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NEW ANALYTICAL TECHNIQUE FOR DETERMINATION OF TRACE AMOUNT OF FE (III) BY USING UV-VISIBLE SPECTROPHOTOMETRIC METHOD WITH PHOTOMETRIC REAGENT

V. S. More*, R. S. Lokhande[1], S. P. Janwadkar[2], D. K. Yadav, P. K. Rana, C. Y. Patil, C. L. Pamale and Y. K. Patil

[1]Jaipur National University Jaipur.
[2]S.D. Arts, V.S. Apte Commerce and M.H. Mehta Science College, Palghar, Dist- Palghar (M.S.).

***Corresponding Author: Dr. V. S. More**
Jaipur National University Jaipur.

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ABSTRACT
3-nitrosalicylaldehyde thiosemicarbazone (3-NSTS) is proposed as a new photometric reagent for the extractive spectrophotometric determination of Fe (III) (3-NSTS) reacts with Fe (III) and form a stable coloured complex in the pH range 2.0 to 3.0.This was well extracted in n-butanol. The absorption spectrum of Fe (III) (3-NSTS) complex in n-butanol shows maximum absorbance at 380 nm. The system obeyed Beer's law up to 2-10 µg / cm^3. The molar extinction coefficient was found to be 3.957 x 10^3 lit $mol^{-1}cm^{-1}$ and the sensitivity of the method as defined by Sandell's sensitivity was 3.33 x 10^{-2} µg/cm^2. The composition of the extracted species was determined by Job's Continuous variation method, Mole ratio method and slope ratio method and it was found to be 1:2. The proposed reagent is satisfactorily applied for the determination of trace amount of Fe (III) from industrial waste water as well as synthetic and commercial samples.

KEYWORDS: Iron,n-butanol,3-Nitro-salicylaldehyde thiosemicarbazone derivative(3-NSTS) etc.

INTRODUCTION
Iron is a chemical element with the symbol Fe and atomic number 26. Iron belongs to group 8 and period 4. Iron is most widely used among all the metals, accounting for 95% of worldwide metal production. Its low cost and high strength make it indispensable in engineering applications, such as the construction of machinery and machine tools, automobiles, the hulls of large ship, and structural components for buildings. Since pure iron is quite soft, it is most commonly used in the form of steel.

Iron is essential to nearly all known organisms. In cell, iron is generally stored in the center of metalloproteinase, because free iron which binds nonspecifically to many cellular components can catalyze production of toxic free radicals. Iron deficiency can lead to iron deficiency anemia.

Owing to the significance of iron, it's determination from associated elements by extractive spectrophotometry has been of considerable importance. Several compounds are known to react with the metal ions to give coloured complexes and have been employed for the quantitative extraction and spectrophotometric determination of metals at trace level.

EXPERIMENTAL
Procedure for the Extraction
An aliquot of solution containing 1mL of 100ppm of Iron was taken. To this 1mL of (3-NSTS) reagent is mixed. The pH of the solution adjusted to 2.0, & noted that the total volume should not exceed than 10mL.The solution was transfer to the 125mL of separating funnel & equilibrated with 10mL of n-butanol solution. The separating funnel was shaken vigorously and allowed to stand for some time to separate the two phases. The aqueous phase is separated and the organic phase is passed through anhydrous sodium sulphate in order to absorb water and then collected in 10mL volumetric flask and dilute up to the mark with n-butanol. The absorbance was measured at λmax = 380nm on a Shimadzu UV-Visible 2100 Spectrophotometer with 1cm quarts cells.

RESULT AND DISCUSSION
The results of various studies were discussed as given below:

Effect of solvent on extraction
n-Butanol is chosen as solvent, since it was found that the metal complex Fe (III) (3-NSTS) complex in n-butanol shows maximum absorbance at 380nm.

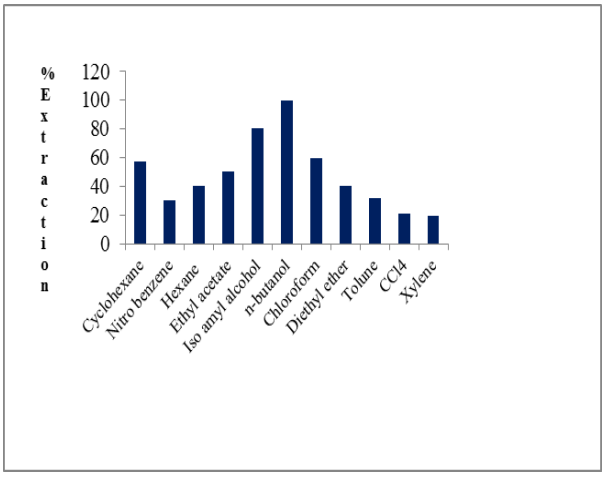

Fig. 1: Effect of solvent on extraction.

Effect of pH on extraction

The absorbance the complex Fe (III) (3-NSTS)was measured as a function of pH of the aqueous phase. The complexation of Fe (III) was carried out at pH 1-10. From which pH range 2.0-3.0 is selected. The data obtained shows maximum absorbance at pH 2.0.

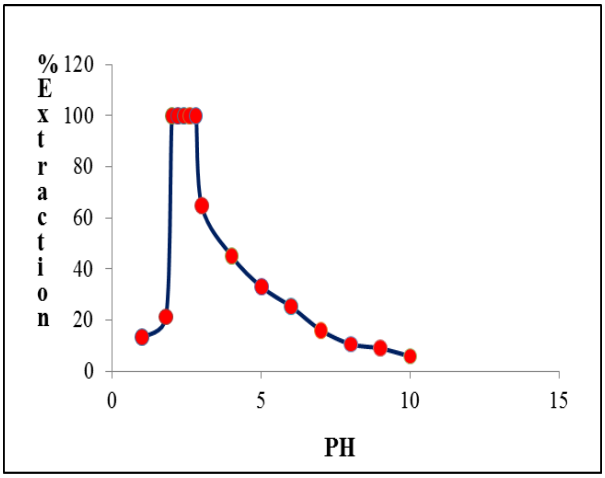

Fig. 2: Effect of pH on extraction.

Effect of Reagent concentration:

The results obtained from the plot of absorbance versus concentration of (3-NSTS) shows that 1mL of 8.45 x10^{-4} M reagent sufficient for the quantitative extraction and spectrophotometric determination of100ppm of Fe (III) in n-butanol. Addition of excess reagent did not interfere with complexation and extraction of complex, so further study was carried out by using 1mL of 2.08 x 10^{-3} M reagent.

Effect of equilibration time and stability of the complex

The equilibration time of 1 minute was sufficient for the quantitative extraction of Iron and the complex Fe (III) (3-NSTS) is stable for 48 hours, after which slight decrease in absorbance is observed.

Calibration plot

The system obeys Beer's law in the concentration range up to 2-10 µg / cm^3 at 380nm.The molar absorptivity and Sandell's sensitivity were calculated and found to be 3.957 x 10^3 lit $mol^{-1}cm^{-1}$ and 3.33 x 10^{-2} µg/cm^2 respectively. (Fig.3)

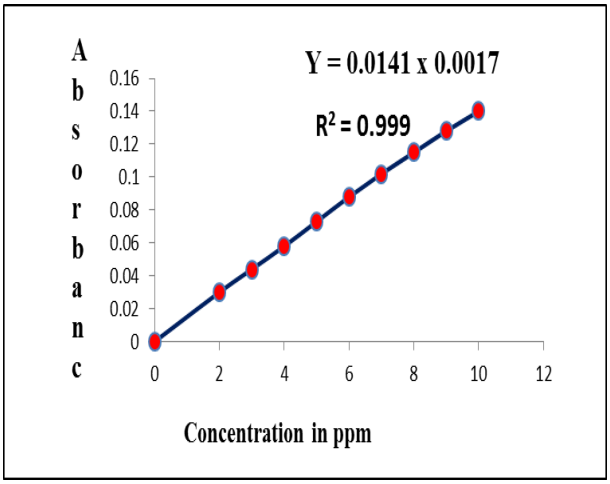

Fig. 3: Calibration Curve.

Effect of divalent ions and foreign ions

The effect of diverse ions on the Fe (III) was studied, in the presence of foreign ions. The ions which show interference in the spectrophotometric determination of Iron were overcome by using appropriate masking agent as given in table 1.

Table no: 1.

Sr.no	Interfering ions	Masking agent
1	Co(II)	Ammonium Sulphite
2	Cu(II)	Sodium Thiosulphate
3	Ni(II)	DMG
4	CN^{-1}	Boiled with concentrated HNO_3 and formaldehyde.
5	EDTA	Boiled with concentrated HNO_3

Precision and Accuracy

The precision and accuracy of the spectrophotometric method were tested by analyzing ten solutions containing 2.0 µg of Iron in 10 cm^3.The average of ten determinations was 2.1197µg which is varies between 2.1197µg to 0.1403µg at 95% confidence limit.

Limit of detection

Standard deviation of blank solution and slope of calibration curve is used for calculating limit of detection, which found to be 0.153 µg / mL.

Nature of extracted species

The composition of extracted Fe (III) (3-NSTS)complex has been determined by Job's continuous variation method, Slope method and Mole ratio method. It shows that the composition of Fe (III) (3-NSTS)complex is 1:2 (Fig.4)

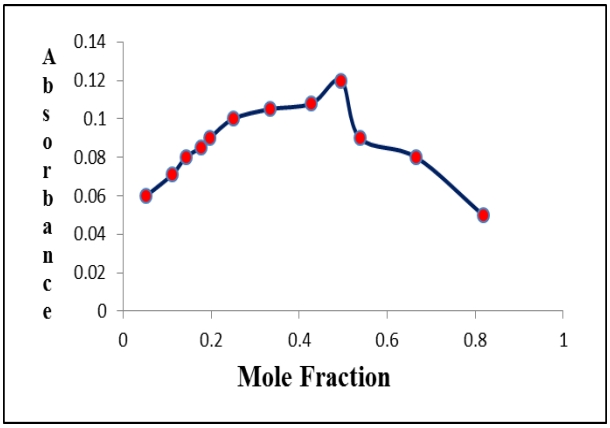

Fig. 4: Job Continuous Variation.

Application

The proposed method was successfully applied for the determination of Iron from various synthetic mixtures, industrial waste and alloys and commercial samples etc. The results obtained were found to be in good agreement with those obtained by the standard method as given in below table.

Table no: 2.

Sr.No.	Sample	Certified value(µg)	Present method(µg)
Iron alloys:			
1	Hematite ore	25	24.8
3	Elinver alloys	12	11.9
Synthetic mixture:			
1	Ni+ Zn+Fe	6	5.9
2	V+ Fe+ Co	5	5
3	Fe +Cu +Mn	8	7.8
Multi Vitamin Tablet	Supradyne Tablet	32.24	32.25

Industrial effluents are collected from the following industries

1. Camelin Fine chemical 2.Viraj Ltd.
3.Lupin Ltd. 4. Neon pharma
5. Genisis pharma 5. Rank Organic chemical

Industrial effluents collected Bimonthly Analysis

TABLE6.22.

Sr.No	Industrial effluent (Concentration in ppm)	January February March (Concentration in ppm)	April May June (Concentration in ppm)	July August September (Concentration in ppm)	October November December (Concentration in ppm)
1	Camelin Fine chemical	15.63	15.95	14.22	15.19
2	Viraj Ltd.	14.38	14.80	13.95	14.31
3	Lupin Ltd.	12.45	12.88	11.25	12.13
4	Neon pharma	10.95	11.58	10.11	10.45
5	Genisis pharma	13.55	14.89	13.22	13.31
6	Rank Organic chemical	11.38	12.80	11.15	11.31

1) Each result is average of three independent determinations.
2) Compared with EDTA method.

CONCLUSIONS

The method required simple apparatus which have low cost. This method offer several silent features such as rapidity, selectivity and simplicity. The other associated elements do not interfere in the determination. Hence the proposed method is recommended for the determination of Fe (III) (3-NSTS) by spectrophotometric method, at trace level analysis of various alloys, synthetic mixture and industrial waste.

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