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Spectrophotometric Determination of Trace Amount of Th(IV)metal ion by Using 3-(2-HydroxyPhenylimino)indolin-2-one (HPI2O) As An Photometric Reagent

Vijay J. Ghodvinde*, S. M. Pitale, S.P.Janwadkar, Prasant P.Lohani, P.K.Rana
Department of Chemistry,
S.D. Arts, V.S.Apte. Commerce and M.H.Mehta, science College, Palghar, 401404
Mumbai University

Abstract: A 3-(2-Hydroxylphenylimino)indolin-2-one [HPI2O] is use as a reagent for the extractive spectrophotometric determination of Thorium. The reagent HPI2O gave instantaneous and stable violet coloured complex with Thorium at pH 7.6. The colour reaction in detail has been explored and the possibility of photometric determination of the micro amounts of Thorium is established with necessary conditions. A linear calibration graph over the concentration range 1 ppm to 10 ppm was obtained by applying the spectrophotometric method at wavelength 430 nm. The stoichiometry of the complex is established as 1:2 (M:L) by Job's method of continuous variation and confirmed by mole ratio method. The Sandell's Sensitivity is 0.09302 µg cm^{-2} with molar absorptivity 4988.8 L mol^{-1} cm^{-1}. The results of the prescribed procedure applied for the determination of the micro amounts of Th(IV) in various synthetic samples are presented.

Keywords:- Spectrophotometric method, Th(IV) metal, reagent HPI2O, complex, n-butanol etc.

1.INTRODUCTION:-

Thorium is a chemical element with symbol Th and atomic number 90. Thorium is an inner transition element placed in group III B and seventh period of periodic table. Thorium is also known as 5f block element. Thorium metal is silvery and tarnishes black when exposed to air, forming the dioxide. Thorium is weakly radioactive: all its known isotopes are unstable. Thorium-232 (^{232}Th), which has 142 neutrons, is the most stable isotope of thorium and accounts for nearly all natural thorium, with six other natural isotopes occurring only as trace radioisotopes. Thorium has the longest half-life of all the significantly radioactive elements, 14.05 billion years. thorium and uranium are the only significantly radioactive elements with major commercial applications that do not rely on their radioactivity. Thorium is predicted to be able to replace uranium as nuclear fuel in nuclear reactors, but only a few thorium reactors have yet been completed. Thorium was once commonly used as the light source in gas mantles and as an alloying material, but these applications have declined due to concerns about its radioactivity. Thorium is still widely used as an alloying element in TIG welding electrodes ,at a rate of 1–2% mix with tungsten. (Tata McGraw-Hill Education ,2013)

2.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.

2.1 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.

2.2 Th (IV) standard solutions

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

3. PROCEDURE FOR THE EXTRACTION:

1 mL of aqueous solution containing 10 μg of Thorium metal and 1 mL of reagent was mixed in a 50 mL beaker (S.P.Janwadkar,2011). The pH of the solution adjusted to 7.6, 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(Vogel,1957). The amount of Thorium 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; Yadav D.K.,2012).

4. RESULTS AND DISCUSSION:

The results of various studies are discussed below.

4.1 Extraction as a function of pH:

The extraction of Thorium with 3-(2-Hydroxylphenylimino)indolin-2-one has been studied over the pH range 1-10 and was observed that percentage extraction of Thorium is maximum at pH range 7.2-7.8. Hence, further extraction and determination carried out at pH 7.6 (Fig I).

4.2 Absorption spectrum:

The absorption spectrum of Thorium**:**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 II).

4.3 Influence of diluents:

The suitability of solvent was investigated using various organic solvents and the extraction of Thorium**:**HPI2O was quantitative in n-Butanol. Hence, n-Butanol was used for further extraction studies as it gave better and quicker phase separation.

4.4 Effect of reagent concentration:

It was found that 1 mL of 0.1% reagent is sufficient for the colour development of the metal Thorium in 10 mL of aqueous solution at pH 7.6.

4.5 Effect of equilibration time and stability of the complex:

The equilibration time of 1 minute is sufficient for the quantitative extraction of Thorium. The stability of colour of the Thorium:HPI2O complex with respect to time shows that the absorbance due to extracted species is stable up to 49 hours, after which slight decrease in absorbance is observed.

4.6 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 4988.8 L mol^{-1} cm^{-1} and 0.09302 µg cm^{-2} respectively (Fig III).

4.7 LOD:

LOD[10] (Limit Of Detection) of the present method was calculate at 98.3 % confidence level, it was 0.2085 ppm

4.8 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 Thorium. The ions which show interference in the spectrophotometric determination of Thorium were overcome by using appropriate masking agents. (Table I)

4.9 Precision and accuracy:

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing six solutions each containing 6 μg of Thorium in the aqueous phase. The average of ten determinations was 6.006 and variation from mean at 95% confidence limit was 6.006 ± 0.02321.

4.10 Nature of extracted species:

The composition of extracted Thorium:HPI2O complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Thorium:HPI2O complex is 1:2. (Fig IV).

5. APPLICATION:

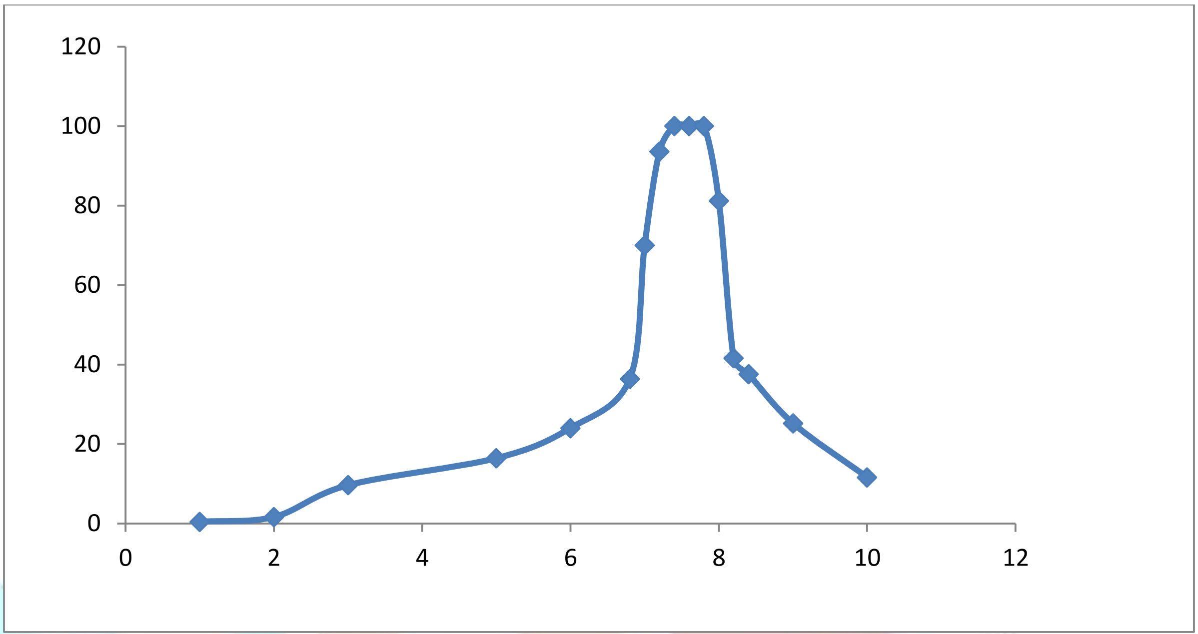
The proposed method was successfully applied for the determination of Thorium from various synthetic samples. The results found to be in good agreement with those obtained by the standard known method. (Table II)

Synthetic mixture:- The separation of Thorium(IV) from synthetic mixture of associated metals containing Al(III), Cr(III), Mn(II), and Zn(II) with varying combination was carried out. A definite aliquot of this solution was taken and after the adjustment of the aqueous solution to pH 7.6 and addition of 1 ml of 0.1% HPI2O solution, the Thorium complex formed was extracted into 10 ml of n-Butanol. The amount Thorium present was computed using the calibration curve method. The result obtained is compared with those obtained by standard method.

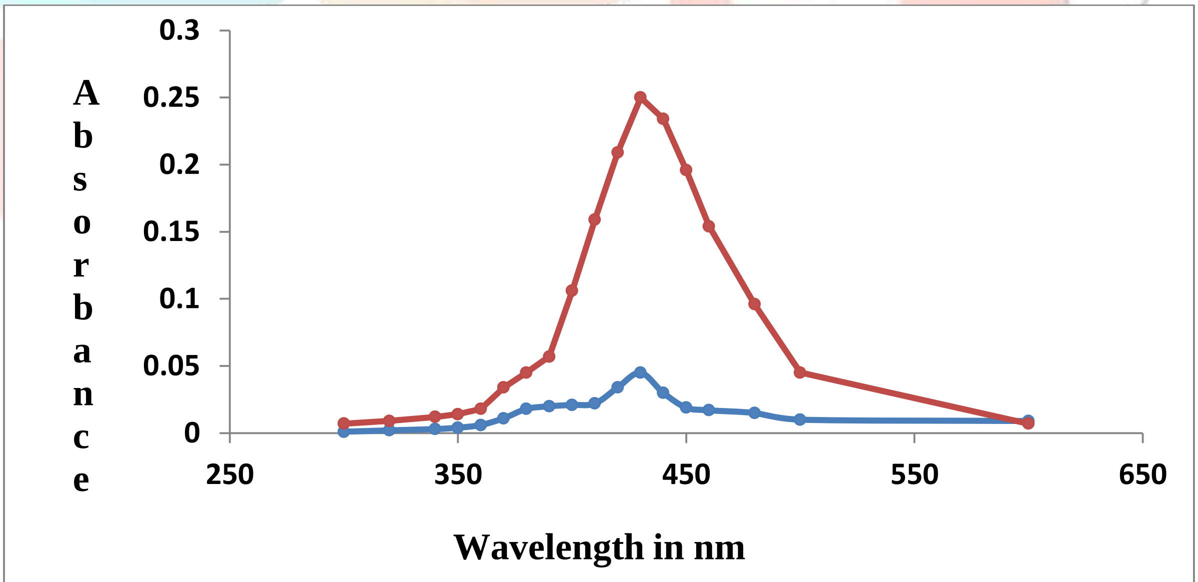
6. CONCLUSION

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 Thorium from aqueous media. The proposed method is quick and requires less volume of organic solvent. The results show good agreement with the standard method. The method is very precise, faster and simpler than other methods.

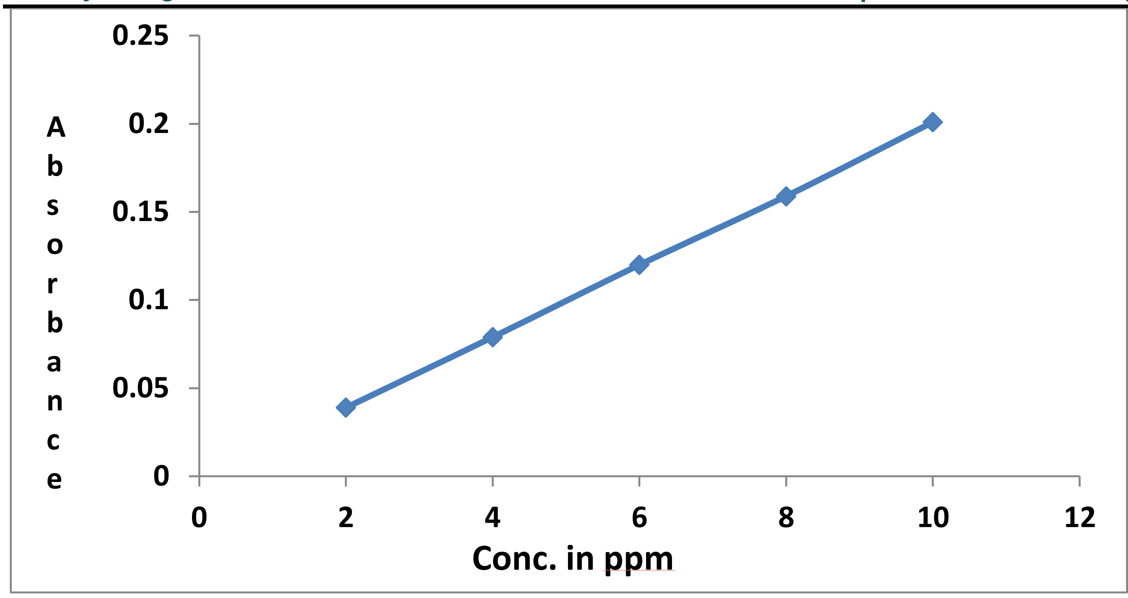
The method is accurate, less time consuming and easily employed anywhere, even in small laboratories as it requires only uv – visible spectrophotometer and not much sophisticated and costly measurement devices or instrumentation.



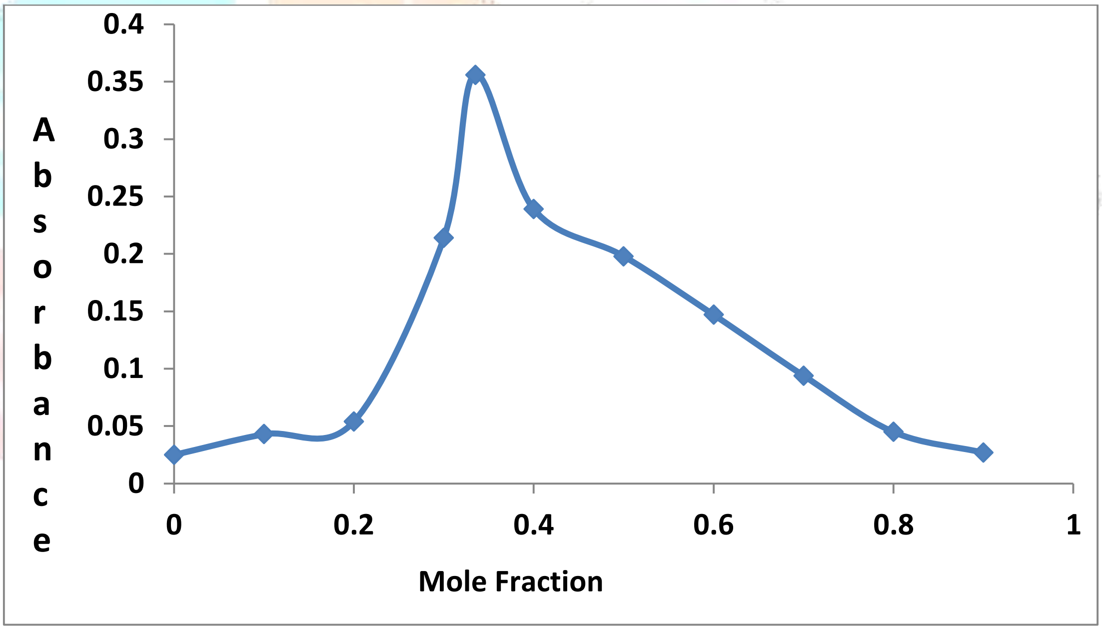
Extraction as a function of pH (Fig I)



Absorption spectrum (Fig. II)



Calibration plot (Fig III)



Job continuous variation

Nature of extracted species (Fig IV)

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

Use of masking agent (Table I)

Sr.No	Mixtue Taken (ppm)	Thorium added (ppm)	Thorium found (ppm) by present , method	Thorium found (ppm) by known , method
1	**Th(IV) (5) + Al (III) (5)**	**4.97 ppm**	**4.97 ppm**	**Th(IV) (5) + Al (III) (5)**
2	**Th(IV) (5) + Zn (II) (5)**	**4.89 ppm**	**4.85 ppm**	**Th(IV) (5) + Zn (II) (5)**
3	**Th(IV) (5) + Mn (II) (5)**	**4. 92ppm**	**4.89 ppm**	**Th(IV) (5) + Mn (II) (5)**
4	**Th(IV) (5) + Cr(III) (5)**	**4.95 ppm**	**4.92 ppm**	**Th(IV) (5) + Cr(III) (5)**

Applications (Table II)

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