C8—C13—N2	−59.3 (3)	C15—C14—N2—C13	−179.2 (3)
N3—C15—C16—C17'	30.0 (9)	C16—C15—N3—C18	−5.9 (4)
N3—C15—C16—C17	−10.9 (5)	C8—N1—S1—C5	67.4 (3)
C16—C17'—C18—N3	40.6 (16)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.85 (1)	2.14 (1)	2.961 (4)	163 (3)
N2—H2···N3	0.86 (1)	2.15 (3)	2.660 (4)	118 (2)
N2—H2···O2	0.86 (1)	2.61 (2)	3.288 (4)	137 (2)
N3—H3*A*···O1^{ii}	0.86 (1)	2.61 (2)	3.411 (4)	155 (3)
C13—H13···O1^{iii}	0.98	2.55	3.506 (4)	164
C15—H15···O2^{ii}	0.98	2.52	3.344 (4)	142

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$.

Atom C17 of pyrrolidine is disordered over two sites (C17 and C17'). They were refined with occupancies of 0786 and 0.204, respectively, which were set at 0.80 and 0.20 for the final refinement. The positional disorder of the H atoms bonded to C16 and C18 were also considered. The H atoms bonded to the N atoms were located in difference-density maps, and refined with an N—H distance restraint of 0.86 (1) Å. The methyl H atoms were constrained to an ideal geometry with C—H distances of 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C). The other H atoms bonded to C atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93–1.00 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

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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