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SHORT COMMUNICATION

# CHARACTERISATION OF NICKEL(II) SCHIFF BASE COMPLEXES DERIVED FROM ACETYLACETONE OR SALICYLALDEHYDE WITH TRIPOD AMINE

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## ABSTRACT

Novel complexes of the type [Ni(TAA-sal)($H_2O$)]X (TAA-sal = Schiff base derived from tris(2-aminoethyl)amine – TAA and salicylaldehyde–sal, X= Cl) or [Ni(TAA-acac)($H_2O$)]$BPh_4$ (TAA-acac=Schiff base derived from TAA and acetylacetone-acac) have been prepared and studied by elemental analysis, IR and magnetic measurements. Physicochemical studies account for the deformed octahedral coordination mode.

**Keywords**: Schiff base, salicylaldehyde, amine.

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## INTRODUCTION

A number of hexamino cryptands have been prepared from aromatic aldehydes and tripod amines[1]. The cryptands containing transition metal ions are expected to show catalytic activity, which is one of the major goals in supramolecular chemistry. Some macrocyclic complexes are known to be prepared from acetylacetone and polyamines. In analogy with these known reactions, sato et al[2]. intended to prepare a new cryptand by condensation of acetylacetone with tris(2-aminoethyl)amine. Although every attempt did not afford the targent cryptand, two kinds of complexes have been prepared for various metal ions: one containing two tridentate ligands which have ketoimine moieties at all primary amino groups of TAA and the other has a pentadentate ligand having a ketoimine at one of the three amino groups of TAA. The $Sr^{2+}$, $Ca^{2+}$, $Mg^{2+}$ and $Mn^{2+}$ give the former type complexes and $Cu^{2+}$, $Co^{2+}$ and $Zn^{2+}$ give the latter. The aim of this work was to study the influence of nickel(II) on composition of such type of complexes.

## EXPERIMENTAL

All the chemicals are analar grade. C,H and N was analysed with the help of a perkin-Elmer model-540 CHN analyzer. IR spectra were recorded in KBr on a Jasco IR-700 spectrophotometer, UV-visible spectra were recorded on a UNIDEC-340 double beam spectrophotometer, conductivities were measured with a electrolytic resistance bridge. Magnetic susceptibilities were measured by Guoy method using Hg[Co$(SCN)_4$] as the calibrent.

### Preparation of [Ni(TAA-acac)($H_2O$)]$BPh_4$ (1)

Exactly 0.39g of methanolic(10ml) solution of $Ni(ClO_4)_2.6H_2O$ with 0.11ml of acetylacetone were added and after 5minutes 0.16ml of tris(2-aminoethyl)amine and 0.28ml of triethylamine was added. The pale brown solution was stirred at room temperature overnight. 0.34g of $NaBPh_4$ in methanol solution was added to this solution and the precipitate deposited after evaporation of a part of solvent was collected and washed with ethanol and ether. Yield ≈ 75%.

### Preparation of [Ni(TAA-sal)($H_2O$)]Cl(2)

Exactly 0.39g of methanolic(10ml) solution of $Ni(ClO_4)_2.6H_2O$ with 0.16ml of acetylacetone were added and after 5minutes 0.28ml of tris(2-aminoethyl)amine and 0.22ml of triethylamine was added. The brown solution was stirred at room temperature overnight and then left to stand for several days. After the solution was evaporated to dryness, the pulverized substance was obtained by stirring bin methylcyanide, then it was washed with ethanol and ether.Yield ≈ 70%.

## RESULTS AND DISCUSSION

From the observation of elemental analysis the complexes were formulated as Ni(SB)($H_2O$)X, Where SB = Schiff base and X = $BPh_4$ and Cl. The magnetic moments of these complexes are typical for octahedral Ni(II) complexes and their values are 3.38BM (1) and 3.09BM (2) respectively.
The electronic spectra shows[3] (Table-1) three distinct bands at 10,000cm$^{-1}$($3A_{2g}$< --- $3T_{2g}$), 18000 cm$^{-1}$($3A_{2g}$< --- $3T_{1g}$) and at 27000cm$^{-1}$($3A_{2g}$< --- $3T_{1g}$(P)) which are characteristic for octahedral Ni(II) coordination.
The IR spectra of all complexes show characteristic strong band corresponding to the vibration of C-N at ca 1585cm$^{-1}$. The sharp bands at 3290 – 3298cm$^{-1}$ are attributed to the N-H stretching vibration, the band at 1450cm$^{-1}$ is assigned to the $CH_2$-bending vibration and $NH_2$- twisting vibrations appear at 1040 cm$^{-1}$. The maxima of middle or strong intensity[4] at 1170 cm$^{-1}$ or 1186 cm$^{-1}$ respectively, may be NH-wagging vibrations. The band at 830 cm$^{-1}$ belong to[5] anion $NO_3^{-}$. The frequencies corresponding to acetylacetone vibrations are given in the Table-1

Table-1: Infrared and Electronic spectral data (cm$^{-1}$)

| Complexes | Electronic spectra | IR spectra |
|---|---|---|
| | Band maxima | Bands |
| [Ni(TAA-acac)($H_2O$)]$BPh_4$ | 10600,12300,18000, CT | 3288s,1592s,<br>1444s,1290m,<br>1260m,1196m |
| [Ni(TAA-sal)($H_2O$)]Cl | 11600,12500,18000,<br>26800 | 3280s,1652s,<br>1596s,1444s,<br>1335s, 1290s |

The ionic character of $NO_3$ group was confirmed by measurement of the molar conductivity. The value of 90.85 S.cm$^2$.mol$^{-1}$ obtained for [Ni(TAA-acac)($H_2O$)]$BPh_4$ in 5.10M$^{-4}$
Solution in methanol is typical[6] for 1:1 electrolyte. Analogical, the value of 87.7 S.cm$^2$.mol$^{-1}$ for [Ni(TAA-sal)($H_2O$)]Cl is characteristic for 1:1 electrolyte.

Consequently, based on the composition of these complexes and on the results of physico-chemical methods. We can concluded that these compounds are octahedral nickel(II) complexes with Schiff bases obtained by template reaction from (1:1) tris(aminoethyl)amine and salicylaldehyde or acetylacetone.

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