



Development of Extractive Spectrophotometric Method For The Determination of Ruthenium (III) With Schiff Base 2-[(2-Hydroxyphenylimino) Methyl]-4-Nitrophenol

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ABSTRACT

A simple spectrophotometric method has been developed for the determination of ruthenium (III) by using Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP]. HPIMNP extracts Ru (III) quantitatively (99.01%) into Xylene from an aqueous solution of pH 4.0 to 6.0 followed by digestion on boiling water bath for 20-25 minutes. The xylene extract shows an intense peak at 570 nm (λ max). Beer's law is obeyed over the Ru (III) concentration range 1.0 - 14.0 $\mu\text{g mL}^{-1}$. The molar absorptivity and Sandell's sensitivity of coloured species are 1587.46 $\text{L mole}^{-1} \text{cm}^{-1}$ and 0.067 $\mu\text{g.cm}^{-2}$ respectively. The composition of extracted species is found to be 1:3 (Ru: HPIMNP) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method had been applied for determination of Ru (III) in titanium alloy samples.

Keywords: Extractive Spectrophotometry, Ruthenium (III), Alloy samples, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP], Schiff Base.

INTRODUCTION

Various reagents [1] are available for the spectrophotometric determination of Ruthenium of which oximes, Schiff bases and its derivatives constitutes an important class [2,3]. Synthesis and Antimicrobial Activity of Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] has been reported [4]. However Analytical application of HPIMNP was not studied. In the present communication, we describe the extractive spectrophotometric determination of Ru (III) with 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP].

MATERIALS AND METHODS

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO - LI 127 pH meter was employed for pH measurements. The reagent HPIMNP was synthesized by condensation of 5-Nitro salicylaldehyde with 2 - amino phenol as per reported procedure [4]. The resulting product was recrystallized by using ethanol [5] and characterized by elemental and spectral analysis. Its 0.5 % solution was prepared in dimethylformamide

(DMF). A stock solution of Ru (III) was prepared by dissolving Ruthenium Chloride in double distilled water containing dilute Hydrochloric acid. It was standardized by thiourea method [6]. Working solutions of Ru (III) were made by suitable dilution. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extraction and separation of Ru (III): To an aliquot of aqueous solution containing 500 μg of Ru (III) and 2 mL of 0.5 % solution of HPIMNP prepared in DMF were mixed in 25 mL beaker. The pH of solution was adjusted to desired value with dilute solution of HCl/ NaOH, keeping the total volume to 10 mL with distilled water. Resulting solution digested on boiling water bath for 20-25 min, cooled and then transferred into 125 mL separatory funnel. The beaker was then washed with 5 mL portion of organic solvent twice and each washing was added to the solution in separatory funnel. The two phases were equilibrated for one min 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 determined by thiourea method [6]. The extraction was carried out with different solvents to find out the best extracting solvent. On the basis of ruthenium content in aqueous and organic phase, extraction coefficient and percent extraction was calculated.

Extractive Spectrophotometric Determination of Ru (III): To an aliquot of aqueous solution containing 10-140 μg of Ru (III), 2 mL of potassium hydrogen phthalate (KHP) and sodium hydroxide buffer solution of pH 5 and 1 mL of 0.5 % solution of HPIMNP prepared in DMF were added. The volume of solution was made up to 10 mL with distilled water followed by heating on boiling water bath for 20-25 min. The solution was first cooled at room temperature and then equilibrated for 30 sec with 10 mL of xylene and the phases were allowed to separate. The xylene extract was collected in a 10 mL measuring flask and made up to mark with xylene. The absorbance of xylene extract was measured at 570 nm against a reagent blank prepared under identical conditions. The Ru (III) content of the sample solution was determined from 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 ruthenium in alloy sample: Accurately weighed 0.2 - 0.4 g alloy sample was dissolved in boiling 10 mL aquaregia. The resulting solution was evaporated to almost dryness. The residue was then dissolved in 10 mL of 1 M HCl, filter if required and resulting solution was diluted to 100 mL with doubly distilled water. To an aliquot of this solution 1 mL was analyzed for ruthenium by the procedure as described earlier.

RESULTS AND DISCUSSION

Ruthenium (III) could be extracted quantitatively (99.01%) by HPIMNP into Xylene from an aqueous solution of pH range 4.0 – 6.0 followed by heating on boiling water bath for 20-25 min. Organic solvents used for extraction of Ru (III) can be arranged on the basis of their extraction coefficient values as xylene > toluene > benzene > nitro benzene > chloroform > carbon tetrachloride > n- butanol > n-amyl alcohol > benzyl alcohol > ethyl acetate. Xylene was found to be the best extracting solvent; hence, it was selected for extraction throughout the work.

The xylene extract of Ru - HPIMNP complex showed an intense peak at 570 nm. 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 Ruthenium concentration range 1.0 to 14 $\mu\text{g mL}^{-1}$ (Fig- 1). The molar absorptivity and Sandell's sensitivity of the extracted species on the basis of Ru (III) content were calculated to be 1587.46 $\text{L mol}^{-1} \text{cm}^{-1}$ and 0.067 $\mu\text{g. cm}^{-2}$ respectively. It was found that 1 mL of 0.5 % solution of HPIMNP prepared in DMF was sufficient to extract 140 μg of Ru (III). The color of the xylene extract was found to be stable at least 24 h at room temperature.

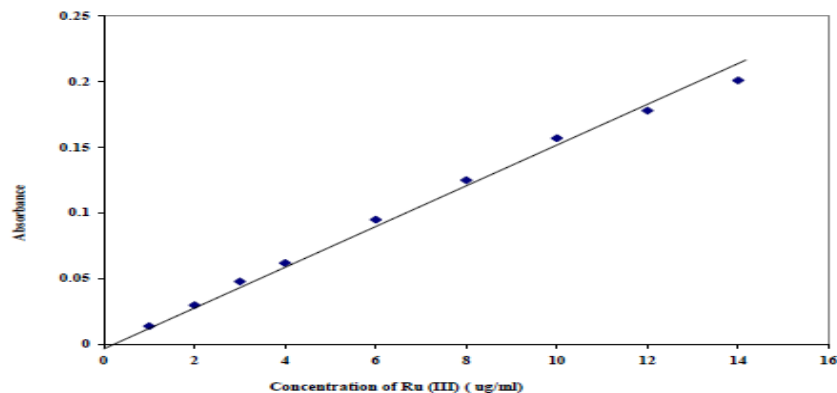


Fig. (1) - CALIBRATION CURVE FOR RUTHEMIUM

Effect of Other Ions: Ru (III)(100 μ g) 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)(100 μ g) : 10 mg each of, Li (I), Be (II), Ba (II), Ca (II), Sr (II), Al (III), Ti(III), V (V), Mo (VI), U(VI) & Ni (II). 100 ppm of each Os (IV), Pd (II), Pt (IV) and Rh (III). And 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphide, phosphates, tartrate, acetate, citrate and thiosulphate, thiocyanide, triethanol amine, ascorbic acid.

Interference due to iron removed before the extraction by conventional method [6]. Interference by various ions was removed by using appropriate masking agent (Table 1).

Table 1. Masking agents required for suppressing the interference by other ions

Interfering ion	Amount added in mg	Masking agent added 1ml of 2M solution
Cr (III)	10	Tri ethanol Amine
Ag (I)	10	Potassium bromide
Cu (II)	10	Sodium thiosulphate
Co (II)	10	Ascorbic Acid
Thiourea	10	Tartarate

Composition of the Extracted Complex: The composition of the extracted complex was found to be 1:3 (Ru: HPIMNP) by Job's continuous variation and Mole ratio methods (Fig 2 & Fig 3).

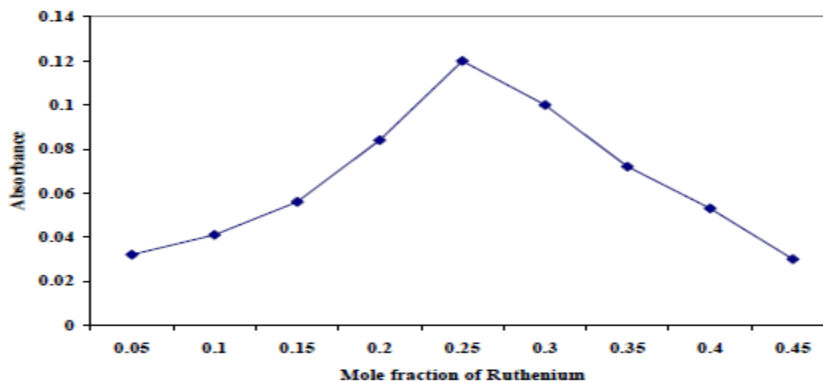


Fig.-(2) JOB'S CONTINUOUS VARIATION METHOD

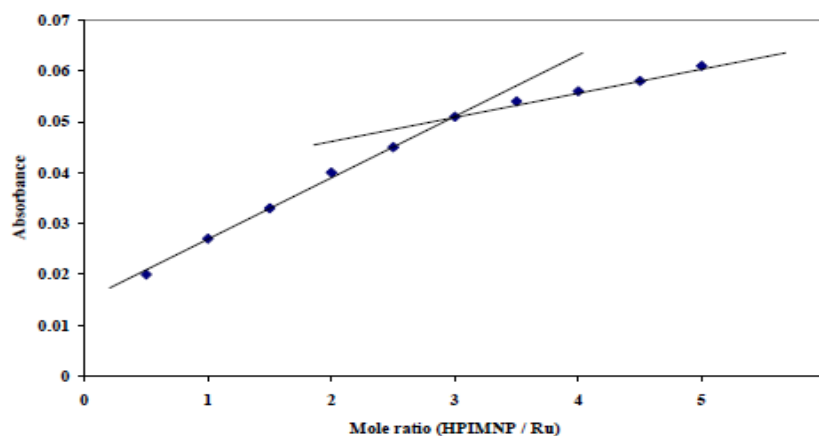


Fig.-(3) MOLE RATIO METHOD

Precision, Accuracy and Sensitivity 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 40 µg of Ru (III) in 10 cm³ solutions was 40 µg, which is varied between 40.67 and 39.33 at 95% confidence limit and standard deviation is ± 0.94.

APPLICATIONS

The proposed method has been applied for the determination of Ru (III) in titanium alloy sample. The results of the analysis of the sample were comparable with those obtained by the thiourea method (Table 2).

Table 2. Determination of Ru (III) in Alloy sample

Alloy Sample	Ru (III) found %	
	Present method	Thiourea method
Titanium based alloy	0.098	0.099

Results are the average of three independent determinations.

CONCLUSIONS

From the above discussions, it is found that Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] is a good sensitive reagent for development of rapid and sensitive extractive spectrophotometric method for the determination of Ru (III) and it has been satisfactory applied for determination of ruthenium in titanium alloy sample.

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