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NOVEL SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF TH(IV) USING 2,4 - DMBINH

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ABSTRACT

A simple, selective and accurate method was proposed for the determination of Th(IV) in microgram quantities using 2,4-DMBINH. The 2,4-DMBINH give a yellow coloured solution in basic medium with Th(IV). The maximum absorbance is at 398.4 nm. The reagent forms a 1:1 complex with Th(IV). The stability constant of the complex is $3.3x10^6$.

KEYWORDS: Spectrophotometric method, 2,4-DMBINH and Th(IV).

1) INTRODUCTION

Thorium occurs in solutions exclusively in +4 oxidation state. It is a silvery white, soft metal. Thorium is soluble in acids and reacts so slowly with oxygen forming Thorium dioxide.

2,4 - dimethoxy benzaldhyde isonicotinoy hydrazone (2,4 - DMBINH) play and important role in medicinal chemistry. They are used as their complexes with metal ions for the treatment of number of diseases. As there organic compounds contain nitrogen as well as oxygen, they have ability to form complexes easily with many metal ions. The hetero atoms, nitrogen as well as oxygen can form co-ordinate bonds with many metal ions and thus, form stable, highly coloured, soluble or insoluble complexes.

2) EXPERIMENTAL

2.1) Reagent and Materials

Acetic acid, Ammonium chloride, Sodium acetate, Sulphuric acid, Hydrochloric acid, methanol, acetone, urea, EDTA, 2,4-DMBINH, all chemicals and solvents used were of analytical grade and spectroscopic grade.

2.2) Equipment

ELICO digital pH meter (M/s Elico Private Limited), spectrophotometer (Kyoto UV-160A), analytical balance, Thermostated water bath.

2.3) Experimental Solution

0.01M Th(IV) was prepared from 0.48g of $Th(NO_3)_46H_2O$ with double distilled water containing one drop of concentrated HNO_3 and made up to 100ml. The solution is standardized.

2,4-DMBINH solution is prepared in dimethyl formamide 0.285gm of the reagent (2,4-DMBINH) is transferred into a 100 ml volumetric flask and made up to the mark with DMF.

1ml of metal ion solution of $1x10^{-3}$M concentration of 5ml of buffer solution of pH-8 and 1ml of reagent of $1x10^{-2}$M concentration are taken in volumetric flask and made up to the mark with distilled water. The absorption spectra are recorded in the wavelength a region of 398.4nm.

3) RESULTS AND DISCUSSION

3.1) Effect of Ph

The effect of pH on the complexation is studied. The absorbance values increase from pH^{-2} to pH^{-10}, the absorbance value is maximum in a solution of pH-8. Further the solution is highly stable and the spectrum is reproducible. Therefore a solution of pH-8 is chosen for further studies. The maximum absorbance is noticed at 398.4 nm. The data is presented in Fig-1.

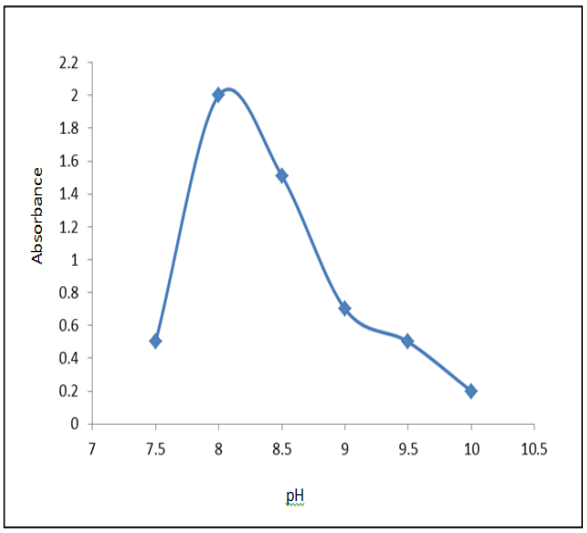

Fig-1: Effect of pH on Absorbance of Th(IV) - 2,4-DMBINH complex.
[Th(IV)] = 1 x 10^{-4}M
[2,4-DMBINH] = 1 x 10^{-3}M

3.2) Effect Of Metal Ion Concentration

The effect of Th(IV) ion concentration on the absorbance is studied. The concentration of metal ion is increased from 0.1 to 0.9x10^{-4}M. The concentration of reagent is kept constant. The pH of the solution is maintained at pH-8. From the result it may be noted that Th(IV) can be determined in the range 1.16 to 10.44 µg/ml. The data is presented in Fig-2.

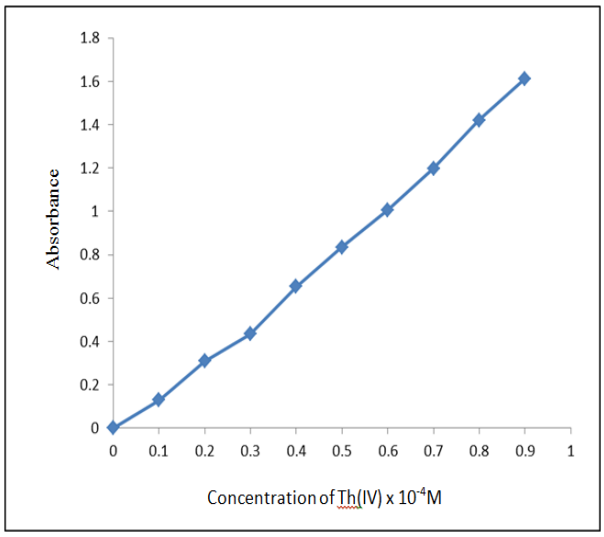

Fig-2: Applicability of Beer's Law on Th(IV) - 2,4-DHBINH complex.
[Th(IV)] = 1 x 10^{-4}M
[2,4-DMBINH] = 1 x 10^{-3}M
λ_{max} = 398.4 nm
pH = 8.0

2,4-DMBINH

The effect of reagent is studied in the range from 0.5 to 1.5x10^{-3}M. By keeping the concentration of the metal ion constant the absorbance values are measured at 398.4 nm. The concentration of Th(IV) is fixed. The present method the reagent can be determined in the range of 0.5 to 1.5x10^{-3}M. The data is presented in Fig-3.

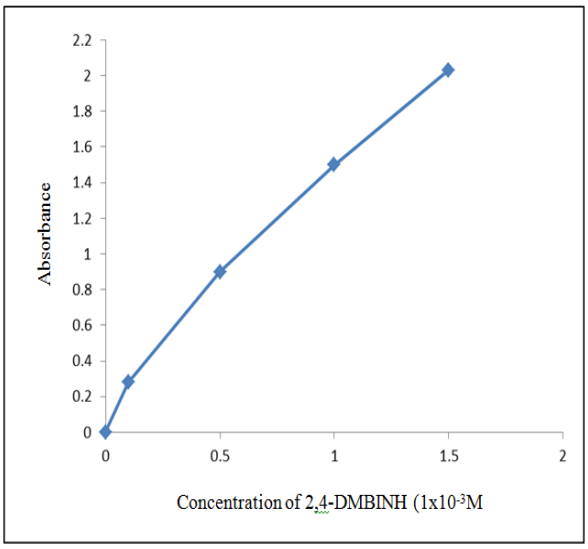

Fig-3: Effect of Reagent concentration on Th(IV) - 2,4-DMBINH complex.
[Th(IV)] = 1 x 10^{-4}M
λ_{max} = 398.4 nm
pH = 8.0

3.4) Effect of Time on The Reaction

The effect of time on absorbance is studied for period of one hour for the same solution. It is found that the absorbance values remains constant for an hour. The complex stable for a considerable period of time. The data is presented in Table-1.

Table-1: Effect of time
[Th(IV)] = 1 x 10^{-4}M
[2,4-DMBINH] = 1 x 10^{-3}M
λ_{max} = 398.4 nm
pH = 8.0

Time (in minutes)	Absorbance
10	1.901
20	1.853
30	1.841
40	1.811
50	1.792
60	1.776

3.5) Effect Of Organic Solvent

Organic solvents generally influence a complexation reaction. Therefore effect of Methanol, Acetone, 1,4 dioxane, DMF, DMSO, DMF 50% by volume are investigated. An analysis of data reveals that in presence of all solvents there is variation in absorbance values. The maximum absorbance value is noticed in DMF. The data is presented in Table-2.

Table-2: Effect of organic solvent
[Th(IV)] = 1 x 10^{-4}M

[2,4-DMBINH] = 1 x 10^{-3}M
λ_{max} = 398.4 nm
pH = 8.0

Solvent (50% v/v)	Absorbance
None	0.301
DMF	0.774
DMSO	0.183
Methanol	0.188
Acetone	0.241
1.4-Dioxane	0.215

4) COMPOSITION OF THE COMPLEX

The Stoichiometry of the Th(IV) and 2,4-DMBINH complex is determined by Job's method of continuous variation. In the Job's method a series of solution containing varying volumes metal ion and 2,4-DMBINH solution of required concentration were taken in 10ml volumetric flask. The absorbance values of these solutions were recorded in each case and it is clear from that Th(IV) forms 1:1 complex with reagent. The data is presented in Fig-4.

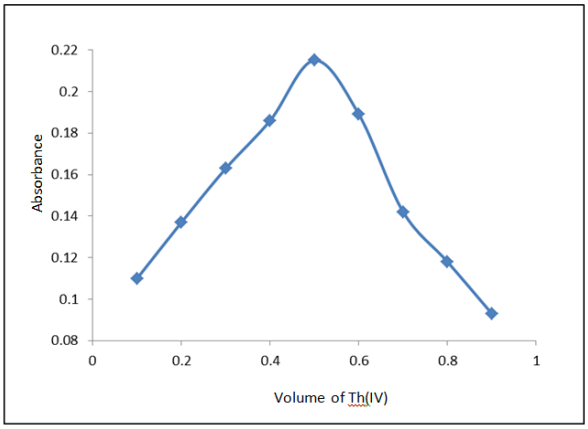

Fig-4: Job's method of variation of Th(IV) - 2,4-DMBINH complex.

[Th(IV)]= [2,4-DMBINH] = 1 x 10^{-4}M
λ_{max} = 398.4 nm
pH = 8.0

5) INTERFERENCE OF DIVERSE IONS

The effect of interfering ion on the determination of Th(IV) was investigated. The data is presented in Table-3. Except phosphate and acetate most of the anions do not interfere. The metal ions show serious interference.

Table-3: Tolerance limit of foreign ions.

Tolerance limit of foreign ions in the determination of Th(IV)
[Th(IV)] = 1 x 10^{-4}M
λ_{max} = 398.4 nm
pH = 8.0

Foreign ion	Tolerance limit (µg/ml)	Foreign ion	Tolerance limit (µg/ml)
Citrate	2700	U9VI)	673
Sulphate	63.5	W(VI)	275
Acetate	Interfere	Fe(III)	525
Phosphate	Interfere	Ti(IV)	343
Thicynate	650	Se(IV)	Interfere
Thiosulphate	570	Cr(III)	120
Tartarate	2560	Co(II)	460
EDTA	1600	Zn(II)	Precipitate
Chloride	24.3	Pb(II)	Precipitate
Carbonate	63.5	Zr(IV)	450

6) CONCLUSION

The proposed method is simple and sensitive are for the determination of thorium using 2,4-DMBINH in basic solution. The present method is convenient to determine the metal ion and 2,4-DMBINH in the range 1.16 to 10.44 mg/ml and 0.5 to 1.5x10^{-3} mg/ml.

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