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STUDIES ON ZR(IV)- 2,4 - DIMETHOXYBENZALDHYDE ISONICOTINOYL HYDRAZONE SYSTEM

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ABSTRACT

2,4-DMBINH reagent is used for the determination of Zr(IV). The reagent gives yellow coloured solution in basic medium with Zr(IV). The maximum absorbance is at 391nm. The present method is rapid, selective and sensitive for the determination of zirconium.

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

1) INTRODUCTION

Zirconium is a rare metal. It is a very strong malleable, ductile, lustrous silvery-grey metal. Zirconium is used in alloys such as Zircaloy, which is used in nuclear applications since it does not readily absorb neutrons. Also used in catalytic converters, perucssion caps and furnace bricks. Baddeleyite and Impure zirconium are used in lab crucibles, zirconium occurs in +4 state. This state has a tendency to add on the negative ions, thus making the determination of zirconium.

2,4 - dimethoxy benzaldhyde isonicotinoy hydrazone (2,4 - DMBINH) is play an importance role in medicinal chemistry. They are used as their complexes with metal ions for the treatment of number of diseases. As these organic compounds contain nitrogen as well as oxygen, they have ability to form complex 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

2,4-DMBINH, Methanol, Acetic acid, Sodium acetate, Ammonium chloride, Ammonia, Sulphuric acid, Hydrochloric acid, acetone, urea, EDTA, all chemicals and solvents used were of analytical reagents 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) Preparation of Stock Solutions

2.4) Preparation of 2,4-Dmbinh Solution

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

2.5) Preparation of Zirconium (Iv) Solution

0.01M Zr(IV) was prepared from 0.322 gr of $ZrOCl_2.8H_2O$ with distilled water and made up to 100ml. The solution is standardized.

2.6) Experiment

5ml of buffer solution of required pH, 1ml of standard solution and 1ml of reagent solution are transferred in to a 100 ml flask and made up to the mark with DMF.

3) RESULTS AND DISCUSSION

3.1) Effect Of Ph

The absorption spectrum obtained with Zr(IV) and 2,4-DMBINH is presented in Table-1. An analysis of the spectrum reveals that the wavelength corresponding to the maximum is 391nm, at pH-8.0 (Fig-1).

Table-1: Effect of pH on the absorbance of Zr(IV) - 2,4-DMBINH complex.

Zr(IV) = 1×10^{-4} M

[2,4-DMBINH] = 1×10^{-3} M

S.No	pH	Colour of the complex	λ_{max}	Absorbance
1	2	No colour reaction	---	---
2	3	No colour reaction	---	---
3	4	No colour reaction	---	---
4	5	No colour reaction	---	---
5	6	No colour reaction	---	---
6	7	No colour reaction	---	---
7	8	Yellow	391	1.362
8	9	Light yellow	391	0.432
9	10	No colour reaction	391	0.131

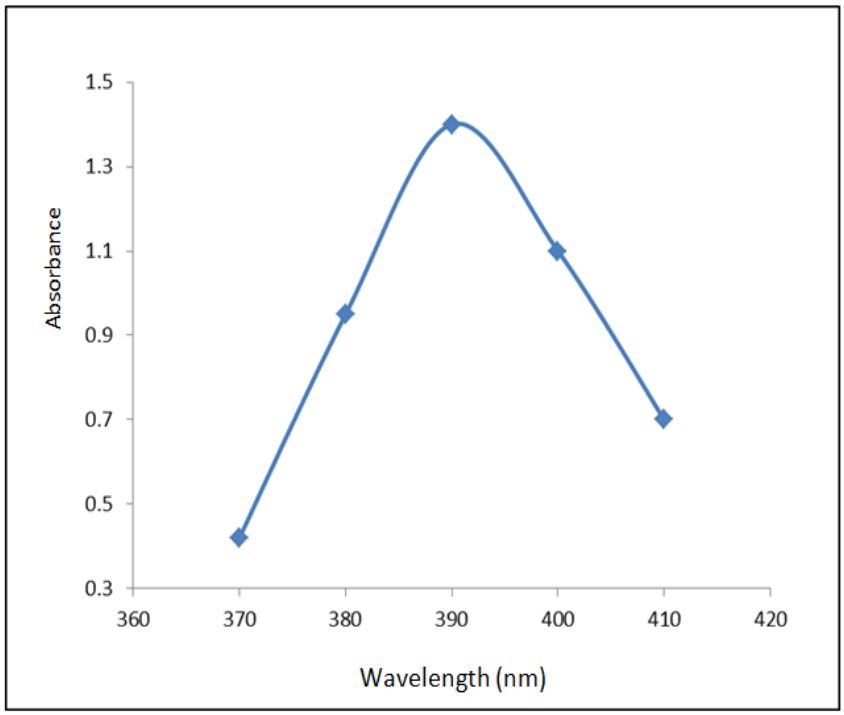

Fig-1: Absorption Spectrum Of Zr(Iv) - 2,4-Dmbinh.

Zr(IV) = $1x10^{-4}$M

[2,4-DMBINH] = $1x10^{-3}$M

pH = 8.0

3.2) Effect of Metal Ion Concentration

Zr(IV) ion concentration is varied and its effect on the absorbance value is studied. The linear calibration plots are shown in Fig-2. The Zr(IV) can be determined in the range of 0.912 to 7.297 µg/ml.

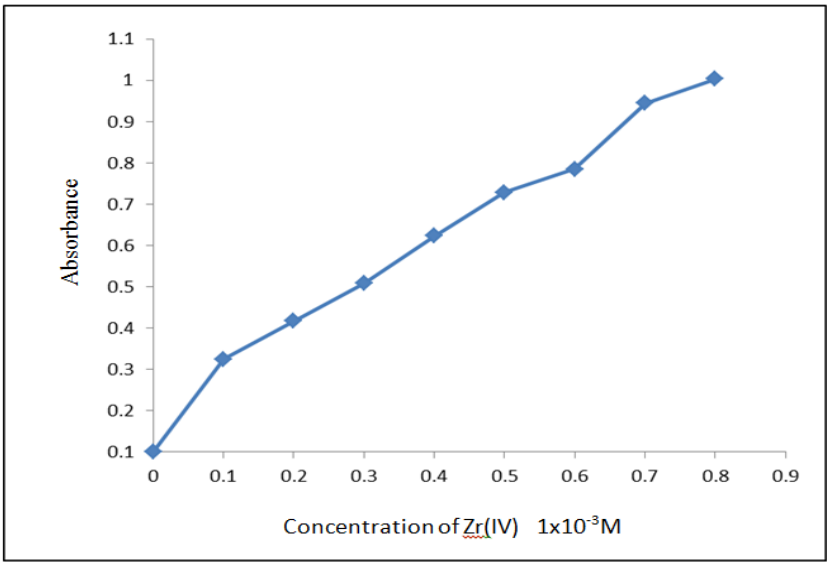

Fig-2: Applicability of Beer's law on Zr(IV) - 2,4-DMBINH complex

[2,4-DMBINH] = 1 x 10^{-3}M

λ_{max} = 391 nm

pH = 8.0

3.3) Effect of 2,4-Dmbinh

The concentration of Zr(IV) is kept constant. The absorbance values are measured by varying the 2,4-DMBINH concentration. The linear calibration plots are shown in Fig-3. From the data the reagent can be determined in the range of 0.5 to 1.5x10^{-3}M.

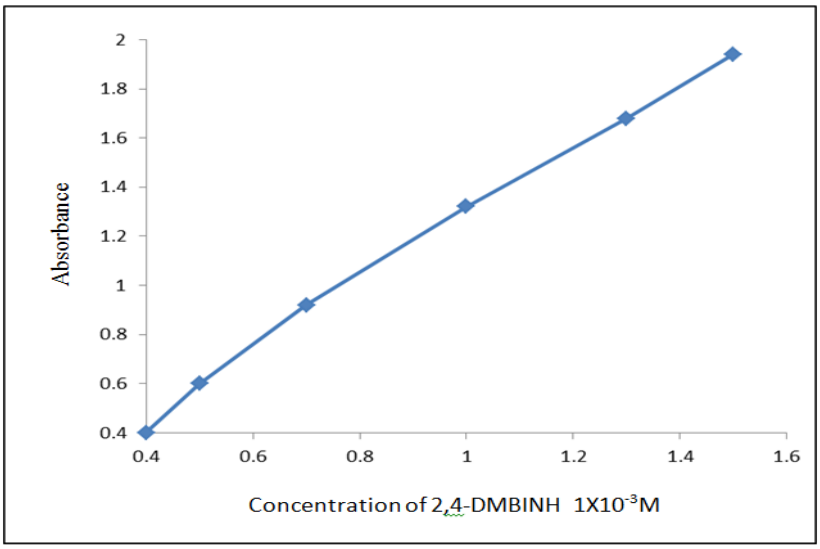

Fig-3: Effect of Reagent concentration on the absorbance of complex

Zr(IV) = 1 x 10^{-4}M

λ_{max} = 391 nm

pH = 8.0

3.4) Effect Of Time

The effect of time on absorbance values are measured at regular intervals of time of 10 minutes each. The data is presented in Table-2. From the data the complex remained stable for a considerable period of time.

Table 2: Effect of time.

Zr(IV) = 1×10^{-4}M

[2,4-DMBINH] = 1×10^{-3}M

λ_{max} = 391 nm

pH = 8.0

Time (in minutes)	Absorbance
10	1.352
20	1.351
30	1.279
40	1.241
50	1.139
60	1.062

3.5) Effect of Organic Solvent

Generally the non-aqueous solvent influence a complexation reaction. Studies were made in presence of organic solvents (50% by volume). Methanol, Acetone, 1-4 Dioxane, DMF, DMSO. The data is presented in Table-3. From the results revealed that there is variation in absorbance values in presence of all solvents. Solvents used did not alter the characteristics of the medium.

Table 3: Effect of organic solvent

Zr(IV) = 1×10^{-4}M

[2,4-DMBINH] = 1×10^{-3}M

λ_{max} = 391 nm

pH = 8.0

Solvent (50% v/v)	Absorbance
None	0.412
DMF	0.187
DMSO	0.252
Methanol	0.345
Acetone	0.82
1.4-Dioxane	0.181

4) Composition of The Complex

Zr(IV) forms an yellow coloured solution with the reagent. The absorbance values are measured. The data is presented in Fig-4. From the results reveal that Zr(IV) forms a 1:1 complex with 2,4-DMBINH. The stability constant of the complex is $1.5x10^{-6}$.

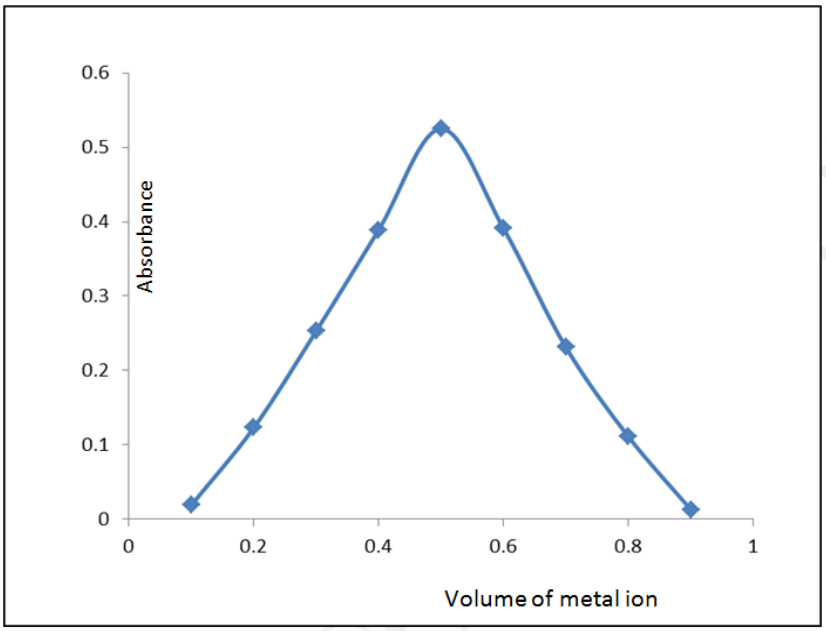

Fig 4: Job's method of continuous variation of Zr(IV) - 2,4-DMBINH complex.

Zr(IV)= [2,4-DMBINH]	=	$1x10^{-4}$M
λ_{max}	=	391 nm
pH	=	8.0

5) CONCLUSION

The proposed method is rapid, simple and sensitive for the determination of Zr(IV) and 2,4-DMBINH in the range of 0.912 to 7.297 µg/ml and 0.5 to $1.5x10^{-3}$M, respectively.

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