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(1*R*,2*S*)-3-Methyl-2-(*p*-methylphenylsulfonylamino)-1-phenylbutan-1-ol

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.089
Data-to-parameter ratio = 19.0

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

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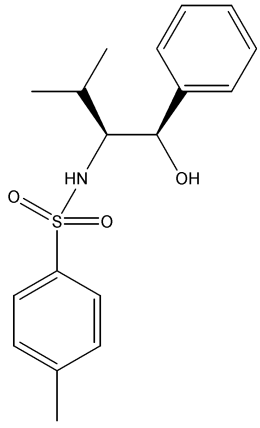
In the crystal structure of the title compound, $C_{18}H_{23}NO_3$, the molecules are linked by intermolecular N—H···O and O—H···O hydrogen bonds to form sheet-like structures perpendicular to the *c* cell direction.

Comment

Chiral sulfonamides, derived from amino alcohols, have been investigated as chiral ligands for the reduction of prochiral ketones (Hu *et al.*, 2001; Otsuka *et al.*, 1995), enantioselective copper-catalyzed 1,4-addition of diethylzinc to cyclohexenone (Wendisch & Sewald, 1997), asymmetric trialkylaluminium and diethylzinc addition to aldehydes catalyzed by titanium complexes (You *et al.*, 2001; Ito *et al.*, 1992), ruthenium-catalyzed asymmetric transfer hydrogenation of functionalized ketones (Everaere *et al.*, 2001), and enantioselective trimethylsilylcyanation of aldehydes (You *et al.*, 2000). It is known that differences in ligand structures strongly influence the enantioselectivity of the reaction. For amino alcohols and their derivatives, ligands with two stereocentres usually give better enantioselectivity than ligands with one stereocentre. Furthermore, ligands with an *R,S* configuration are more effective for asymmetric reaction than the *S,S* diastereomers (You *et al.*, 2000, 2001).



(I)

As there is only one stereocentre in most amino alcohols derived from natural amino acids, it is of interest to develop new amino alcohols and their derivatives with two stereocentres for studying their structure and application in asymmetric reactions. Here we report the preparation and crystal structure of (1*R*,2*S*)-3-methyl-2-(*p*-methylphenylsulfonylamino)-1-phenylbutan-1-ol, (I).

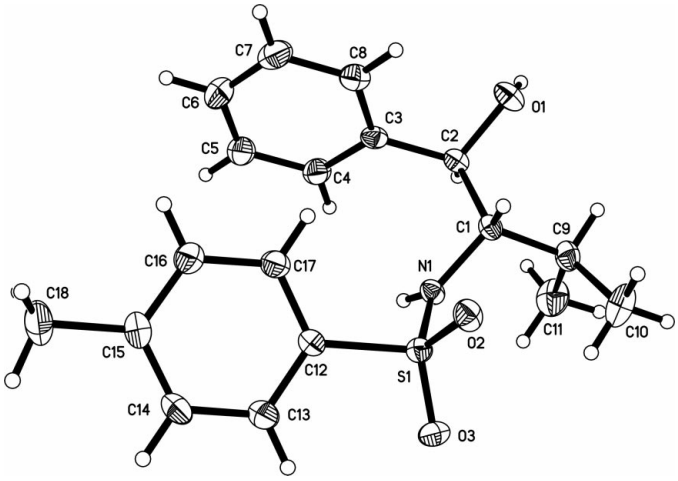


Figure 1
The molecular structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The structure of (I) is shown in Fig. 1. As the starting material used for the synthesis of (I) is L-valine, it is reasonable that the configuration of C1 is *S*. The new chiral C atom, C2, is in the *R* configuration. An intramolecular C—H···π contact is observed in the structure so that C17—H17*A* is 2.74 Å from the ring centroid of C3–C8, with an angle at H17*A* of 127°. In the crystal, the molecules translated along the *a* cell axis are linked through N—H···O hydrogen bonds (Table 1) to form infinite one-dimensional molecular chains and these are interlinked by O—H···O hydrogen bonds to form a two-dimensional molecular network (Fig. 2).

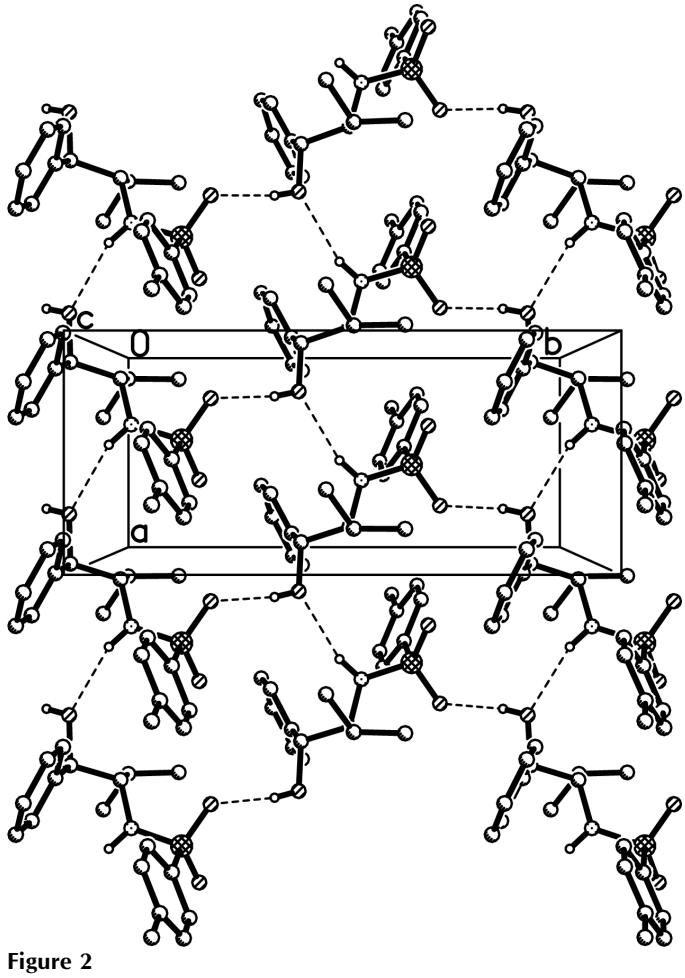


Figure 2
Packing of the molecules viewed down the *c* axis.

Experimental

A mixture of (*R,S*)- and (*S,S*)-2-amino-3-methyl-1-phenyl-butan-1-ol was prepared, using the route described in the literature (Reetz *et al.*, 1987), and was used without separation (the ratio of *R,S* to *S,S* is 87 to 13, estimated by 1 HNMR analysis). *p*-Toluenesulfonyl chloride (0.21 g, 1.1 mmol) and triethylamine (1.5 mmol) were added to the solution of the amino alcohol (0.18 g, 1 mmol) in 30 ml of dichloromethane. After the mixture was stirred at 273 K for 2 h and then at room temperature for 6 h, water (15 ml) was added and the organic layer was separated. The aqueous layer was extracted with dichloromethane (3 × 15 ml). The combined organic phase was washed with brine and 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 (*R,S*)-2-amino-3-methyl-1-phenyl-butan-1-ol (0.21 g, 63% yield).

Crystal data

$C_{18}H_{23}NO_3S$
M_r = 333.43
Orthorhombic, $P2_12_12_1$
a = 5.7407 (6) Å
b = 13.0939 (14) Å
c = 22.846 (2) Å
V = 1717.3 (3) Å^3
Z = 4
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3782 reflections
θ = 1–27.5°
μ = 0.20 mm^{-1}
T = 294 (2) K
Block, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.953, T_{max} = 0.976
11860 measured reflections
3952 independent reflections
2225 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.6°
$h = -7 \rightarrow 7$
$k = -16 \rightarrow 17$
$l = -24 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.089
S = 0.89
3952 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.06 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*A*···O2^{i}	0.82	2.07	2.884 (3)	172
N1—H1*B*···O1^{ii}	0.86	2.36	3.162 (3)	156

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

All H atoms were geometrically placed and allowed to ride on the atoms to which they are attached.

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

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