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Key indicators

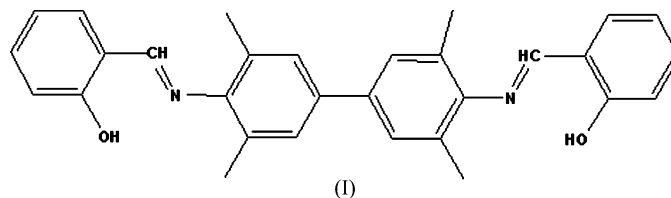
Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.064
 wR factor = 0.173
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3,5,3',5'-Tetramethyl-*N,N'*-bis(salicylidene)-
biphenyl-4,4'-diamine

In the molecule of the title Schiff base compound, $\text{C}_{30}\text{H}_{28}\text{N}_2\text{O}_2$, the dihedral angles between the phenol rings and the adjacent dimethylbenzene rings [60.14 (11) and 77.57 (11)°] are distinctly larger than that formed by the dimethylbenzene rings [39.65 (11)°], possibly as a result of the steric hindrance of the methyl groups and the presence of intramolecular O—H···N hydrogen bonds.

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Comment

Multidentate Schiff base ligands and their metal complexes have been studied for many years (Daier *et al.*, 2004; Munro & Camp, 2003; Weber, 1967). These complexes play an important role in the development of coordination chemistry related to magnetism, catalysis and molecular architectures. As a continuation of our previous work (Xu *et al.*, 2001), we report here the structure of the title compound, (I).



Selected bond distances and angles are listed in Table 1, and the molecular structure is shown in Fig. 1. The dihedral angles formed by the C1–C6 and C25–C30 phenol rings with the adjacent C8–C13 and C16–C21 dimethylbenzene rings are 60.14 (11) and 77.57 (11)°, respectively. These angles are remarkably larger than that formed by the dimethylbenzene rings [39.65 (11)°], possibly due to the concomitant effects of the steric hindrance of the methyl groups and the presence of two intramolecular O—H···N hydrogen-bond interactions (Table 2). There are neither π – π stacking nor weak intermolecular hydrogen-bond interactions, and the crystal packing (Fig. 2) is controlled by van der Waals forces.

Experimental

The title compound was synthesized by the condensation of salicylaldehyde and 3,3',5,5'-tetramethylbenzidine. A mixture of 3,3',5,5'-tetramethylbenzidine (0.59 mmol) in MeOH (10 ml) and salicylaldehyde (1 mmol) in MeOH (10 ml) was refluxed for 3 h under an N_2 atmosphere and allowed to stand at room temperature overnight. The yellow solid product was isolated by filtration and washed with MeOH (yield: 61%). Pale-yellow single crystals suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution (m.p. 480 K). ^1H NMR (300 MHz, CDCl_3): 2.29 (s, 12H, CH_3), 6.95–7.10 (m, 4H, Ph), 7.22–7.75 (m, 8H, Ph), 8.41 (s, 2H, CH).

Crystal data

$C_{30}H_{28}N_2O_2$
 $M_r = 448.54$
 Monoclinic, $P2_1/c$
 $a = 10.693$ (5) Å
 $b = 12.896$ (6) Å
 $c = 17.673$ (8) Å
 $\beta = 90.871$ (9)°
 $V = 2436.7$ (19) Å³
 $Z = 4$

$D_x = 1.223$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 661 reflections
 $\theta = 2.5$ – 18.1 °
 $\mu = 0.08$ mm⁻¹
 $T = 273$ (2) K
 Block, pale yellow
 $0.12 \times 0.09 \times 0.06$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 12159 measured reflections
 4289 independent reflections

1897 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.102$
 $\theta_{max} = 25.0$ °
 $h = -12 \rightarrow 12$
 $k = -8 \rightarrow 15$
 $l = -20 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.173$
 $S = 0.93$
 4289 reflections
 314 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.23$ e Å⁻³
 $\Delta\rho_{min} = -0.21$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0088 (13)

Table 1

Selected geometric parameters (Å, °).

C1–O1	1.351 (4)	C19–N2	1.447 (4)
C6–C7	1.467 (4)	C24–N2	1.274 (4)
C7–N1	1.273 (4)	C24–C25	1.460 (4)
C8–N1	1.441 (4)	C30–O2	1.357 (4)
C7–N1–C8	120.7 (3)	C24–N2–C19	120.5 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1 \cdots N1	0.82	1.89	2.616 (3)	147
O2–H2 \cdots N2	0.82	1.89	2.609 (2)	146

H atoms were included in calculated positions ($C-H = 0.93$ – 0.96 Å and $O-H = 0.82$ Å) and refined using a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. The crystals showed poor diffraction quality which resulted in a rather high R_{int} value (0.102) and a low ratio of observed to unique reflections (44%).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

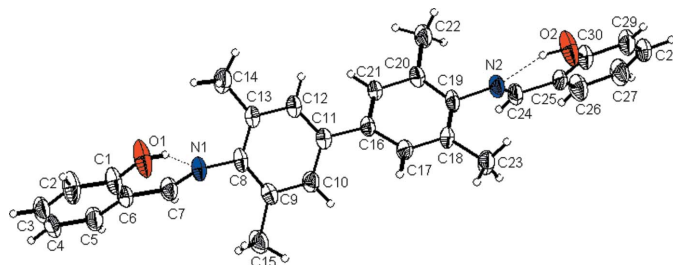


Figure 1

The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The $O-H\cdots N$ hydrogen-bond interactions are indicated by dashed lines.

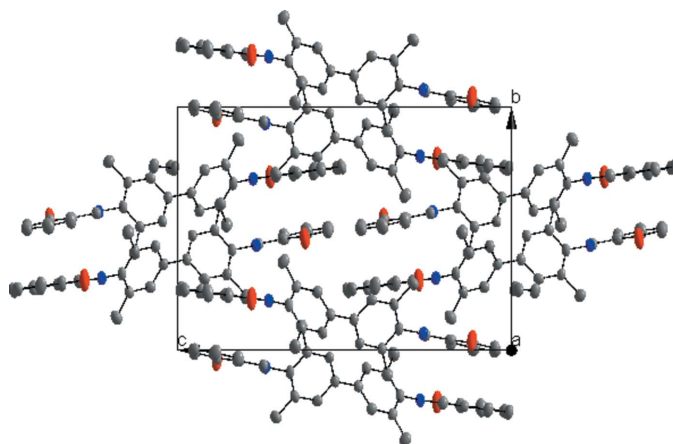


Figure 2

The crystal packing of the title compound, viewed down the [100] direction. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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