

Solvent extraction and spectrophotometric determination of Fe(III) by using 5-bromo salicylidene-2-aminothiophenol (BSATP) as an analytical reagent

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Abstract : A spectrophotometric method has been developed for the determination of Fe (III) using 5-bromo salicylidene-2-aminothiophenol (BSATP) as an extractive reagent.

The reagent forms a colored complex which has been quantitatively extracted into chloroform at pH 8.2. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is $16460 \text{ L mol}^{-1}\text{cm}^{-1}$ and Sandell's sensitivity is $0.01639 \mu\text{g cm}^{-2}$ respectively. The proposed method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.

Key words: Spectrophotometric determination, 5-bromo salicylidene-2-aminothiophenol (BSATP), chloroform, molar absorptivity.

Introduction:

The cursory look at the literature survey reveals the fact that Iron reacts with many organic reagents^{1,2}. It also indicates that some of the reagents recommended in literature are suffering through limitations such as interference of Ag(I)^3 , V(VI)^4 , Co(II)^5 , Ni(II)^6 , Mn(II)^6 , Zn(II)^6 , complex formation takes place after several minutes, moreover some of the reagents are not selective^{7,8} and sensitive. In this article, a new method has been developed using 5-bromo salicylidene-2-aminothiophenol (BSATP) for extraction and spectrophotometric determination of Fe(III), which is simple, selective and sensitive.

Experimental:

The reagent 5-bromo salicylidene-2-aminothiophenol (BSATP), was synthesized by the given procedure. 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 ceric (IV) sulphate method. The absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode respectively.

Procedure for the extraction:

1 ml of aqueous solution containing 0.1mg of iron metal and 2 ml of reagent was mixed in a 50 ml beaker. The pH of the solution adjusted to 8.2. It must be noted that total volume should not exceed 10 ml. The solution was transferred to 100 ml separatory funnel. The beaker was washed twice with chloroform and

transferred to the same funnel. The two phases were shaken for one minute and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from it 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 385 nm and that in the aqueous phases was determined by ferrous ammonium sulphate method.

Results and discussion:

The results of various studies are discussed below.

Extraction as a function of pH:

The extraction of iron with 5-bromo salicylidene-2-aminothiophenol (BSATP) has been studied over the pH range 2 to 10 and was observed that percentage extraction of Fe (III) is maximum at pH 8.2. (Fig. 1)

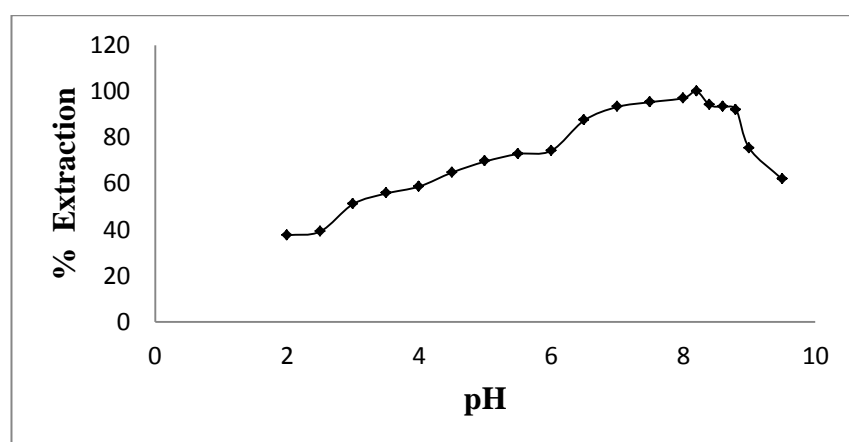


Fig. 1 Effect of pH on % extraction

Absorption spectrum:

The absorption spectrum of Fe (III): 5-bromo salicylidene-2-aminothiophenol (BSATP) in chloroform shows the maximum absorption at 385 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 385 nm.

Influence of diluents:

The suitability of solvent was investigated using various organic solvents and the extraction of Fe (III) was quantitative in chloroform. Hence, chloroform was used for further extraction studies as it gave better and quicker phases separation.

Effect of reagent concentration:

It was found that 2 ml of 0.05% reagent is sufficient for the colour development of the metal Fe(III) in 10 ml of aqueous solution at pH 8.2.

Effect of equilibration time and stability of the complex:

The equilibration time 1 minute is sufficient for the quantitative extraction in chloroform. The stability of colour of the Fe(III) complex with respect to time shows that the absorbance due to extracted species is stable up to 60 minutes, after which slight decrease in absorbance is observed.

Calibration plot:

The Beer's law is obeyed from 1 to 10 ppm. The molar absorptivity and Sandell's sensitivity were calculated to be $16460 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $0.01639 \mu\text{g cm}^{-2}$ respectively. (Fig. 2)

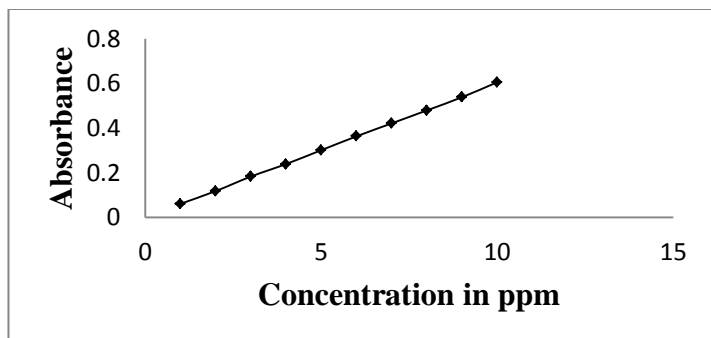


Fig. 2 Calibration plot

Effect of divalent ions and foreign ions:

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 100 ppm of iron. The ions which show interference in the spectrophotometric determination of iron were overcome by using appropriate masking agents. (Table no.1)

Table no. 1

Sr. No.	Interfering ions	Masking agents added (mg)
1	Ce ⁴⁺	Sodium fluoride
2	Mo ⁶⁺	Citrate
3	Cr ²⁺	Ammonium acetate
4	Ag ⁺	Potassium iodide
5	Pd ²⁺	Thiourea
6	Citrate, Tartarate, EDTA	Sodium molybdate
7	Cyanide	Boiling with HNO ₃ and formaldehyde
8	Pb ²⁺	Sodium sulphate

Precision and accuracy:

The precision and accuracy of the spectrophotometric method have been studied by analyzing ten solutions each containing 5 ppm of iron in the aqueous phase. The average of ten determinations was 5.023 and variation from mean at 95% confidence limit was 5.023 ± 0.0449 .

Nature of extracted species:

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

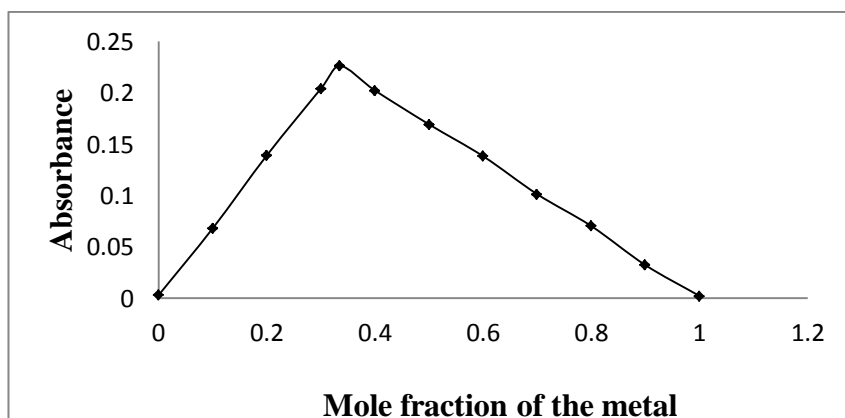


Fig. 3 Job's continuous variation curve

Applications:

The proposed method successfully applied for the determination of iron from various alloys, ores, industrial waste water and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method (Table no.2).

Table no. 2: Estimation of iron in alloys, ores, pharmaceutical samples and industrial waste water

Estimation of iron in	Sample	% Fe(III) certified value	% Fe(III) observed value
Alloys and ores	Steel	67.00	66.45
	BAS-85	1.20	1.18
	Hematite ore	20.50	20.54
Pharmaceutical samples	Vitamin B ₁₂	14.7	15.01
	Dexorange plus syrup	19.9	19.8
	Autrin capsule	32.66	33.0
Industrial waste water	Industrial waste water at Vashi Creek	0.578 ppm	0.592 ppm

Each result is average of three independent determinations.

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