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Extractive spectrophotometric determination of rhodium (III) with isonitroso p-methyl acetophenone phenyl hydrazone

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ABSTRACT

Isonitroso p-Methyl Acetophenone Phenyl Hydrazone (HIMAPH) extracts Rhodium (III) quantitatively (99.29%) into toluene from on aqueous solution of pH 4.0-6.0 in the presence of 1 ml of 2 M solution of sodium acetate followed by digestion on boiling water bath for 50-60 minutes. The toluene extract shows an intense peak at 465 nm (λ max). Beer's law is obeyed over the Rh (III) concentration range 0.5-16.0 µg/m. The molar absorptivity and Sandell's sensitivity of coloured species are 2631.58 L $mole^{-1}$ cm^{-1} and 0.039 µg. cm^{-2} respectively. The composition of extracted species is found to be 1:3 (Rh: HIMAPH) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method has been applied for determination of Rh (III) in Nickel-Rhodium alloy sample.

Key words: HIMAPH, Rhodium, spectrophotometery.

INTRODUCTION

Various reagents[1] are available for the spectrophotometric determination of Rhodium of which isonitrosoketone and it derivatives constitutes an important class[2-3]. Isonitroso p – Methyl Acetophenone Phenyl Hydrazone (HIMAPH) has been used fro extractive spectrophotometric determination of Pt (IV)[4]. In the present communication, we describe the extractive spectrophotometric determination of Rh (III) with Isonitroso p – Methyl Acetophenone Phenyl Hydrazone (HIMPAH).

EXPERIMENTAL

ELICO- SL 159 spectrophotometer with optically matched quartz cells of 1cm path length were used fro absorbance measurement. An ELICO LI 127 pH meter was employed for pH measurements. The reagent HIMAPH was synthesized by condensation of isonitroso p – Methylacetophenone with phenyl hydrazine as procedure recommended by Vogel[5] and characterized by elemental and spectral analysis. Its 1.5% solution was prepared in Dimethylformamide (DMF). A stock solution of Rh (III) was prepared by dissolving Rhodium chloride in double distilled water containing dilute hydrochloric acid. The solution was standardized gravimetrically[6]. Working solutions of Rh (III) were made by suitable dilution. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extractive Spectrophotometric Determination of Rh (III)

For the extractive spectrophotometric determination of Rh (III), and aliquot of aqueous solution containing 5-160 µg of Rh (III), 1 ml of 2M-sodium acetate, 2ml of buffer solution (pH-5) and 1ml of 1.5% solution of HIMAPH prepared in DMF

added. The volume of solution was made up to 10 ml with distilled water followed by heating on boiling water bath for 50-60 minutes. The solution was first cooled at room temperature and then equilibrated for 1 min with 10 ml of toluene and the phases were allowed to separate. The toluene extract was collected in a 10 ml measuring flask and made up to mark with toluene. The absorbance of toluene extract was measured at 465 nm against a reagent blank prepared under identical conditions. The Rhodium 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.

RESULTS AND DISCUSSION

Rhodium (III) could be extracted quantitatively (99.29%) by HIMAPH into toluene from an aqueous solution of Ph 4.0 to 6.0 in the presence of 1ml of 2M sodium acetate followed by digestion on boiling water bath for 50-60 minutes. Organic solvents used for extraction of Rh (III) can be arranged on the basis of their extraction coefficient values as toluene > benzene > chloroform > n-butanol > n-amyl alcohol > carbon tetrachloride > ethyl acetate > benzyl alcohol > xylene > isobutanol. Toluene was found to be the best extracting solvent; hence, it was selected for extraction throughout the work.

The toluene extract of Rh- HIMAPH complex showed an intense peak at 465 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 rhodium concentration range 0.5 to 16 µg/ml (Fig. 1). The molar absorptivity and Sandell's sensitivity of the extracted complex on the basis of Rh (III) content were calculated to be 2361.58 L mol-1 cm-1 and 0.039 µg. cm-2 respectively. It was found that 1 ml of 1.5% DMF solution of HIMAPH was sufficient to extract 160 µg of Rh (III). The colour of the toluene extract was found to be stable at least 48 hrs at room temperature.

Effect of Other ions

Rh (III) (20 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) (20 µg) : 10 mg each of, Li (I), Be (II) Ba (II), Ca (II), Sr(II), Al (III), Ti (III), V (V), Ni (II), Mo (IV), U (VI), 0.01 mg of Pt (IV), Ru (III), 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphide, phosphates, tartrate, acetate, citrate and thiosulphate, thiocyanide.

The masking agent required for suppressing the interference by other ions in the determination of 20-µg rhodium are shown in table 1.

Composition of the extracted complex

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

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

Interfering ion	Amount Added in mg	Masking agent 1 ml of 2M solution
Fe (II) and Fe (III)	10	Citric acid
Cr (III) and Zn (II)	10	Sodium fluoride
Co (II), Hg (II), W (VI) and Zr (IV)	10	Potassium iodide
Pd (II), Cd (II), Pb (II), Th (IV) and Mg (II)	10	Ammonium bromide
Cu (II) and Ce (IV)	10	Sodium thiosulphate
Mn (II)	10	Thiocyanide
Thiourea	10	

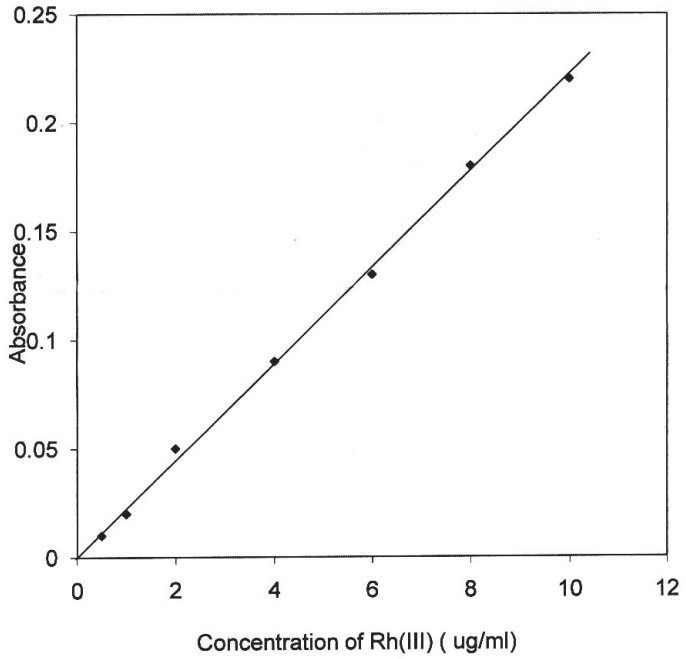

Fig. 1: Calibration curve for rhodium

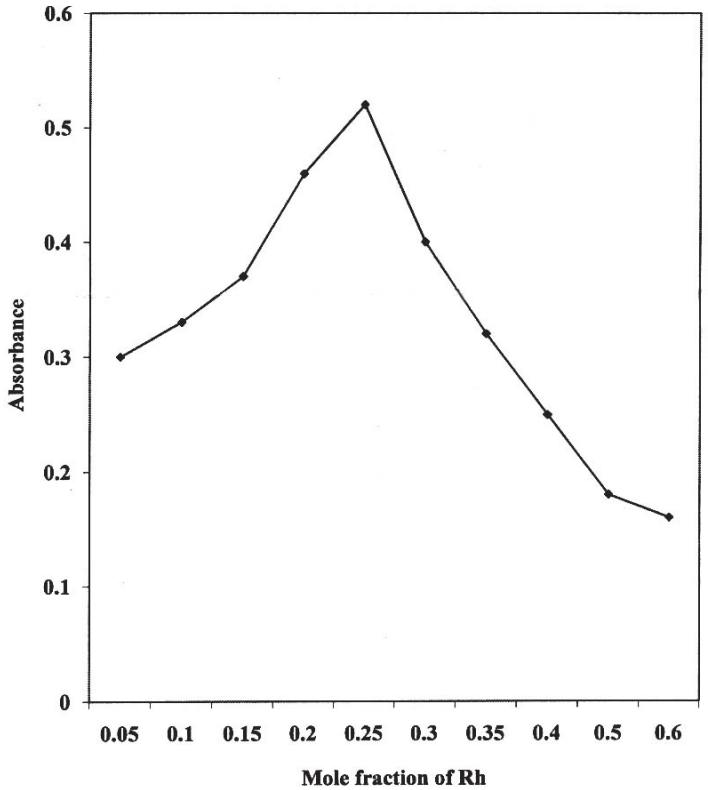

Fig. 2: Job's continuous variation method

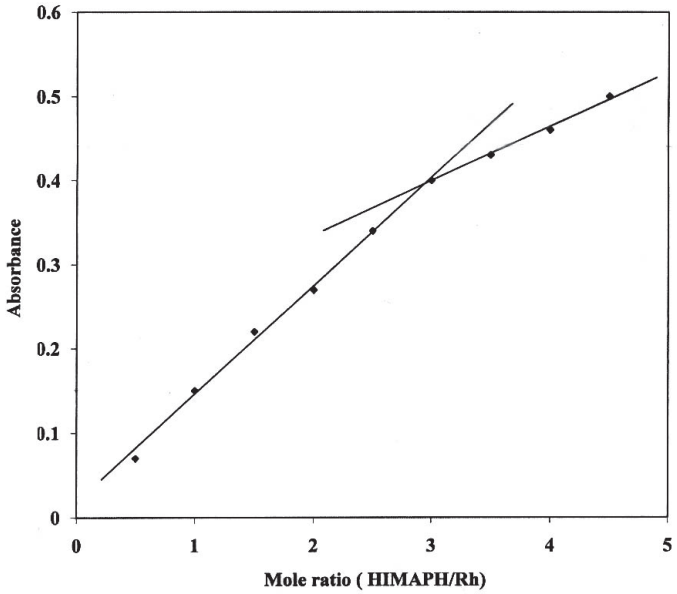

Fig. 3: Mole ratio method

Table 2: Determination of Rh (III) in Alloy sample

Ally Sample	Rh (III) found %	
	Present method	**Stannous Chloride method**
Nickel-Rhodium alloy	5.950	5.955

Precision, Accuracy, Sensitivity and Application 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 20 µg of Rh (III) in 10 cm^3 solutions was 19.9 µg, which is varied between 20.76 and 19.04 at 95% confidence limit and standard deviation was ±0.86. The proposed method has been applied for the determination of Rh (III) in Nickel-Rhodium alloy sample. The results of the analysis of the sample was comparable with those obtained by the Stannous Chloride method[3] (Table 2).

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