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Extractive Spectrophotometric Determination Of Ruthenium (Ii) With [N-(O-Methoxy Benzaldehyde) -2 – Amino Phenol]

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Abstract: A simple, rapid and sensitive spectrophotometric method has been developed for the determination of Ru (III) by using N - (o - methoxy benzaldehyde) 2 - aminophenol (NOMBAP) as an analytical reagent. NOMBAP has been synthesized and characterized by elemental and spectral analysis. NOMBAP extracts Ru (III) quantitatively (99.30%) into chloroform from an aqueous solution of pH range 4.3 -5.5. The chloroform extract shows an intense peak at 490 nm (λ max).Beer's law is obeyed over the Ru (III) concentration range of 0.5 - 7.0 ml. The sandell's sensitivity and molar absorptivity for Ru - NOMBAP system is 0.00713 µgcm^{-2} and 14177.21 L mole^{-1}cm^{-1} respectively. The composition of extracted species is found to be 1:3 (Ru: NOMBAP) by Job's Continuous Variation and Mole Ratio Method. The average of 10 determination of 10 µg of Ru (III) in 10 cm^3 of solution is 10.05 µg which is varied between 9.79 µg and 10.31 µg at 95% confidence limit and standard deviation is ±0.369. Interference by various ions has been studied. The proposed method has been successfully applied for determination of Ru (III) in alloy samples.

Key words: Extractive Spectrophotometry, Ruthenium (III), N-(o-methoxy benzaldehyde) 2-aminophenol (NOMBAP), alloy Sample.

INTRODUCTION

Ruthenium has derived its name from Ruthenia.. Ruthenium together with rhodium, palladium, platinum, osmium and iridium forms a group of elements referred to as the platinum group metals (PGM). Ruthenium is the best hardeners for addition to palladium or platinum.. All ruthenium compounds are regarded as highly toxic and as carcinogenic. Compounds of ruthenium stain the skin, eyes and respiratory tract (mouth, throat, and lungs) very strongly. The primary uses of ruthenium are in alloys and as catalysts for industrial processes. Ruthenium catalysts may provide a way of changing light energy into electrical energy. The process is similar to photosynthesis, in which green plants change sunlight into stored chemical energy. Ruthenium - 106 is used for medical purposes. When ruthenium - 106 breaks down, it gives off a form of radiation called beta rays. These beta rays act somewhat like X rays. They attack and kill cancer cells. As an example, ruthenium-106 has been used to treat certain forms of eye cancer.

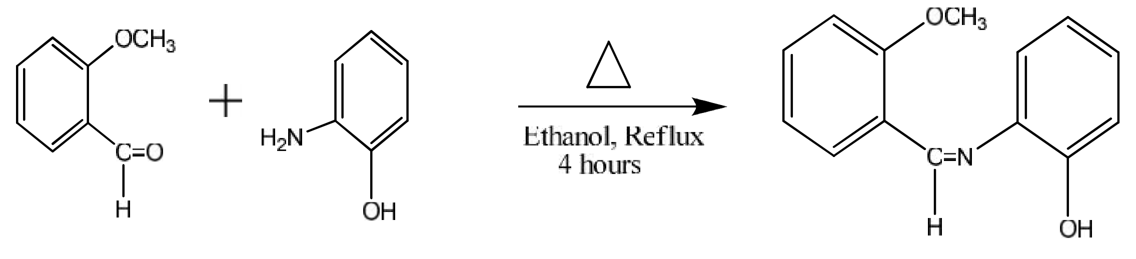
A solvent extraction is becoming important separation technique in chemistry. It has grown into one of the most promising method in the separation of metal ions at trace level because of its simplicity, rapidity and varsality[1-2]. Solvent extraction as a separation technique with spectrophotometrically 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 Ru (III). In the present communication, we describe the extractive spectrophotometric determination of Ru (III) with [N - (o - methoxy benzaldehyde) 2- amino phenol]

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. A stock solution of Ruthenium was prepared by dissolving accurately weighed Ruthenium chloride in concentrated hydrochloric acid by heating on boiling waterbath and the solution was then diluted to 250 mi with double distilled water. Its solution was standardized by thiourea method[14-15]. Working solutions of Ru (II) 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.

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

The reagent NOMBAP was synthesized by refluxing equimolar amount of ethanolic solution of o -methoxy benzaldehyde with 2 – amino phenol for 6 hours. On cooling the reaction mixture, a sharp yellow crystal product separated out (yield 80%, m.p.87^0-88^0C) which was collected by filtration. The resulting NOMBAP was recrystalised using aqueous ethanol as the procedure recommended by Vogel[13].



o-methoxy benzaldehyde 2-aminophenol N-(o-methoxybenzaldehyde)-2-aminophenol

The product obtained was characterized by elemental and spectral analysis. Its solution was prepared in Dimethylformamide (DMF).

Procedure for the Extraction and separation of Ruthenium (III):

An aliquot of aqueous solution containing 500ug of Ru (III) and 2 ml of 2%solution of NOMBAP prepared in DMF were mixed in 25 ml beaker . The pH of solution was adjusted to the desired value with dilute solution of HCl /NaOH, keeping the total volume to 10 ml with distilled water followed by digestion on boiling water bath for 15 - 20 minutes. The resulting solution was then transferred into 125 ml separatory funnel. The beaker was then washed with 5 ml portion of organic solvent and each washing was added to the solution in the separatory funnel. The two phases were equilibrated for one minute and allowed to separate. After the separation of two phases, pH of the equilibrated aqueous phase was measured and Ruthenium content in each phase was estimated by thiourea method[15]. The extraction was carried out with different solvents to find out the best extracting solvent.

Procedure for the Extractive Spectrophotometric Determination of Ru (III)

To an aliquot of aqueous solution containing 1- 100 µg of Ru (III), 2ml of buffer solution of pH 5.0 and 2 ml of 2% solution of NOMBAP prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water followed by digestion on boiling water bath for 10 - 15 minutes. On cooling, the solution was then equilibrated for one minute with 10 ml of chloroform and the phases were allowed to separate. The chloroform extract was collected in a 10 ml standard measuring flask and made up to mark with chloroform, if necessary. The absorbance of chloroform extract was measured at 490 nm against a reagent blank prepared under identical conditions. The measured absorbance was used to compute the amount of Ru (II) 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.

Determination of Ru (III) in Alloy Sample

0.1 to 0.2 gm alloy 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 1M 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 Ru (III) by the procedure as described earlier.

RESULTS AND DISCUSSION

Ru (III) could be extracted quantitatively (99.30%) by NOMBAP into chloroform from an aqueous solution of pH range 4.3--5.5 . Organic solvents used for extraction of Ru (III) can be arranged on the basis of their extraction coefficient values as chloroform > ethyl acetate > toluene > benzene > carbon tetrachloride > n-amyl alcohol > benzyl alcohol > nitrobenzene > xylene.> chloro benzene. Chloroform was found to be the best extracting solvent hence; it was selected for the extraction throughout the work. The chloroform extract of Ru: NOMBAP complex showed an intense peak at 490 nm [fig -III]. 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 a Ru (III) concentration range of 0.1 to 7.0 µg/ml [Fig -IV]. The molar absorptivity of the extracted complex on the basis of Ru (II) content was calculated to be 14177.21 L mol $^{-1}$ cm^{-1}. It was found that 2 ml of 2.0% DMF solution of NOMBAP was sufficient to extract 100 µg of Ru (III). The colour of the Chloroform extract was found to be stable at least 24 hrs. at room temperature.

Fig - I

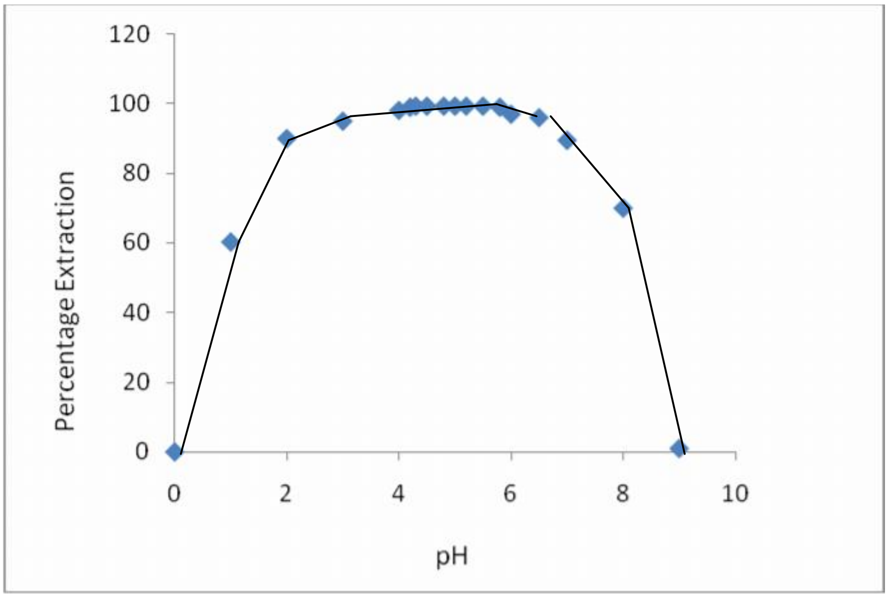


Fig II

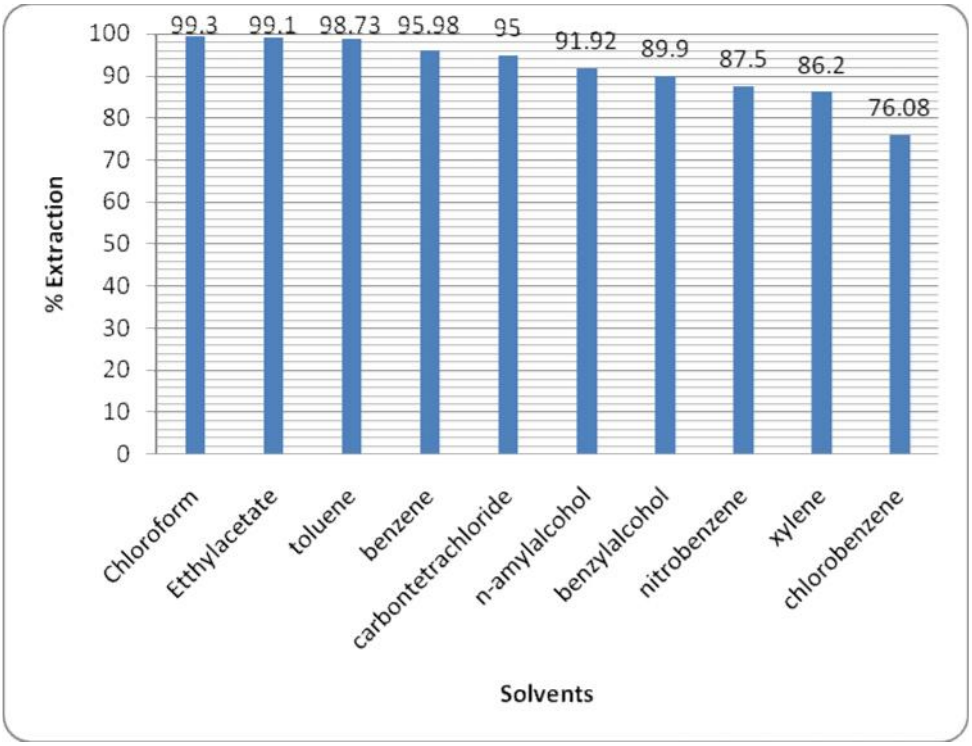
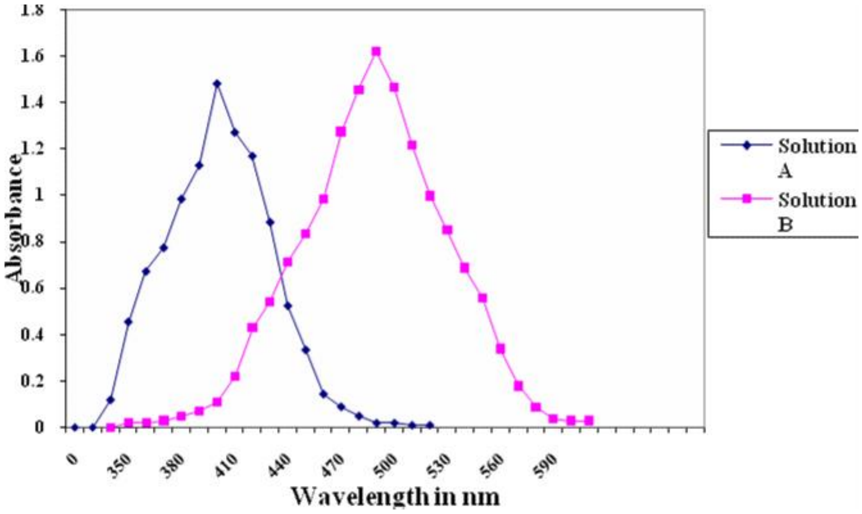
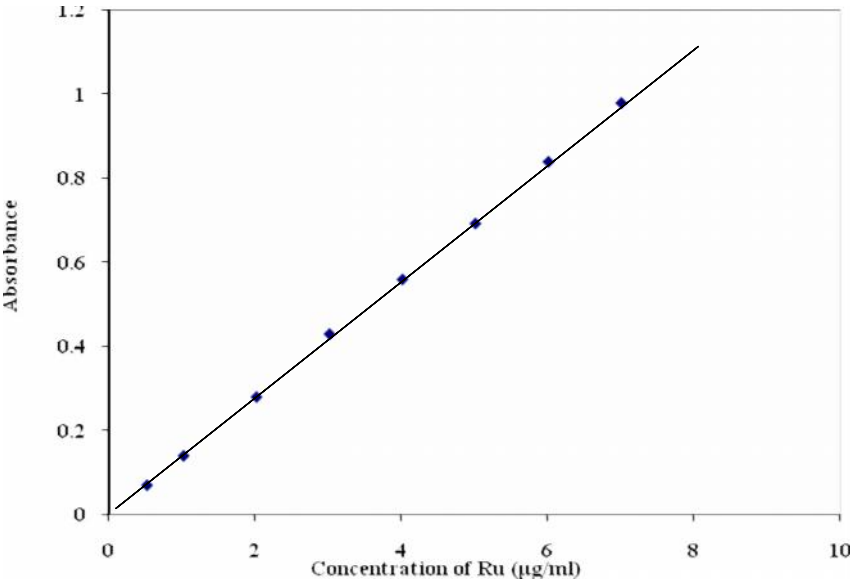


Fig III



Solution A: Absorption spectra of NOMBAP
Solution B: Absorption spectra of Ru-NOMBAP complex

Fig - IV **Calibration curve for Ru (III)**



Effect of other Ions

Ru (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 Ru (III) (40 µg): .
a) 10 mg each of Li(I),Be(II),Mg(II),Ca(II),Sr (II),Ba(II),Sn(II),Bi(III), Mo(VI), W(VI),Ce(IV), Th(IV) and Zr(IV).
b) 2 mg each of Pb(II),Cd(II) and Zn(II),
c) 1 mg each of Pd (II), Pt(IV) and Hg(II),
d) 20 mg each of chloride, iodides, bromide, fluoride,, sulphate, nitrate, thiocyanate, phosphates, acetate, citrate, thiourea and tri ethanol amine.

Interference by the various ions were removed by using appropriate masking agent.

Sr. No.	Interferring Ions	Amount added in mg	Masking agent added 1ml of 0.5M solution	Absorbance
1.	Fe (II)	10	Tri ethanol amine	0.56
2.	Cu(II)	10	NaH_2PO_4	0.56
3.	Ag(I)	10	Potassium thiocyanate	0.56
4.	Mn(II)	10	Potassium Thiocyanate	0.56
5.	Cr(III)	10	Tri ethanol amine	0.56
6.	V(V)	10	Tri ethanol amine	0.56
7.	Ni(II)	10	5-sulphosalicylic acid	0.56
8.	Oxalate	10	Sodium molybdate	0.56

Gomposition of the Extracted Complex

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

Fig - V Job's Continuous Variation Method

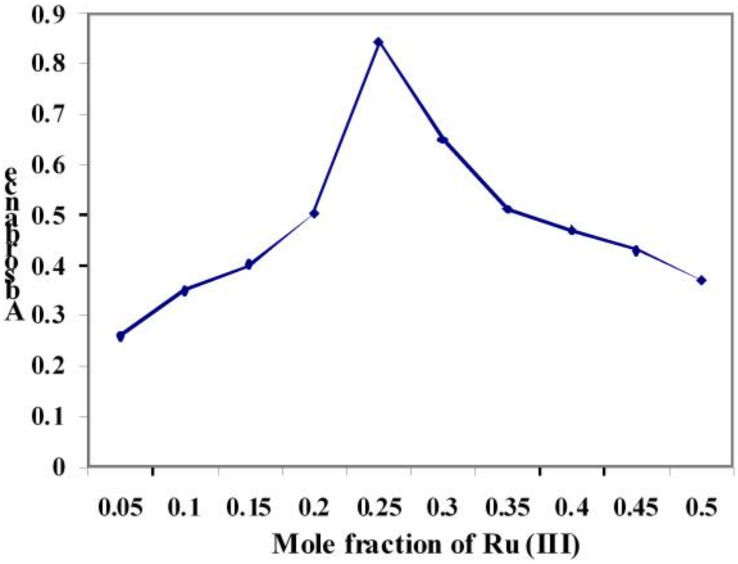
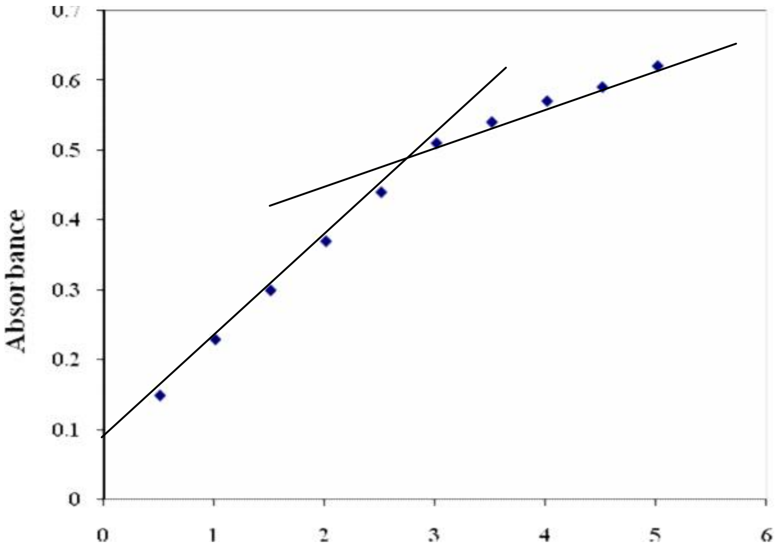


Fig - VI Mole Ratio Method



Mole ratio [NOHBPA] / Ru (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 Ru (III) following the recommended procedure. The average of 10 determination of 10 µg of Ru (III) in 10 cm^3 solutions was 10.05 µg, which is varied between 10.31 and 979 at 95% confidence limit. Standard deviation and Sandell's sensitivity of the extracted species is found to be ±0.735 and 0.0174 $\mu g.cm^{-2}$ respectively. The proposed method has been successfully applied for the determination of Ru (III) in alloy samples.

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

Table – II

Determination of Ru (III) from alloy Sample

Sample	Ru (III) found (%) * Present method	Ru (III) found (%) Standard method[15]
Ru - Titanium Alloy	0.096	0.100

*Average of three determinations

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