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Determination of Trace Amount of Fe (III) Using 3', 5'-Dinitro Salicylaldehyde Semicarbazone as an Analytical Reagent by Solvent Extraction and Spectrophotometric Method

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ABSTRACT

The trace amount of Fe (III) was detected by new sensitive, Analytical reagent viz.3',5'-Dinitro Salicylaldehyde Semicarbazone [3',5'DNSAS]. The reagent 3',5' DNSAS is synthesized in the laboratory and characterized by NMR, IR and elemental analysis. A selective spectrophotometric method is presented for the trace determination of Fe(III) using 3',5'DNSAS as spectrophotometric reagent (λ_{max} = 410 nm) in acidic aqueous solution (pH = 1.0). The Beer's law is obeyed in the concentration range from 1 to 10 ppm. The 3',5' DNSAS forms a 1:2 purple coloured complex. The Sandell's Sensitivity is 0.0375 µg cm^{-2} with molar absorptivity 2489.75 L mol^{-1} cm^{-1}. The proposed method has been successfully applied to the determination of Iron in various real and synthetic samples. The precision and the accuracy obtained were satisfactory.

Keywords: Iron, reagentconcentration, Spectrophotometric determination, n-Butanol, 3',5'-Dinitro Salicylaldehyde Semicarbazone derivative.

INTRODUCTION

Iron and its compounds are widely used in alloys and various biological samples. Trace concentrations of Iron can also affect the physical and mechanical properties of metal and alloys. Iron is essential to nearly all known organisms. The use of iron metal fillings in organic synthesis is mainly used for the reduction of nitro compounds. Therefore, precise knowledge of the Iron present in a various samples is required for an accurate assessment of the Iron, which has resulted in an increasing need of analytical methods for determination of micro-trace or ultra-trace level. Many methods have been reported for the determination of Ironsuch as inductively coupled plasma atomic emission or mass spectrometry [1- 3] requires expensive instruments and well-trained operators. Some of the reagents used for the spectrophotometric determination of iron are Bathophenanthroline [3], Mercapto pyridine-1-oxide [5], Pyridazine-3,6-diol [4], etc. However, most of these methods suffer from certain limitation, such as interference by number of ions [6, 7], of low sensitivity [8]. In this paper a new method has been developed using 3',5'-Dinitro Salicylaldehyde Semicarbazonefor extraction and spectrophotometric determination of Iron, which is simple, selective and sensitive.

MATERIALS AND METHODS

The reagent3',5'-Dinitro Salicylaldehyde Semicarbazone was synthesized by the given procedure [9]. The stock solution of Fe (III) was prepared by dissolving a weighed amount of ferric ammonium sulphate in double distilled water and then diluted to the desired volume with double distilled water and standardized by thiocyanate method. The absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with1 cm quartz cells and digital pH meter with combined glass electrode respectively.

Procedure for the Extraction: 1 mL of aqueous solution containing 0.5µg of iron metal and 2mL of reagent was mixed in a 50 mL beaker. The pH of the solution adjusted to 1.0, 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 Iron present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 410 nm and that in the aqueous phase was determined by thiocyanate method.

RESULTS AND DISCUSSION

Extraction as a function of pH: The extraction of Iron with 3',5'-Dinitro Salicylaldehyde Semicarbazonehas been studied over the pH range 1-10 and was observed that percentage extraction of Fe (III) is maximum at pH range 1.0-1.8. Hence, further extraction and determination was carried out at pH 1.0(Fig 1).

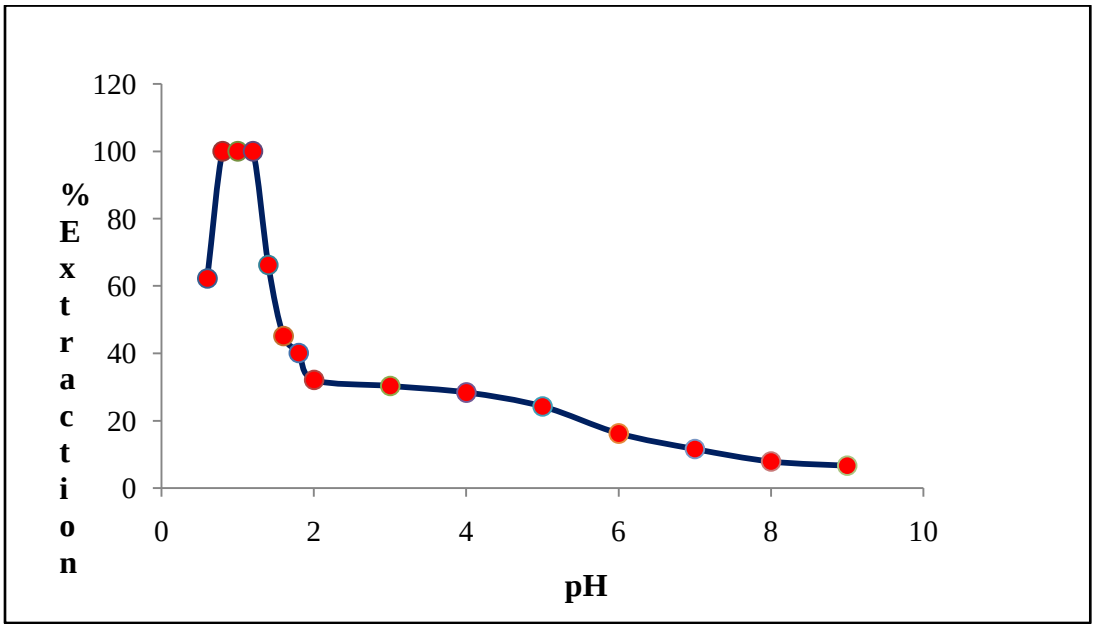

Fig 1. Effect of pH on extraction of Fe (III): (3',5'-DNSAS) complex

Absorption spectrum: The absorption spectrum of Fe (III): 3',5'-DNSAS in n- Butanol shows the maximum absorption at 410 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 410nm.

Influence of diluents: The suitability of solvent was investigated using various organic solvents and the extraction of Fe (III):3',5'-DNSAS was quantitative inn-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 1mL of 0.1% reagent is sufficient for the colour development of the metal Fe (III) in 10 mL of aqueous solution at pH 1.0

Effect of equilibration time and stability of the complex: The equilibration time of 1 min is sufficient for the quantitative extraction of Iron. The stability of colour of the Fe (III): 3',5'-DNSAS complex with respect to time shows that the absorbance due to extracted species is stable up to 24 h, after which slight decrease in absorbance is observed.

Calibration plot: The Beer's law is obeyed from 1 to 10ppm. The molar absorptivity and Sandell's sensitivity were calculated to be is2489.75 L $mol^{-1}cm^{-1}$ and 0.0375 µg cm^{-2} respectively (Fig 2).

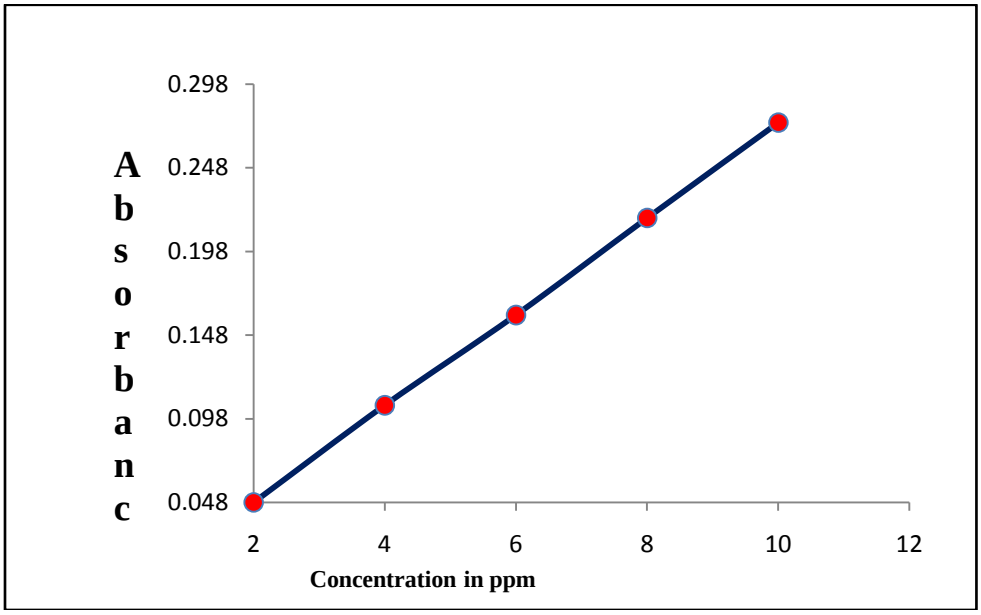

Fig 2. Calibration plot of Fe (III): (3',5'-DNSAS)complex

Solvent Study: Various solvents were tried to get the maximum extraction of iron, Fig 3, n-Butanol was found to be the most suitable solvent as it showed the maximum extraction. The extraction of iron varied from maximum to minimum for the solvent in the order of:

n-Butanol > Ethyl acetate> Isoamyl alcohol > Hexane > Chloroform > Cyclohexanone > Diethyl ether > Toluene >Nitrobenzene> Xylene> Carbon tetrachloride

Precision and accuracy: The precision and accuracy of the spectrophotometric method were tested by analyzing the solution containing known amount of iron (III) .Average of ten determinations of 10µg Fe (III) in 10 cm^3 solution is 9.9982µg which varies between 9.960µg to 10.020µg at 95% confidence limit.
Nature of extracted species: The composition of extracted Fe (III): 3',5'DNSAS complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Fe (III): 3',5' DNSAS complex is 1:2. (Fig4).

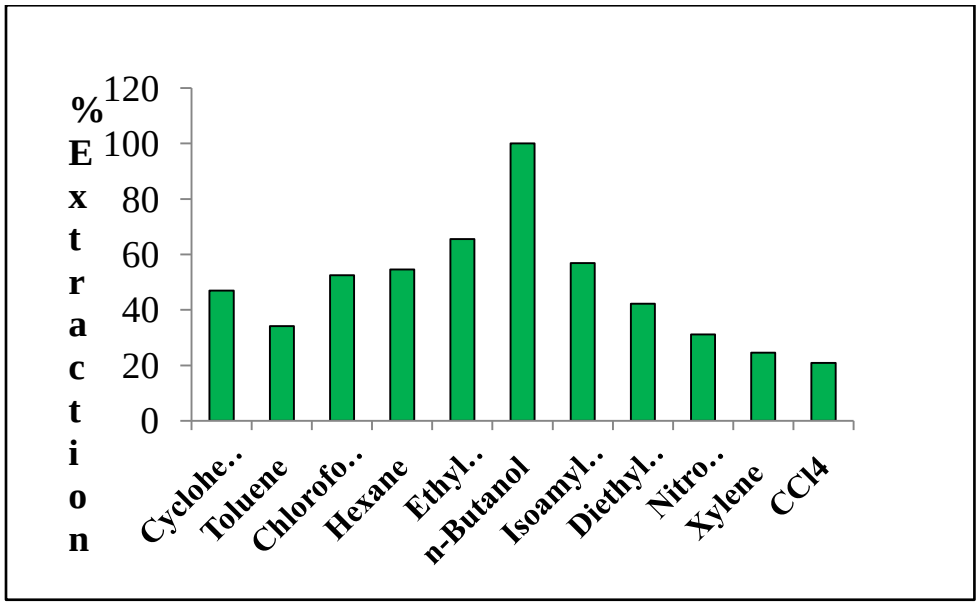

Fig 3. Effect of various solvents on Fe (III): (3',5'-DNSAS) complex

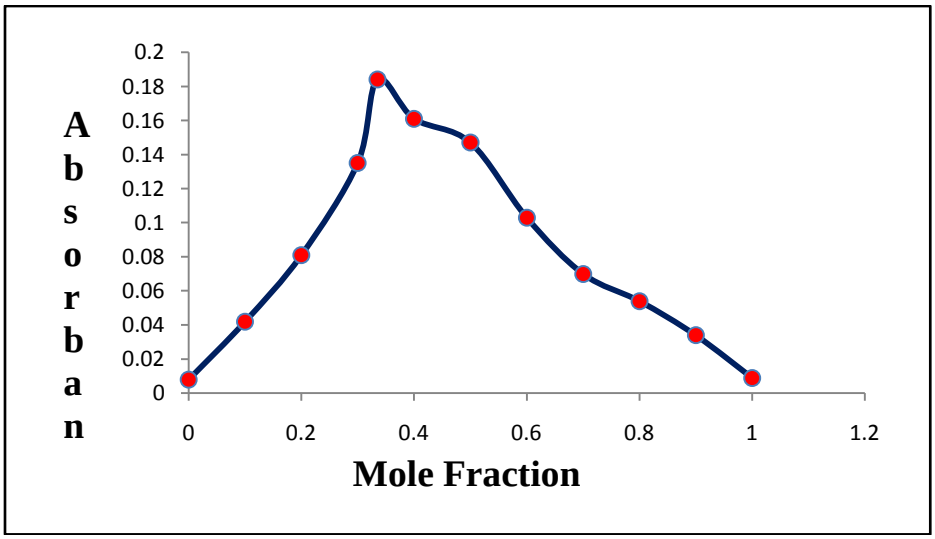

Fig 4. Job's continuous variation curveNature of extracted species

APPLICATIONS

The proposed method was successfully applied for the determination of iron from various alloys and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method. Each result is average of three independent determinations. Each result is compared with standard thiocyanate method as shown in Table 1.

Table 1. Observation Table for determination of Fe (III) using3',5'- DNSAS from different Samples

Sr.No.	Sample	Certified value	Present method
Iron alloys:			
1	Hematite ore	25 µg	24.8 µg
3	Elinver alloys	12 µg	11.9 µg
Synthetic mixture:			

1	Fe +Zn	10ppm	9.89ppm
2	Fe+ Mg	5ppm	4.98ppm
Industrial Waste	From Kalu river	25ppm	27ppm
Multi Vitamin Tablet	Supradyne Tablet	32.24 µg	32.23µg

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