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(*R*)-(–)-[1-(1-Naphthyl)ethyl]salicylaldimine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.050
wR factor = 0.130
Data-to-parameter ratio = 7.5

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

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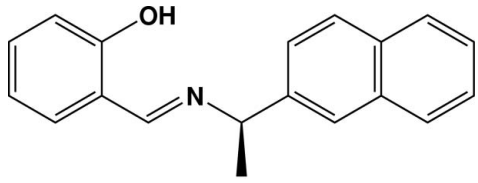
In the title compound, $C_{19}H_{17}NO$, the π-conjugated system formed by the phenol–imine tautomer is essentially planar. An intramolecular O—H···N hydrogen bond is formed between the phenol OH group and the Schiff base N atom. The crystal packing is stabilized by van der Waals forces.

Comment

Metal-chelate Schiff base complexes have played an important role in developing stereochemical models in main group and transition metal coordination chemistry, mainly owing to their stability and ease of preparation and the structural variability of the ligands (Serron *et al.*, 1997). The burgeoning field of asymmetric synthesis relies mainly on the use of chiral ligands for transition metal-based catalysis. Metal complexes of chiral Schiff bases derived from salicylaldehyde and chiral amines have been used successfully for a myriad of reactions, including asymmetric cyclopropanation, oxidation of sulfides, epoxidation of olefins and silylcyanation of aldehydes (Antonov *et al.*, 1995).



(I)

As part of our research with optically active imine-based metal complexes in homogeneous catalysis, a series of chiral Schiff base–copper(II) complexes was synthesized and applied to the asymmetric cyclopropanation of olefins (Iglesias *et al.*, 2004). With the aim of gaining further insight into the struc-

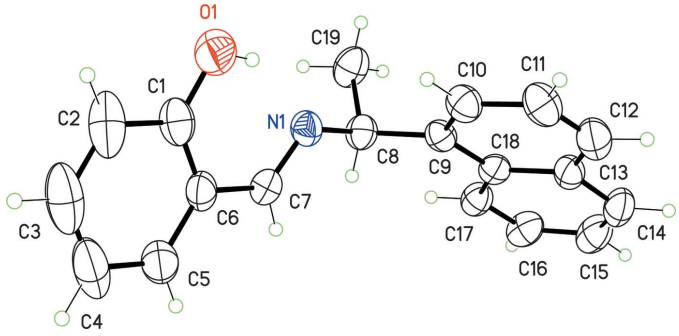


Figure 1
The molecular structure of (I), showing the atom–labelling scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

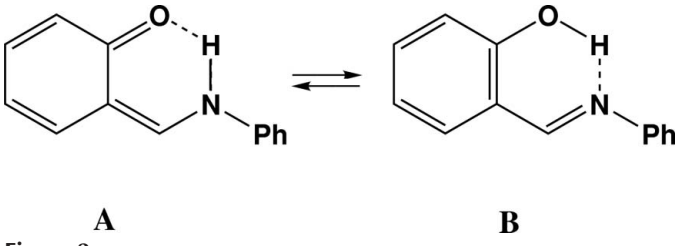


Figure 2
Tautomerism between keto–imine (*A*) and phenol–imine (*B*) forms.

tural aspects of the ligands and steric aspects in the catalytic reaction, the X-ray crystal structure determination of the title compound, (I), has been carried out.

The molecular structure of (I), with the atom-numbering scheme, is shown in Fig. 1. The molecule adopts an *E* configuration with respect to the C=N double bond, with a C6—C7=N1—C8 torsion angle of −177.4 (4)° and a C7=N1—C8 angle of 118.0 (3)°. Thus, the π-conjugated phenol–imine system is essentially planar and similar to an analogous compound (Akitsu *et al.*, 2004). Schiff bases exhibit tautomerism between the keto–imine (or quinoid) form, *A*, and the phenol–imine (or benzenoid) form, *B* (Fig. 2; Percy & Thornton, 1972), the preferred form for arylimines derived from salicylaldehyde being the benzenoid form (Garnovskii *et al.*, 1998). The C1—O1 bond distance in (I) is 1.326 (5) Å, which is rather short compared with other *N*-salicylaldimines (1.34–1.35 Å; Inabe *et al.*, 1994). The C7=N1 and N1—C8 bond distances are 1.260 (4) Å and 1.469 (5) Å, respectively (Table 1), in agreement with the mean literature values of similar structures (Calligaris & Randaccio, 1986; Liu *et al.*, 1997).

An intramolecular O—H···N hydrogen bond (Table 2) determines the overall geometry of the molecule (Calligaris & Randaccio, 1986; Tümer *et al.*, 1997) and corresponds to those observed in other chiral *N*-salicylaldimines (Akitsu *et al.*, 2004; Liu *et al.*, 1997). The relatively short intermolecular distance between atom H14*A* and the centroid of the C9–C13/C18 ring, of 2.85 Å, indicates a possible C—H···π interaction in the structure. The crystal packing (Fig. 3) is mainly stabilized by van der Waals forces.

Experimental

Under argon, to a solution of (*R*)-(−)-[1-(1-naphthyl)ethyl]amine (0.150 g, 0.925 mmol) in dry methanol (25 ml) were added salicylaldehyde (0.113 g, 0.925 mmol), and anhydrous sodium sulfate (2 g) as a drying agent. The reaction mixture was refluxed at 338 K for 2 h and stirred overnight (Iglesias *et al.*, 2004). The solution was filtered and the solvent removed under reduced pressure to afford (I) as a yellow solid (0.251 g, 98.5%). M.p. 356–357 K; IR (cm^{-1}, KBr): 3435, 3044, 2974, 1626, 1576, 1493, 1277, 1125, 761; ^{1}H NMR (200 MHz, $CDCl_3$, δ, p.p.m.): 13.60 (*s*, 1H), 8.35 (*s*, 1H), 8.05 (*dd*, *J* = 8.5 and 1.4 Hz, 1H), 7.70 (*d*, *J* = 8.2 Hz, IH), 7.56 (*dd*, *J* = 6.9 and 1.0 Hz, 1H), 7.52–7.34 (*m*, 2H), 7.22 (*t*, *J* = 8.1 and 1.8 Hz, 1H), 7.10 (*dd*, *J* = 7.7 and 1.7 Hz, 1H), 6.90 (*d*, *J* = 8 Hz, 2H), 6.77 (*t*, *J* = 7.6 and 1.2 Hz, 2H), 5.33 (*q*, *J* = 6.6 Hz, 1H), 1.71 (*d*, *J* = 6.6 Hz, 3H); ^{13}C{^{1}H} NMR (50 MHz, $CDCl_3$, δ, p.p.m.): 164.3, 161.3, 139.6, 134.1, 132.4, 131.6, 129.4, 128.2, 126.3, 125.8, 124.2, 123.9, 123.4, 119.1, 117.1, 64.3, 24.7. Analysis found: C 82.72, H 5.93, N 4.93%; calculated for $C_{19}H_{17}NO$: C 82.87, H 6.22, N 5.08%. Single crystals suitable for X-ray structure analysis were obtained by slow evaporation of a concentrated solution in methanol at 298 K.

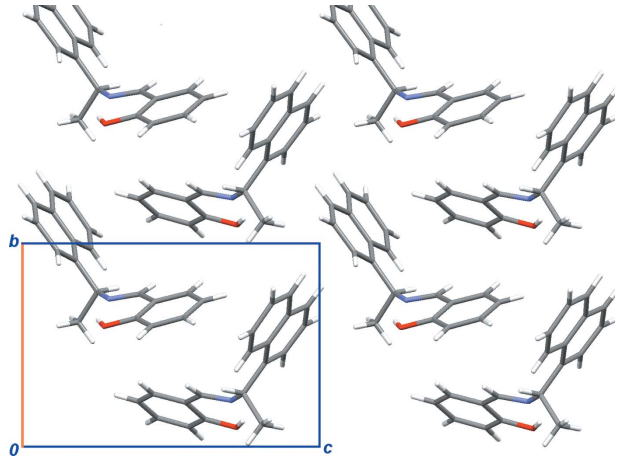


Figure 3
A molecular packing diagram for (I), viewed along the *a* axis.

Crystal data

$C_{19}H_{17}NO$	D_x = 1.203 Mg m^{-3}
M_r = 275.34	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 38 reflections
a = 7.9887 (11) Å	θ = 5.1–12.4°
b = 8.0574 (7) Å	μ = 0.07 mm^{-1}
c = 11.8420 (11) Å	T = 294 (2) K
β = 94.223 (10)°	Prism, yellow
V = 760.18 (14) Å^3	0.62 × 0.48 × 0.26 mm
Z = 2	

Data collection

Bruker *P*4 diffractometer	θ_{max} = 25.0°
$2\theta/\omega$ scans	$h = 0 \rightarrow 9$
Absorption correction: none	$k = 0 \rightarrow 9$
1548 measured reflections	$l = -14 \rightarrow 14$
1442 independent reflections	3 standard reflections
968 reflections with $I > 2\sigma(I)$	every 97 reflections
R_{int} = 0.015	intensity decay: 5.3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.130	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
1442 reflections	Extinction correction: *SHELXTL*
191 parameters	Extinction coefficient: 0.050 (10)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.260 (4)	C6—C7	1.464 (6)
N1—C8	1.469 (5)	C8—C9	1.511 (6)
O1—C1	1.326 (5)		
C7—N1—C8	118.0 (3)	N1—C7—C6	123.0 (4)
O1—C1—C6	122.4 (4)	N1—C8—C9	111.6 (3)
C5—C6—C7	120.6 (5)	N1—C8—C19	108.6 (4)
O1—C1—C6—C7	−2.9 (6)	C7—N1—C8—C9	−108.0 (4)
C8—N1—C7—C6	−177.4 (4)	C7—N1—C8—C19	129.6 (4)
C1—C6—C7—N1	6.7 (6)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.82	1.86	2.595 (4)	148

We could not assign the hydroxy H atom (H1*A*) bonded to atom O1 from a difference map, so we located it in an idealized position, as all other H atoms. Refinement for H atoms was carried out using a riding model, with distances constrained to 0.82 Å for the OH group, 0.93 Å for aromatic CH, 0.98 Å for methine CH and 0.96 Å for methyl CH_3. U_{iso}(H) parameters were set to $1.2U_{eq}$(carrier atom) for aromatic CH and methine CH, and $1.5U_{eq}$(carrier atom) for OH and methyl CH_3. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXTL-NT* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-NT*; molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

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