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Extraction Spectrophotometric Determination of Rhodium (III) using 2, 4-dimethyl -3H- 1, 5 benzodiazepine as Fresh Analytical Reagent

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Abstract: *In the present experimental investigations, a novel reagent 2, 4-dimethyl -3H- 1, 5 benzodiazepine (DBA) has been developed for the extractive estimation of rhodium (III) from given sample and by use of extractive spectrophotometric analysis. In the Laboratory, the fresh reagent was prepared and its characterization has been done with the help of mass spectrophotometer and IR, NMR, elemental analysis. The developed analytical reagent (DBA) when reacts with rhodium (III) produces red complex, this complex can be extracted by using n-butanol as a selected solvent, maintained at constant pH 8.9. The Beers law is followed in the concentration of 1-10 µg L^{-1} of rhodium (III), the optimum values of maximum absorption, molar extinction coefficient and sandell's sensitivity to the red complex, observed to be 510 nm, 4863 Lit $mol^{-1}cm^{-2}$ and 0.01102µg cm^{-2}.The proposed analytical reagent is found to be best, simple and superiorfor extraction of rhodium (III) metals from syntheticsamples at optimized conditions.*

Keywords: Rhodium (III), Spectrophotometric determination, DBA reagent, molar absorptivity.

I. INTRODUCTION

Rhodium is a precious element usually found in ores mixed with other elements viz. platinum, palladium, silver and gold. Its abundance in earth's crust is only 0.001 ppm. It has application in catalysis, corrosion, electrical and electronic apparatus.

Various analytical techniques are available for the determinations of rhodium still most of them have some drawbacks. Comparatively solvent extraction technique is the most efficient method of separation technology with high simplicity, speed and applicability to both tracer as well as macro amount of metal ions. N-n-octylaniline is used for the quantitative extraction of rhodium (III) from sodium malonate media (Anuse and Kolekar, 2002). Extraction of rhodium (III) was carried out from bromide media in the presence of stannous chloride using cyanex 471X and cyanex 923. (Duche et al., 2002). N, N'dimethylN, N'diphenyltetradecyl malonamide (DMDPHTDMA) extracts rhodium (III) in the presence of tin (Malik and Ana paula, 2008). Rhodium (III) was separated from iridium (III) and ruthenium (III) using different concentrations of alamine336 in kerosene using hydrochloric acid media (Goralsa et al., 2007).Rhodium (III) complexes with water soluble porypyrin 5, 10, 15, 20 – tetrakis(4-N-methylpyridyl)Porphine(TMPYM) in acetate buffer at pH 3.9, complexation enhanced in the presence of ethanol by heating at 100 ^{o}C for 15 min (Kunio et al.,2006).

In the present investigation we have reported a novel method for extractive spectrophotometric determination of rhodium (III), using novel reagent 2, 4-dimethyl -3H- 1, 5 benzodiazepine (DBA). The method is proved to be selective and sensitive as compared to other existing extractive spectrophotometric determination methods for rhodium(III) determination.

II. EXPERIMENTAL METHODOLOGY

2.1 Instrumentation

During the experimentation the absorbance was measured by using calibrated UV visible spectrophotometer (Model:Shimadzu 2450 UV-Visible with 10 mm quartz cell, Make: Shimadzu Europe, Supplier: Scientific

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Engineering works, Tamilnadu, India) and pH was maintained by using calibrated digital pH meter (Elico LI-120). The parameters maintained during experimentation are depicted in Table 1

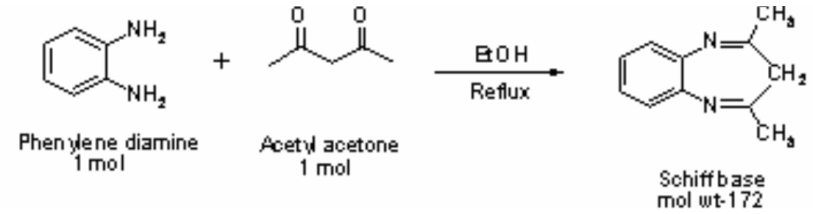
Parameter maintained	Remark
Maximum Absorbance	510 nm
Solvent used	n- butanol as best among studied
pH required	8.9
Equilibration time observed	60 seconds
Stability of rhodium(III)- reagent	45hrs
Optimum Beer's concentration limit	1 to 10 mg cm^{-3}
Molar exction coefficient	4863 L $mol^{-1}cm^{-2}$
Sandell's sensitivity	0.01102 mg cm^{-2}
Mole ratio of rhodium(III) : DBA	1:1

Table 1: Experimental parameters

2.2 Preparation of Analytical Reagent

The analytical reagent is prepared by mixing 1 mole of o-phenylenediamine and 2 moles of Acetyl acetone in the Ethanol as solvent. The mixture obtained is then refluxed for the duration of 2 hrs in the round bottom glass flask. The obtained solution from above process is poured in ice to get crystals. This results a solid product, which is recrystallized by using ethanol as a solvent. Further, it is then synthesized and characterized and used as analytical reagent for spectrophotometric identification ofrhodium (III) ions. All chemicals and reagents used in the present experimental investigations and preparation of novel fresh regent were of Analytical grade. A stock solution of above analytical reagent with concentration of 0.05% was made ready in a methanol. The reaction scheme is as indicated below.

Reaction



Reaction Scheme: Preparation of analytical Reagent 2, 4-DIMETHYL -3H- 1,5 BENZODIAZEPINE (DBA)

2.3 Stock Solution

An accurately measured amount of rhodium chloridewas dissolved in small quantity of purified water, and then diluted to 100 ml solution; this resulted into 100 ppm stock solution. Further, in the experimentation 1-10 ppm solution was prepared from the stock solution.

2.4 Experimental Procedures

In a beaker mix 1 ml (1 ppm) to 10 ml (10 ppm) in the increment of 1 ml solution of rhodium chloride, and 0.05% reagent in methanol are thoroughly mixed. The pH is then adjusted to 8.9 by adding the buffer solution. The above solution is then added to separating glass funnel with 10 ml n-butanol. The organic and aqueous phases were separated. The organic phase is then subjected to spectrophotometer at wavelength of 510 nm.

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III. RESULTS AND DISCUSSIONS

3.1 Effect of pH on Extraction

In this study, different Buffer solutions were used for rhodium (III)extraction with varied range of pH from 1 to 11, maintaining constant molar ratio 1:1 between organic phase and aqueous phase. Figure 1 indicates the variation of absorbance with pH. It is observed from this figure that absorbance increases with increasing pH and touches maximum, to corresponding value of pH 8.9 and thereafter rise in pH, reduces absorbance significantly. Hence, the value of pH 8.9 was used in further investigations

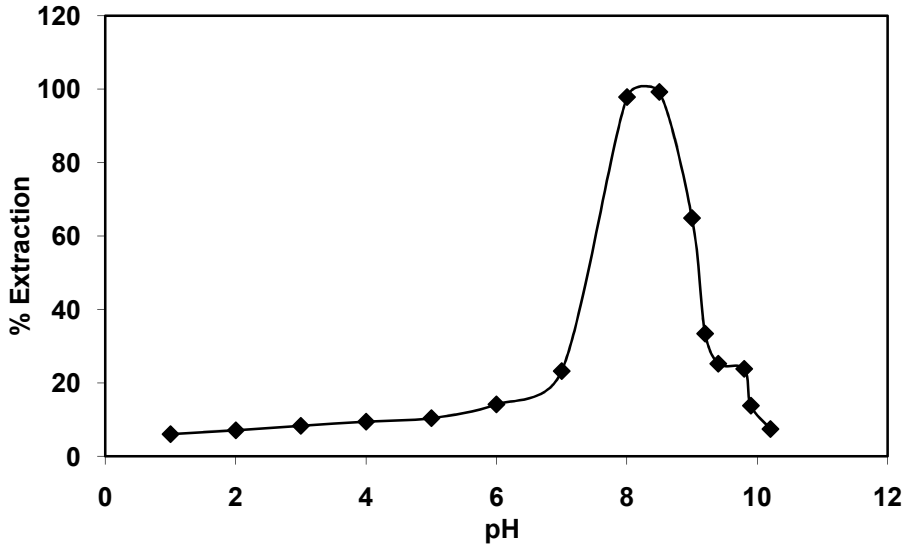

Figure 1: Variation of Absorbance with pH

3.2 Solvent Selection

Different organic solvents were tested in this experimental work to identify the suitability of solvent and presented in Figure 2. The. n-butanol is seems to be suitable solvent as indicated in the same figure.

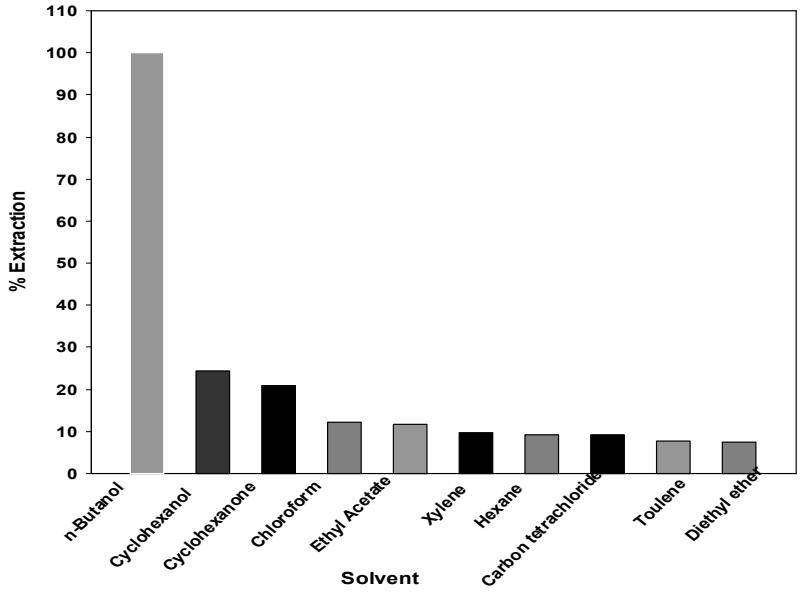

Figure 2: Selection of solvent

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3.3 Selection of Wavelength

Fig.3 shows that absorbance increases with increasing wavelength and attains a maximum value of absorbance at corresponding wavelength of 510 nm for n-butanol as a solvent. Further, increase in wavelength reduces absorbance. This value of wavelength was recommended for further investigations

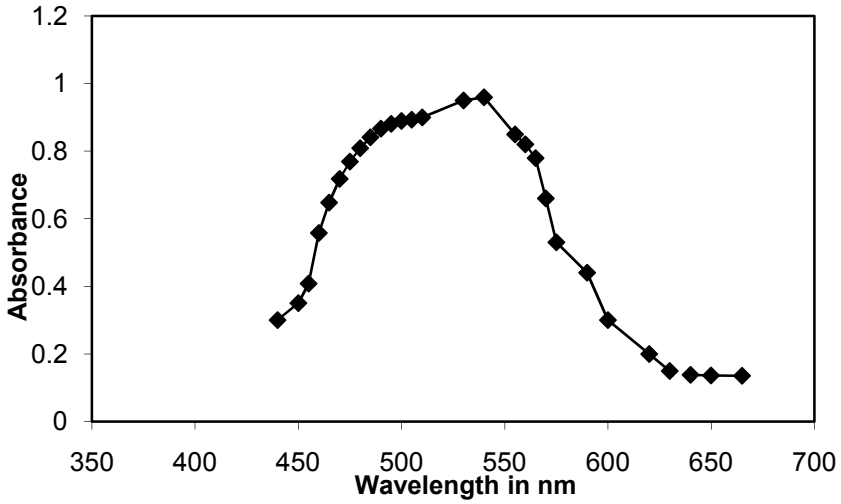

Figure 3: Variation of Absorbance with Wavelength

3.4 Development of Calibration Curve

Figure 4 shows the variation of absorbance with concentration of rhodium (III) ions. By following the same method as demonstrated in experimental procedure, the samples of rhodium (III) with different concentrations were prepared and its absorbance was measured for development of Calibration curve.

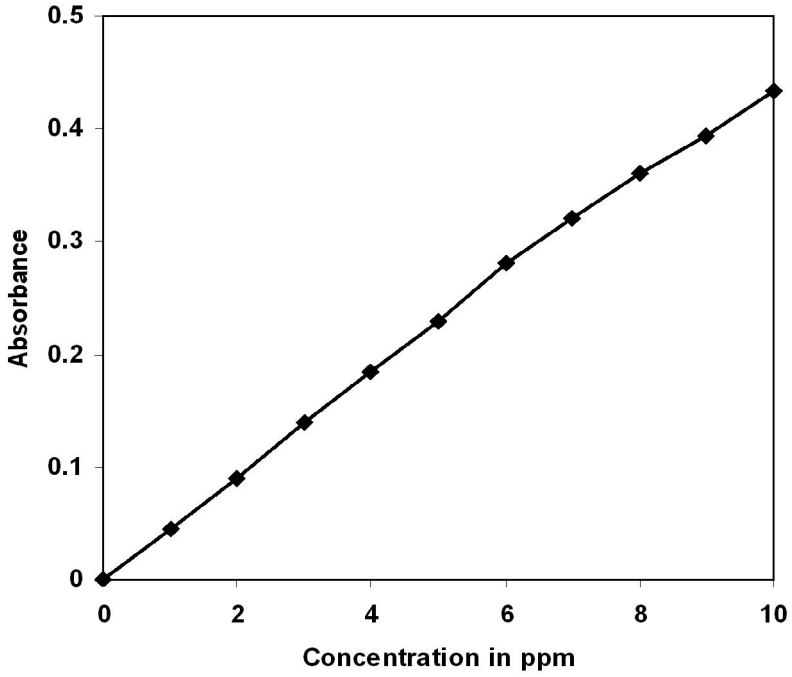

Figure 4: Development of Calibration Curve

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3.5 Selection of Molar Ratio Between Rhodium (III) and Analytical Reagent

Figure 5 shows the selection of molar ratio between rhodium (III) and analytical reagent using various methods. The Job's continuous variation method was extensively used for fixation of composition ratio of the extracted species, and same results were further validated by using, mole ratio method and slope ratio method. From, these methods a molar ratio between rhodium (III) and analytical reagent to be fixed as1: 1

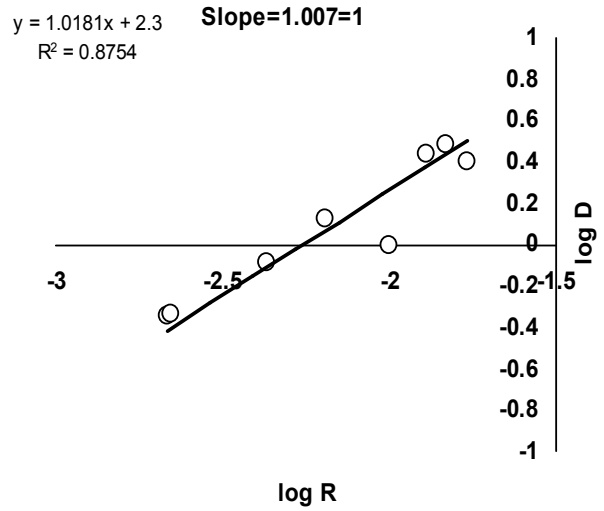

Figure 5: Selection of molar ratio between rhodium (III) and analytical reagent

IV. USES OF PRESENT STUDY

The selectivity of a method was confirmed by applying it in the determination of rhodium (III) in synthetic mixtures to alloys, catalyst and thermocouple wire.

V. CONCLUSION

2, 4-dimethyl -3H- 1, 5 benzodiazepine (DBA)has been proved to be a sensitive and selective spectrophotometric reagent for extraction of rhodium (III) metal ions. The method developed is simple, sensitive, selective, reproducible and rapid with low reagent concentration. Thequantitative extraction was carried out in a single step. Methodis free from interferences from a large number of cations and anions. Separation of rhodium(III) from synthetic mixtures corresponding to iridium alloy, platinum-palladium catalyst and thermocouple wire is carried out

VI. ACKNOWLEDGEMENT

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VIII. CONFLICT OF INTERESTS

The author hereby state that no sort of conflict of interest associated to this work.

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