Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

A new chiral Schiff base: (*R*)-*N*,*N*′-bis(3,5-di-*tert*-butylsalicylidene)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-naphthyl-2,2′-diamine

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Received 4 January 2002
Accepted 15 January 2002
Online 25 January 2002

Key indicators

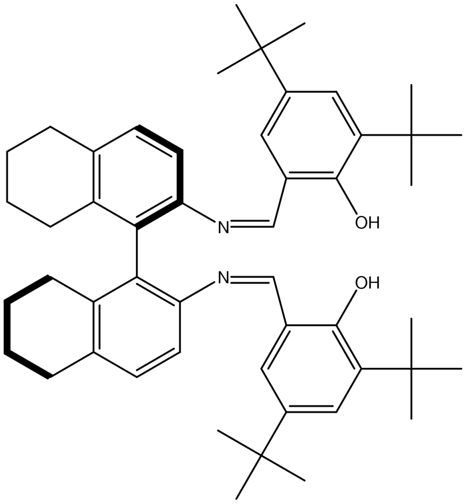
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.149
Data-to-parameter ratio = 21.1

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

The synthetic route to and the chemical structure of a new chiral Schiff base, $C_{50}H_{64}N_2O_2$, derived from (*R*)-H8-BINAM [(*R*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyl-2,2′-diamine] are reported.

Comment

Chiral Schiff bases have been extensively investigated as ligands in asymmetric catalytic synthesis (Belokon *et al.*, 1997; Bandini *et al.*, 1999, 2000). Recent studies (Jacobsen *et al.*, 1991; Zhang & Chan, 1997; Chan *et al.*, 1997) have shown that chiral catalysts derived from 5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl ligands (H_8-BINOL) exhibited higher efficiency and enantioselectivity for many asymmetric reactions than those obtained from BINOL because of the steric and electronic modulations in the binaphthyl backbone from BINOL. On the basis of previous research, a new Schiff base derived from H_8-BINAM [(*R*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyl-2,2′-diamine], namely (*R*)-*N*,*N*′-bis-(3,5-di-*tert*-butylsalicylidene)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyl-2,2′-diamine, (I), was synthesized and its structure was confirmed. The application of the compound in asymmetric synthesis is still under investigation.



(I)

Experimental

For the preparation of (*R*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-binaphthyl-2,2′-diamine [(*R*)-H8-BINAM], (*R*)-1,1′-binaphthyl-2,2′-diamine [(*R*)-BINAM] (100 mg, 0.35 mmol), PtO_2 (20 mg, 0.09 mmol) and glacial acetic acid (20 ml) were added to a 50 ml autoclave equipped with a magnetic stirring bar. The autoclave was closed and

DOI: 10.1107/S160053680200082X

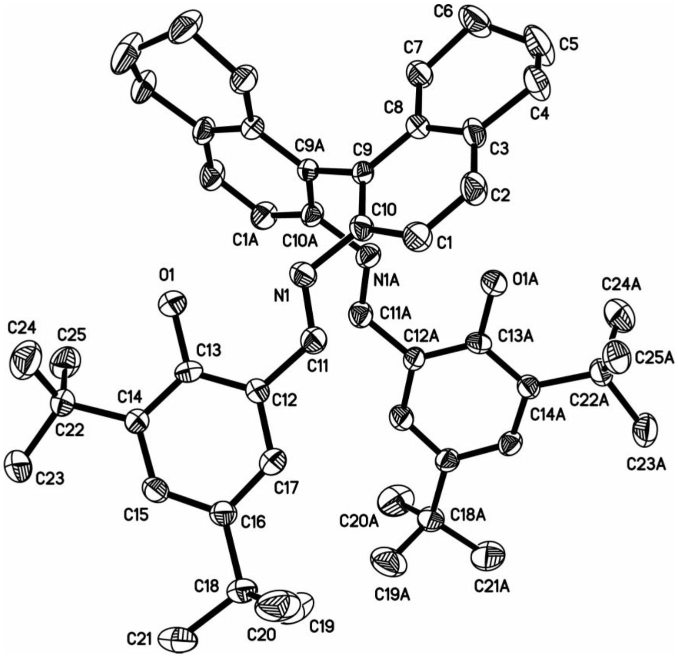


Figure 1

The molecular structure of (I) showing 30% probability ellipsoids (Siemens, 1995).

charged with 10 atm of H_2. The solution was stirred for 24 h at 323 K. The solid catalyst was removed by filtration; the filtrate was neutralized with aqueous $NaHCO_3$ solution followed by extraction with ethyl acetate (3 × 20 ml). The organic phases were combined and dried with sodium sulfate and the solvent was removed with a rotary evaporator to give the crude product, which was purified by crystallization from ethyl acetate and hexane (1:3) to give white crystals of (*R*)-H_8-BINAM (90 mg, 87.5% yield). For the preparation of the title compound, 2,4-di-*tert*-butylsalicylaldehyde (0.70 g, 3 mmol) and magnesium sulfate (1.13 g, 8 mmol) were added to a solution of (*R*)-H_8-BINAM (0.29 g, 1 mmol) in ethanol (40 ml). The mixture was refluxed under nitrogen for 24 h, and filtered through celite when cooled to room temperature. The solvent was removed under reduced pressure, the solid was recrystallized from ethanol, and colorless crystals (0.47 g) of the product were obtained (65% yield).

Crystal data

$C_{50}H_{64}N_2O_2$
$M_r = 725.03$
Tetragonal, $P4_32_12$
$a = 10.6183\ (11)$ Å
$c = 39.629\ (6)$ Å
$V = 4468.1\ (9)$ Å^3
$Z = 4$
$D_x = 1.078$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1782 reflections
$\theta = 1$–27.5°
$\mu = 0.06$ mm^{-1}
$T = 294\ (2)$ K
Block, colorless
0.20 × 0.18 × 0.18 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.987$, $T_{\max} = 0.989$
30648 measured reflections

5130 independent reflections
1824 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.103$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -51 \rightarrow 47$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.149$
$S = 1.00$
5130 reflections
243 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0014 (5)
Absolute structure: Flack (1983)
Flack parameter = 4 (3)

Table 1

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.91	2.643 (3)	148
O1—H1···N1	0.82	1.91	2.643 (3)	148

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *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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