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Research Article

Solvent Extraction and Spectrophotometeric Determination of U (VI) By Using Acetophenone 2', 5'-Dihydroxy, Semicarbazone as an Analytical Reagent

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ABSTRACT

A spectrophotometric method has been developed for the determination of U(VI) using Acetophenone 2',5'dihydroxy, semicarbazone[1] as an extractive reagent .The reagent form a colored complex which has been quantitatively extracted into n-Butanol at pH 6.0. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is 6785.7 L $mol^{-1}cm^{-1}$ and Sandell's sensitivity is 0.03508 µg cm^{-2} respectively. The propose method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.

Keywords: Uranium, Spectrophotometric determination, n-Butanol, Acetophenone 2', 5'-dihydroxy, semicarbazone derivative.

1. INTRODUCTION

The cursory look at the literature survey reveals the fact that Uranium reacts with many organic reagent it also indicate that some of the reagent recommended suffering through limitations such as interference of Cr(VI), Mo(VI), [1, 2] complex formation takes place after several minutes [3], also some of the regents are not selective [4-6] and sensitive, tedious [7]. In this paper a new method has been developed using Acetophenone 2',5'dihydroxy, semicarbazone [ADHS] for extraction and Spectrophotometric determination of uranium U(VI), which is simple, selective and sensitive.

2. MATERIAL AND METHODS

The reagent Acetophenone 2',5'-dihydroxy semicarbazone was synthesized by the given procedure. The stock solution of U (VI) was prepared by dissolving a weighed amount of uranyl nitrate in double distilled water and then diluted to the desired volume with double distilled water and standardized by oxine method. Absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode respectively.

2.1. Procedure For The Extraction

1.0 ml of aqueous solution containing 1µg of uranium metal and 2 ml of reagent were mixed in a 50 ml beaker. The pH of the solution adjusted to 6.0, it must be noted that the total volume should not exceed 10 ml. The solution was transferred to 100 ml separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 ml measuring flask and made up to the mark with organic solvent if required. The amount of uranium present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 380 nm and that in the aqueous phase was determined by oxine method.

3. RESULTS AND DISCUSSION

The results of various studies are discussed below.

3.1. Extraction as a function of pH

The extraction uranium with Acetophenone 2',5'-dihydroxy, semicarbazone has been studied over the pH range 1-10 and was observed that percentage extraction of U (VI) is maximum at pH 6.0. (Fig. 1)

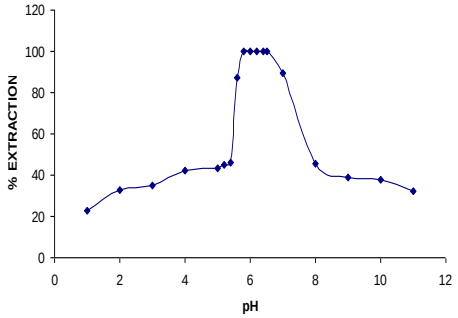

Fig. 1: Effect of pH on % Extraction

3.2. Absorption spectrum

The absorption spectrum of U (VI): Acetophenone 2',5'-dihydroxy semicarbazone in n-butanol shows the maximum

absorption at 380 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 380nm.

3.3. Influence of diluents

The suitability of solvent was investigated using various organic solvents and the extraction of U(VI):ADHS was quantitative in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

3.4. Effect of reagent concentration

It was found that 2 ml of 0.1% reagent is sufficient for the colour development of the metal U (VI) in 10 ml of aqueous solution at pH 6.0.

3.5. Effect of equilibration time and stability of the complex

The equilibration time of 1 minute is sufficient for the quantitative extraction of uranium. The stability of colour of the U (VI): ADHS complex with respect to time shows that the absorbance due to extracted species is stable up to 22 hours, after which slight decrease in absorbance is observed.

3.6. Calibration plot

The Beer's law is obeyed from 1 to 10 ppm. The molar absorptivity and sandell's sensitivity were calculated to be is 6785 L .mol^{-1}cm^{-1} and 0.03508 µg cm^{-2} respectively. (Fig. 2)

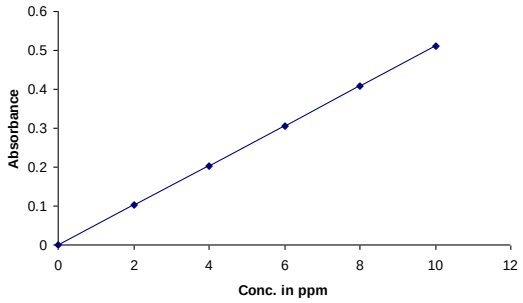

Fig. 2: Calibration curve

3.7. Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10 ppm of uranium. The ions which show interference in the spectrophotometric determination of uranium were overcome by using appropriate masking agents.

3.8. Precision and accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10µg of uranium in the aqueous phase. The average of ten determinations was 10.00351 and variation from mean at 95% confidence limit was ±0.01846.

3.9. Nature of extracted species

The composition of extracted U(VI):ADHS complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of U (VI): ADHS complex is 1:2. (Fig. 3).

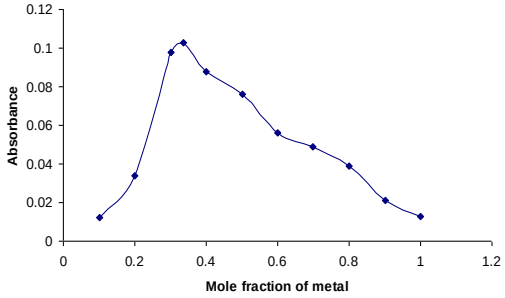

Fig. 3: Jobs variation method

Table 1: Use of masking agent

Interfering Ion	Masking agent
Cu (II)	Sodium thiosulphate
Fe(III)	Thiourea
Ce(IV)	Sodium fluoride
Cr (II)	Ammonium acetate
Mo (VI)	Citrate
EDTA	Boiled with concentrated HNO_3
CN-	Boiled with concentrated HNO_3 and formaldehyde

4. APPLICATION

The proposed method was successfully applied for the determination of uranium from various synthetic mixtures. The results found to be in good agreement with those obtained by the standard known method.

5. REFERENCES

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