

## Synthesis and characterization of complexes of nickel (II) with N,N-AlkanllidenBissAmideligand

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**Abstract:** In this study the process of synthesis in some complexes of AlkanllidenBiss Amide ligand with transition metal of nickel was investigated. N ligands and N -AlkanllidenBiss Amide are made by the direct reaction of Aldehyde with type I amides. The structure of these complexes and ligands were identified by UV-Visb, FT-IR and H-NMR spectroscopy and some physical properties.

**Keywords:** AlkanllidenBiss Amide ligand, transition metal of nickel, synthesis

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### I. Introduction

Schiff bases, named after Hugo Schiff [1], are formed when any primary amine reacts with an aldehyde or a ketone under specific conditions. Structurally, a Schiff base (also known as imine or azomethine) is a nitrogen analogue of an aldehyde or ketone in which the carbonyl group (C=O) has been replaced by an imine or azomethine group. Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilizer's [2]. Schiff bases have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, anti-malarial, anti-proliferative, anti-inflammatory, antiviral, and antipyretic properties [2-5].

Amides are the most important components of biological compounds and in general they are useful for the synthesis of peptide amides (proteins). Alkane is an amide with a nitrogen atom and these compounds contain 2 absorption bands around 1700-1600  $\text{cm}^{-2}$  which is mainly related to *def, C<sup>110</sup> str* N-H, but due to the nature of pairing in them their vibration changes. It can be said that nickel is the only transition metal for which a biological role is not considered and it has many reasons, because nickel ions after complexation with the ligand in vivo show a considerable amount of light absorption, even spectroscopy of Mossbauer can't be used for nickel isotopes. Also, Nickel is just one of the components of complicated enzymes which often contain different coenzyme such as other minerals. N ligands and AlkanllidenBiss Amide will be obtained by the direct reaction of first type amides with Aldehyde, with ratios of one to two and in the presence of a strong acid as a catalyst and dichloromethane as a solvent.

### II. Methodology

Used materials: All solvents are used without purification. Materials such as Formamide, Benzamide, benzaldehyde, acetanilide, thioacetamide, acetamide, N , Bromosuccinimide, Tiubanzanamide, sulfonamide, Hydrochloric acid, nicotinamide dichloromethane, have been prepared by Merck company and were used without purification.

#### Used machine

Electronic spectra were recorded by Camspec UV-Visible spectrophotometer model Wpa bio Wave S2 100. The IR spectra were recorded using FT-IR Bruker Tensor 27 spectrometer. <sup>1</sup>H NMR and <sup>13</sup>C-NMR were recorded on a Bruker AVANCE DRX 500 spectrometer at 500 and 125 MHz respectively. All the chemical shifts are quoted in ppm using the high-frequency positive convention; <sup>1</sup>H spectra was referenced to external SiMe<sub>4</sub>. The percent composition of elements was obtained from the Microanalytical Laboratories, Department of Chemistry, OIRC, Tehran.

#### Ligand synthesis

##### Amino benzyl amine c

To prepare, 10 mmol of acetamide should be added into a balloon containing 5 mmol benzaldehyde and 10 ml dichloromethane and 10 ml of concentrated HCl. The reaction is refluxed for 24 hours, after cooling to room temperature a white deposit will be obtained and after washing it with toluene and recrystallization action with ligands in a ratio of 4 to 1 (Hot acetonitrile in water 4: 1), a good crystal will be obtained. Ligand's melting point is around 238-235°C. Ligands is in the form of soluble in water in solvents such as propanol, ethanol, methanol, dichloromethane and pure acetonitrile and they are strong due to hydrogen bonds and it is in the form of insoluble in solvents such as ether, hexane, benzene and petroleum.

IR( $\lambda_{\max}$ ,  $\text{cm}^{-1}$ ): 3279 (N-H), 1666 (C=O), 1381 (C=N), 690.87 (C-H)

$^1\text{H NMR}$  ( $\delta$  ppm  $\text{CDCl}_3$ , 300MHz): 7, 7.12 and 7.08 [3q, 2d, 5H, arom]; 8.15 [d, 1H, NH]; 6.13 [m, 1H, CH]; 3.23 [d, 2H,  $\text{CH}_2$ ].

### Benzoic acid amide Biss( $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_2$ )

The synthesis method is similar to the previous method and melting point of ligand is around 158 to 161 ° C. Benzoic acid amide Biss is in the form of soluble in water in solvents such as methanol, ethanol, propanol and hot dichloromethane, acetonitrile and chloroform and insoluble in solvents such as petroleum ether, benzene and hexane.

— IR( $\lambda_{\max}$ ,  $\text{cm}^{-1}$ ): 3283 (N-H), 1653 (C=O), 1494 (C=N), 701 (C-H)

$^1\text{H NMR}$  ( $\delta$  ppm  $\text{CDCl}_3$ , 300MHz): 7.21, 7.12 and 8.28 [3q, 2d, 5H, arom]; 9.1 [d, 1H, NH]; 6.08 [m, 1H, CH]; 4.03 [d, 2H,  $\text{CH}_2$ ].

### Synthesis of nickel (II) complex; overall approach

To amino benzyl amine ligands -2 and benzoic acid amide Biss metal salt  $\text{NiCl}_2$  will be added which is solved in ethanol with the ratio of 1:1, we should add ligand metal and the mixture is stirred until the precipitate formed, then it should be filtered by paper, we wash it with hexane then it will be dried.

### Synthesis of -2 amino-benzyl amine nickel II chloride complex( $\text{C}_{11}\text{H}_{14}\text{N}_2\text{O}_2\text{NiCl}_2$ )

Green complex was melted at temperature of 112-116.

— IR (KBr,  $\text{cm}^{-1}$ ): 3241 (N-H), 1674 (C=O), 1274 (C=N), 590 (C-H)

— UV- vis (MeCN):

$\lambda_{\text{C.T}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$	$\lambda_{\text{CT(LMCT)}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$	$\lambda_{\text{CT(LMCT)}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$
272	386	599
136	77	67

### Synthesis of (biss-benzoic acid amide) Nickel Chloride II complex( $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_2\text{NiCl}_2$ )

Milky yellow complex was melted around the temperature of 131-135 C. NMR ( $\text{CDCl}_3$ ,  $\text{Me}_4\text{Si}$ ) (ppm): 3.298; 2.787; 2.623; 2.474. IR (KBr,  $\text{cm}^{-1}$ ): 3413 (N-H), 1662 (C=O), 1393 (C=N), 665 (C-H), Ni-N (230-400). UV- vis (MeCN):

$\lambda_{\text{C.T}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$	$\lambda_{\text{CT(LMCT)}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$	$\lambda_{\text{CT(LMCT)}}$ $\epsilon, \text{M}^{-1}\text{CM}^{-1}$
281	400	600
800	40	57

## III. Discussion and conclusion

Nickle complexes with Biss-amide ligand are attached to the metal via the nitrogen, the similarity of the number and location of the stretching band C = N in the infrared spectrum and similarity in change of intensity and maximum wavelength in electronic spectrum refers to the fact that, the manner of coordination in ligand is similar in all of them. Types of nickel complex depending on their R sort, create a tetragonal structure, or something similar to tetragonal and flat square structure and it is the most stable form of energy for  $\text{T}_1(\text{F})\text{NiL}_2\text{X}_2$  complex. Often the charge transfer is from metal to the ligand and absorption bands for tetrahedron structures compared with octahedral symmetry mostly appears in lower energy parts and complex due to the lack of symmetry center, is stronger than octagon. (In analyzing the spectrum in the region below 1000, 3 transmissions can be seen and just two spectrums of them are visible in the visible region).

By considering IR spectrum of ligand and complex, it was determined that nickel wasn't attached to carbonyl group C = O by oxygen, because particular stretch strips were specified around the range of 16000-1 and for the spectrum of IR, (ligand and complexes) C = S stretch is weaker and is placed in non-spectrum area.

In complexes, UV-VIS shows three electron transport.

$$\begin{array}{lll}
 {}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_1(\text{p}) & \nu_3 = & 12000 - 17000 \text{cm}^{-1} \quad \epsilon = 100 - 200 \\
 {}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{A}_2(\text{F}) & \nu_2 & 6500 - 1000 \text{cm}^{-1} \quad \epsilon = 50 - 150 \\
 {}^3\text{T}_1(\text{f}) \rightarrow {}^3\text{T}_2(\text{F}) & \nu_1 & 3000 - 5000 \text{cm}^{-1} \quad \epsilon =
 \end{array}$$

V1 refers to the absorption of organic part in the molecule or the solvent absorption.

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