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(1*R*,2*S*)-1-(6-Methoxy-2-naphthyl)-3-methyl-2-(*p*-methylphenylsulfonylamino)-butan-1-ol

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.091
Data-to-parameter ratio = 18.3

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

The title compound, $C_{23}H_{27}NO_4S$, is U-shaped, with the phenyl and naphthalene rings almost parallel, having a dihedral angle of 15.34 (13)°. In the crystal structure, a hydrogen-bonded chain-like structure is observed, extending in the **a** direction.

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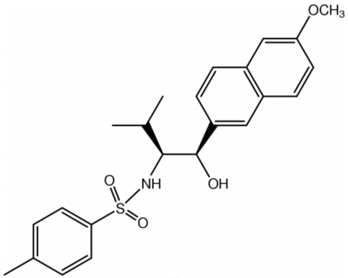
Comment

Chiral amino alcohols and their sulfonamides are important ligands in asymmetric synthesis (Itsuno *et al.*, 1985; Corey *et al.*, 1987, 1998; Deloux & Screbnik, 1993; Ager *et al.*, 1996; Otsuka *et al.*, 1995; Hu *et al.*, 2001). In some instances, ligands with two stereocentres give better enantioselectivity than ligands with one stereocentre (You *et al.*, 2000, 2001). Recently, we have prepared a series of amino alcohols with two stereocentres, using L-valine as the starting material, and found that the groups adjacent to the new chiral centres have strongly influenced the enantioselectivity of the reaction (Li, 2003). Here we report the preparation and crystal structure of the new chiral sulfonamide (1*R*,2*S*)-1-(6-methoxy-2-naphthyl)-3-methyl-2-(*p*-methylphenylsulfonylamino)butan-1-ol, (I).



(I)

Fig. 1 shows the structure of compound (I), which is U-shaped with the substituted phenyl and naphthalene rings almost parallel. An intramolecular π–π contact is observed between a ring of the naphthalene group (atoms C3, C4, C5, C10, C11 and C12) and the phenyl ring (atoms C5, C6, C7, C8, C9 and C10). These rings are inclined at an angle of 14.6 (2)°, and the ring centroids are separated by a distance of 3.81 (5) Å. A weak C—H···π contact, C23—H23*A*···*CgB* (*CgB* is the centroid of plane through atoms C5, C6, C7, C8, C9 and C10), is also found, with a distance of 3.49 (5) Å.

In the crystal structure, two hydrogen bonds are present. One involves the hydroxyl and a sulfonamide O-atom [O1—H1*A*···O4^{ii} with a *D*···*A* distance of 3.033 (3) Å and angle of 149°], and the other involves the amine and the hydroxyl group, N1—H1···O1^{i}, with a *D*···*A* distance of 3.343 (3) Å and an angle of 144° (see Table 1 for symmetry codes). A hydrogen-bonded polymer chain is formed, with the molecules stacking along the *a* axis (Fig. 2).

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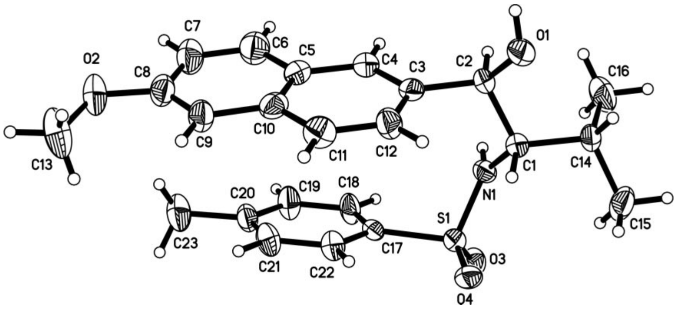

Figure 1
Molecular structure of (I), with displacement ellipsoids at the 30% probability level, and the atom-numbering scheme.

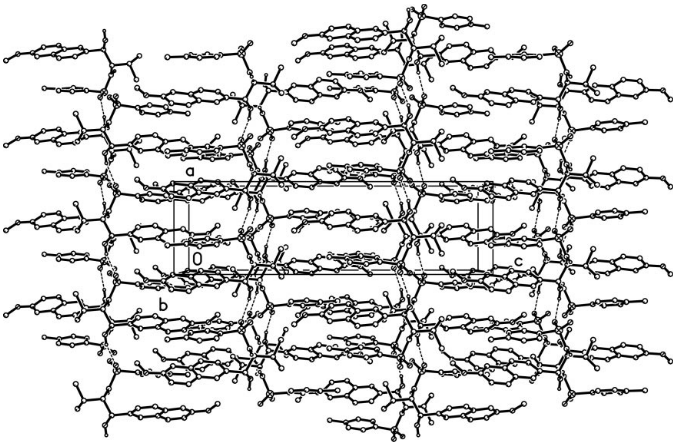

Figure 2
Crystal packing viewed down the *b* axis; dashed lines indicate hydrogen bonding.

Experimental

A mixture of (*R,S*)- and (*S,S*)-2-amino-3-methyl-1-(6-methoxy-2-naphthyl)butan-1-ol were prepared, as described in the literature (Reetz *et al.*, 1987), and were used without separation (the ratio of *R,S* to *S,S* was about 87 to 13 as estimated by ^{1}H NMR analysis). Toluenesulfonyl chloride (0.63 g, 3.3 mmol) and triethylamine (5.0 mmol) were added to a solution of 0.78 g (3.0 mmol) of the amino alcohol in 50 ml of tetrahydrofuran. The mixture was stirred at 273 K for 2 h and then left at room temperature overnight. Water (30 ml) was then added and the organic layers separated. The aqueous layer was extracted with ethyl acetate (3 × 25 ml). The combined organic phase was washed with brine, dried over Na_2SO_4, and the solvents were removed under reduced pressure. The crude products were purified by flash chromatography and crystallized from dichloromethane and hexane to afford the title compound (0.87 g, 71%).

Crystal data

$C_{23}H_{27}NO_4S$
$M_r = 413.52$
Orthorhombic, $P2_12_12_1$
$a = 7.3107$ (13) Å
$b = 11.5702$ (19) Å
$c = 25.117$ (4) Å
$V = 2124.6$ (6) Å^3
$Z = 4$
$D_x = 1.293$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3575 reflections
$\theta = 1$–27.5°
$\mu = 0.18$ mm^{-1}
$T = 294$ (2) K
Prism, colourless
0.32 × 0.28 × 0.24 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.944$, $T_{\max} = 0.958$
14469 measured reflections
4879 independent reflections
2113 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.072$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 13$
$l = -23 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.091$
$S = 0.80$
4879 reflections
266 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.03(9), 2113 Friedel pairs

Table 1
Hydrogen-bonding geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*B*⋯O1$^{\rm i}$	0.86	2.61	3.343 (3)	144
O1—H1*A*⋯O4$^{\rm ii}$	0.82	2.30	3.033 (3)	149

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

The absolute configuration of the molecule indicates that atom C1 has an *S* configuration and atom C2 an *R* configuration. The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding-model approximation.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* and *SHELXTL-NT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

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