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SOLVENT EXTRACTION AND SPECTROPHOTOMETERIC DETERMINATION OF Ce(IV) 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 Ce(IV) 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 4.0. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is 2564.1 L $mol^{-1}cm^{-1}$ and Sandell's sensitivity is 0.02484 µg cm^{-2} respectively. The propose method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.

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

INTRODUCTION

The cursory look at the literature survey revels the fact that Cerium reacts with many organic reagent it also indicate that some of the reagent recommended suffering through limitations such as interference of Mn(VII), Ti(V), V(V), Thorium[2] etc. In some cases complex formation takes place after several minutes [3, 4] also some of the regents are not selective[5,6] and sensitive. In this paper a new method has been developed using Acetophenone 2',5'dihydroxy, semicarbazone [ADHS] for extraction and Spectrophotometric determination of Cerium, Ce (IV), which is simple, selective and sensitive.

EXPERIMENTAL

The reagent Acetophenone 2', 5'- dihydroxy semicarbazone was synthesized by the given procedure. The stock solution of Ce (IV) was prepared by dissolving a weighed amount of ammonium cerric sulphate in double distilled water and then diluted to the desired volume with double distilled water and standardized by Arsenic (III) oxide method. The 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.

Procedure for the extraction

0.1 ml of aqueous solution containing 1µg of Cerium metal and 2 ml of reagent was mixed in a 50 ml beaker. The pH of the solution adjusted to 4.0, 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 Cerium 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 arsenic (III) oxide method.

RESULTS AND DISCUSSION

The results of various studies are discussed below.

Extraction as a function of pH

The extraction of Cerium with Acetophenone 2',5'-dihydroxy ,semicarbazone has been studied over the pH range 1-10 and was observed that percentage extraction of Ce (IV) is maximum at pH 4.0.(Fig-1).

Absorption spectrum

The absorption spectrum of Ce (IV): 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.

Influence of diluents

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

Effect of reagent concentration

It was found that 2 ml of 0.1% reagent is sufficient for the colour development of the metal Ce (IV) in 10 ml of aqueous solution at pH 4.0.

Effect of equilibration time and stability of the complex

The equilibration time of 1 minute is sufficient for the quantitative extraction of Cerium. The stability of colour of the Ce (IV): ADHS complex with respect to time shows that the absorbance due to extracted species is stable up to 45 hours, after which slight decrease in absorbance is observed.

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 2564.1 L $mol^{-1}cm^{-1}$ and 0.02484 µg cm^{-2} respectively. (Fig.-2).

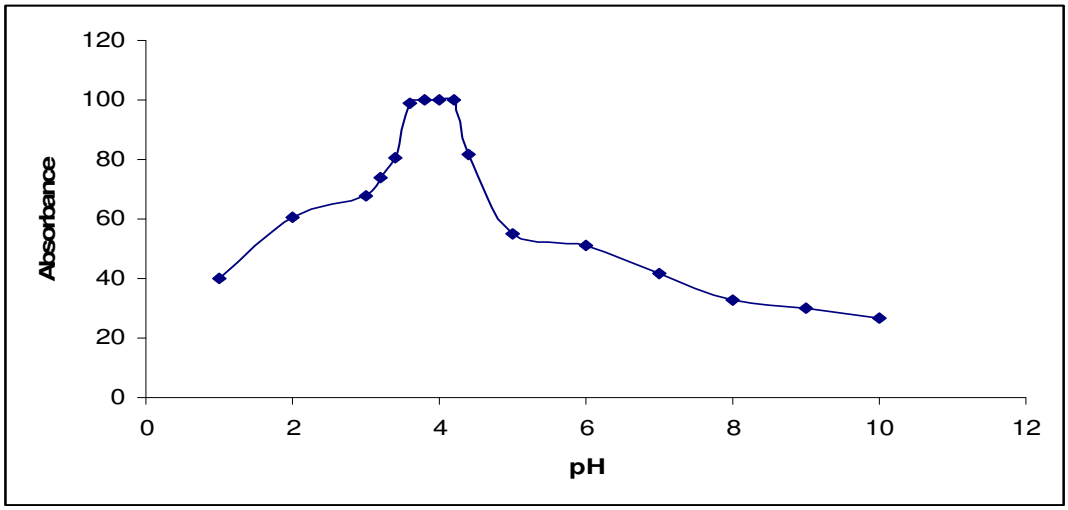

Fig.-1: Extraction as a function of pH

Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10ppm of Cerium. The ions which show interference in the spectrophotometric determination of Cerium were overcome by using appropriate masking agents. (Table-1)**.**

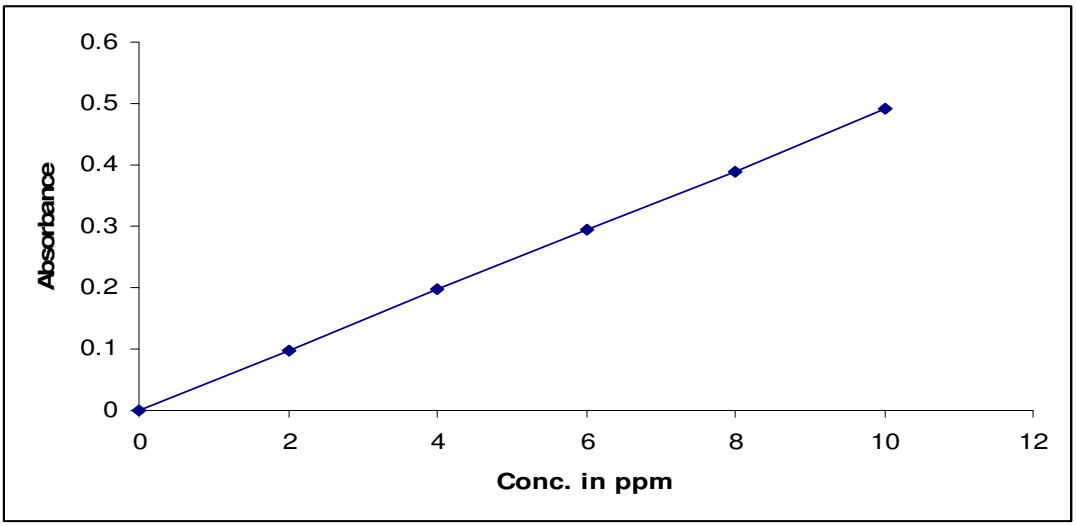

Fig.-2: Calibration plot

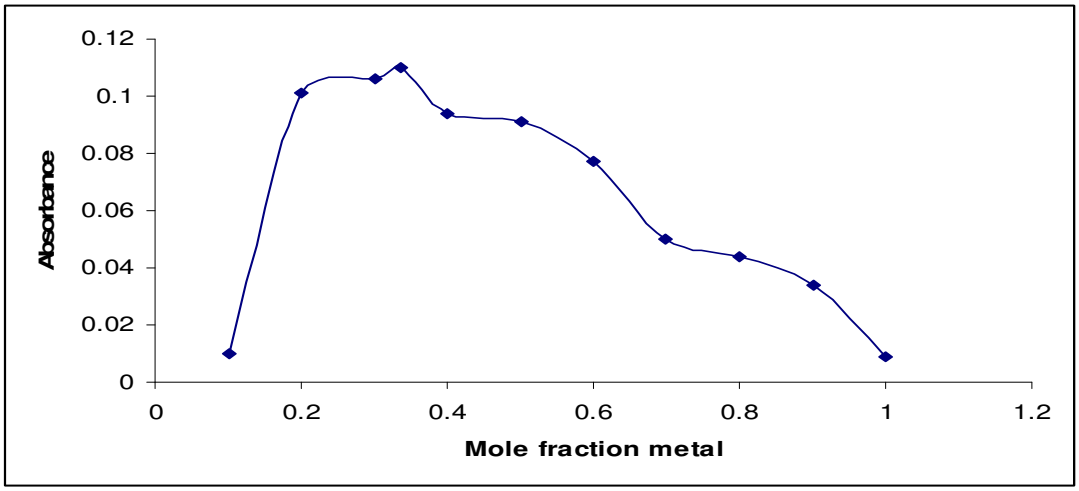

Fig.-3:Nature of extracted species

Precision and accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10µg of Cerium in the aqueous phase. The average of ten determinations was 10.0019 and variation from mean at 95% confidence limit was ± 0.01007.

Nature of extracted species

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

Application

The proposed method was successfully applied for the determination of Cerium from various alloys, ores and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method.

Table-1: Use of masking agent

S. No.	Interfering Ion	Masking agent
1	Cu (II)	Sodium thiosulphate
2	Fe(III)	Thiourea
3	U (VI)	8-Hydroxy quinoline

4	Cr (II)	Ammonium acetate
5	Mo (VI)	Citrate
6	EDTA	Boiled with concentrated HNO_3
7	CN^-	Boiled with concentrated HNO_3 and formaldehyde

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