CHAPTER-7

SUMMARY AND CONCLUSION AND RECOMMENDATIONS

Metal ions are using in different field's, because analyzing the metal ions at minute level is very important. Absorbance photometry plays an important role than other available analytical methods because of simplicity and elegance. As Ultra Violet–Visible spectrophotometry is within the research of many analytical laboratories and is proved to the simple and versatile technique.

One or more than one functional group containing organic reagents are used for the metal analysis in different areas. According to the literature survey to the determination of metal ions hydrazone seem to be very useful. In this context, by the condensation reaction the author has developed new reagent such as α-Amyl Cinnamaldehyde Isonicotinoyl Hydrazone (ACINH) was synthesized. By using IR, ^{1}H-NMR and Mass spectroscopy techniques, the reagent is characterized. The data supports the synthesis and structures of ligands. The metal ions forms colour reactions with ligands. Hence for the determination of metal ions by spectrophotometric these ligands were used as good analytical reagents in different samples. By employing developed spectrophotometric methods the colour reactions of metal ions were studied in detail in aqueous medium. The following factors such as amount of reagent required for full colour development, order of addition of reagents, effect of foreign ions, absorbance maximum (λ_{max}), Beer's law verification, molar absorptivity (ε), composition and stability constant of the complex, optimum concentration range for accurate determination of metal ions (Ringbom's plots), time stability have been studied. Various analytical and Physico-chemical characteristics of Hg (II) and Cd (II) complexes of ACINH were summarized in Table 7.1.

7.1 Determination of wavelength maximum

For various metal ions absorbance maximum were studied in aqueous solutions. It is observed that the absorbance maxima of Hg (II)-ACINH complex shows maximum wavelength at (λ_{max}=391nm) and Cd (II)-ACINH complex shows minimum wavelength at (λ_{max}=380nm).

7.2 Study of Optimum pH

For the development of constant colour of the metal complexes pH of the buffer solutions played an important role. At pH 10.0 Hg (II) show constant colour development with ACINH and Cd (II) show constant colour development with ACINH at pH 9.0.

7.3 Study of Amount of reagent necessary for full colour development

For the full colour development of Hg (II) and Cd (II) with ACINH, ten-fold molar excess of reagent is required.

7.4 Study of Order of addition of reagent concentration

Order of addition of metal ion solution or buffer solution or reagent solution has no adverse effect on the absorbance of the species in the present investigation.

7.5 Study of Time stability

The colour development between reagent solution and metal ions is instantaneous and no incubation period or heating is necessary. The metal complexes are stable for more than five hours, to record absorbance of Hg (II) and Cd (II) metal complexes this time is sufficient.

7.6 Study of Extinction coefficient

All the metal complexes obeyed Beer's law over a broad range of concentrations. Cd(II) with ACINH shows high molar absorptivity ($\varepsilon=2.94x10^4$ $L.mol^{-1}.cm^{-1}$) where as Hg(II)-ACINH shows minimum molar absorptivity ($\varepsilon=2.01x10^4 L.mol^{-1}.cm^{-1}$). Hence the reagent is more sensitive with Cd(II).

7.7 Study of Optimum concentration range

By the data obtained in Beer's law verification and Ringbo's plots the quantity range for the accurate estimation of metal ions was determined. The values are presented in Table 7.1.

7.8 Study of Effect of various anions and cations

The effect of various foreign ions (anions and cations) has been examined and incorporated in chapter 4.0.

7.9 Study of Composition and stability constant of the complex

In this present investigation the reagent ACINH forms 1:1 complexes with Hg(II) and Cd(II). By using the Job's method data stability constant of the complexes were calculated. Among the two complexes Cd(II)-ACINH was more stable ($\beta=8.58x10^7$) and Hg(II)-ACINH less stable ($\beta=6.45x10^5$).

7.10 Conclusion and Recommendations

For the estimation of metal ions by spectrophotometry has proved to be a powerful analytical technique in different samples. The main aim of the study was for the estimation of selected metal ions in the given samples to develop simple, sensitive, specific and time effective methods. For this, it is necessary to have scientific and analytical skill to synthesis reagent, preparation and separate complexes using analytical tools. For the preparation of new organic reagent development and validation of analytical methodology work was describes in this thesis, preparation of new complexes with the metal ions of our interest and the determination of metal ions in their trace levels. The developed methods are reliable, rapid, simple and validated. For the estimation of metal ions in various samples these methods are useful. A detailed account of analytical and Physico-chemical properties of metal complexes employing ACINH was presented in Table 7.1.

Hydrazones are very important chromogenic reagents for the determination of metal ions spectrophotometricaly in microgram levels than the other reagents. The literature survey reveals that α- Amyl Cinnamaldehyde Isonicotinoyl Hydrazone (ACINH) is not exploited for determination of metal ions by the spectrophotometricaly.

Based on the lacuna identified in the literature the new hydrazone α-Amyl Cinnamaldehyde Isonicotinoyl Hydrazone (ACINH) was synthesised and characterised by using the spectral data such as Infrared, proton NMR and Mass spectras. The reagent with different metal ions gives intense colours. Hence, ACINH is potential analytical reagent for the determination of metal ions by spectrophotometric method.

The colour reactions are instantaneous and methods do not involve heating of the reaction mixture or pre-extraction of the components. The reagent with metal ions in buffer medium gave colour reactions. Tolerance limit values for certain metal ions are small. For the determination of Mercury (II) and Cadmium (II) metal ions derivative spectrophotometric technique is employed.

The methods are applied for the determination of metal ions in appropriate samples. In the analysis obtained results are satisfactory. The present methods are sensitive, selective, simple, reasonably and rapid without need of extraction or heating. The reagent is very easy to synthesize and purify with available chemicals. The synthesised reagent is water soluble which facilitate the determination of metal ions in aqueous medium.

Table 7.1: A detailed account of Physico-chemical and analytical properties of metal complexes using ACINH

CHARACTERISTICS	**Hg(II) - ACINH**	**Cd(II) – ACINH**
Maximum Absorbance (Zero order)(λ_{max})	391	380
Optimum pH range	10.0	9.0
Colour of the Complex	Yellow	Yellow
First order derivative(λ_{max})	440	435
Molar absorptivity (ε) $l.mol^{-1}.cm^{-1}$	2.01×10^4	2.94×10^4
Sandell's sensitivity ($\mu g.cm^{-2}$)	0.0099	0.0034
Beer's law validity range (µg/ml)	1.0029-10.029	0.5031-5.0531
Optimum concentration range (µg/ml)	2.009-10.045	0.482-4.9254
Composition (M:L)	1: 1	1: 1
Stability constant (β)	6.45×10^5	8.58×10^7
R.S.D (%)	0.02	0.1