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(*R*)-2,2′-Dimethoxy-3,3′-bis{[(1*S*)-1-phenylethyl]iminomethyl}-1,1′-binaphthalene, a new chiral Schiff base

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The synthesis and crystal structure of the title new chiral Schiff base, $C_{40}H_{36}N_2O_2$, derived from BINOL (1′1-bi-2-naphthol), are presented.

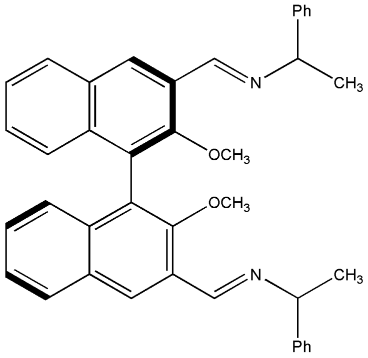
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.118
Data-to-parameter ratio = 10.7

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

Comment

The search for new chiral ligands for asymmetric synthesis is an important task in organic chemistry. Various chiral Schiff bases are widely used in asymmetric reactions (Jiang *et al.*, 1995; Belokon *et al.*, 1997; Bandini *et al.*, 1999, 2000; Kureshy *et al.*, 2001; Cozzi, 2003). The successful resolution of racemic 1′1-bi-2-naphthol (BINOL) provides an economic production of (*S*)- or (*R*)-BINOL and excellent opportunity for their exploitation in asymmetric synthesis. Both enantiomers of BINOL can be used as chiral inducing agents for catalytic asymmetric reactions such as the Diels–Alder reaction, ene reaction, Lewis acid-catalysed reactions, enantioselective reduction of ketones, synthesis of chiral macrocycles, *etc.* (Bao *et al.*, 1993; Terada *et al.*, 1994; Sakane *et al.*, 1985; Noyori & Tomino, 1984; Sogah & Cram, 1979; Miyano *et al.*, 1980).



(I)

We report here the synthesis and crystal structure (Fig. 1) of a new Schiff base, *viz.* (*R*)-2,2′-dimethoxy-3,3′-bis{[(1*S*)-1-phenylethyl]iminomethyl}-1,1′-binaphthalene, (I), derived from BINOL.

Experimental

To a solution of (*R*)-2,2′-dimethoxy-1,1′-binaphthyl (6.28 g, 20.0 mmol) and *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TMEDA) (15.8 ml, 105 mmol) in diethyl ether (300 ml) was added dropwise a solution of *n*-BuLi (2.0 *M* in hexanes, 43 ml, 86 mmol) at 273 K over a period of 30 min. The mixture was stirred for 1 h at this temperature and was then warmed to reflux. After being refluxed for 16 h, the resulting mixture was cooled to 273 K and dimethylformamide (DMF) (25 ml, 160 mmol) was added dropwise; the mixture was stirred at 273 K for 90 min and then 4 *N* HCl (60 ml) was added with

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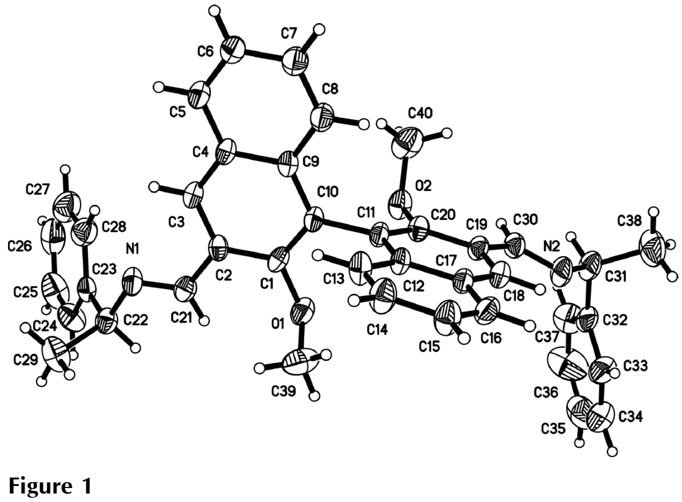

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

vigorous stirring for 1 h. The organic layer was separated, washed with 0.5 *N* HCl, saturated $NaHCO_3$ and brine, dried over Na_2SO_4, and concentrated and purified by chromatography to afford 5.8 g of 2,2′-dimethoxy-1,1′-binaphthyl-3,3′-dicarbaldehyde; the ^{1}H NMR data were in accord with the literature (Stock & Kellogg, 1996). For the preparation of the title compound, (*S*)-α-methylbenzylamine (6.6 mmol) and magnesium sulfate (1.13 g, 8 mmol) were added to a solution of 2,2′-dimethoxy-1,1′-binaphthyl-3,3′-dicarbaldehyde in ethanol. The mixture was refluxed for 18–24 h under nitrogen and filtered through celite after cooling to ambient temperature. The solvent was removed and the residue recrystallized from ethanol to afford colorless crystals (60% yield).

Crystal data

$C_{40}H_{36}N_2O_2$
$M_r = 576.71$
Orthorhombic, $P2_12_12_1$
$a = 9.614$ (2) Å
$b = 15.270$ (4) Å
$c = 22.838$ (5) Å
$V = 3352.8$ (14) Å^3
$Z = 4$
$D_x = 1.143$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3352 reflections
$\theta = 1–17.5°$
$\mu = 0.07$ mm^{-1}
$T = 294$ (2) K
Plate, colorless
0.40 × 0.28 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.973$, $T_{\max} = 0.993$
22 940 measured reflections
4309 independent reflections
1718 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.100$
$\theta_{\max} = 27.6°$
$h = -12 \rightarrow 12$
$k = -19 \rightarrow 14$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.118$
$S = 1.01$
4309 reflections
402 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0022 (4)

All H atoms were positioned geometrically and refined in the riding-model approximation, with C—H distances of 0.93 (aromatic) and 0.96 Å (methyl), and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C_{aromatic}})$ and $1.5U_{\rm eq}({\rm C_{methyl}})$. In the absence of significant anomalous scattering, the absolute configuration is indeterminate and has been assigned arbitrarily; Friedel pairs were merged. The crystal structure contains a void of 52 Å^3 positioned at (0.29, 0.19, 0.38). However, the Fourier difference map shows no peaks higher than 0.29 e Å^{-3}, indicating the absence of solvent.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SHELXTL* (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*; software used to prepare material for publication: *SHELXTL*.

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References

Bandini, M., Cozzi, P. G., Melchioree, P. & Umani-Ronchi, A. (1999). *Angew. Chem. Int. Ed.* **38**, 3357–3359.
Bandini, M., Cozzi, P. G. & Umani-Ronchi, A. (2000). *Angew. Chem. Int. Ed.* **39**, 2327–2330.
Bao, J., Wulff, W. D. & Rheingold, A. L. (1993). *J. Am. Chem. Soc.* **115**, 3814–3815.
Belokon, Y., Flego, M., Ikonnikov, N., Moscalenko, M., North, M., Orizu, C., Tararov, V. & Tasinazzo, M. (1997). *J. Chem. Soc. Perkin Trans.* 1, pp. 1293–1295.
Cozzi, P. G. (2003). *Angew. Chem. Int. Ed.* **42**, 2895–2898.
Jiang, Y., Zhou, X., Hu, W., Wu, L. & Mi, A. (1995). *Tetrahedron Asymmetry*, **6**, 405–408.
Kureshy, R. I., Khan, N. H., Abdi, S. H. R., Patel, S. T. & Jasra, R. V. (2001). *Tetrahedron Lett.* **42**, 2915–2918.
Miyano, S., Tobita, M., Nawa, M., Sato, S. & Hashimoto, H. (1980). *J. Chem. Soc. Chem. Commun.* pp. 1233–1234.
Noyori, R. & Tomino, I. (1984). *J. Am. Chem. Soc.* **106**, 6709–6716.
Sakane, S., Maruoka, K. & Yamamoto, H. (1985). *Tetrahedron Lett.* **26**, 5535–5538.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1995). *SMART* and *SAINT* (Versions 4.0), and *SHELXTL* (Version 5.10). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sogah, G. D. Y. & Cram, D. J. (1979). *J. Am. Chem. Soc.* **101**, 3035–3042.
Stock, H. T. & Kellogg, R. M. (1996). *J. Org. Chem.* **61**, 3093–3105.
Terada, M., Motoyama, Y. & Mikami, K. (1994). *Tetrahedron Lett.* **35**, 6693–6696.