



Synthesis and crystal structure of a new Schiff base 2-(naphthalene-2-ylimino methyl) phenol

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ABSTRACT

The structure of the title compound, [C₁₇H₁₃NO] the Schiff base, 2-(naphthalene-2-ylimino methyl) phenol was elucidated by H¹, C¹³ NMR, and IR spectroscopic techniques. The X-ray structure was determined in order to establish the conformation of molecule. The compound crystallizes in the orthorhombic space group Pca2₁, with a= 13.6460(3), b= 5.8732(1), c =15.8729(3)Å, α = β = γ = 90° and Z=4. The two benzene rings (naphthyl and salicylaldehyde) and the azomethine group are practically coplanar, as a result of O-H...N hydrogen bond with graph-set notation S(6).

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Introduction

Schiff base complexes have been studied extensively, due to their interesting structures and broad applications [1,2]. Studies of these compounds are of great interest in various aspects of chemistry, such as antimicrobial drugs, functional coordinated complex, photo electric materials, catalytic materials [3,4] etc. The presence of the amino group in this compound leads to condensation with aldehyde, thus enlarging the number of functional groups. Schiff bases with azomethine linkage were used as anti infectious agents. The synthesis and crystal structure of a new Schiff base 4-[(2-hydroxy-benzylidene)-amino-N-(5-methyl-isoxazol-3-yl)-benzene sulfonamide [5], synthesis of metal complexes chelated with N-naphthalenyl aminomethyl phenol and their application to OELD [6] have already been reported. The present study has been undertaken to study, the synthesis and crystal structure of a new Schiff base 2-(naphthalene-2-ylimino methyl) phenol. Schiff bases derived from substituted naphthalenes and salicylaldehyde are of interest because of their metal complexing behaviour.

Experimental

The title compound was synthesized by the condensation of 2-naphylamine (NA) and 2-hydroxy benzaldehyde (SA) (standard procedure) [7]. An ethanol solution (25ml) of NA (0.25mole) was mixed with SA (0.25mole) and the contents were refluxed for 4 hours. After air cooling, the reaction mixture was kept for crystallization. After three days yellow prisms of compound (I) were obtained (scheme a & b). IR spectrum was recorded on a Perkin- Elmer RXI FT-IR spectrophotometer with range 4000-400cm⁻¹ using KBr pellets. H¹ and C¹³ NMR spectra of Schiff base were recorded with a Bruker 200 MHZ instrument using TMS as an internal standard and DMSO-d⁶ as solvent.

X-ray crystallography

A crystal with dimensions of 0.04 × 0.05 × 0.05 mm³ was used for X-ray data collection. All measurements were made on

a Bruker SMART APEXII CCD area-detector diffractometer with graphite monochromated MoK_α (0.71073Å) radiation at 293K, using the ω scan technique. Data collection: APEX2 [8]; cell refinement: SAINT [8]; data reduction: SAINT [8]. The programs used to solve and refine the structure were SHELXS-97 and SHELXL-97 [9]. Molecular graphics: PLATON [10]; software used to prepare material for publication: PLATON [10]. The refinement was carried out by using the full-matrix least square on F². All non-hydrogen atoms were refined anisotropically. All hydrogen atoms have been geometrically fixed and refined with isotropic thermal parameters. Crystallographic details are shown in Table 1.

Structure solution and refinement

The data of the title compound, 2NAS had the following systematic absences.

- i) h0l type of reflections h odd absent
- ii) 0kl type of reflections l odd absent
- iii) h0l type of reflections h odd absent
- iv) 00l type of reflections l odd absent.

Hence the space group Pca2₁ was assigned.

Results and Discussion

Spectral studies

The H¹ spectrum of the Schiff base in DMSO exhibits signals at δ13.29 and 9.14 ppm, attributed to –OH and –CH=N– protons respectively. The multi signals within the (δ) 6.80-8.02 ppm range are assigned to the aromatic protons of both rings. The signal at δ2.50 is assigned to –CH protons. C¹³ – NMR: (DMSO- d⁶): 105.83, 116.62, 118.38, 120.82, 129.22, 131.91, 132.56, 133.80, 155.35 ppm (aromatic C), 163.52 ppm (– C– OH) and 136.56ppm (– C= N–) respectively. The IR spectrum of the Schiff base has a broad absorption band at 3298cm⁻¹ which is assigned to the phenolic OH. The breadth of this band indicates the presence of hydrogen bond. The

observation of bands in the range 1560, 1328 and 3043 cm^{-1} are $\nu(\text{C}=\text{N})$, $\nu(\text{C}-\text{O})$ and $\nu(\text{C}-\text{H})$ respectively. On condensation, the $\nu(\text{C}=\text{O})$ frequency vanishes and is replaced by band at 1656 cm^{-1} which corresponds to (azomethine, $-\text{HC}=\text{N}-$) carbon-nitrogen double bond stretching frequency.

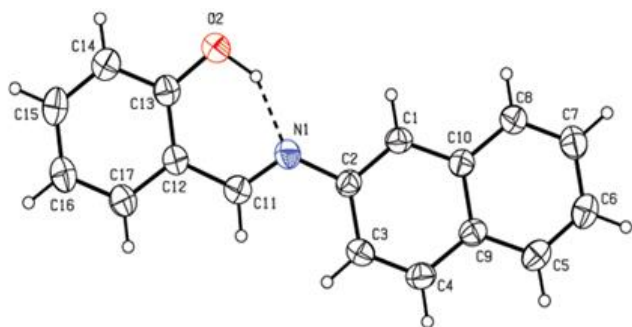
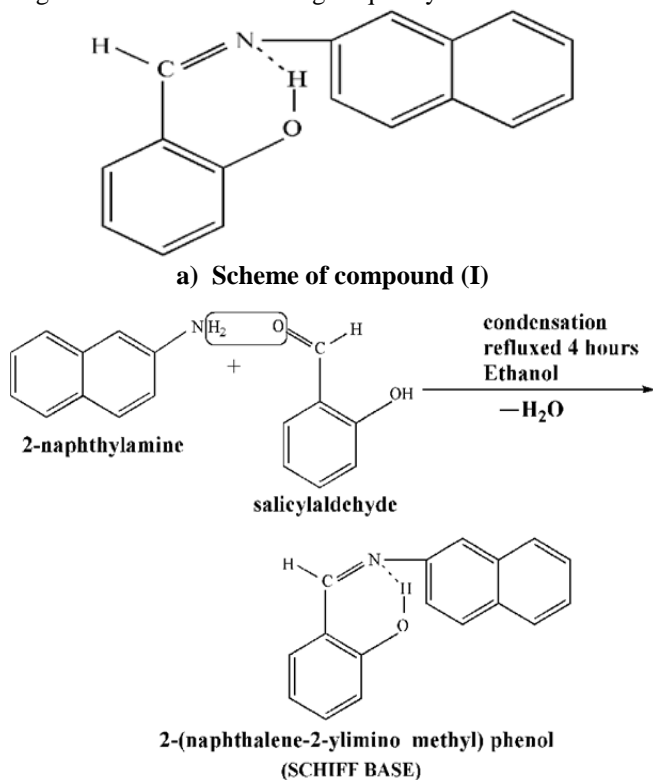


Figure 1. ORTEP view of the title compound (I) showing 50% probability displacement ellipsoids

An ORTEP [11] view of the asymmetric unit is shown in Figure 1. The compound crystallizes in the orthorhombic space group $\text{Pca}2_1$, with $a = 13.6460(3)$, $b = 5.8732(1)$, $c = 15.8729(3)$ Å, $\alpha = \beta = \gamma = 90^\circ$ and $Z = 4$. The dihedral angle between the salicylaldehyde moiety and amino phenyl plane is 59° . The two torsion angles τ_1 (N-C-C-C) and τ_2 (C-N-C-C) defining the confirmation of the molecule. In the present crystal structure the torsion angles are $177.5(2)^\circ$ ($\text{N1}-\text{C11}-\text{C12}-\text{C17}$), $-176.07(2)^\circ$ ($\text{C2}-\text{N1}-\text{C11}-\text{C12}$) and $-4.1(3)^\circ$ ($\text{C3}-\text{C2}-\text{N1}-\text{C11}$). The $\text{N1}-\text{C11}$ distance of $1.281(3)$ Å is normal double bond values and agree well with those observed in other azomethines. The $\text{C2}-\text{N1}-\text{C11}$ bond angle of $122.16(2)^\circ$ in the Schiff base ligand has a normal value. The bond lengths and angles of the title compound are shown in Table 2.

Hydrogen bonding

The two benzene rings (naphthyl and salicylaldehyde) and the azomethine group are practically coplanar, as a result of intramolecular $\text{O}-\text{H}\cdots\text{N}$ ($\text{O2}-\text{H2A}\cdots\text{N1} = 2.621(2)$ Å) hydrogen bond involving the hydroxy O-atom and azomethine N-atom with graph-set notation $\text{S}(6)$ as shown in Figure 2. Similar intramolecular hydrogen bonds are reported for the crystal structures of N-acetyl-4-[(2-hydroxybenzylidene)-amino] benzene sulfonamide monohydrate and N-acetyl-4-[(5-bromo-2-hydroxybenzylidene)amino]benzene sulfonamide monohydrate [12]. The hydrogen bonding geometries are shown in Table 3.

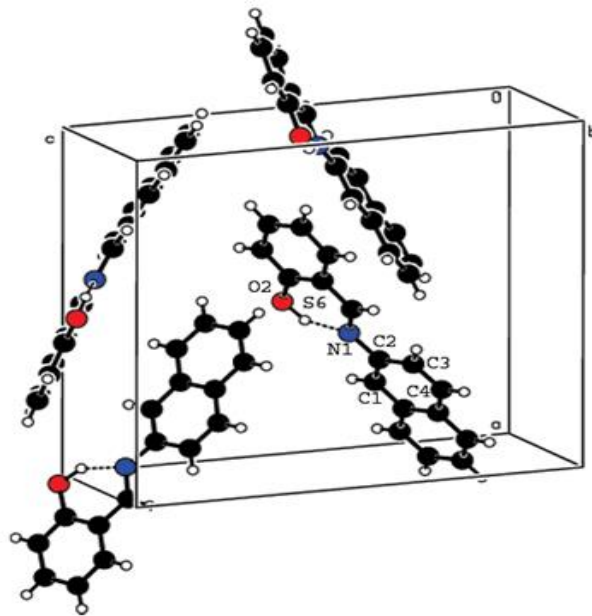


Figure 2. Hydrogen bonding interaction of compound (I).

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Supplementary material

CCDC- 880779 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 (0) 1223-336033; e-mail: deposit@ccdc.cam.ac.uk].

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Table 1. Crystallographic Data

Crystal Data	
Formula	C ₁₇ H ₁₃ NO
Formula Weight	247.28
CCDC deposit no.	880779
Crystal System	Orthorhombic
Space group	Pca21 (No. 29)
Shape, colour	prism, yellow
Cell dimensions	
a = 13.6460(3) [Å]	
b = 5.8732(1) [Å]	
c = 15.8729(3) [Å]	
V [Å ³]	1272.15(4)
Z	4
Density (calc) [g/cm ³]	1.291
Absorption coefficient	0.080 [mm ⁻¹]
F(000)	520
Crystal Size [mm]	0.04 x 0.05 x 0.06
Temperature (K)	296
Radiation [Å]	MoKα 0.71073
Min-Max [°]	2.6, 23.1
Dataset	-15: 15 ; -6: 6 ; -17: 17
Tot., Uniq. Data, R(int)	18405, 1788, 0.036
Observed data [I > 2.0 σ(I)]	1533
Refinement method	Full-matrix least squares on F ²
Solution method	Direct methods
Weighing scheme	w=1/[σ ² (Fo ²)+(0.0381P) ² +0.0738P]
where P=(Fo ² +2Fc ²)/3	
R, wR2, S	0.0291, 0.0733, 1.06
Flack x	-0.6(16)
Min. and Max. Resd. Dens. [e/Å ³]	-0.09, 0.09

Table 2 Bond Distances (Å) and angles (°) of Schiff base

O2	-C13	1.356(3)	C12	-C13	1.405(3)		
C13	-C14	1.392(3)	N1	-C2	1.420(3)		
C14	-C15	1.375(3)	N1	-C11	1.281(3)		
C15	-C16	1.382(3)	C1	-C10	1.411(3)		
C16	-C17	1.376(3)	C1	-C2	1.368(3)		
C2	-C3	1.409(3)	C3	-C4	1.365(3)		
C4	-C9	1.412(3)	C5	-C9	1.420(3)		
C5	-C6	1.356(3)	C6	-C7	1.400(3)		
C7	-C8	1.360(3)	C8	-C10	1.407(3)		
C9	-C10	1.413(3)	C11	-C12	1.450(3)		
C12	-C17	1.390(3)					
C15	-C16	-C17	119.3(2)	C2	-N1	-C11	122.08(17)
C12	-C17	-C16	121.46(18)	C2	-C1	-C10	122.13(17)
N1	-C2	-C1	116.94(16)	C1	-C2	-C3	118.69(18)
N1	-C2	-C3	124.36(17)	C2	-C3	-C4	120.61(18)
C3	-C4	-C9	121.51(17)	C6	-C5	-C9	120.8(2)
C5	-C6	-C7	120.5(2)	C6	-C7	-C8	119.9(2)
C7	-C8	-C10	121.5(2)	C4	-C9	-C5	123.14(18)
C4	-C9	-C10	118.26(17)	C5	-C9	-C10	118.61(17)
C1	-C10	-C9	118.80(16)	C8	-C10	-C9	118.62(17)
C1	-C10	-C8	122.57(17)	N1	-C11	-C12	121.95(17)
C11	-C12	-C17	119.43(17)	C13	-C12	-C17	118.59(17)
C11	-C12	-C13	121.95(17)	O2	-C13	-C14	118.8(2)
C12	-C13	-C14	119.69(19)	O2	-C13	-C12	121.51(18)
C13	-C14	-C15	120.1(2)	C14	-C15	-C16	120.8(2)

Table 3 – Hydrogen bonding geometries (Å, °)

D–H...A	D–H(Å)	H...A(Å)	D...A(Å)	D–H...A(°)
O2–H2A...N1	1.02(3)	1.71(3)	2.621(2)	147(2)

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