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(*S*,*S*)-6,6′-Dimethyl-2,2′-[*o*-phenylenebis(nitrilomethylidyne)]diphenol benzene solvate

Ming-Hua Yang,* Guo-Bing Yan and Yun-Fa Zheng

Department of Chemistry, Lishui University, Lishui 323000, People's Republic of China

Correspondence e-mail:
ymhxraylab@hotmail.com

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Key indicators

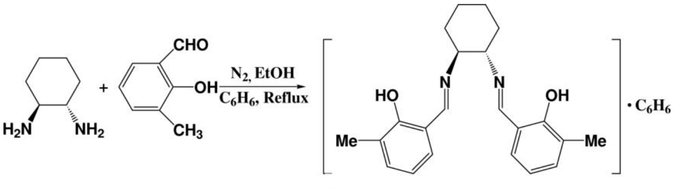
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.006 Å
R factor = 0.057
wR factor = 0.151
Data-to-parameter ratio = 9.7

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

The asymmetric unit of the title compound, $C_{22}H_{26}N_2O_2 \cdot C_6H_6$, contains two molecules of *N*,*N*′-bis(3-methylsalicylidene)cyclohexane-1,2-diamine {or 6,6′-dimethyl-2,2′-[*o*-phenylenebis(nitrilomethylidene)]diphenol} and two benzene molecules. There are two intramolecular O—H···N bonds in each of the independent salicylidene molecules; each of them closes a six-membered pseudo-ring. The packing of the crystal structure features stacks of alternating cyclohexane fragments and benzene molecules running along the short *a* axis.

Comment

The Schiff bases formed from salicylaldehyde and ethylenediamine derivatives are collectively known as salen compounds. Chiral salen compounds containing the 1,2-cyclohexanediamine motif constitute an important family of chiral ligands which are widely used in asymmetric catalytic synthesis (Canali & Sherrington, 1999; Jacobsen, 2000). The structure of chiral salen compounds has a crucial effect on their activity and enantioselectivity in asymmetric catalytic reactions (Nicewicz *et al.*, 2004; Yao *et al.*, 2001). Nevertheless, only relatively few single-crystal structures of chiral salen compounds have been reported to date. Our research is focused on asymmetric synthesis catalyzed by chiral salen–metal complexes (Yang *et al.*, 2005). As part of our study of the relationship between the structure and properties of such salen compounds, we have synthesized the new chiral ligand (*S*,*S*)-*N*,*N*′-bis(3-methylsalicylidene)-1,2-cyclohexanediamine and we describe here its crystal structure as the title benzene solvate, (I).



(I)

The asymmetric unit of (I) contains two molecules of (*S*,*S*)-*N*,*N*′-bis(3-methyl-salicylidene)-1,2-cyclohexanediamine and two molecules of benzene (Fig. 1). Both salicylidene molecules have very similar geometries. Each of them has two rather strong intramolecular hydrogen bonds, which involve the hydroxyl groups of the salicyl unit as a donor and the corresponding Schiff base N atom as an acceptor (Table 1). Each of the hydrogen bonds closes a six-membered ring.

The crystal packing of (I) features infinite stacks formed of alternating benzene molecules and cyclohexane fragments of

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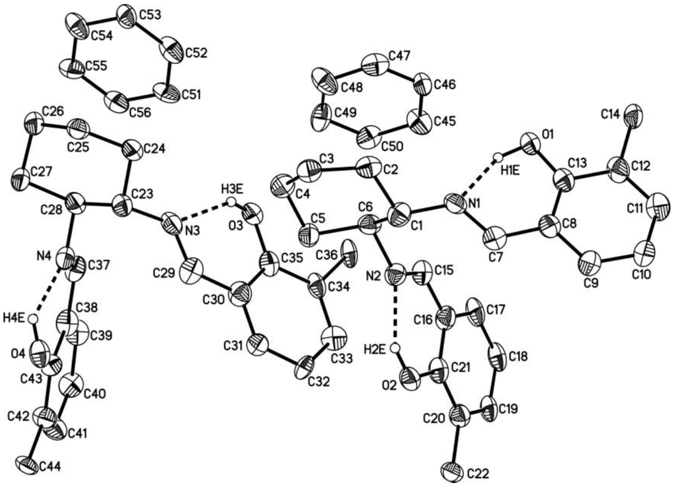

Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms bonded to C atoms have been omitted for clarity. Intramolecular hydrogen bonds are shown as dashed lines.

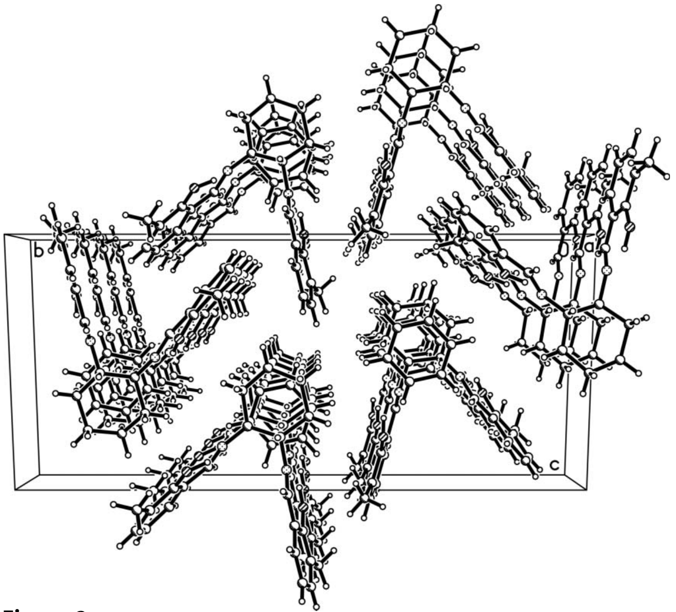

Figure 2
The molecular packing of (I), viewed down the *a* axis.

the salicylidene molecules (Fig. 2). It is noteworthy that there are two symmetry-independent stacks in the structure, as all molecules belonging to one stack are derived from one of the two independent benzene molecules and one of the two independent salicylidene molecules.

Experimental

A mixture of (*S*,*S*)-1,2-cyclohexanediamine (1.14 g, 10 mmol), Na_2SO_4 (3.0 g) and 3-methyl-2-hydroxybenzaldehyde (2.72 g, 20 mmol) in absolute ethanol (40 ml) was refluxed for about 5 h under a nitrogen atmosphere. The yellow precipitate was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2 × 10 ml) and brine (10 ml). It was then dried over Na_2SO_4, the solvent was removed under vacuum, and the yellow solid was isolated (3.33 g; yield 95%). Single crystals of compound (I) suitable for X-ray analysis were grown from a solution in benzene by slow evaporation of the solvent at room temperature over a period of about three weeks.

Crystal data

$C_{22}H_{26}N_2O_2 \cdot C_6H_6$
$M_r = 428.56$
Monoclinic, $P2_1$
$a = 8.2760$ (18) Å
$b = 26.636$ (6) Å
$c = 12.250$ (3) Å
$\beta = 105.250$ (3)°
$V = 2605.4$ (10) Å^3
$Z = 4$
$D_x = 1.093$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 291$ (2) K
Block, yellow
0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.978$, $T_{\max} = 0.984$
15086 measured reflections
5788 independent reflections
4361 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.151$
$S = 1.06$
5788 reflections
597 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0844P)^2 + 0.1157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4*E*···N4	0.88 (5)	1.74 (5)	2.438 (5)	135 (4)
O3—H3*E*···N3	0.85 (5)	1.84 (5)	2.550 (4)	140 (5)
O2—H2*E*···N2	0.87 (6)	1.81 (6)	2.577 (5)	146 (5)
O1—H1*E*···N1	0.86 (5)	1.91 (5)	2.604 (4)	137 (4)

C-bound H atoms were positioned geometrically, with C—H = 0.93–0.96 Å, and were included in the refinement in the riding-model approximation, with $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$, or $1.5U_{\rm eq}(C)$ for methyl H atoms. The hydroxyl H atoms were located in a difference Fourier map and refined in the isotropic approximation [O—H = 0.85 (5)–0.88 (5) Å]. Due to the lack of significant anomalous scatterers, determination of the absolute configuration based on anomalous scattering was impossible and Friedel pairs were merged. The absolute configuration was assigned on the basis of the known *S*,*S*-configuration of the starting (*S*,*S*)-1,2-cyclohexanediamine.

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*.

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