IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
Volume 6, Issue 2, June 2021
Impact Factor: 4.819

Development of Extractive Spectrophotometric Determination of Rhodium (III) using Schiffs Base as An Analytical Reagent

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Abstract: *Rh (III) was determined by simple, sensitive & feasible spectrophotometric method by using analytical reagent N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP) . Elemental analysis was used to characterize synthesized Schiff's base NOMBAP. Rh (III) is extracted quantitatively (99.66%) by NOMBAP into carbon tetra chloride from an aqueous solution having pH range 5.1 – 5.9. An intense peak at 480 nm (λ max) was observed in the extract of n-amyl alcohol. Beer's law is obeyed over the concentration range 1.0 – 10.0 µg/ml for Rh (III). The molar absorptivity and Sandel's sensitivity for Rh - NOMBAP system is 8248.23 dm^3 $mole^{-1}cm^{-1}$and 0.08332 μgcm^{-2} respectively. Mole Ratio & Job's Continuous Variation and Method show that the extracted species has composition 1:3 (Rh-NOMBAP). Study has been done for interference by various ions. For determination of Rh (III) in alloy the proposed method has been used.*

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

I. INTRODUCTION

Rhodium is silvery white, hard, corrosion-resistant, and chemically inert transitionmetal having atomic number 45. Rhodium being the best hardeners for is alloyed with palladium or platinum to make electrical contacts with extreme wear resistance. Solvent Extraction also popular as Liquid –Liquid Extraction is one of the successful method of separation techniques for the separation of metal ion at trace level. Solvent extraction as a separation technique coupled with spectrophotometrically plays key role in industry. Various reagents [1-9] are used as tools for the Spectrophotometric determination of Rhodium. [N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP) has been successfully used for the determination of Fe(II)[10] , Cu(II) [11] and Ni (II)[12] at the trace level by extractive spectrophotometric methods . This communication speaks about the extractive spectrophotometric determination of Rh (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.

2.1 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]

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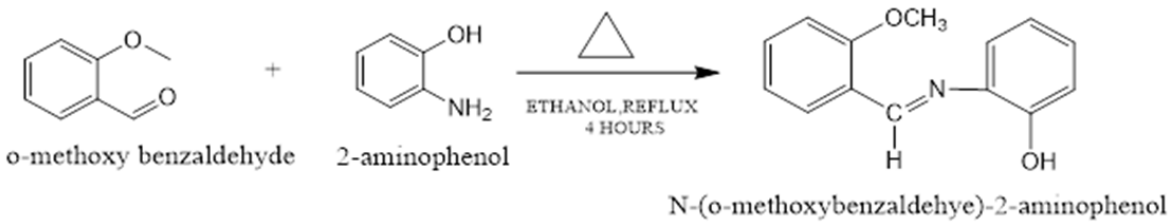

Figure 1: Synthesis of Schiffs Base

2.2 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 1: 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)

2.3 Preparation of Stock Solution

A stock solution of Rh (III) was prepared by dissolving accurately weighed rhodium chloride supplied by E. Merck Co. Ltd. in concentrated hydrochloric acid on the boiling water bath then diluted with doubly distilled water. It was standardized gravimetrically[14] An appropriate dilution gives the working solution of Rh (III). All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

2.4 Extractive Spectrophotometric Determination of Rh (III)

2.0 cm^3 of 2% NOMBAP prepared in DMF and 3.0 cm^3 of pH 5.5 (sodium acetate-acetic acid buffer) solution of was added to an aqueous solution containing 10.0 to 100.0 µg of Rh (III). followed by digestion on boiling water bath for 20 - 25 minutes. Using Distilled water, the volume of the solution was made up to 10.0 cm^3. By equilibrating the solution with 10.0 cm^3 of carbon tetrachloride for 1 min the phases were separated. The extract of carbon tetrachloride was measured at 480 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 studythe effects of the other ions, foreign ions were added to the aqueous phase before the extraction and pH was adjusted accordingly.

2.5 Determination of Rh(III) in Synthetic Sample

0.1 gm synthetic sample of Rh (III) was dissolved in boiling with 10 ml of aqua regia. The resulting solution was evaporated to dryness and the residue was then dissolved in 10 ml of 1N HCl filtered, diluted to 100 ml . The working solution was prepared by appropriate dilution of stock solution. According to the procedure described earlier Rh(III) was analysed using an aliquot (1ml) of this solution

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III. Results And Discussion

NOMBAP extraction Rh (III) quantitatively (99.66%) by into carbon tetra chloride from an aqueous solution of pH 5.1 to 5.9. The solvent carbon tetra chloride having highest extraction coefficient was used for the entire extraction process. As shown in [Table-II]

Table 2: The Percentage of extraction coefficient of solvent

Sr. No	Solvent	Percentage of extraction coefficient
1	Carbon tetrachloride	99.66
2	Chloroform	93.89
3	Chlorobenzene	90.21
4	N Amyl alcohol	87.45
5	N-butanol	76.32
6	Ethyl acetate	65.56
7	Chlorobenzene	59.25
8	Benzene	53.67

An intense peak at 480 nm is exhibited by the carbon tetrachloride extract of Rh-NOMBAP [Fig 2] complex. The Schiff's base shows negligible absorbance at this wavelength, so the entire experiment was done at this wavelength.

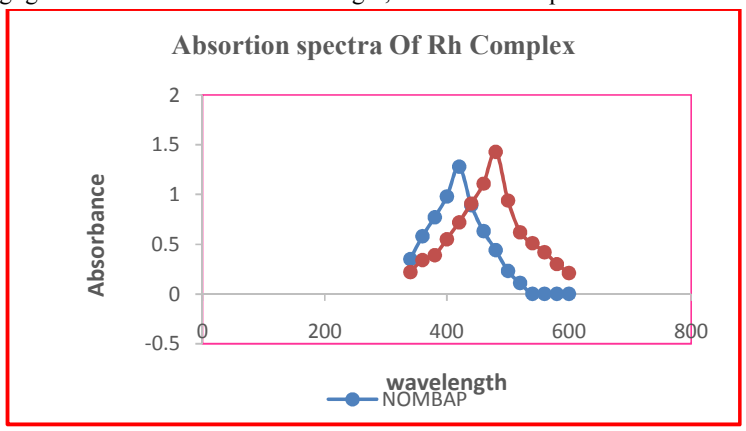

Figure 2: Absorption Spectra

Beer's law is obeyed at 480 nm over a Rh (III) concentration range of1.0-10.0 µg/cm^3 [Fig-3]. Molar absorptivity of the extracted complex was calculated to be 8248.23 dm^3 mole^{-1}cm^{-1}. It was found that to extract 40 µg of Rh (III), 1.0 cm^3 of 2% solution of NOMBAP prepared in DMF was sufficient. At room temperature and for at least 18 hrs the color of carbon tetrachloride extract remained stable.

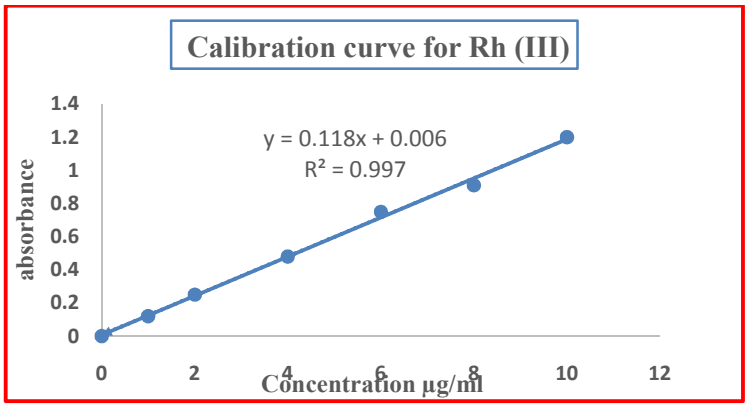

Figure 3: Calibration Curve for Ir (III)

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3.1 Effect of Other Ions

Inspectrophotometric estimation of Rh(III) (50µg) ions the
10 mg each of Cd(II), Li(I),Ba (II), Ca(II),Mg(II), Zn(II) and Sr(II)
0.1 mg each of Pt(IV), Os(IV), Pd(II), Cr(III) and Ni(II)
20 mg each of - citrate, chloride, bromide, nitrate, thiocyanate and acetate. did not interfere
In the presence of Thiourea, Ru (III) do not interfere.
Interference due to the Cu (II) could be masked by adding EDTA or Sodium dihydrogen phosphate Interference due to the Fe (II) & Fe(III) could be masked by adding Triethanol amine.

3.2 Composition of the Extracted Complex

Job's continuous variation [Fig.4] & Mole ratio method. [Fig. 65] suggest the composition of the extracted complex to be 1:3 (Rh : NOMBAP)

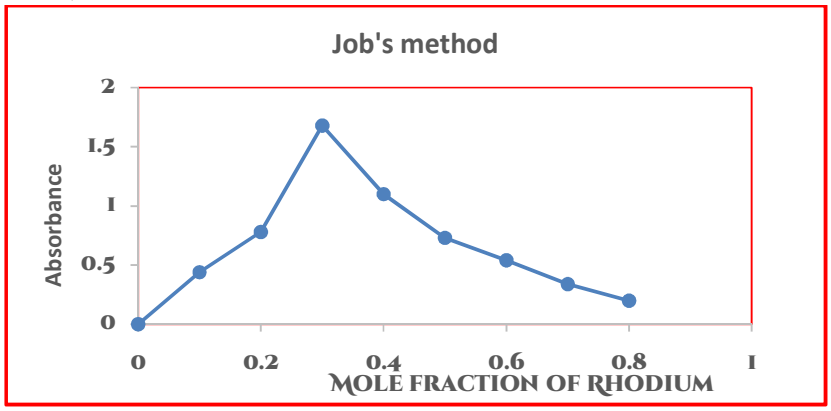

Figure 4: Job's method

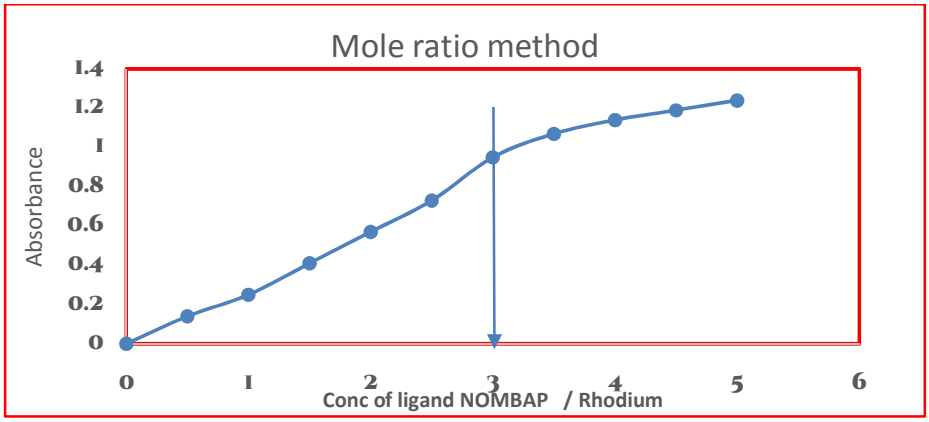

Figure 6: Mole Ratio Method

3.3 Precision, Accuracy, Sensitivity and Applications of Method

The average of 10 determination of 40 µg of Rh (III) in 10 cm^3 solutions was 40.19 µg, which is varied between 40.7358 and 39.8542 at 95% confidence limit. Sandell's sensitivity of the extracted species is found to be 0.08332 μgcm^{-2} respectively. The proposed method has been used for the determination of Rh (III) in synthetic sample. The result obtained are comparable with standard method [15] [Table III].

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Table 3: Determination of Iridium (III) in synthetic sample

Sample	Platinum- Iridium mixture
Rh (III) found by present Method(µg)*	9.986
Reported value(µg)	10.0

*The average of three determinations

IV. CONCLUSION

NOMBAP extracts Rh (III) into carbon tetra chloride successfully. This method is fast, easy and efficient, the method was applied for the analysis of synthetic sample to determine the Rhodium content. The outcomes of the method were in very much agreement the standard method[15].

ACKNOWLEDGEMENT

Author expresses gratitude to the Principal Dr. (Mrs). Anita Kanwar, VES. College of Arts, Science & Comm., Chembur, Mumbai – 71 for facilitating necessary research facilities. The author gratefully acknowledges the use of central instrumentation facilities at Vivekanand Education Society's College of Arts, Science and Commerce funded by FIST-DST (Department of Science & Technology, Government of India) and DBT Star College Scheme.

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