

Journal of Molecular Structure 323 (1994) 7-14

Journal of MOLECULAR STRUCTURE

The structure and tautomeric properties of 2-(3-pyridylmethyliminomethyl)phenol¹

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(Received 26 July 1993)

Abstract

The structure and properties of 2-(3-pyridylmethyliminomethyl)phenol have been determined by X-ray crystallography, NMR, UV and IR spectroscopy and compared with the characteristics of the Schiff bases derived from salicylaldehyde and aminopyridines. Crystal data: $C_{13}H_{12}N_2O$, $M_r = 212.250$, monoclinic, space group $P2_1/n$, a = 10.49(1) Å, b = 9.002(4) Å, c = 12.42(1) Å, $\beta = 109.10(4)^\circ$, V = 1109(2) Å³, Z = 4, $D_x = 1.271$ g cm⁻³, MoK α ($\lambda = 0.71073$ Å), $\mu = 0.8$ cm⁻¹, F(000) = 448, T = 297 K, R = 0.049, $R_W = 0.053$ for 886 reflections with $I > 2\sigma(I)$. Bond distances and bond angles are similar to those of 2-(pyridyliminomethyl)phenols. In the title molecule the dihedral angle between the pyridine and benzene least-squares planes is $89.9(2)^\circ$. There is an intramolecular $O1-H \cdots N1$ hydrogen bond of 2.572(5) Å. In solutions of polar solvents tautomeric interconversion of the Schiff base into the ketoamine is observed. Corresponding equilibrium constants are estimated in a variety of solvents on the basis of UV spectral data.

1. Introduction

Familiarity with the structural characteristics and equilibria of Schiff bases is the core to an understanding of their function in biological systems. Considerable attention has therefore been paid to the Schiff bases of amino- and aminoalkylpyridines which are structurally related to compounds participating in vitamin B_6 chemistry. Their additional attractive characteristics, pharma-

We have presented data previously about the characteristics of the Schiff bases of salicylaldehyde with 2-amino-3-aminomethylpyridine and 2,3-diaminopyridine [9, 10]. We have pointed to a potential ring-chain tautomerization ability of mono Schiff bases and considered this ability in relation to the structure. Here, we report spectroscopic characteristics, tautomeric properties and the structure of 2-(3-pyridylmethyliminomethyl)phenol. Bearing in mind the different behaviours of the Schiff bases derived from 2-amino-3-aminomethylpyridine and 2,3-diaminopyridine [9, 10], we have tried to compare the

¹The authors are grateful to the late Dr. Nikola Galešić for valuable long-lasting cooperation and dedicate this jointly started work to his memory.

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cological activity, and chromic properties, and the possibility of analytical application, have stimulated many investigations [1-8].

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Table 1 Crystal data and summary of experimental details and refinement

Molecular formula	$C_{13}H_{12}N_2O$
M _r	212.250
Crystal size (mm)	0.09 imes 0.34 imes 0.36
<i>a</i> (Å)	10.49(1)
$b(\mathbf{A})$	9.002(4)
$c(\mathbf{\dot{A}})$	12.42(1)
β (deg)	109.10(4)
$V(\text{\AA}^3)$	1109(2)
Crystal system	Monoclinic
Space group	$P2_1/n$
$D_{\rm r} (\rm gcm^{-3})$	1.271
Ζ	4
μ (MoK α) (cm ⁻¹)	0.8
F (000)	448
<i>T</i> (K)	297
No. of reflections used for cell	23
Parameters and θ range (deg)	4-16
θ range (deg) for intensity measurement	2-25
hkl range	(0, 10; 0, 8; -11, 11)
Scan	$\omega/2\theta$
$\Delta \omega$	$0.8 + 0.35 \tan \theta$
No. of measured reflections	2411
No. of symmetrical independent	
reflections	886 $(I > 2\sigma(I))$
No. of variables	156
R	0.049
$R_{\rm w}, w^{-1} = k(\sigma F_0^2 + gF_0)$	0.053
Final shift/error	0.029 (C4, x)
S	0.49
Residual electron density	
$(\Delta \rho)_{\rm max}, (\Delta \rho)_{\rm min} ({\rm e} {\rm \AA}^{-3})$	0.12, -0.19

properties of the title compound with 2-(pyridyliminomethyl)phenols.

2. Experimental

2.1. Preparation of Schiff bases

Salicylaldehyde was obtained from Merck, and the amines were purchased from Fluka. The title compound was prepared by mixing equimolar amounts of 3-aminomethylpyridine and salicylaldehyde, both dissolved in absolute ethanol, and refluxing for 2 h on a water bath. The Schiff base, which crystallized slowly from the reaction solution after the solvent had been removed by evaporation, was recrystallized from the mixture cyclohexane/petroleum ether 1/1 (m.p. 49–52°C, elemental analysis for $C_{13}H_{12}N_2O$: calculated: C, 73.53; H, 5.70; N, 13.20; found: C, 73.17; H, 5.50; N, 13.17%).

The same procedure was used for preparation of 2-(2-pyridyliminomethyl)phenol from salicylaldehyde and 2-aminopyridine (m.p. $60-62^{\circ}$ C, elemental analysis for C₁₂H₁₀N₂O: calculated: C, 72.71; H, 5.09; N, 14.13; found: C, 73.17; H, 5.53; N, 13.4%).

2.2. Spectroscopy

Absorption spectral data were obtained using a Varian Cary 3 spectrometer. IR spectra of liquid samples and of KBr discs were recorded with Perkin–Elmer 783 and FT 1725 X IR spectrometers. The spectra were measured at 2 cm^{-1} resolution. NMR spectra were recorded on a Varian XL Gemini 300 spectrometer with tetramethylsilane as internal standard. Digital resolution was 0.01 ppm. In solvents containing water, hydrolysis of Schiff bases took place, hence spectra were recorded in a very short time or extrapolated to zero time.

2.3. X-ray structure analysis

Crystallographic data and details of data collection and refinement are listed in Table 1. Data reduction was performed by the Enraf-Nonius SDP/VAX package [11]; Lorentz and polarization effects were corrected. Reference reflections showed a loss of intensity of about 2% during data collection. The structure was solved by direct methods, using the SHELX86 program [12]. The scattering factors were those included in the SHELX77 program [13]. The structure was refined by a fullmatrix least-squares method on the F values: the non-hydrogen atoms were refined anisotropically and the hydrogen atoms isotropically. The hydrogen atoms were introduced and refined under the constraints of pivot carbon atom geometry. The hydrogen atoms of the OH group (phenol moiety) and C7 were located from a difference Fourier map. The overall thermal parameters were assigned and refined to the hydrogen atoms.

Atom	x	у	Ζ	$U_{\rm eq}({ m \AA}^2)^{ m a}$
01	0.5107(3)	0.5886(3)	0.1490(3)	0.093(1)
NI	0.6977(3)	0.6616(3)	0.0782(3)	0.079(1)
N2	0.6118(4)	0.8551(6)	-0.2985(3)	0.106(2)
Cl	0.5788(4)	0.6173(4)	0.2516(4)	0.073(2)
C2	0.5278(5)	0.6032(5)	0.3415(4)	0.091(2)
C3	0.5944(6)	0.6667(5)	0.4453(4)	0.098(2)
C4	0.7132(5)	0.7444(5)	0.4638(4)	0.099(2)
C5	0.7653(4)	0.7573(5)	0.3765(4)	0.086(2)
C6	0.6999(4)	0.6948(4)	0.2687(3)	0.068(2)
C7	0.7567(5)	0.7119(4)	0.1775(4)	0.077(2)
C8	0.7627(4)	0.6818(5)	-0.0082(4)	0.090(2)
C9	0.6849(4)	0.7877(4)	-0.1002(3)	0.069(2)
C10	0.6750(5)	0.7632(5)	-0.2119(4)	0.093(2)
C11	0.5559(5)	0.9758(6)	-0.2725(4)	0.094(2)
C12	0.5601(5)	1.0078(5)	-0.1657(4)	0.098(2)
C13	0.6246(5)	0.9139(4)	-0.0774(4)	0.090(2)

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters of the non-hydrogen atoms

^a $U_{eq} = (1/3) \Sigma_i \Sigma_j U_{ij} a_i^* a_j^* a_i \cdot a_j$

Bond lengths and angles, torsion angles, and the preparation of a packing diagram were performed by the EUCLID package [14]. The molecular structure with anisotropic thermal ellipsoids (scaled at a 30% level) was drawn with the ORTEPII program [15]. Calculations were carried out on a micro-VAX II computer in the X-ray Laboratory of the Ruđer Bošković Institute, Zagreb. Final atomic coordinates of the non-hydrogen atoms and equivalent isotropic thermal parameters are listed in Table 2.

A list of structure factors, anisotropic thermal parameters, and hydrogen coordinates is available as supplementary data, deposited with BLLD as Supplementary Publication number SUP26492 (6 pages).



Fig. 1. ORTEP drawing of 2-(3-pyridylmethylimidomethyl)phenol showing atom numbering. Intramolecular hydrogen bond $O1-H\cdots N1$ is shown.



Fig. 2. Molecular packing in 2-(3-pyridylmethylimidomethyl)phenol.

3. Discussion

The solid state structure of 2-(3-pyridylmethyliminomethyl)phenol established by X-ray diffraction analysis is given in Figs. 1 and 2. Interatomic distances, bond and selected torsion angles are listed in Tables 3 and 4.

The geometry of the molecule differs from those of the Schiff bases obtained by condensation

Table 3 Bond lengths (Å) and angles (deg)

01-C1	1.348(6)	C7-N1-C8	118.3(4)
N1-C7	1.269(6)	C10-N2-C11	116.8(4)
N1-C8	1.459(6)	O1-C1-C2	119.9(4)
N2-C10	1.347(6)	O1-C1-C6	120.8(4)
N2-C11	1.324(7)	C2-C1-C6	119.4(4)
C1-C2	1.393(7)	C1-C2-C3	120.2(5)
C1-C6	1.404(6)	C2-C3-C4	121.4(5)
C2-C3	1.374(7)	C3-C4-C5	118.9(4)
C3-C4	1.381(8)	C4-C5-C6	121.5(4)
C4-C5	1.370(7)	C1-C6-C5	118.6(4)
C5-C6	1.407(6)	C1-C6-C7	121.2(4)
C6-C7	1.451(6)	C5-C6-C7	120.3(4)
C8-C9	1.507(6)	N1-C7-C6	121.8(4)
C9-C10	1.375(6)	N1-C8-C9	111.7(4)
C9-C13	1.374(6)	C8-C9-C10	120.5(4)
C11-C12	1.344(7)	C8-C9-C13	122.6(4)
C12-C13	1.375(6)	C10-C9-C13	116.9(4)
		N2-C10-C9	124.2(4)
		N2-C11-C12	122.9(5)
		C11-C12-C13	120.3(5)
		C9-C13-C12	118.9(4)

of salicylaldehyde with 2-aminopyridines [2, 5], 3-aminopyridine [3] and 2,3-diaminopyridine [10], due to the methylene group inserted between the N atom of the imino group and the pyridine ring.

Moustakali-Mavridis et al. [3] have shown that 2-(2-pyridyliminomethyl)phenols are almost planar molecules, with dihedral angles between the benzene and pyridine least-squares planes 4.6°, 4.4°, 4.6°, and -3.1° in 2-(2-pyridyliminomethyl)phenol, 3-methoxy-2-(3-methyl-2-pyridyliminomethyl)phenol, 5-bromo-2-(2-pyridylimino-5-methoxy-2-(5-chloro-2methyl)phenol, and pyridyliminomethyl)phenol, respectively. In other 2-(2-pyridyliminomethyl)phenols: 2-(4,6-dimethyl-2-pyridyliminomethyl)phenol, 2-(4-methyl-2-pyridyliminomethyl)phenol, and 4-bromo-2-(2-pyridyliminomethyl)phenol, reported by Garland and co-workers [5], the corresponding dihedral angles are $5.4(9)^{\circ}$, $9.4(9)^{\circ}$, and 5.2° , respectively.

2-(3-Pyridyliminomethyl)phenols do not have planar structures: deviation from planarity is the

Table 4 Selected torsion angles (deg)

······································		
C7-N1-C8-C9	-112.1(4)	
C8-N1-C7-C6	-179.2(4)	
C1-C6-C7-N1	2.1(6)	
C5-C6-C7-N1	-177.4(4)	
N1-C8-C9-C13	39.4(6)	
N1-C8-C9-C10	-142.6(4)	

result of repulsive interactions of the exocyclic hydrogen atoms from the methine groups with ortho positioned protons on the nuclei. The dihedral angle between the pyridine and phenol least-squares planes is $14.8(2)^{\circ}$ in 2-(3-pyridyliminomethyl)phenol [3]. In the case of 2-(2amino-3-pyridyliminomethyl)phenol this angle is $51.5(2)^{\circ}$ [10], but there is an additional reason for distortion of planarity: an H–H repulsion of two hydrogen atoms that are included in two hydrogen bonds with a common acceptor nitrogen atom.

Accordingly, in the molecules of mono and bis Schiff bases of 2,3-diaminopyridines, the 2-positioned salicylidene moiety is in the plane of the pyridine ring, whereas the 3-positioned salicylidene moiety is inclined, the corresponding dihedral angles amounting to $51.1(4)^{\circ}$ and $51.8(4)^{\circ}$ (two crystallographically independent molecules of 2,3-bis(salicylideneamino)pyridine), respectively. Other reasons for deviation from planarity of the molecule of the bis Schiff base of 2,3-diaminopyridine are the H–H steric repulsions: hydrogen atoms of both hydroxy groups are bonded by hydrogen bonds to the nitrogens of the imino groups.

case of 2-(3-pyridylmethylimino-In the methyl)phenol, the benzene π system delocalization is extended over C8-N1-C7-C6 bonds (Table 3), so that the N1, C7, and C8 atoms deviate slightly from the benzene least-squares plane. the deviation amounting to 0.04(2)Å, 0.06(2)Å, and 0.01(2)Å, respectively. They deviate more from the pyridine least-squares plane, bv 0.06(2)Å, -0.076(2)Å, and -0.18(2)Å respectively. All these deviations are of the same order as those of the Schiff bases of aminopyridines reported by Moustakali-Mavridis et al. [3] and Cimerman et al. [10]. However, the C8-C9 bond makes an angle of $61.0(3)^{\circ}$ with the benzene leastsquares plane, whereas the benzene and pyridine rings are almost perpendicular, and the dihedral angle is $89.9(2)^{\circ}$. The latter angle is significantly greater than those in the Schiff bases of 3-aminopyridines and 2,3-diaminopyridines. There is some H-H repulsion which determines the position of the pyridine ring with respect to the phenol moiety such as in H10-H82 and H81-H7 contacts (Fig. 1) with distances of 2.33(7) Å and 2.15(4) Å,

respectively. The exocyclic C8 and H7 atoms are in the cis position.

The bond length N1-C7 is 1.269(6) Å, which corresponds to a double bond, indicating an enolimino character of the molecule. The short bond lengths O1-C1, C2-C3, C4-C5, and C6-C7 are due to a quinoid resonance structure (Table 3). The molecular conformation is stabilized by intramolecular hydrogen bonding between the hydroxy and imino groups, with the geometry O1...N1, O1-H, H...N1 of 2.572(5) Å, 1.02(5) Å and 1.73(5) Å, respectively, and the O1-H...N1 angle 137(4)° (Fig. 1). The supplementary ring formed as the result of hydrogen bonding is tilted with respect to the pyridine ring by 86.8(2)°. The crystal packing is determined by van der Waals interactions only.

The characteristics of 2-(3-pyridylmethyliminomethyl)phenol in solution have been studied using NMR, IR and UV spectroscopic methods. Attention has been turned to ketoaminoenolimino tautomerism.

In the ¹H NMR spectrum of CDCl₃ solution (Table 5) the signals of the methine proton at 8.48 ppm and of the hydroxy proton at 13.06 ppm point at predominance of the enolimino form with the intramolecular bond OH \cdots N=C. No splitting of the methine proton signal is observed, nor are the aromatic protons displaced from their normal benzenoid positions as would be implied by the quinoid form. Accordingly, in the IR spectrum of CCl₄ solution, bands at 1630, 1280 and 2650 cm⁻¹,

Table 5

Proton chemical shifts and splitting constants in the ${}^{1}H$ NMR spectrum of a CDCl₃ solution of 2-(3-pyridylmethylimino-methyl)phenol

Proton	Chemical shift δ (ppm)	Splitting constant J (Hz)
HI	13.06	$J_{11,12} = 4.1$
H10	8.59	$J_{12,13} = 7.8$
H7	8.48	$J_{3,4} = 7.4$
H11	8.55	$J_{4.5} = 7.4$
H13	7.64	$J_{56} = 8.2$
H12	7.29	
H3	7.34	
Н5	7.30	
H4	6.90	
H6	6.96	
H8	4.80	

Solvent	ν(OH)	ν (C=N)	ν(C–O)	ν(C=O)	ν (NHC=CC=O)
2-(3-pyridylmethylimin	omethyl)phenol				
CCl ₄	2650vw	1630vs	1280s		
CH ₃ OH/H ₂ O 9/1	a	1630vs	1280s	1650w	1530vw
CH ₃ OH/H ₂ O 8/2	a	1630vs	1280s	1652w	1530vw
KBr	2640vw	1630vs	1275s		
2-(2-pyridyliminomethy	l)phenol				
CCl ₄	2750vw	1612s 1558s	1283m		
CH ₃ OH/H ₂ O 9/1	а	1618s 1560s	1280m		
CH ₃ OH/H ₂ O 8/2	а	1619s 1562m	1280m		
KBr	2730vw	1612s 1556s	1280s		

Table 6 Characteristic IR bands (cm⁻¹) of 2-(3-pyridylmethyliminomethyl)phenol and 2-(2-pyridyliminomethyl)phenol

^a Overlapped with solvent absorption.

s, strong; v, very; m, medium; w, weak.

characteristic of C=N, C-O, and OH stretching of enolimine, are present (Table 6). In polar solvents, e.g. methanol/H₂O, weak absorption at 1650 and $1530 \,\mathrm{cm}^{-1}$ occurs. Corresponding bands have been assigned to ν (C=O) (the former absorption) and to quinoid ring skeletal stretching ν (NHC=CC=O) (the latter absorption) [16, 17]. Consistent with the results of X-ray analysis, these bands reflecting the presence of ketoamine are missing in the spectrum recorded in KBr pellets. Shown in Table 6 are the IR spectra of 2-(2-pyridyliminomethyl)phenol. This compound does not exhibit an observable degree of ketoamine structure even in polar solvents. Comparing values of OH stretching frequencies of 2-(3-pyridylmethyliminomethyl)phenol with 2-(2-pyridyliminomethyl)phenol (Table 6) reveals a lower frequency value for the former compound. On the assumption that no other effects are present, a shift of OH stretching frequency to lower values reflects a stronger intramolecular H bond [18]. It can be deduced that the increase in imino nitrogen basicity, caused by insulation from the ring by a CH_2 group, results in a strengthening of the intramolecular H bond $OH \cdots N=C$, followed by a shift of the tautomeric equilibrium to the ketoamine form.

The tautomeric equilibrium has been systematically studied by analysing electronic absorption spectra. The parameters of spectra of 2-(3-pyridylmethyliminomethyl)phenol in various solvents are listed in Table 7. The spectra, measured in non-

Table 7

UV/Vis absorption spectra of 2-(3-pyridylmethyliminomethyl)phenol in various solvents

Solvent	$\lambda \text{ (nm) } [(\epsilon/L \text{ mol}^{-1} \text{ cm}^{-1}) \cdot 10^{-4}]$					
Diethyl ether	217 (2.64)	256 (1.57)		318 (0.46)		
Dichloromethane	. ,	257 (1.59)		318 (0.46)		
Chloroform		259 (1.57)		318 (0.46)		
Dioxane		257 (1.57)		318 (0.46)		
Ethanol	214 (2.58)	256 (1.61)		317 (0.44)	405 (0.02)	
Methanol	215 (2.56)	256 (1.57)		316 (0.42)	405 (0.03)	
Dioxane/H ₂ O 1/1		256 (1.50)	279 (shoulder)	316 (0.37)	405 (0.08)	
Methanol/H ₂ O 1/1	213 (2.12)	256 (1.33)	279 (0.46)	315 (0.30)	403 (0.14)	

 Table 8

 Values of tautomeric constants in various solvents

Solvent		Ketoamine (%)	K
Diethyl ether		0	_
Dichloromethane		0	_
Chloroform		0	-
Dioxane		0	-
Ethanol		4	0.04
Methanol		8	0.09
Dioxane/H ₂ O	4 /1	7	0.08
· -	3/2	10	0.11
	1/1	20	0.25
	2/3	26	0.35
	1/4	44	0.78
Methanol/H ₂ O	4/1	17	0.21
. –	3/2	28	0.39
	1/1	33	0.49
	2/3	41	0.69
	1/4	51	1.04

polar solvents, contain two bands above 240 nm in the solvent transparent region, at 318 and 256-257 nm. These bands are due to $\pi - \pi^*$ transitions. By analogy with 2-(phenyliminothe methyl)phenols, the former band can be associated with excitation of the whole molecule and the latter one with excitation of the salicylidene fragment [19, 20]. In polar solvents one additional pair of bands emerges at approximately 400 and 279 nm and has been logically linked with the shift of tautomeric equilibrium to the ketoamine form. Similar UV spectral data have been obtained in the case of a tautomeric interconversion to ketoamine of the Schiff bases of the o-hydroxyaromatic aldehydes with aliphatic amines and amino acids [21, 22]. Assuming that the wavelength shift from 405 and 318 nm (in diethyl ether) to 398 and 314 nm (in methanol/ $H_2O(2/8)$ is due to the solvent effect, and that the molar absorptivities are not changed markedly by variation of solvent, the percentage of the ketoamine form can be deduced from the values of ϵ at 314-318 and 398-405 nm. Table 8 contains values of the tautomeric constants $K_{\rm r}$ = [ketoamine]/[enolimine] in different solvents. In non-polar solvents only enolimine is present. An increase of solvent dielectric constant results in a

continuous increase of K_t , which reaches the value of about unity in methanol/water 1/1.

Császár and Balog [23] have observed the appearance of a shoulder between 430 and 450 nm in the spectrum of a methanolic solution of 2-(2-pyridyliminomethyl)phenol and supposed on the basis of this observation the presence of the ketoamine. A corresponding absorption has not been noted in the case of 2-(3-pyridyliminomethyl)phenol. We have estimated the approximate values of tautomeric constants for 2-(2-pyridyliminomethyl)phenol from the values of ϵ at 442 nm (ketoamine) and 305 nm (enolimine) in methanol, methanol/ H_2O 4/1 and methanol/ H_2O 1/1. The obtained values of K_t are 0.02, 0.09 and 0.14, respectively, showing that this Schiff base also tends to tautomerism, although to a lesser extent than 2-(3-pyridyliminomethyl)phenol.

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