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Synthesis of Mn(II) and Fe(II) Complexes with Ethylenediamine and Acetylacetonate Ligands

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Abstract
Synthesis of acetylacetonato, ethylenediamine manganese (II) complex and acetylacetonato, ethylenediamine iron (II) complex were carried out, a pale yellow and brown solids were obtained from the reaction. The solubility tests of the complexes were carried out, in which manganese (II) complex was found to be soluble in water, methanol and ethanol and insoluble in chloroform and petroleum ether while the iron (II) complex was found to be slightly soluble in water, methanol and ethanol and insoluble in chloroform and petroleum ether. The UV visible spectra of manganese (II) complex showed absorption maximum at around 480nm, and infrared spectra of manganese (II) complex showed absorption band at 1725cm^{-1}.
Keywords: Acetylacetone, manganese, iron, ethylenediamine.

1. INTRODUCTION

Selection of ligand is a critical consideration in many practical areas, including bioinorganic and medicinal chemistry, homogeneous catalyst and environmental chemistry. Sender and Marsh (2011)

Acetylacetone is an inorganic compound that famously exists in two tautomeric forms that rapidly interconvert. The less stable tautomer is a diketone named pentan-2,4-dione. Acetylacetone is prepared by rearrangement of isopropyl acetate. Sender and Marsh (2011) substituted effect of keto-enol equilibra using NMR spectra.

Ethylenediamine is prepared by the reaction of 1,2-dichloroethene and ammonia under pressure at 180^{0}c in an aqueous media, Hanju Arfer (2007). It has been known that different metals ion on interaction with polydentate ligands yields chelates, for example; Tsumaki (1983) reported [Co(sal2-en)] complex which receive a great attention owing to its ability to undergo reverssible adduct formation with molecular oxygen.

Nishikawa and Nakamura reports the redox reaction between tris (acetylacetonato) manganese (III) and several aliphatic amine and also on the characterization of manganese (II) complexes produced. In the infrared spectra of these complexes, the metal oxygen vibrational mode was found at 650cm^{-1} and the three low frequency vibrational mode of the acetylacetonate anion was found at 543cm^{-1} , 449cm^{-1} and 399cm^{-1}. Dismuske, Jones and Bailar (1961)

Dwyer and Sargeson synthesized bis (acetytlacetonato) diamminemanganese (II) and similar adduct of 2,2-bipyridine and 1,10-phenanthroline either by substitution reaction between bases and sodium tris (acetylacetonato) manganese (II) complex or by direct addition reaction, Dwyer and Sargeson (1956).

In another report, Mosoarca synthesized mononuclear complex of iron (III) with N,N-tetra (4-antipyryl-methly) ethylenediamine in which two complex compounds are obtained as binuclear and mononuclear iron(III) complex of N,N- tetra(4-antipyryl-methyl)-1,2-diaminoethane and ethylenediamine, Costisor, *et,al;(*1994). The complex was found to be octahedral and the IR spectra of the complex shows the band at 1556cm^{-1} is assigned to a combination of (C=O) and (C=N) stretching mode. The Fe-N vibration was identified at 412cm^{-1} for iron (III) complex, Lever inorganic Electronic Spectroscopy (1968)

Efforts have been made to synthesize manganese (II) and iron (II) complexes with acetylacetone and ethylenediamine ligand due to their application as bioactive molecule. Robert, *et,al;* also synthesized chromium (III) ion complex using acetylacetonate ligand, William (2001). Lowk synthesized copper acetylacetonate compound used spectroscopic analysis to identify the complex

Transition metal complexes of polydentate ligands are used in various fields, such as medicine, agriculture, industries e.t.c. for example [Co (salen)] in dimethylformide, pyridine and substituted pyridine proved to be involved in oxygen metabolism, Hanna and Mona (2001).

2. MATERIALS AND METHODS

2.1 Materials

In preparation of reagents, chemicals of analytical grade purity were used. All weighing are observed on electrical meter balance. Melting point, decomposition temperature was determined on melting point apparatus. IR spectral measurements were recorded using Fourier Transformed Infrared. UV – Vis spectra were recorded using UV – Vis spectrophotometer.

2.2 Synthesis of [Mn (acac)(en) Cl_2]

To 25ml of methanolic solution $MnCl_2.4H_2O$ (0.0042mm), acetylacetone (0.0042mm) and ethylenediamine (0.0042mm) were added respectively and stirred. To the resulting pale yellow solution three drops of aqueous sodium hydroxide (NaOH) solution were added while heating and stirring continued on hot plate with magnetic stirrer for 30minutes. The resulting yellow solution was allowed to evaporate.

2.3 Synthesis of [Fe(acac)(en) Cl_2]

To 25ml of methanolic solution of $FeCl_2.4H_2O$ (0.0042mm), acetylacetone (0.0042mm) and ethylenediamine (0.042mm) were added respectively and stirred. To the resulting brown solution three drops of aqueous sodium hydroxide (NaOH) solution were added while heating and stirring were continued on hot plate with magnetic stirrer for 30minutes. The resulting brown solution was removed and air dried.

2.4 Solubility Tests

The solubility tests were carried out in chloroform, ethanol, methanol, water and petroleum ether. The tests was conducted by dissolving 0.1g of each complex in each of the test tube containing solvent, the results obtained are shown in table 2.

2.5 Uv Spectroscopy of [Mn(acac)(en)]Cl_2 and [Fe(acac)(en)Cl_2] complexes

Millimolar solution of [Mn(acac)(en)Cl_2] and [Fe(acac)(en)Cl_2] complexes were prepared by dissolving 0.0895g each of the complexes in 250cm^3 volumetric flask and filled with water up to the mark. The analysis was carried out at the wavelength range of (320-500cm^{-1}). The results obtained are shown in table 3.

3. RESULTS AND DISCUSSION

Our effort to prepare [Mn(acac)(en)Cl_2] complex was successful as this was investigated through the use of infrared spectroscopy, Uv-vis spectra and other techniques, but for the [Fe(acac)(en)Cl_2] complex was unsuccessful.

Manganese complex formed yellow solution which is hygroscopic. Iron complex gave a brown crystalline solid with a relatively high decomposition temperature. Mn (II) complex prepared were soluble in ethanol, methanol and water but insoluble in chloroform and petroleum ether and Fe (II) complex were slightly

Table1. Color and melting point of the complexes

Complex	Color	Melting point
[Mn(acac)(en)Cl_2]	Yellow	170^{o}c
[Fe(acac)(en)Cl_2]	Brown	Hygroscopic

Table2. Solubility tests

Complex	Ethanol	Methanol	H_2O	Chloroform	Petroleum ether
[Mn(acac)(en)Cl_2]	S	S	S	IS	IS
[Fe(acac)(en)Cl_2]	SS	SS	SS	IS	IS

S=soluble SS=slightly soluble IS=insoluble

The IR spectra of Mn (II) complex shows absorption band at (1725-1700cm^{-1}) with greater intensity indicating the presence of carbonyl (C=O) group in acetylacetone.

The infrared spectra of manganese ethylenediamine, the band at 4587.11cm^{-1} and 3270.42- 3060.17cm^{-1} shows the absorption of (Mn-N) and (N-H) stretching vibration respectively, confirming the coordination of ethylenediamine to the manganese metal ion. Byeong – Goo et al (1996). Therefore [Mn(acac)(en)Cl_2] was formed.

But for the [Fe(acac)(en)]Cl_2 complex, the carbonyl absorption were not found and in the iron ethylenediamine metal complex, the band at 458.11cm^{-1} indicating the presence of (Fe-N) stretching the (N-H) band at absorption of 2962.76cm^{-1} does not correspond to what is expected within the range.

Also in the Uv-vis spectra of the complexes, the analysis shows that as the wavelength increases the absorption decreases, therefore indicating the formation of the complex.

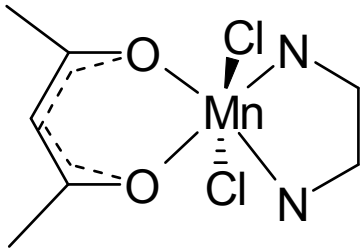

Fig. 1 Depicted structure of the prepared [Mn(acac)(en)Cl_2]

CONCLUSION

In conclusion this paper reports the synthesis of [Mn(II) and Fe(II) acetylacetonato ethylenediamine complexes. The complexes were found to be in conformity with the previous literature.

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