

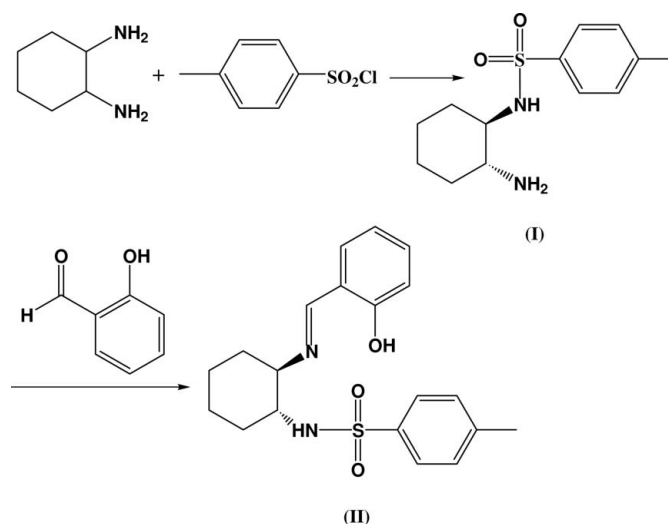
Hong-Wei Yang,<sup>a</sup> Yi-Zhi Li,<sup>b</sup> Xu Pan,<sup>a</sup> Jiang-Tao Sun<sup>a</sup> and Cheng-Jian Zhu<sup>a\*</sup><sup>a</sup>State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, People's Republic of China, and <sup>b</sup>Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

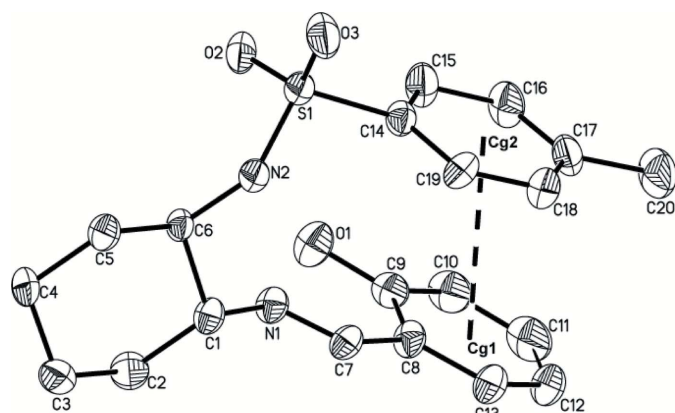
Correspondence e-mail: llyyz@nju.edu.cn

## Key indicators

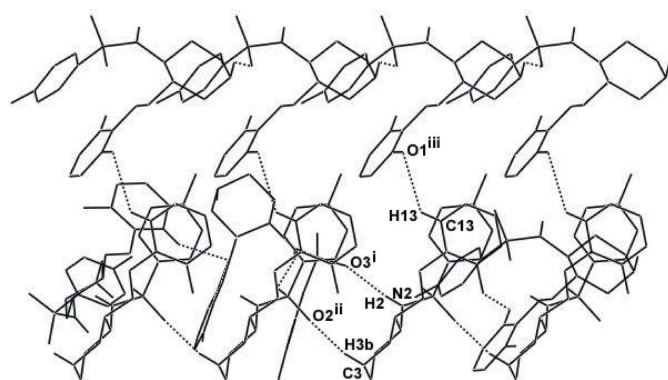
Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.055  
 $wR$  factor = 0.123  
Data-to-parameter ratio = 15.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(1*R*,2*R*)-(+)-1-(2-Hydroxybenzylideneamino)-2-(*p*-tolylsulfonylamino)cyclohexane**The title compound,  $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$ , was synthesized by a two-step reaction. In the crystal structure, chains of molecules are formed through  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds and weak  $\text{C}-\text{H}\cdots\text{O}$  interactions, and these chains are further linked together through weak  $\text{C}-\text{H}\cdots\text{O}$  interactions to form a two-dimensional network structure.Received 14 March 2006  
Accepted 17 April 2006

## Comment

The title compound, (II), was prepared from 1,2-(*R,R*)-cyclohexanediamine by reaction with *p*-toluenesulfonyl chloride to give an intermediate amino-sulfonamide compound, which was then condensed with salicylaldehyde (Balsells & Walsh, 1998). Such sulfonamide Schiff base compounds are found to be efficient chiral auxiliaries for the asymmetric cyclopropanation reaction (Balsells & Walsh, 2000) and for asymmetric addition of diethylzinc to aldehydes (Kitajima & Katsuki, 1997). We have carried out an X-ray structural study of (II) in order to provide a basis for discussing the mechanism for asymmetric synthesis.The molecular structure of (II) is shown in Fig. 1. The  $\text{N1}-\text{C7}$  and  $\text{N2}-\text{S1}$  bond lengths are 1.247 (4) and 1.586 (3)  $\text{\AA}$ , respectively, indicative of a standard  $\text{C}=\text{N}$  double bond and  $\text{N}-\text{S}$  single bond (Tang & Gu, 2005). Atom S1 adopts a distorted tetrahedral environment of N, C and two O atoms, with *cis* angles in the range 105.6 (2)–119.6 (2) $^\circ$ . The average  $\text{S}=\text{O}$  bond length is 1.421 (5)  $\text{\AA}$ .Within the molecule of (II), there is an  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond, together with a weak  $\pi-\pi$  interaction (the  $\text{Cg1}\cdots\text{Cg2}$  distance is 3.906  $\text{\AA}$ , where  $\text{Cg1}$  and  $\text{Cg2}$  are the centroids of



**Figure 1**  
The molecular structure of (II), with 30% probability displacement ellipsoids. The dashed line shows the weak  $\pi$ - $\pi$  interaction.



**Figure 2**  
The two-dimensional network structure, with hydrogen bonds and C-H...O interactions shown as dashed lines. [Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{1}{2}, -z$ ; (ii)  $x + 1, y, z$ ; (iii)  $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$ ]

the rings C8–C13 and C14–C19, respectively). A chain of molecules is formed through intermolecular hydrogen bonds and weak C–H...O interactions (Table 1), and these chains are linked together through another weak interaction to give a two-dimensional network structure.

## Experimental

Dichloromethane (20 ml) was added to a stirred solution of (*R,R*)-1,2-diaminocyclohexane (7.5 mmol) in a 2 N NaOH solution (8 ml). The mixture was cooled to 273 K and a solution of *p*-toluenesulfonyl chloride (2.5 mmol) in dichloromethane (20 ml) was added dropwise over a period of 20 min. After the addition was complete, the mixture was allowed to warm to room temperature and stirred overnight. The resulting solution was washed with water and the solvent was removed at reduced pressure. An intermediate, (I), was isolated in 99% yield (2.49 mmol). Salicylaldehyde (0.5 mmol) in  $\text{CH}_2\text{Cl}_2$  (4 ml) was added to a stirred solution of (I) (0.5 mmol) in  $\text{CH}_2\text{Cl}_2$  (6 ml) at room temperature. The mixture was stirred at room temperature overnight. Anhydrous sodium sulfate was added, the solution filtered and the solvent removed at reduced pressure to obtain 0.5 mmol (100%) of (II) as a yellow solid.  $[\alpha]_{\text{D}}^{25} = 76.1^\circ$  ( $c = 0.5$ , acetone).

## Crystal data

$\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$   
 $M_r = 372.47$   
Orthorhombic,  $P2_12_12_1$   
 $a = 8.134$  (2) Å  
 $b = 11.043$  (3) Å  
 $c = 21.385$  (6) Å  
 $V = 1920.9$  (9) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.288$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
 $\mu = 0.19$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Block, yellow  
 $0.30 \times 0.24 \times 0.22$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Bruker, 2000)  
 $T_{\text{min}} = 0.952$ ,  $T_{\text{max}} = 0.963$

10001 measured reflections  
3717 independent reflections  
2909 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.049$   
 $\theta_{\text{max}} = 26.0^\circ$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.055$   
 $wR(F^2) = 0.123$   
 $S = 1.07$   
3717 reflections  
236 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.39$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
with 1651 Friedel pairs  
Flack parameter: 0.26 (11)

**Table 1**

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1A...N1	0.96	1.85	2.603 (4)	133
C3—H3B...O2 <sup>i</sup>	0.97	2.61	3.357 (5)	134
N2—H2...O3 <sup>ii</sup>	0.86	2.16	2.953 (4)	153
C13—H13...O1 <sup>iii</sup>	0.93	2.67	3.349 (5)	130

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

H atoms were treated as riding, with O—H = 0.96 Å, N—H = 0.86 Å and C—H = 0.85–0.98 Å;  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$  or  $1.2U_{\text{eq}}(\text{N}, \text{C})$ . The refined Flack (1983) parameter indicates partial inversion twinning for this particular crystal, and the optical rotation measurement shows that the compound is not completely racemized.

Data collection: SMART (Bruker, 2000); cell refinement: SMART; data reduction: SAINT (Bruker, 2000); program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL and MERCURY (Bruno *et al.*, 2002); software used to prepare material for publication: SHELXTL.

This project was supported by the National Natural Science Foundation of China (grant Nos. 20102002, 20332050).

## References

- Balsells, J. & Walsh, P. J. (1998). *Tetrahedron Asymmetry*, **9**, 4135–4142.  
Balsells, J. & Walsh, P. J. (2000). *J. Org. Chem.* **65**, 5005–5008.  
Bruker (2000). SMART (Version 5.625), SAINT (Version 6.01), SHELXTL (Version 6.10) and SADABS (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.  
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Kitajima, H. & Katsuki, T. (1997). *Bull. Chem. Soc. Jpn.* **70**, 207–217.  
Tang, G. P. & Gu, J. M. (2005). *Acta Cryst.* **E61**, o3140–o3141.