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International Journal of Current Research
Vol. 9, Issue, 04, pp.49215-49217, April, 2017

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

ISSN: 0975-833X

RESEARCH ARTICLE

SPECTROPHOTOMETRIC DETERMINATION OF TRACE AMOUNT OF CE (IV) METAL ION BY USING 3-(2-HYDROXYPHENYLIMINO) INDOLIN-2-ONE AS AN PHOTOMETRIC REAGENT, HPI2O

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ARTICLE INFO

Article History:
Received 16th January, 2017
Received in revised form
18th February, 2017
Accepted 05th March, 2017
Published online 30th April, 2017

Key words:
Spectrophotometric Method,
Ce (IV) Metal,
Reagent HPI2O,
Complex,
N-Butanol Etc.

ABSTRACT

A 3-(2-Hydroxylphenylimino) indolin-2-one [HPI2O] is use as a reagent for the extractive spectrophotometric determination of Cerium. The reagent HPI2O gave instantaneous and stable brown colour complex with Cerium at pH 5.0. The colour reaction in detail has been explored and the possibility of photometric determination of the micro amounts of Cerium is established with necessary conditions. A linear calibration graph over the concentration range 1 ppm to 12 ppm was obtained by applying the spectrophotometric method at wavelength 430 nm. The stoichiometry of the complex is established as 1:1 (M:L) by Job's method of continuous variation and confirmed by mole ratio method. The Sandell's Sensitivity is 0.0303 µg cm^{-2} with molar absorptivity 4623.92 L mol^{-1} cm^{-1}. The results of the prescribed procedure applied for the determination of the micro amounts of Ce (IV) in various synthetic and standard steel samples are presented.

Citation: Vijay J. Ghodvinde, Pitale, S. M., Janwadkar, S.P., Prashant P. Lohani, Rana, P.K. and Yadav, D.K. 2017. "Spectrophotometric Determination of Trace Amount of Ce (IV) metal ion by Using 3-(2-HydroxyPhenylimino) indolin-2-one as an Photometric Reagent, HPI2O", *International Journal of Current Research*, 9, (04), 49215-49217.

INTRODUCTION

Cerium is a chemical element with symbol Ce and atomic number 58. Cerium is an inner transition element placed in group III B and sixth period of periodic table. Cerium is also known as 4f block element. It is a soft, silvery, ductile metal which easily oxidizes in air. Cerium was named after the dwarf planet Ceres (itself named for the Roman goddess of agriculture). Commercial applications of cerium are numerous. They include catalysts, additives to fuel to reduce emissions and to glass and enamels to change their color. Cerium oxide is an important component of glass polishing powders and phosphors used in screens and fluorescent lamps. It is also used in the "flint". Cerium can act as similar to calcium in organism so accumulate in bone in small amount. It can stimulate metabolism. It is found in tobacco plants, barely etc.

EXPERIMENTAL

The pH measurements were made using a pH meter Elico, Model LI-129, India in conjugation with a combined glass and calomel electrode. Shimadzu UV-Visible 2100 spectrophotometer with 1.0 cm matched quartz cells were used for all absorbance measurements.

Reagent and chemicals

0.1% HPI2O reagent is prepared by dissolving the requisite amount of HPI2O in a known volume of ethanol. All chemicals used were of analytical-reagent grade or the highest purity available. Doubly distilled de-ionized water and A.R. grade ethanol, which is were used throughout.

Ce (IV) standard solutions

The stock solution of cerium(IV) was prepared by dissolving weighed amount of ammonium cerric sulphate in doubly distilled de-ionized water. More dilute standard solutions were prepared from this stock solution as and when required.

PROCEDURE FOR THE EXTRACTION

1 mL of aqueous solution containing 10 µg of cerium metal and 1 mL of reagent was mixed in a 50 mL beaker (Janwadkar, 2011). The pH of the solution adjusted to 5.0, it must be noted that the total volume should not exceed 10 mL. The solution

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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 (Vogel, 1957). The amount of cerium present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 430 nm and that in the aqueous phase was determined by EDTA method (Vogel, 1991; Patil, 2016).

RESULTS AND DISCUSSION

The results of various studies are discussed below.

Extraction as a function of pH

The extraction of cerium with 3-(2-Hydroxylphenylimino)indolin-2-one has been studied over the pH range 1-10 and was observed that percentage extraction of Ce(IV) is maximum at pH range 4.0-6.0. Hence, further extraction and determination carried out at pH 5.0(Fig 1).

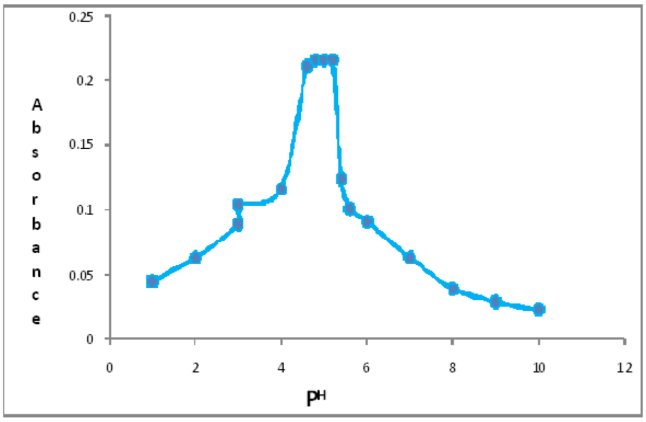


Fig. 4. Extraction as a function of pH

Absorption spectrum

The absorption spectrum of Ce (IV): HPI2O in n- Butanol shows the maximum absorption at 430 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 430 nm (Fig 2).

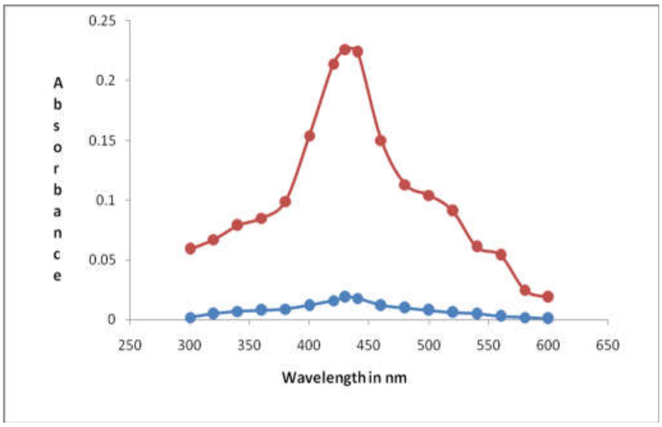


Fig. 4. Absorption spectrum

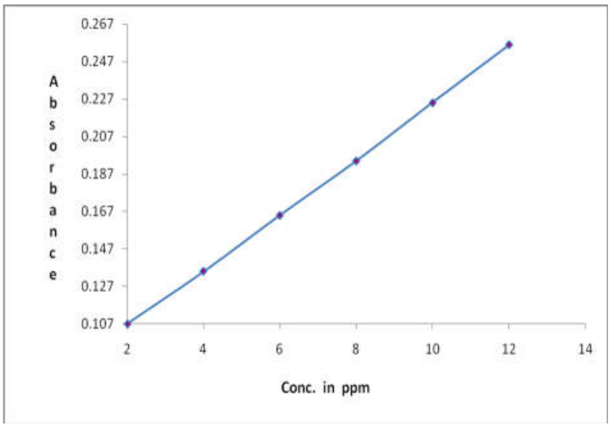
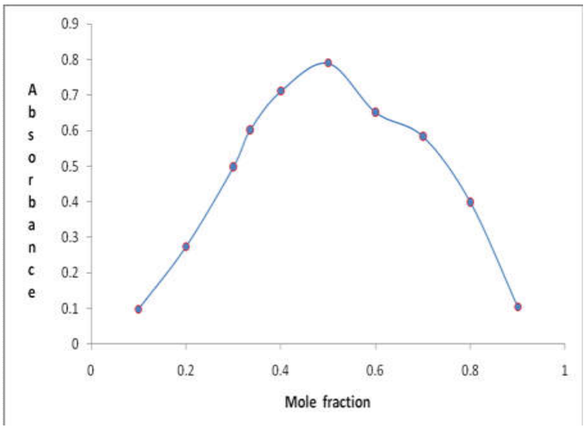


Fig. 3. Calibration plot



Job continuous variation

Fig. 4. Nature of extracted species

Table 1. Use of masking agent

Sr. No.	Interfering Ion	Masking agent
1	Cu(II)	Sodium thiosulphate
2	Ni (II)	DMG
3	Fe(III)	Thiourea
4	EDTA	Boiled with concentrated HNO_3
5	CN^-	Boiled with concentrated HNO_3 and formaldehyde

Table 2. Applications

Sr.No	Mixtue Taken (ppm)	Cerium added (ppm)	Cerium found (ppm) by present, method	Cerium found (ppm) by known, method
1	Ce(5)+La(40)+Nd(10) + Sm (15)	5.0	5.02	4.99
2	Ce (5) +Pr(15) + Zr (4)	5.0	5.02	5.03
3	Ce (4) + Cd (15) + Mg (10)	4.0	3.96	4.02

Influence of diluents

The suitability of solvent was investigated using various organic solvents and the extraction of Ce(IV)**:**HPI2O 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 1 mL of 0.1% reagent is sufficient for the colour development of the metal Ce(IV) in 10 mL of aqueous solution at pH 5.0.

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):HPI2O complex with respect to time shows that the absorbance due to extracted species is stable up to 60 hours, after which slight decrease in absorbance is observed.

Calibration plot

The Beer's law is obeyed from 1 to 12 ppm. The molar absorptivity and sandell's sensitivity were calculated to be is 4623.92 L $mol^{-1}cm^{-1}$ and 0.0303 µg cm^{-2} respectively (Fig III).

LOD

LOD[10] (Limit of Detection) of the present method was calculate at 98.3 % confidence level, it was 0.2032 µg/mL.

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 Cerium. The ions which show interference in the spectrophotometric determination of cerium were overcome by using appropriate masking agents. (Table 1)

Precision and accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10 µg of cerium in the aqueous phase. The average of ten determinations was 10.005 and variation from mean at 95% confidence limit was 10.005±0.02378.

Nature of extracted species

The composition of extracted Ce(IV):HPI2O 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):HPI2O complex is 1:1. (Fig IV).

APPLICATION

The proposed method was successfully applied for the determination of cerium from various synthetic samples. The results found to be in good agreement with those obtained by the standard known method. (Table II)

Synthetic mixture:- The separation of cerium(IV) from synthetic mixture of associated metals containing La(III), Nd(III), Sm(III), Cd(II) and Mg(II) with varying combination was carried out. A definite aliquot of this solution was taken and after the adjustment of acidity of the aqueous solution to pH 5.0 and addition of 1 ml of 0.1% HPI2O solution, the cerium complex formed was extracted into 10 ml of n-Butanol. The amount cerium present was computed using the calibration curve method. The result obtained is compared with those obtained by standard method.

Conclusion

The results obtained show that the newly developed method in which the reagent HPI2O was used, can be effectively used for quantitative extraction and estimation of Ce(IV) from aqueous media. The proposed method is quick and requires less volume of organic solvent. The result show good agreement with the standard method. The method is very precise, faster and simpler than other methods. The developed method is compared with result obtained with the arsenic (III) oxide method for the estimation cerium (IV) and observed to be comparable. The method is precise, accurate, less time consuming and easily employed anywhere, even in small laboratories as it requires only uv – visible spectrophotometer and not much sophisticated and costly measurement devices or instrumentation.

Acknowledgement

I take this opportunity to thank my research guide Dr. S. M. Pitale also Dr.Hemant Pednekar Principal of S.D.S.M, College Palghar who provide me laboratory facilities. I am also thankful to my research co-guide Dr. Suhas Janwadkar who helped me to enter the research world. I thankful to Dr. Paritosh Rana, Dr. D. K. Yadav, Dr. Vaibhava More who helped me in many ways. Last but not the least I thank my family members who stood behind me in each and every situation.

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