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Extractive Spectrophotometric Method for determination of Rhodium (III) using [N - (O - Hydroxy benzylidene) pyridine - 2 - amine] (NOHBPA) as an Analytical reagent

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Abstract: N - (o - hydroxy benzylidene) pyridine - 2 - amine (NOHBPA) extracts Rh (III) quantitatively (99.20%) into n-amyl alcohol from an aqueous solution of pH range 4.5 - 5.7 followed by digestion on boiling water bath for 20 - 25 minutes. The n-amyl alcohol extract shows an intense peak at 510 nm (λ max). Beer's law is obeyed over the Rh (III) concentration range of 1.0 - 8.0 µg/ml. The sandell's sensitivity and molar absorptivity for Rh - NOHBPA system is 0.0174 $\mu g cm^{-2}$ and 5917.16 L $mole^{-1} cm^{-1}$ respectively. The composition of extracted species is found to be 1:3 (Rh: NOHBPA) by Job's Continuous Variation and Mole Ratio Method. Interference by various ions has been studied. The proposed method has been satisfactorily applied for determination of Rh (III) in alloy samples.

Key words: Extractive Spectrophotometry, Rhodium (III), NOHBPA, Alloy Sample.

INTRODUCTION

Rhodium is a chemical element that is a rare, silvery-white, hard and chemically inert transition metal. Rhodium together with Ruthenium, palladium, platinum, osmium and iridium forms a group of elements referred to as the platinum group metals (PGM). Rhodium is the best hardeners for addition to palladium or platinum. It is alloyed with these metals to make electrical contacts with extreme wear resistance. Rhodium compounds are encountered relatively rarely by most people. All rhodium compounds should be regarded as highly toxic and as carcinogenic. Compounds of rhodium stain the skin, eyes and respiratory tract (mouth, throat, and lungs) very strongly. The primary uses of rhodium are in alloys and as catalysts for industrial processes.

A solvent extraction is becoming important separation technique in chemistry. It has grown into one of the most promising method in the separation of metal ions at trace level because of its simplicity, rapidity and varsality[1-2]. Solvent extraction as a separation technique with spectrophotometrically using different organic reagent have significant role in pharmaceutical science[3]. Literature survey reveals that various reagents[4-12] are available for the spectrophotometric determination of Rh (III). In the present communication, we describe the extractive spectrophotometric determination of Rh (III) with [N - (o - hydroxy benzylidene) pyridine - 2- amine].

EXPERIMENTAL

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

General procedure for preparation of [N - (o - hydroxy benzylidene) pyridine - 2 - amine] (NOHBPA)

The reagent NOHBPA was synthesized by refluxing equimolar amount of ethanolic solution of o - hydroxy benzaldehyde with 2 - amino pyridine for 6 hours. On cooling the reaction mixture, a sharp yellow crystal product separated out (yield 80%, m.p. 70^0-71^0C) which was collected by filtration. The resulting NOHBPA was recrystalised using aqueous ethanol as the procedure recommended by Vogel[13].

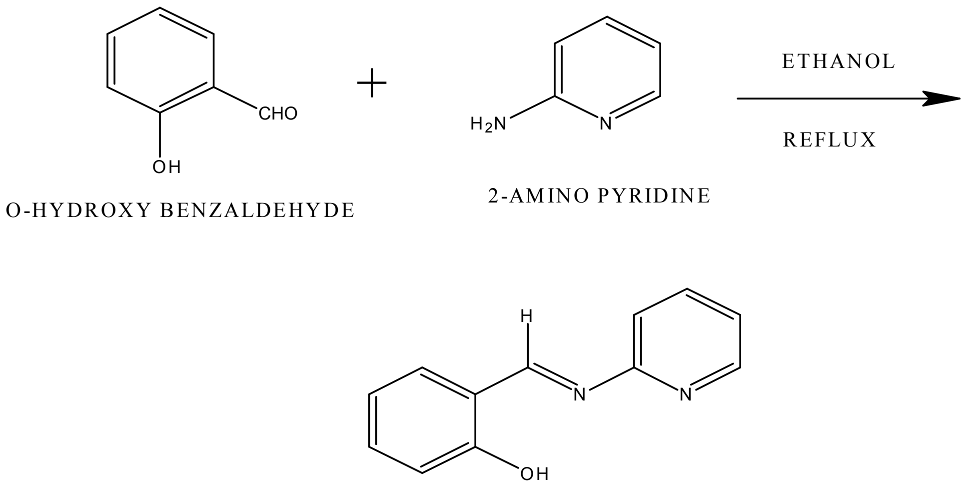
The purity of product was checked by TLC and characterized by elemental and spectral analysis. Its solution was prepared in Dimethylformamide (DMF). A stock solution of Rh (III) was prepared by dissolving accurately weighed rhodium chloride in conc. hydrochloric acid by heating on a boiling water bath and the solution was then diluted to 250 ml with double distilled water. Its solution was standardized by gravimetrically[14]. Working solutions of Rh (III) were made by diluting the stock solution to an appropriate volume. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

EXTRACTIVE SPECTROPHOTOMETRIC DETERMINATION OF Rh (III)

To an aliquot of aqueous solution containing 10 - 80 µg of Rh (III), 2ml of buffer solution (sodium acetate and acetic acid) of pH 5.5 and 2 ml of 3% solution of NOHBPA prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water. The solution was then equilibrated for 30 seconds with 10 ml of n - amyl alcohol and the phases were allowed to separate. The n - amyl alcohol extract was collected in a 10 ml measuring flask and made up to mark with n - amyl alcohol, if necessary. The absorbance of n - amyl alcohol extract was measured at 510 nm against a reagent blank prepared under identical conditions. The measured absorbance was used to compute the amount of Rh (III) present in the sample solution from predetermined calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH or acidity.

DETERMINATION OF Rh (III) IN ALLOY SAMPLE

0.1 to 0.2 gm sample was dissolved in boiling with 10 ml of aquaregia**.** The resulting solution was evaporated to dryness and the residue was then dissolved in 10 ml of 1N HCl filter, if required and solution was diluted to 100 ml with doubly distilled water. The working solution was prepared by appropriate dilution of stock solution. To an aliquot of this solution 1ml was analyzed for Rh (III) by the procedure as described earlier.



[N- (O - HYDROXY BENZYLIDENE) PYRIDENE - 2 - AMINE]

RESULTS AND DISCUSSION

Rh (III) could be extracted quantitatively (99.20%) by NOHBPA into n - amyl alcohol from an aqueous solution of pH 5.5 followed by digestion on boiling water bath for 20 - 25 minutes. Organic solvents used for extraction of Rh (III) can be arranged on the basis of their extraction coefficient values as n-amyl alcohol > ethyl acetate > benzene > toluene > chloroform > carbon tetrachloride > xylene > n-butanol > nitrobenzene > benzyl alcohol. n - amyl alcohol was found to be the best extracting solvent hence; it was selected for the extraction throughout the work. The n - amyl alcohol extract of Rh: NOHBPA complex showed an intense peak at 510 nm [Fig - I]. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength. The result shows that the system confirmed to Beer's law at this wavelength over Rh (III) concentration range of 1.0 to 8.0 µg/ml [Fig - II]. The molar absorptivity of the extracted complex on the basis of Rh (III) content was calculated to be 5917.16 L mol $^{-1}$ cm^{-1}. It was found that 2 ml of 3.0% solution of NOHBPA prepared in DMF was sufficient to extract 80 µg of Rh (III). The colour of the n - amyl alcohol extract was found to be stable at least 12 hrs. at room temperature.

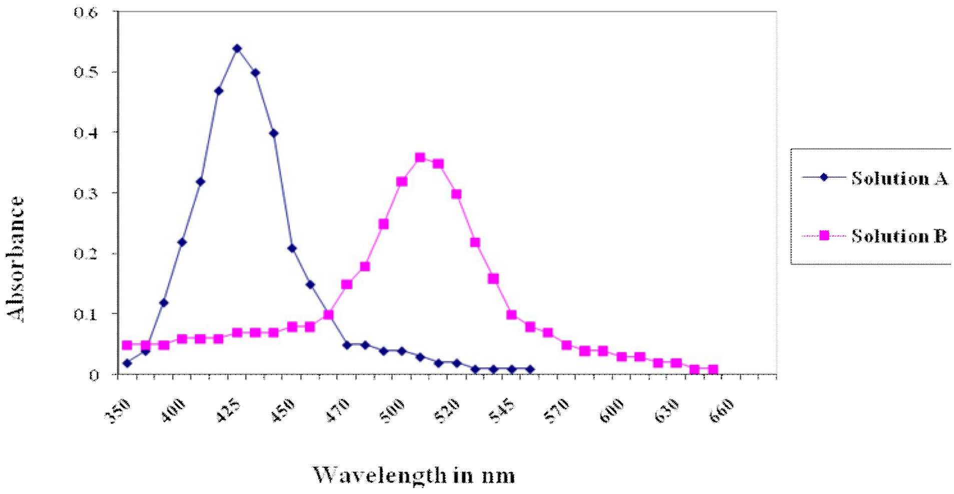
EFFECT OF OTHER IONS

Rh (III) (40 µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Rh (III) (40 µg): 10 mg each of Mg(II), Ca(II), V(V), Ni(II), Co(II), Zn(II), Mo(VI), Ce(IV), Be(II), Ba(II), Bi(III), Li(I), Ti(III), Zr(IV), Pd(II), Sn(III), Th(IV), W(VI), 5mg each of Mn(II), and Cu(II), 1mg each of Ag(I), Fe(II), Fe(III) and Ru(III); 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, carbonate, sulphate, thiocyanate, phosphates, acetate, persulphate and thiosulphate and 1mg of oxalate. In the presence of tartarate, Cr (III) and citrate do not interfere. Interference due to the Pt (IV) and Hg (II) could be masked by adding potassium iodide.

COMPOSITION OF THE EXTRACTED COMPLEX

The composition of the extracted complex was found to be 1:3 (Rh: NOHBPA) by Job's continuous variation [Fig - III] and Mole ratio methods. [Fig - IV]

Fig - I



Solution A: Absorbance spectra of NOHBPA
Solution B: Absorbance spectra of Rh - NOHBPA complex

Fig - II Calibration Curve for Rh (III)

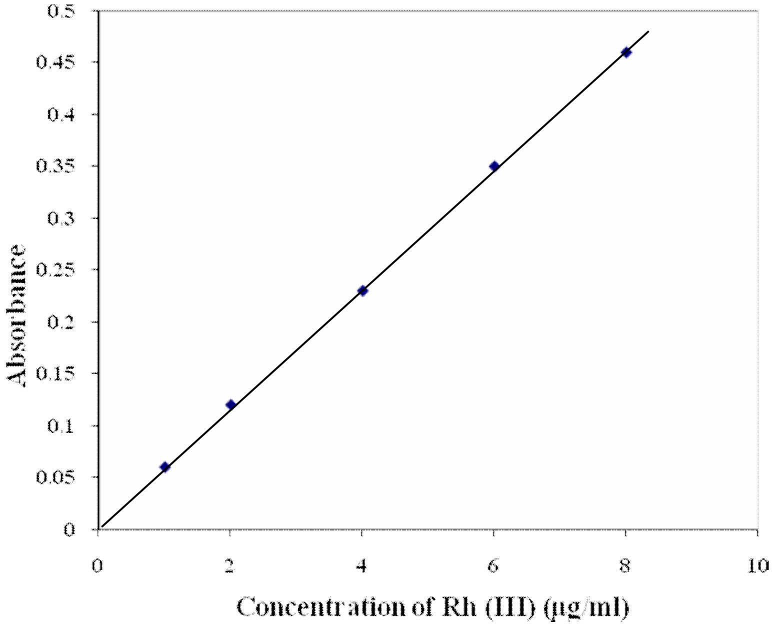


Fig - III Job's Continuous Variation Method

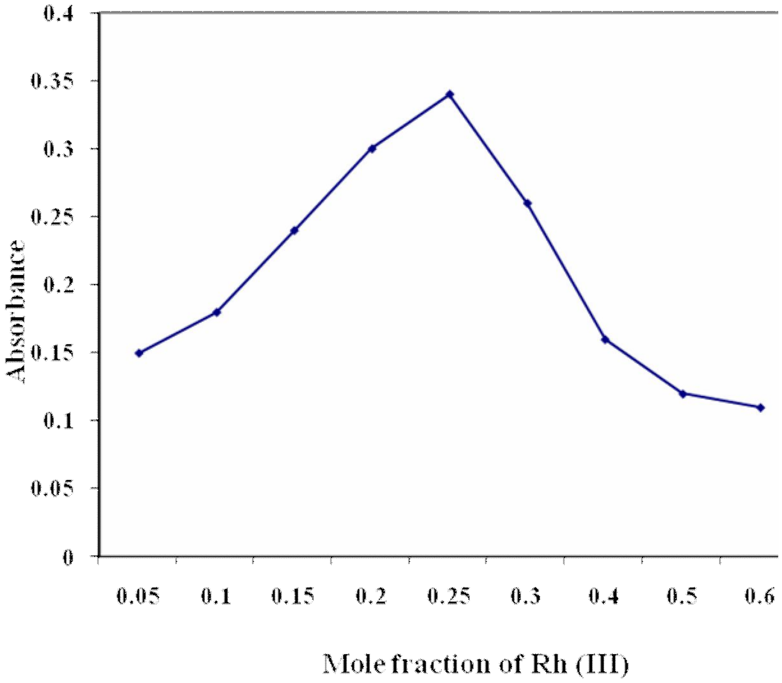
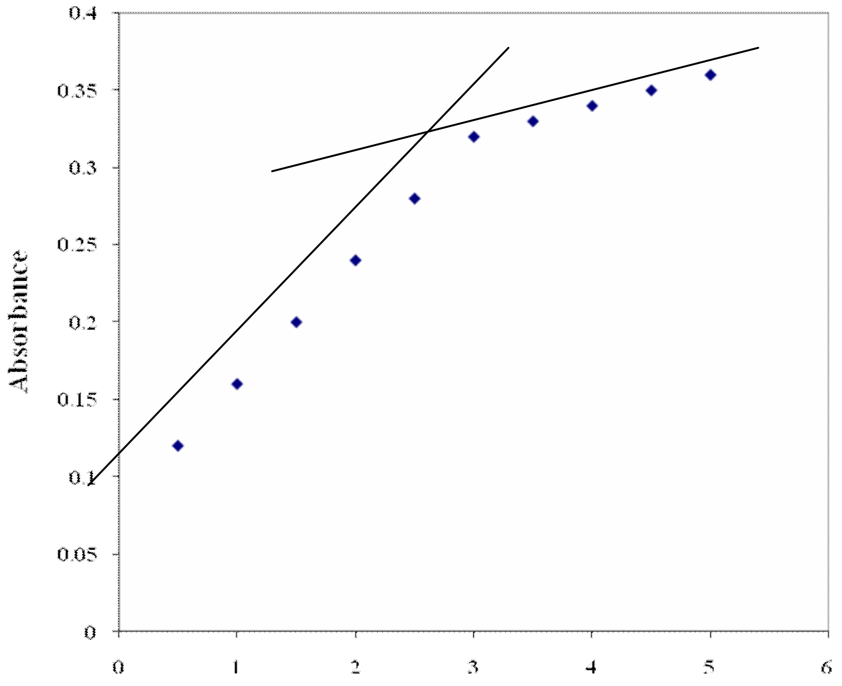


Fig - IV Mole Ratio Methods



Mole ratio [NOHBPA] / Rh (III)

PRECISION, ACCURACY, SENSITIVITY AND APPLICATIONS OF METHOD

The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Rh (III) following the recommended procedure. The average of 10 determination of 40 µg of Rh (III) in 10 cm^3 solutions was 39.80 µg, which is varied between 40.25 and 39.35 at 95% confidence limit. Standard deviation and Sandell's sensitivity of the extracted species is found to be ±0.633 and 0.0174 μgcm^{-2} respectively. The proposed method has been successfully applied for the determination of Rh (III) in alloy samples.

The results of the analysis of the samples were comparable with those obtained by the standard method[15] for Rh (III) (Table - I).

Table – I: Determination of Rh (III) from alloy Sample

Sample	Rh (III) found (%) * Present method	Rh (III) found (%) Standard method
Nickel – Rhodium Alloy	5.945	5.950

*Average of three determinations

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