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Extractive Spectrophotometric Determination of Palladium (ii) with 2-hydroxy-5-methylacetopheneoneisonicotinoylhydrazone (HMAINH)

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ABSTRACT

2-hydroxy-5-methylacetophenoneisonicotinoylhydrazone (HMAINH) is used as new reagent for spectrophotometric determination of palladium(II). The method is simple, rapid and highly sensitive and reagent forms a 1:1 yellow coloured complex with Pd (II). The complex shows absorption maximum at 385 nm, where as absorption due to reagent is negligible. The extraction was carried out at 0.010-0.015M sulphuric acid into chloroform. The Beer's law is obeyed over the concentration range of 2.0 - 9.0 ppm of palladium (II) at 385 nm. Optimum range as defined by Ringbom's plot was 3.0 – 7.0 ppm. The molar absorptivity and Sandell's sensitivity of extracted species are 5.320 x 10^3 dm^3 $mol^{-1}cm^{-1}$ and 0.02 µg cm^{-2} respectively. The standard deviation is 0.8755. The effect of various diverse ions on the estimation of Pd (II) has been studied.

Keywords: HMAINH, Extraction, Palladium determination, Spectrophotometry.

INTRODUCTION

Palladium is a lustrous silver-white metal. It forms many compounds and several complex salts. Palladium is the least dense and has lowest melting point of the platinum group metals. It is chemically attacked by sulphuric, nitric and hydrochloric acid in which it dissolves slowly. Many sensitive methods, such as spectrofluorometry, X-ray fluorescence spectrometry, neutron activation analysis, atomic absorption spectrophotometry have been used for determination of palladium. However spectrophotometric methods have gained popularity for palladium determination as advantageous in respect of simplicity and low operating costs. A wide variety of spectrophotometric reagent, such as 2-arylthio-p- nitroacetophenone[1] , 2-(2-quinolylazo)-5-

diethylaminobenzoic acid[2], *p*-Anisaldehyde thiosemicarbazone[3], 2-(2-Benzothiazolylazo)-5-dimethylamino-4-tolylarsonic acid[4] , 4-(2,6-diamino-4-pyrimidylazo)phenol[5] have been developed for determination of palladium.

Palladium has many applications and its separation and estimation at trace level of considerable importance. Determination by solvent extraction and spctrophotometric methods are ideal techniques and widely used for determination of palladium[6-11]. But to the best of our knowledge no work seems to have been done using 2 hydroxy-5-methyl - acetophenoneiso - nicotinoylhydrazone (HMAINH). Therefore, it was thought of interest to develop suitable and economical viable method for the determination of palladium with the above reagent. In the present paper the extraction and spectrophotometric determination of Pd(II) with HMAINH is reported. The use of this reagent may prove to be advantageous as it can be synthesized at low cost with high yield and of best purity.

MATERIAL AND METHODS

Instruments and Chemicals

A digital Systronics 335 *p*H meter with combined glass electrode and a Systronics 106 spectophotometer were used for *p*H and absorbance measurements. All chemicals used were of analytical grade. A stock solution of Palladium(II) (1 mg/mL) was prepared by dissolving Palladium chloride in double distilled water and concentrated hydrochloric acid (1M). Solutions of lower concentration were obtained by appropriate dilution as required. The stock solution of Palladium(II) was standardized[12]. 2-Hydroxy-5-methylacetophenoneisonicotinoylhydrazone (HMAINH) was synthesized by known method[13] and a 0.01 M HMAINH solution was prepared by dissolving HMAINH in dimethylformamide and volume make up by ethanol in volumetric flask.

Recommended method

An aliquot of sample solution containing 70 µg of Pd (II) and 1 mL of 0.01 M reagent was taken and acidity was adjusted to 0.01 – 0.015 M with sulphuric acid in 25 ml volumetric flask. The solution was transferred into 125 mL separating funnel and equilibrated for 60 to 90 sec with 10 ml chloroform solvent. The two phases were allowed to separate and dried over anhydrous sodium sulphate, the absorbance of the organic phase was measured against chloroform at 385 nm. The palladium(II) content was computed from a calibration graph.

RESULTS AND DISCUSSION

Absorption Spectrum

The absorption spectrum of the Pd(II) - HMAINH complex in chloroform shows the absorption maxima at 385 nm, whereas absorption due to reagent is nearly negligible (Figure 1). Therefore, all the absorbance measurement was taken at 385 nm against the reagent blank in spectrophotometric determination of palladium.

Effect of acidity

The extraction of 70 µg of palladium was carried out from different acid media with 1 mL 0.01 M reagent (HMAINH) and chloroform. Since there was no complexation in HNO_3, $HClO_4$ and

HCl media all of the extractions were carried out at 0.01 M H_2SO_4. The extraction of palladium(II) was investigated from 0.005 M to 0.03 M sulphuric acid. The extraction was found to be complete (Figure 2). Hence extraction of palladium (II) was carried out at 0.010 – 0.015 M sulphuric acid.

Choice of extraction solvent

Various organic solvents were examined for the extraction of Palladium- HMAINH complex and it was observed that the extraction of Palladium(II) complex was quantitative in chloroform. The solvents can be arranged in the decreasing order of their extraction coefficients as chloroform 〉 xylene〉 toluene〉 carbon tetrachloride 〉 benzene〉 hexane.

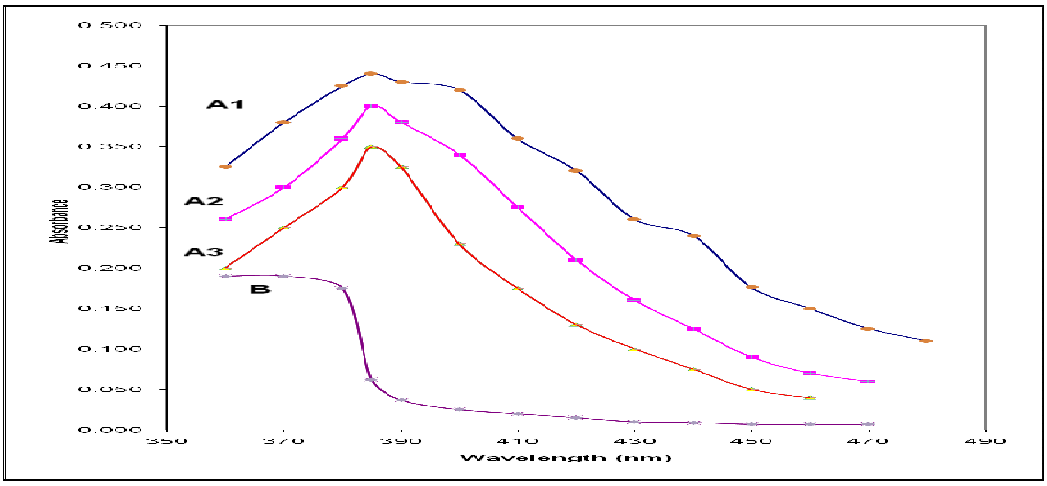

Figure. 1 A – Absorption Spectrum of Pd(II) – HMAINH against Reagent Blank, (A1 - 8.00 ppm, A2 - 7.00 ppm , A3 - 6.0 ppm)
B -- Absorption Spectrum of HMAINH Against chloroform

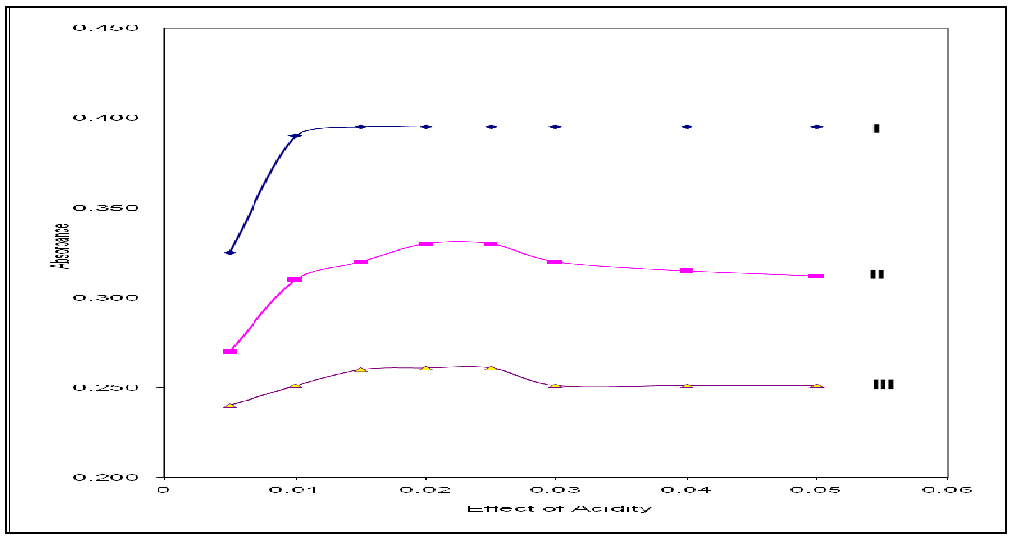

Figure 2 – Effect of Acidity on Extraction of Pd(II) – HMAINH Complex
I - H_2SO_4 , II - $HClO_4$, III – HCl

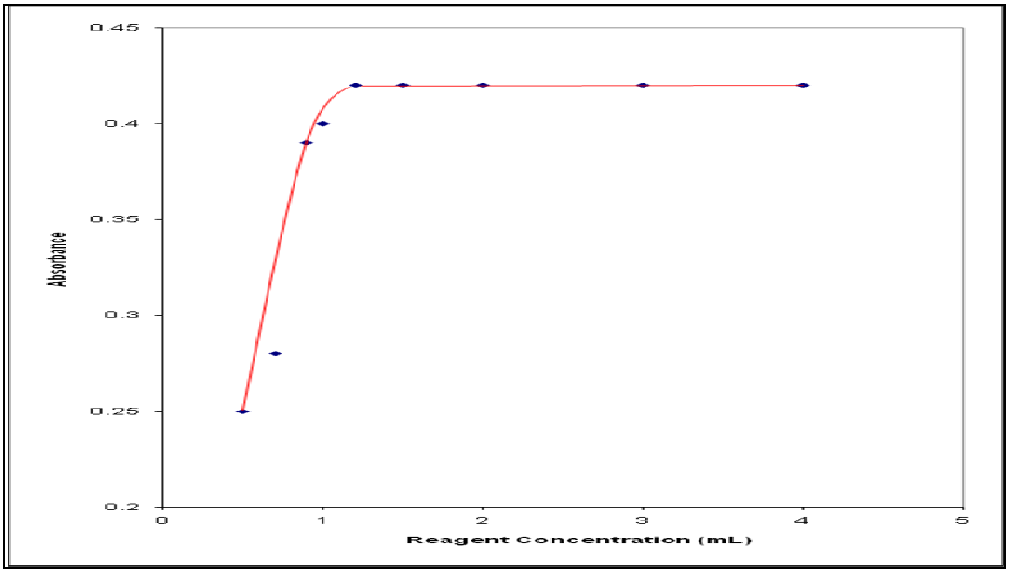

Figure 3 - Effect of Reagent HMAINH Concentration On the Absorbance of Pd(II) in Chloroform

Effect of reagent concentration

The effect of reagent concentration was studied by varying the 0.01 M HMAINH concentration. It was observed that absorbance remains constant from 1 to 4 ml (Figure 3). Hence 1 ml of reagent concentration was recommended for determination of Palladium(II) for the further studies.

Equilibrium time and stability

The study of change in absorbance with variation in equilibrium time required for the complete extraction is 60 s to 90 s. The extracted complex was found to be stable for 24 h.

Beer's range and sensitivity

Beer's law is obeyed over the concentration range of 2.0 - 9.0 ppm of Palladium at 385 nm (Figure 4). Optimum range as defined by Ringbom's plot was 3.0 – 7.0 ppm . The molar absorptivity and Sandell's sensitivity were calculated to be 5.320 x 10^3 dm^3 $mol^{-1}cm^{-1}$ and 0.02 µg cm^{-2} respectively at 385 nm.

Nature of extracted species

The composition of the extracted species was determined by carrying out Job's continuous variation method (Figure 5) and was further confirmed by mole ratio method (Figure 6). It was found to be 1 : 1.

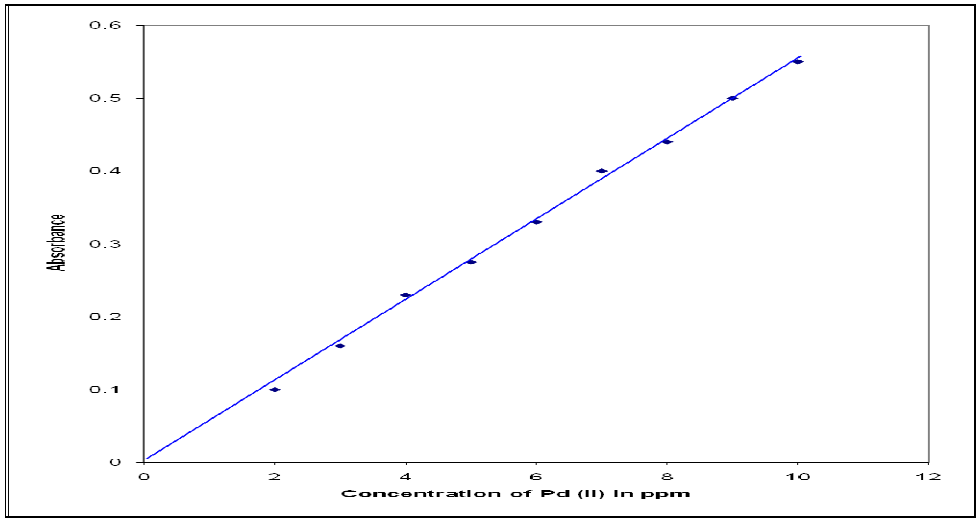

Figure 4 – Calibration Plot Of Pd(II) with HMAINH (Beer's Law)

Effect of diverse Ion

The effect of the various foreign ions were investigated in order to find tolerance limit of these ions in the extraction of Pd(II). No interference was observed in the presence of following ions at the amounts (mg mL^{-1}) shown as Ni(II) (20) , Cu(II) (20), Zn(II) (20), V(V) (10), Co(II) (3), Citrate(200), Acetate(200), Bromide(10).

Precision and accuracy

For the study of reproducibility and accuracy of the method, absorbance measurement with ten different identical solutions containing 70 µg of palladium was determined. Average of these ten readings and standard deviation was calculated. The standard deviation was found to be 0.8755. From standard deviation reproducibility of results with 95% confidence limit was 70.10 ± 0.625.

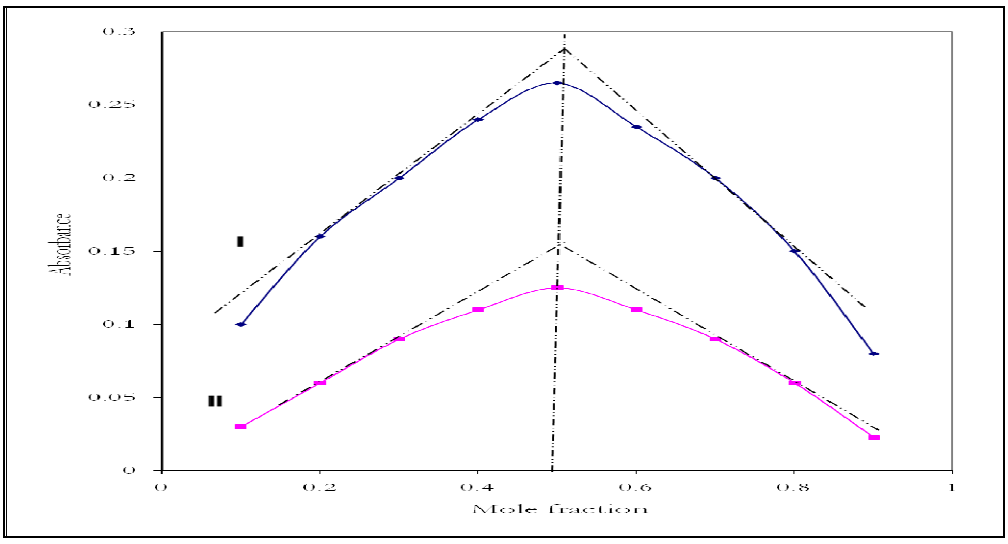

Figure 5: - Pd(II) : HMAINH Species by Job's Continuous Method

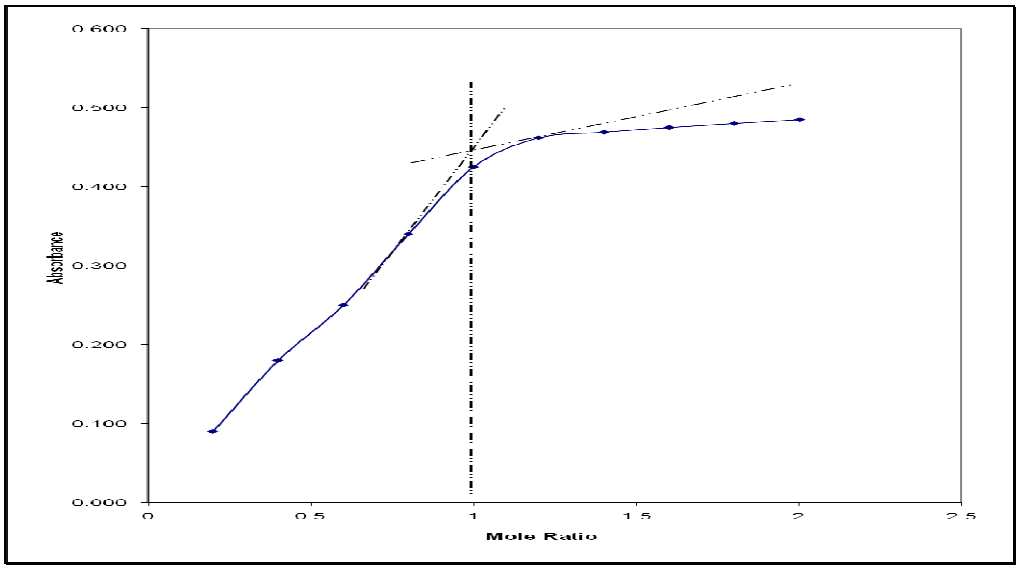

Figure 6- Mole Ratio for Pd(II) - HMAINH Species

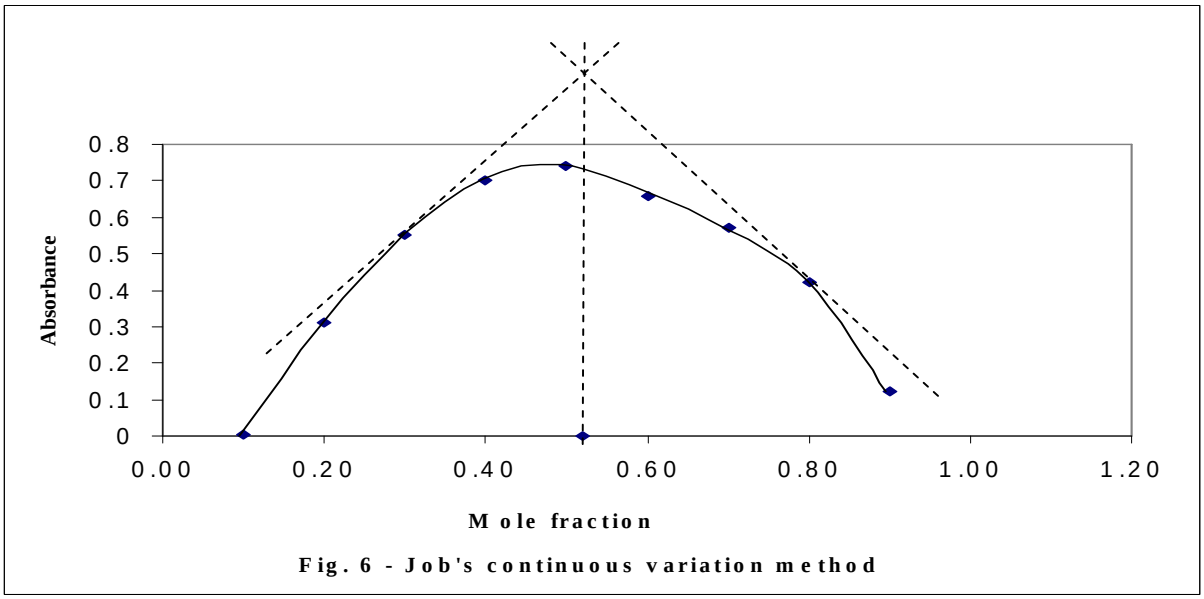

Fig. 6 - Job's continuous variation method

CONCLUSION

The 2-hydroxy-5-methylacetophenoneisonicotinoylhydrazone (HMAINH) was used as the reagent for the extractive spectrophotometric determination of palladium(II) from sulphuric acid media. The above reagent provides a simple, rapid and accurate method for spectrophotometric determination of palladium (II). It has advantages of high sensitivity, selectivity and easy availability.

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