Development Of Extractive Spectrophotometric Determination Of Iridium (III) Using Schiffs base As An Analytical Reagent

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Abstract: Using analytical reagent N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP) a sensitive, simple & rapid spectrophotometric method has been developed for the determination of Ir (III). Elemental analysis was used to characterize synthesized ligand NOMBAP. Ir (III) is extracted quantitatively (99.56%) by NOMBAP into n-amyl alcohol from an acidic aqueous solution having pH range 4.5 – 6.7. An intense peak at 460 nm (λ max) was observed in the extract of n-amyl alcohol. Beer's law is obeyed over the concentration range 1.0 – 14.0 µg/ml for Ir (III). The molar absorptivity and Sandell's sensitivity for Ir - NOMBAP system is 69930 dm^3 mole^{-1} cm^{-1} and 0.00125 µgcm^{-2} respectively. The extracted species gives 1:3 (Ir-NOMBAP) composition suggested by Mole Ratio & Job's Continuous Variation and Method. Study has been done for interference by various ions. For determination of Ir (III) in alloy the proposed method has been used.

Keywords: [N (O - Methoxy Benzaldehyde) 2 – Aminophenol] (NOMBAP), Extractive Spectrophotometry, Iridium (III), Alloy.

I. INTRODUCTION

Iridium is a d-block element in the periodic table, discovered by 'Smithson Tennant'. It is symbolized as 'Ir' with an atomic number 77. It is a transition element which appears to be silvery white, brittle and very hard in nature thereby exists as a metal. Iridium is the second densest element in the periodic table with a density of 22.56 g/cm³ .

Various reagents [1-9] are used as tools for the Spectrophotometric determination of Iridium. Solvent Extraction also known as Liquid –Liquid Extraction is one of the important method of separation techniques. The separation of metal complexes is feasible through this method. The dissolution of the required component in the suitable phase which can be easily separated helps extensively in separation. The Extraction could be done efficiently with high quantitative efficiency.

[N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP) acts as a tool for the determination of iron [10] and Cu(II) [11[and Nickel (II) [12].by using the extractive spectrophotometric methods . In the present communication, we describe the extractive spectrophotometric determination of Ir (III) with [N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP).

II. EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurements. An ELICO LI-127 pH meter was employed for pH measurements.

A General procedure for synthesis of N-(o-methoxy benzaldehyde) 2-aminophenol (NOMBAP):

O-methoxy benzaldehyde and 2-aminophenol in ratio 1:1 ratio in ethanolic solution were refluxed for 4 hours, as shown in [Fig.1.] The reaction mixture was cooled to separate out sharp yellow crystal product (yield 78%, M.P.870-880C) of reagent NOMBAP was filtered & recrystallized by using aqueous ethanol as per reported procedure given in Vogel [13]
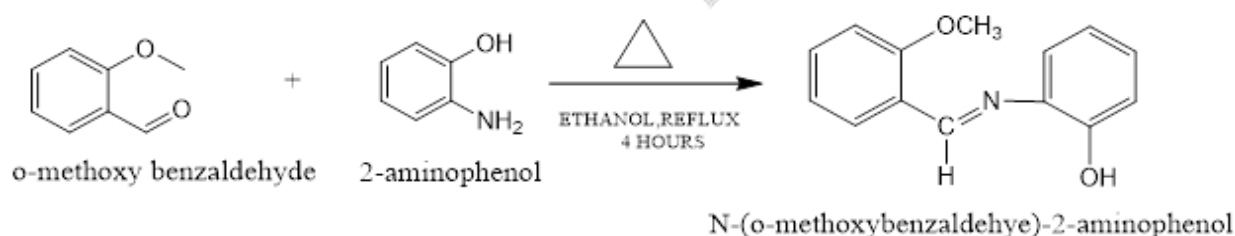

FIG. 1. SYNTHESIS OF SCHIFFS BASE

B. Green synthesis of N-(o-methoxy benzaldehyde) 2-aminophenol

In a beaker, ingredients (0.005 moles of o-methoxy benzaldehyde & 0.005 moles of 2-aminophenol) were mixed and few drops of pure alcohol was added. It was then irradiated in the microwave oven at 180^0 for 2 minutes. The reaction was completed in a short time (1.4 min) with higher yields. (NOMBAP) was characterized by elemental analysis [Table-1]

Table-I: The Analytical and Physical data of ligand

Compound (Colour)	Molecular Weight	Reaction period & %yield Conventional methods	Reaction period & % Yield Micro synthesis	M.P.	% Elemental Analysis Found (Calculated)		
					C	H	N
Ligand NOMBAP	227.28	5 hours 78%	1.4 Minutes 92%	88^0C	73.12 (73.91)	5.14 (5.719)	6.41 (6.159)

C. Preparation of stock solution:

A stock solution of Ir (III) was prepared by dissolving appropriate quantity of A.R. grade Iridium chloride supplied by E. Merck Co. Ltd. in water containing dilute hydrochloric acid to get 10 mg per cm^3. It was standardized gravimetrically [14] Working solutions of Ir (III) were made by suitable dilutions. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

D. Extractive Spectrophotometric Determination of Ir (III):

1.0 cm^3 of 1% NOMBAP prepared in DMF and 3.0 cm^3 of pH 6.5 (sodium acetate-acetic acid buffer) solution was added to an aqueous solution containing 10.0 to 140.0 µg of Ir (III). The resulting solution was kept in boiling water bath for a period of 30 mins for digestion. Using distilled water, the volume of the solution was made up to 10.0 cm^3 after cooling. By equilibrating the solution with 10.0 cm^3 of n-amyl alcohol for 1 min the phases were separated. The extract of n-amyl alcohol was measured at 460 nm against a reagent blank (made under identical conditions). With the help of calibration curve the content of Iridium in sample solution was determined. To study the effects of the other ions, foreign ions were added to the aqueous phase before the extraction and pH was adjusted accordingly.

E. Determination of Ir (III) in synthetic sample

By mixing 9.0 cm^3 of 1000 ppm Platinum (IV) and 1.0 cm^3 of 1000 ppm solution of Iridium (III). Synthetic sample of Iridium and Platinum was prepared. Using Distilled water the resulting solution was diluted up to 100 cm^3. According to the procedure described earlier Ir (III) was analysed using an aliquot (1ml) of this solution.

III. RESULTS AND DISCUSSION

NOMBAP extraction Ir(III) quantitatively (99.56%) by into n-amyl alcohol from an aqueous solution of pH 4.5 to 6.7 On the basis of the Extraction Coefficient value the solvents can be arranged for extraction of Ir (III) as n-amyl alcohol > n-butyl alcohol > ethyl acetate > carbon tetrachloride > Chloroform > chlorobenzene > bromobenzene > benzene > xylene. Hence in this project n-amyl alcohol was used. [Fig.2]
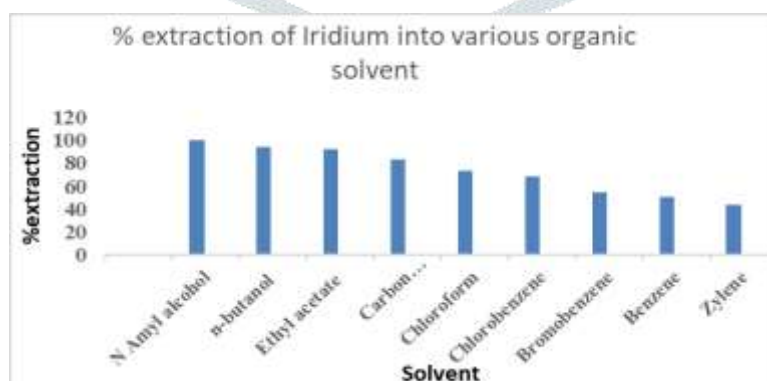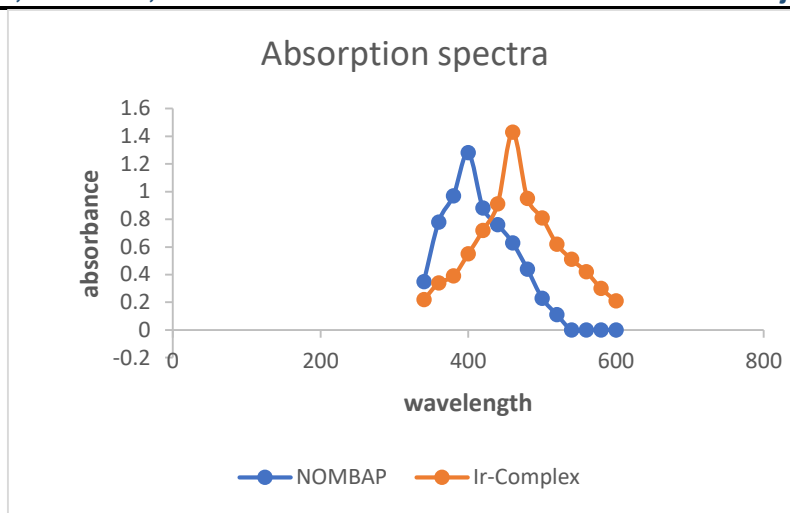

FIG. 2. PERCENTAGE EXTRACTION OF IR (III)

The n-amyl alcohol extract of Ir-NOMBAP complex depicts an intense peak at 460 nm [Fig.3.] The ligand shows negligible absorbance at this wavelength, so the entire extraction was done at this wavelength.

FIG. 3. ABSORPTION SPECTRA

Beer's law is obeyed at 460nm over a Ir (III) concentration range of 1.0-14.0 µg/ cm3 [Fig-4].
Molar absorptivity of the extracted complex was calculated to be 69930 dm3 mole-1cm-1. It was found that to extract 60 µg of Ir (III), 1.0 cm^3 of 1% solution of NOMBAP prepared in DMF was sufficient. At room temperature and for at least 24 hrs the colour of n-amyl alcohol extract remained stable.
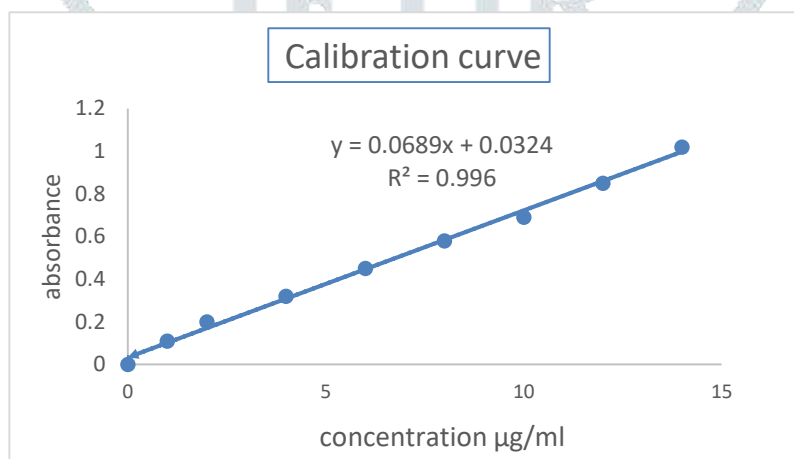
FIG.4. CALIBRATION CURVE FOR IR (III)

A. *Effect of other ions*

In spectrophotometric estimation of Ir(III) (40µg) ions given below did not interfere:

- 10 mg each of Cd(II), Li(I),Ba (II), Ca(II),Mg(II), Zn(II) and Sr(II)
- 0.1 mg each of Rh(III), Pt(IV), Os(IV), Pd(II), Cr(III) and Ni(II)
- 20 mg each of - citrate, chloride, bromide, nitrate, thiocyanate, and acetate.

Interference due to iodide and thiosulphate was removed by boiling solution with concentrated HNO$_3$ before the adjustment of pH. were removed by using. Appropriate masking agents were used to remove the interference by the various ions [Table II]

Table II: Interference ions and masking agent

Sr.No	Amount of interfering ions in mg	1ml of 0.5 M masking agent
1	10 mg of Ru(III)	Thiourea
2	10 mg of Cu (II)	EDTA or Sodium dihydrogen phosphate
3	10 mg of Fe (II) & Fe (III)	Tri ethanol amine
4	10 mg of Mn (II)	Sodium fluoride

B. Composition of the Extracted Complex

Job's continuous variation [Fig.5] & Mole ratio method. [Fig. 6] suggest the composition of the extracted complex to be 1:3 (Ir: NOMBAP)
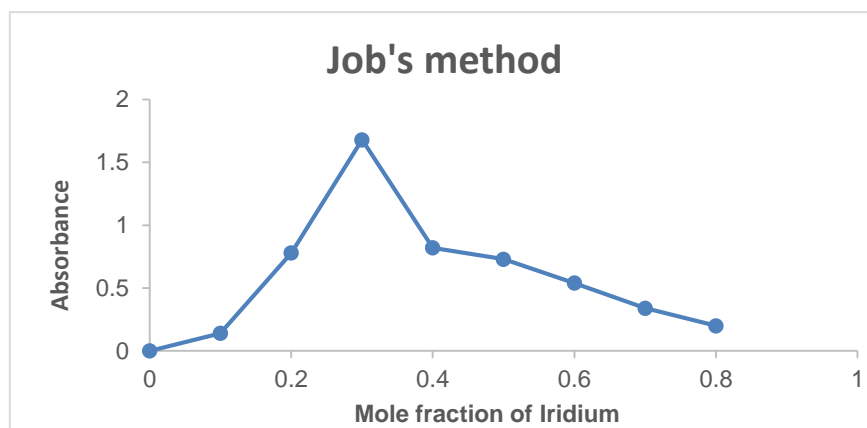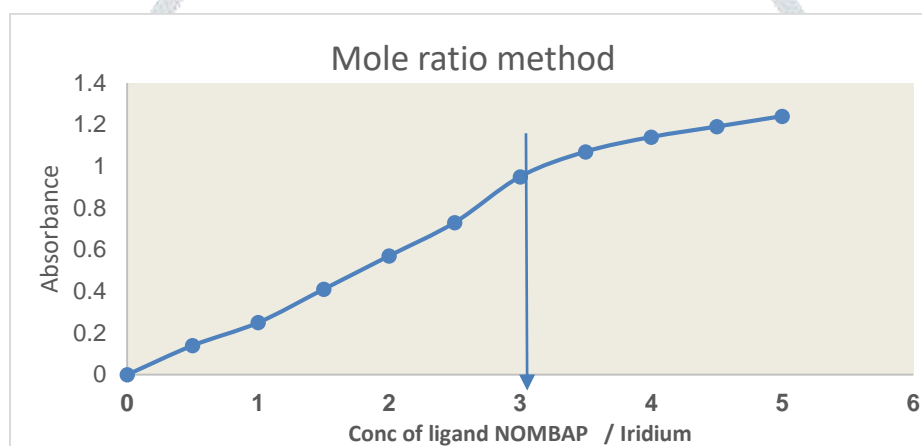

FIG.5. JOBS METHOD

FIG.6. MOLE RATIO METHOD

C. Precision, Accuracy, Sensitivity and Applications of Method

The average of 10 determinations of 40 µg of Ir (III) in 10 cm^3 solutions was 40.25µg, which is varied between 40.6358 and 39.8642 at 95% confidence limit. Sandell's sensitivity of the extracted species is found to be 0.00125µgcm^{-2} respectively. The proposed method has been used for the determination of Ir (III) in synthetic sample. The results obtained are comparable with reported values. [15] [Table III]

Table III: Determination of Iridium (III) in synthetic sample

Sample	Platinum- Iridium mixture
Ir(III) found by present Method(µg)*	9.992
Reported value(µg)	10.0

*The average of three determinations

CONCLUSION

NOMBAP extracts Ir(III) into n amyl alcohol successfully. This method is feasible, easy and efficient, the method was applied for the analysis of synthetic sample to determine the Iridium content. The outcomes of the method were comparable to the reported value.

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