



SPECTROSCOPIC AND POTENTIOMETRIC STUDIES OF CO (II) AND NI (II) COMPLEXES WITH N-(1-MORPHOLINO BENZYL) SEMICARBAZIDE

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ABSTRACT

The Schiff base complexes of cobalt(II) and nickel(II) were synthesized by refluxing the mixture of ethanolic solutions of the Schiff base and metal(II) chlorides, respectively. The complexes were characterized by gravimetry, spectrophotometry, potentiometry, molar conductance and infrared analyses. The N-(1-morpholinobenzyl)semicarbazide Schiff base and its complex compounds are not soluble in water, slightly soluble in most common organic solvents but are readily soluble in methanol and ethanol. The molar conductance of the cobalt (II) and nickel(II) schiff base complexes determined are in the range 5.38 - 6.03 $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$, suggesting their non-electrolytic nature. The dissociation constant, pK_a , of N-(1-morpholinobenzyl)semicarbazide Schiff base determined is 5.40. The standard Gibb's free energy of cobalt(II) and nickel(II) N-(1-morpholinobenzyl)semicarbazide Schiff base complexes determined are -70.78KJmol^{-1} and -68.06KJmol^{-1} , respectively. The ratio of metal ion to Schiff base determined potentiometrically and spectrophotometrically for the complex compounds is 1: 2. The IR spectrum of the Schiff base shows bands observable in the two Schiff base complex compounds, indicating the coordination of the Schiff base to the cobalt(II) and nickel(II) ions.

Key Words: Schiff base, azomethine, benzyldehyde, potentiometry, spectrophotometry, morpholine.

INTRODUCTION

The reaction of primary amines and an aldehyde or a ketone forms a Schiff base. A Schiff base is a chemical compound containing a -C=N- group (Holm *et al.*, 1966; Hobday and Smith, 1972; Pierre, 1987). The carbonyl group of the aldehyde gives aldimines while that of ketone gives ketoimines. It has been known that different metal ions on interaction with Schiff bases yield chelates, for example; Tsumaki, (1983) reported $[\text{Co}(\text{sal}_2\text{-en})]$ complex which received a great attention owing to its ability to undergo reversible adduct formation with molecular oxygen. The oxygenation ability of the complex was first recognized by Hassan *et al.* (1998). However, the mechanism for the oxygenation process was not well understood until recently with the advent of modern physical techniques. Xishi *et al.* (2003) reported the synthesis and characterization of a novel Schiff base ligand formed from the condensation of 2,2-bis (P-methoxyphenylamine) and Salicylaldehyde and its Mn(II), Co(II) and Cu(II) complexes. Ben Saber *et al.*, (2005) reported the synthesis and characterization of Cr(III), Fe(III), Co(II) and Ni(II) complexes with a Schiff base derived from 4-dimethylamino benzaldehyde and primary amines. The chemical analysis data showed the formation of (1:1) metal - ligand ratio and a square planar geometry was suggested for Co(II) and Ni(II) complex while an octahedral structure was suggested for Cr(III) and Fe(III) complexes. In another report, Cu(II), Co(II), Ni(II) and Zn(II) complexes of new heterocyclic Schiff base derived from 1-amino - 5- benzoyl - 4- phenyl - 1H - pyrimidine - 2- one with salicylaldehyde have

been synthesized and investigated by elemental analysis (Sonmez and Sekerel, 2002). An octahedral geometry was suggested for all the complexes. Ben Saber *et al.* (2005), reported the synthesis of a Schiff base derived from salicylaldehyde, and Histidine and its complex compounds with divalent transition metal ions. The complexes were investigated by elemental analysis and were found to be of 1:1 metal to ligand ratio.

Transition metal Schiff base complexes are used in various fields, such as medicine, agriculture, industries etc. For example, $[\text{Co}(\text{acac}_2\text{-en})]$ in dimethylformamide, pyridine and substituted pyridines proved to be involved in oxygen metabolism (Hanna and Mona, 2001). Transition metal complexes with 1, 10 - phenanthroline and 2, 2 - bipyridine are used in petroleum refining (John *et al.*, 1976). Schiff base formed by the condensation of 1-formyl-2-hydroxy-3-naphtholic arylamide with o-hydroxyl or o-methoxy aniline complexes of Co(II), Ni(II), Cu(II) and Zn(II) are useful as pigments (Gupta *et al.*, 2002). Oxovanadium Complexes have been found strongly active, against some type of Leukemia (Dong *et al.*, 2002). Transition metal complexes derived from a number of amino acids have been reported to have biological activity (Zahid *et al.*, 2007). Morad *et al.* (2007), reported the antibacterial activity of Ni(II) with salicylaldehyde and 2-amino-benzoic acid complex. This paper reports the potentiometric and spectroscopic studies of cobalt (II) and nickel(II) N-(1-morpholinobenzyl)semicarbazide complexes, as exhaustive literature search revealed rare information.

MATERIALS AND METHODS

Chemicals of analytical grade purity were used. Electrical metler balance model AB 54, was used in weighing. Melting point, decomposition temperature were determined on Gallenkamp melting point apparatus. IR spectra measurements were recorded using Fourier Transformed IR Genesis series model in Nujol in the region 40000 – 400cm. electrical conductivity measurements were carried out using conductivity meter model 4010. UV-visible spectral measurements were done on a Pye Unicam UV-visible spectrophotometer.

Synthesis of N-(1-Morpholinobenzyl) Semicarbazide

To 20cm³ semicarbazide hydrochloride (11%) solution in ethanol, a 10cm³ of benzaldehyde was added dropwise with constant stirring and then neutralized with ammonia solution. To this solution 10cm³ of morpholine was also added dropwise, while maintaining the stirring for 20minutes. On standing precipitate was observed to form and was separated and recrystallised from hot ethanol (Raman *et al.*, 2004).

Synthesis of the Metal Complexes

A mixture of 5mmol metal (II) chlorides in ethanol and N-(1-morpholinobenzyl)semicarbazide schiff base (1.25g, 5mmol) solution in ethanol/chloroform mixture (1.6) was refluxed for 3 hours. The resulting mixture was concentrated to about a third of its volume and then cooled to 0°C, for 12 hours. The precipitate that formed was filtered, washed with ethanol and dried in vacuo (Raman *et al.*, 2004).

Determination of Dissociation Constant of N-(1-Morpholinobenzyl)semicarbazide

To a 400 cm³ beaker were added 90 cm³ of distilled water, 100cm³ 0.2M KNO₃, 10cm³ of 0.4M N-(1-morpholinobenzyl)semicarbazide and a magnetic stirring bar before the electrodes of a standardized meter were introduced. A 10cm³ of standardized aqueous NaOH was added dropwise with stirring. After each 0.5cm³ addition, the amount of NaOH solution and the corresponding pH were recorded. Points in the 20 to 80 percent titration range were used to calculate the pKa (Gregory *et al.*, 1978).

Determination of Stability Constants of N-(1-Morpholinobenzyl) Semicarbazide Cobalt(II) and Nickel(II) Complexes

Into a 400cm³ beaker 100cm³ of 0.2M KNO₃, 1mmole of metal (II) chloride, 0.1M HNO₃ and 90cm³ of distilled water were added, respectively. A magnetic stirring bar and sodium salt of the Schiff base, prepared by neutralizing a known quantity of the N-(1-morpholinobenzyl)semicarbazide Schiff base with calculated amount of standardized NaOH solution. After each 0.2cm³ aliquot addition, the corresponding pH of the stirred reaction mixture was recorded. From the results obtained, stability constant of the complex compound and the number of the coordinated ligands per metal ion were determined (Gregory, *et al* 1978).

Determination Schiff Base to Metal (II) ion By Continuous Variation Method

The ligands to metal ratio in the complex compounds were determined using "Job's methods" in which one millimolar solutions of the Schiff base and a metal (II) chloride were prepared. By mixing these solutions, mixtures having a total volume of 10cm³ in which the mole fraction of N-(1-morpholinobenzyl)semicarbazide, X, is 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, and 0.9 were prepared. The absorbance of each of these solutions was recorded at the wavelength obtained for a metal salt at maximum absorbance (λ_{max}) at which the metal(II) ion solution absorbs maximally. A plot of absorbance against the ligand's mole fractions was made, from which mole fraction (X) at maximum absorbance was recorded for the mixture containing stoichiometric amount of M²⁺ and ligand from which the number of coordinated Schiff base per a metal ion, n was calculated (Angelici, 1977).

RESULTS AND DISCUSSION

The reaction of morpholine, benzaldehyde and semicarbazide hydrochloride mixture formed N-(1-morpholinobenzyl)semicarbazide Schiff base, a white crystalline product with melting point of 200°C. The metal Schiff base cobalt(II) and nickel(II) complexes synthesized are crystalline pink and green, with yield of 61.30% and 66.34% and have decomposition temperatures 225°C and 240°C, respectively (Table 1). The N-(1-morpholinobenzyl)semicarbazide Schiff base and its metal complexes are not soluble in water, slightly soluble in most common organic solvents but are readily soluble in methanol and ethanol (Table 2). The molar conductance measurement carried out on cobalt(II) and nickel(II) Schiff base complexes are within the range 5.38 – 6.03 ohm⁻¹cm²mol⁻¹, indicating their non-electrolytic nature (Table 3). The IR spectra of the free N-(1-morpholinobenzyl)semicarbazide Schiff base shows bands in the region 1565 - 1600cm⁻¹, which are assigned to $\nu(C=N)$ stretching vibration, a fundamental feature of azomethine group, a confirmation of formation of a Schiff base. Bands within this region are also observable in cobalt(II) and nickel(II) N-(1-morpholinobenzyl)semicarbazide Schiff base complexes an indication that the schiff base, N-(1-Morpholinobenzyl)semicarbazide, has coordinated to the metal ions. The bands around 450-480 and 365-410cm⁻¹ are attributed to $\nu(M-O)$ and $\nu(M-N)$ stretching vibrations, respectively, reaffirming the coordination of the Schiff base to the metal ions (Table 4). The elemental analysis of N-(1-morpholinobenzyl)semicarbazide cobalt(II) and nickel(II) complexes, suggested the molecular formulae, [CoL₂].2H₂O, and [NiL₂].3H₂O, respectively. The dissociation constant, pKa, of N-(morpholinobenzyl)semicarbazide Schiff base (Table 8) determined is 5.40, indicating a weak acid. The stability constants of cobalt(II) and nickel(II) Schiff base complexes determined (Table 7) are 9.37x10¹⁰ and 2.98x10¹⁰, which correspond to the standard Gibb's energies of -70.78KJmol⁻¹ and -68.06KJmol⁻¹, respectively.

These low standard Gibb's free energies of the complex compounds explain their good stability, as buttressed by their high decomposition temperatures. The potentiometric analytical results revealed 1:2 metal to N-(1-morpholinobenzyl)semicarbazide Schiff base in both the complex compounds (Tables 9 and 10). This ratio is supported by the results obtained

from continuous variation studies on cobalt(II) and nickel(II) N-(1-morpholinobenzyl) semicarbazide complex compounds (Tables 5 and 6).

From the analytical results of the two divalent metal N-(1-morpholinobenzyl)semicarbazide complex compounds obtained, the molecular structure in Fig. 1 below, is proposed.

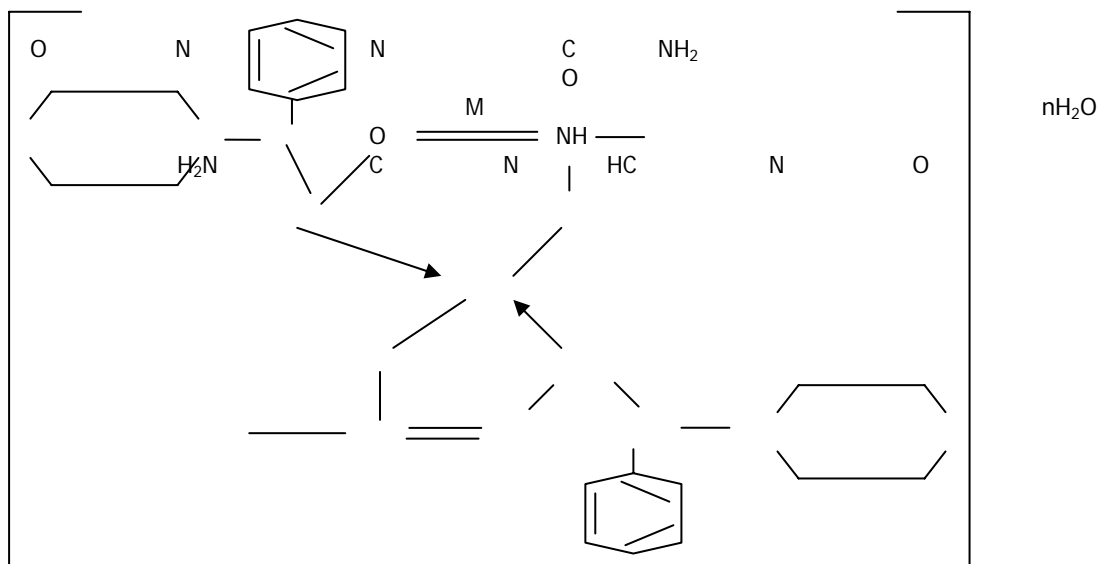


Figure 1: The proposed molecular structure of the complexes, where M is Co(II) or Ni(II) and n, the number of water molecules.

Table 1: Some Physical properties of the Schiff base and Its Co(II) and Ni(II) Complexes

Complex/Compound	Colour	Melting Point (°C)	Decomposition temperature (°C)	Percentage yield (%)
Ligand	White	200	-	78.60
[CoL ₂] ₂ H ₂ O	Pink	-	225	61.30
[NiL ₂] ₃ H ₂ O	Green	-	240	66.34

Table 2: Solubility Test of the Compounds in Some Solvents

Compound	Water	Methanol	Ethanol	Acetone	Chloroform	Benzene	DMSO
Ligand	IS	S	S	SS	SS	SS	IS
[CoL ₂] ₂ H ₂ O	IS	S	S	SS	SS	IS	SS
[NiL ₂] ₃ H ₂ O	IS	S	S	SS	SS	IS	SS

Key: S = Soluble, SS = Slightly Soluble, IS = Insoluble

Table 3: Conductivity Measurement of the Complexes in 10⁻³M Methanol

Complexes	Concentration (mol/dm ³)	Specific conductivity (ohm ⁻¹ cm ⁻¹)	Molar Conductivity (ohm ⁻¹ cm ² mol ⁻¹)
[CoL ₂] ₄ H ₂ O	1 x 10 ⁻³	3.38 x 10 ⁻⁶	3.38
[NiL ₂] ₂ H ₂ O	1 x 10 ⁻³	6.90 x 10 ⁻⁶	6.90

Table 4: Infrared Spectral Data of the ligand and its Metal (II) Complexes

Compound	$\nu(\text{C}=\text{N})$	$\nu(\text{O}-\text{H})$	$\nu(\text{C}=\text{C})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
Ligand	1599	3340-3570	1670	-	-
[CoL ₂] ₄ H ₂ O	1565	3355-3520	1650	450-480	365-390
[NiL ₂] ₂ H ₂ O	1600	3330-3540	1680	455-470	370-410

Table 5: Mole Fraction and Corresponding Absorbance of Cobalt(II) Complex

Mole fraction	Absorbance
0.1	0.04
0.2	0.14
0,3	0.27
0.4	0.34
0.5	0.45
0.6	0.56
0.7	0.69
0.8	0.50
0.9	0.43

Table 6: Mole Fraction and Corresponding Absorbance of Nickel(II) Complex

Mole fraction	Absorbance
0.1	0.04
0.2	0.10
0,3	0.16
0.4	0.27
0.5	0.34
0.6	0.45
0.7	0.49
0.8	0.34
0.9	0.20

Table 7: The Stability Constant and Standard Gibb's Free Energy of the Complexes

Compound	Stability constant	Gibb's free energy (KJmo ⁻¹)
[CoL ₂] ₂ H ₂ O	9.37x10 ¹⁰	-70.78
[NiL ₂] ₃ H ₂ O	2.98x10 ¹⁰	-68.06

Table 8: Determination of Dissociation Constant (pKa) of the Ligand

S/NO	Vol. of NaOH [cm ³]	pH	[H ⁺]	[OH ⁻]	[Na ⁺]	A _{tot} x 10 ⁻²	Pka
1.	-	4.93	786x10 ⁻⁶	2.06x10 ⁻⁹	-	2.00	-
2.	0.5	5.0	6.69x10 ⁻⁶	2.42 x10 ⁻⁹	1.15 x10 ⁻³	1.20	6.14
3	1.0	5.02	6.39x10 ⁻⁶	2.54x10 ⁻⁹	2.29 x10 ⁻³	1.99	6.07
4	1.5	5.04	6.10x10 ⁻⁶	2.66 x10 ⁻⁹	3.42 x10 ⁻³	1.99	5.89
5	2.0	5.06	5.83x10 ⁻⁶	2.78x10 ⁻⁹	4.55 x10 ⁻³	1.98	5.67
6	2.5	5.06	5.83x10 ⁻⁶	2.78x10 ⁻⁹	5.66x10 ⁻³	1.98	5.63
7	3.0	5.07	5.70x10 ⁻⁶	2.85x10 ⁻⁹	6.80x10 ⁻³	1.97	5.62
8	3.5	5.08	5.57x10 ⁻⁶	2.91x10 ⁻⁹	7.91x10 ⁻³	1.97	5.42
9	4.0	6.01	6.54x10 ⁻⁷	2.48x10 ⁻⁸	9.02x10 ⁻³	1.96	6.25
10	4.5	6.01	6.54x10 ⁻⁷	2.48x10 ⁻⁸	1.01x10 ⁻²	1.96	6.15
11	5.0	6.02	6.39x10 ⁻⁷	2.53x10 ⁻⁸	1.12x10 ⁻²	1.95	6.07
12	5.5	6.03	6.24x10 ⁻⁷	2.60x10 ⁻⁸	1.23x10 ⁻²	1.95	5.97
13	6.0	6.05	5.96x10 ⁻⁷	2.72x10 ⁻⁸	1.34x10 ⁻²	1.94	5.87
14	6.5	6.05	5.96x10 ⁻⁷	2.72x10 ⁻⁸	1.45x10 ⁻²	1.94	5.75
15	7.0	6.06	5.83x10 ⁻⁷	2.78x10 ⁻⁸	1.56x10 ⁻²	1.93	5.60
16	7.5	6.08	5.57x10 ⁻⁷	2.91x10 ⁻⁸	1.66x10 ⁻²	1.93	5.46
17	8.0	6.11	5.19x10 ⁻⁷	3.12x10 ⁻⁸	1.77x10 ⁻²	1.92	5.21
18	8.5	6.12	5.08x10 ⁻⁷	3.20x10 ⁻⁸	1.88x10 ⁻²	1.92	4.62

The average dissociation constant, pKa = 5.40

Table 9: Determination of the Number of Coordinated Ligands in Cobalt(II) Complex

S/No.	Vol. of Sodium Liganate (cm ³)	pH	[H ⁺] x 10 ⁻⁴	[OH] x 10 ⁻¹¹	Log[A]	M _{tot} x 10 ⁻³	n
1.	4.2	2.36	2.92	5.55	-5.89	4.90	1.28
2	4.4	2.37	2.85	5.68	-5.87	4.89	1.34
3	4.6	2.38	2.73	5.81	-5.83	4.89	1.40
4	4.8	2.41	2.60	6.23	-5.78	4.88	1.45
5	5.0	2.43	2.49	6.52	-5.74	4.88	1.51
6	5.2	2.45	2.37	6.83	-5.70	4.87	1.55
7	5.4	2.47	2.27	7.15	-5.67	4.87	1.62
8	5.6	2.49	2.17	7.49	-5.63	4.86	1.68
9	5.8	2.51	2.07	7.84	-5.59	4.86	1.75
10	6.0	2.53	1.97	8.21	-5.56	4.85	1.81
11	6.2	2.53	1.97	8.21	-5.56	4.85	1.88
12	6.4	2.54	1.93	8.40	-5.55	4.84	1.96
13	6.6	2.54	1.93	8.40	-5.55	4.84	2.04
14	6.8	2.56	1.84	8,80	-5.51	4.84	2.11
15	7.0	2.57	1.80	9.01	-5.50	4.83	2.17
16	7.2	2.57	1.80	9.01	-5.50	4.83	2.25
17	7.4	2.58	1.76	9.22	-5.48	4.82	2.33
18	7.6	2.59	1.72	9.43	-5.47	4.82	2.39
19	7.8	2.59	1,72	9.43	-5.47	4.81	2.48
20	8.0	2.60	1.68	9.65	-5.45	4.81	2.55
21	8.2	2.61	1.64	9.83	-5.44	4.80	2.63
22	8.4	2.63	1.57	10.3	-5.41	4.80	2.68
23	8.6	2.65	1.50	10.8	-5.38	4.79	2.76
24	8.8	2.67	1.43	11.3	-5,35	4.79	2.83
25	9.0	2.68	1.40	11.6	-5,34	4.78	2.89
26	9.2	2.69	1.37	11.9	-5,33	4.78	2.88
27	9.4	2.70	1.34	12.1	-5.31	4.78	3.05
28	9.6	2.70	1.34	12.1	-5.32	4.77	3.12
29	9.8	2.70	1.34	12.1	-5.32	4.77	3.20
30	10	2.70	1.34	12.1	-5.32	4.76	3.27

The average number of ligands, n = 2.22

Table 10: Determination of the Number of Coordinated Ligands in Nickel(II) Complex

S/NO	Volume of Sodium Liganate (cm ³)	pH	[H ⁺] x 10 ⁻⁴	[OH] x 10 ⁻¹¹	Log[A]	M _{tot} x 10 ⁻³	n
1.	4.2	2.75	1.19	1.36	-5.23	4.90	0.92
2.	4.4	2.77	1.14	1.43	-5.21	4.89	0.99
3	4.6	2.77	1.14	1.43	-5.21	4.89	1.07
4	4.8	2.77	1.14	1.43	-5.21	4.88	1.15
5	5.0	2.79	1.09	1.49	-5.17	4.88	1.22
6	5.2	2.80	1.06	1.53	-5.17	4.87	1.29
7	5.4	2.81	1.04	1.56	-5.16	4.87	1.37
8	5.6	2.81	1.04	1.56	-5.16	4.86	1.46
9	5.8	2.82	1.01	1.60	-5.14	4.86	1.53
10	6.0	2.83	0.99	1.64	-5.13	4.85	1.61
11	6.2	2.82	1.01	1.60	-5.14	4.85	1.68
12	6.4	2.83	0.99	1.64	-5.13	4.84	1.74
13	6.6	2.84	0.97 ¹	1.68	-5.12	4.84	1.84
14	6.8	2.84	0.97	1.68	-5.12	4.84	1.93
15	7.0	2.84	0.97	1.68	-5.12	4.83	1.99
16	7.2	2.85	0.95	1.71	-5.11	4.83	2.07
17	7.4	2.86	0.92	1.76	-5.10	4.82	2.16
18	7.6	2.86	0.92	1.76	-5.10	4.82	2.22
19	7.8	2.87	0.90	1.80	-5.09	4.81	2.30
20	8.0	2.88	0.88	1.84	-5.07	4.81	2.38
21	8.2	2.87	0.90	1.80	-5.09	4.80	2.48
22	8.4	2.87	0.90	1.80	-5.09	4.80	2.54
23	8.6	2.88	0.88	1.84	-5.08	4.79	2.63
24	8.8	2.88	0.88	1.84	-5.08	4.79	2.71
25	9.0	2.89	0.86	1.88	-5.06	4.78	2.78
26	9.2	2.89	0.86	1.88	-5.06	4.78	2.86
27	9.4	2.90	0.84	1.93	-5.05	4.78	2.94
28	9.6	2.91	0.82	1.97	-5.04	4.77	3.00
29	9.8	2.91	0.82	1.97	-5.04	4.77	3.9
30	10	2.91	0.82	1.97	-5.04	4.76	3.16

The average number of ligand, n = 2.04

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