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(*R*,*R*)-4,4′-Dimethyl-2,2′-[cyclohexane-1,2-diylbis(nitrilomethylidyne)]diphenol

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The title compound, $C_{22}H_{26}N_2O_2$, has been prepared by reaction of 1,2-cyclohexanediamine and 5-methyl-2-hydroxybenzaldehyde. The molecular structure is stabilized by intramolecular O—H···N hydrogen-bonding interactions.

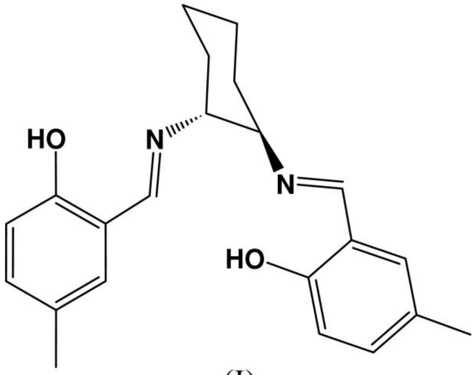
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.072
Data-to-parameter ratio = 11.4

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

Comment

Chiral Schiff base compounds containing the 1,2-cyclohexanediamine scaffold are widely used in asymmetric catalytic synthesis (Canali & Sherrington, 1999). So far, few single-crystal structures of this type of compound have been reported (Yang *et al.*, 2004). Our research is focused on asymmetric synthesis catalysed by chiral Shiff base–metal complexes (Yang *et al.*, 2005). In this context, we have synthesized the title chiral ligand, (I), and present here its crystal structure.



(I)

In (I), the cyclohexane ring displays a chair conformation, as indicated by the puckering parameters q_2 = 0.032 (2), q_3 = 0.560 (2), φ_2 = 124 (3)° and Q_T = 0.561 (2) (Cremer & Pople, 1975). The C8–C13 and C16–C21 benzene rings are oriented to form a dihedral angle of 28.11 (5)°. The molecular structure is stabilized by two intramolecular O—H···N hydrogen bonds (Table 1). The crystal packing is stabilized only by van der Waals interactions.

Experimental

A mixture of (1*R*,2*R*)-1,2-cyclohexanediamine (1.14 g, 10 mmol) and 5-methyl-2-hydroxybenzaldehyde (2.72 g, 20 mmol) in absolute ethanol (20 ml) was refluxed under nitrogen for about 5 h, yielding a yellow precipitate. This was separated by vacuum filtration and washed with ethanol (yield 90%, 3.14 g). Yellow single crystals of (I) suitable for X-ray analysis were grown from a mixture of CH_2Cl_2 and ethanol (1:1 *v*/*v*) by slow evaporation of the solvent at room temperature over a period of about a week.

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Crystal data

$C_{22}H_{26}N_2O_2$
$M_r = 350.45$
Orthorhombic, $P2_12_12_1$
$a = 8.9896\ (5)$ Å
$b = 10.3568\ (6)$ Å
$c = 21.3397\ (13)$ Å
$V = 1986.8\ (2)$ Å^3
$Z = 4$
$D_x = 1.172$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 291\ (2)$ K
Block, yellow
$0.32 \times 0.26 \times 0.24$ mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.98$, $T_{\max} = 0.98$
15010 measured reflections
2782 independent reflections
1636 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.048$
$\theta_{\max} = 28.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.072$
$S = 1.02$
2782 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.09$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$B$$\cdots$N1	0.961 (17)	1.768 (18)	2.6060 (17)	143.7 (16)
O2—H2$D$$\cdots$N2	0.955 (18)	1.783 (19)	2.6065 (17)	142.6 (17)

The hydroxyl H atoms were located in a difference Fourier synthesis and refined with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm O})$. All other H atoms were positioned geometrically and refined using a riding-model approximation, with C—H = 0.93–0.96 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}$(methyl C). In the absence of significant anomalous scattering effects, Friedel pairs were merged in the final refinement. The absolute configuration was assigned from the known configuration of the synthetic precursor.

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

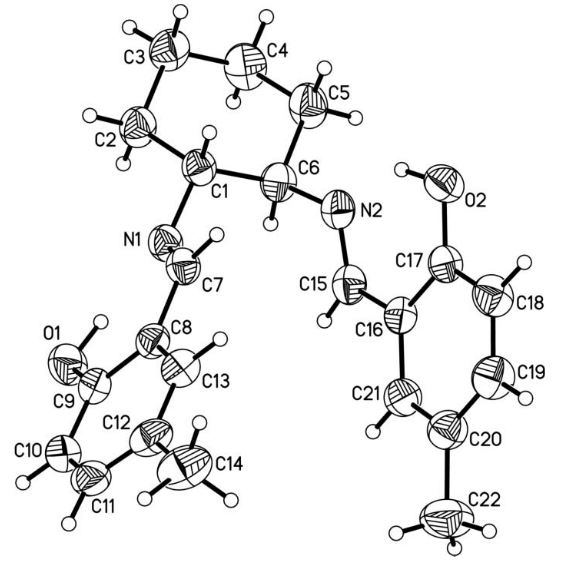

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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References

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.
Canali, L. & Sherrington, D. C. (1999). *Chem. Soc. Rev.* **28**, 85–93.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Yang, M.-H., Li, Y.-Z., Zhu, C.-J., Pan, Y. & Liu, S.-H. (2004). *Acta Cryst.* E**60**, o2397–o2398.
Yang, M. H., Zhu, C. J., Yuan, F., Huang, Y. J. & Pan, Y. (2005). *Org. Lett.* **7**, 1927–1930.