

# Development of method for extractive spectrophotometric determination of Fe(III) with 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone as an analytical reagent

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## ABSTRACT

A spectrophotometric method has been developed for the determination of Fe(III) using 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone as an extractive reagent. The reagent forms a coloured complex, which has been quantitatively extracted into ethyl acetate at pH 6. The method obeys Beer's law over arrange from 1 to 7 ppm. The Molar absorptivity and Sandell's sensitivity were  $0.31 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$  and  $0.13 \mu\text{g cm}^{-2}$  respectively. The proposed method is very sensitive and selective. The method has been successfully applied to synthetic and commercial samples.

**Keywords:** Iron Spectrophotometric determination; ethyl acetate; 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone

## 1. INTRODUCTION

The synthesis of hydrazone derivative and their applications towards metal ions have been reported [1, 2]. Iron is most widely used of all the metals, accounting for the 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 ships, and structural components for buildings. Since pure iron is quite soft, it is most commonly used in the form of steel. 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. A wide variety of reagent has been reported for the spectrophotometric determination of iron. A number of reagents such as hydrazine [3], thiosemicarbazone [4, 6], oxime [6, 7], etc. have been used for the determination of Fe(III). However, these methods suffer from limitations such as critical pH [8]. In continuation to our earlier work [9] the proposed method is a simple, rapid and selective towards spectrophotometric determination of Fe(III) using 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone derivative.

## 2. EXPERIMENTAL

The reagent 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone was synthesized by the given procedure. The stock solution of Fe(III) was prepared by dissolving a weighed amount of sulphate in double distilled water containing dilute sulphuric acid, which was diluted to the desired volume with double distilled water and standardized by o-phenanthroline method [10]. 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.

### 2. 1. Procedure for the extraction

1.0 ml of aqueous solution containing 0.1mg of Iron metal and 1 ml of reagent were mixed in a 50 ml beaker. The pH of the solution adjusted to 6 with 0.2 M dilute solution of  $H_2SO_4$ , keeping the volume 10 ml. The solution was transferred to 100 ml separating funnel. The beaker was washed twice with ethyl acetate and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate.

The organic phase was collected in 10 ml measuring flask and made up to the mark with organic solvent if required. After separation of the two phases, the pH of the aqueous phase was measured and the Fe(III) in each phase was determined by o-phenanthroline method [10].

## 3. RESULTS AND DISCUSSION

The reagent HNPH forms red coloured complex with Fe(III), which was extracted into organic phase. The extraction of Fe(III) forms an aqueous phase by HNPH in ethyl acetate is studied over a wide range of experimental condition. The results of various studies are discussed below.

### 3. 1. Extraction as a function of pH

The extraction of Iron with 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone has been studied over the pH range 1-10 and was observed that percentage extraction of Fe(III) is maximum at pH 6.

### 3. 2. Absorption spectrum

The absorption spectrum of Fe(III): 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone in ethyl acetate shows the maximum absorption at 510 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 510 nm.

### 3. 3. Influence of diluents

The suitability of diluents was investigated using organic solvents such as chloroform, ethyl acetate, isoamyl alcohol, xylene, hexane, diethyl ether, toluene, n-butanol, carbon tetrachloride, MIBK, nitrobenzene, etc.

The extraction of Iron (III) was quantitative with HNPH in ethyl acetate. Hence, ethyl acetate was used for further extraction studies as it gave better and quicker phase separation.

### 3. 4. Effect of salting out agents

The presence of 0.1 M salts of various alkali and alkaline metals does not show any effect over the absorbance value of Fe(III): 1-Naphthalene Carboxaldehyde-2-hydroxy phenyl hydrazone complex extract. Therefore, no salting out agent was required during the extraction.

### 3. 5. Effect of reagent concentration

Various volumes of 0.1 % reagent solution were added to the sample solution containing 40  $\mu\text{g}$  of Iron at respective pH values. The absorbance remained nearly constant when the volume of the reagent solution used was more than 1 ml. Therefore, 1 ml of 0.1 % reagent was chosen for the quantitative determination of the metal.

### 3. 6. Effect of equilibration time and stability of the complex:

The study of change in absorbance with variation in equilibrium time for extraction of extraction of the complex into organic solvent shows that equilibration time of 1 min is sufficient for the quantitative extraction of Iron. The study of stability of colour of the Fe(III): HNPH complex with respect to time shows that the absorbance due to extracted species is stable up to 30 hours, after which slight decrease in absorbance is observed. Throughout the experimental work, for practical convenience, the measurements have been carried out within one hour of extraction of Iron.

### 3. 7. Calibration plot

A calibration plot of absorbance against varying Iron concentration and fixed HNPH concentration gives linear and reproducible graph in the concentration range 1 to 7 ppm of Iron (Fig. 1). This shows that the Beer's law is obeyed in this range. The molar absorptivity and Sandell's sensitivity were calculated to be is  $0.31 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$  and  $0.13 \mu\text{g cm}^{-2}$  respectively.

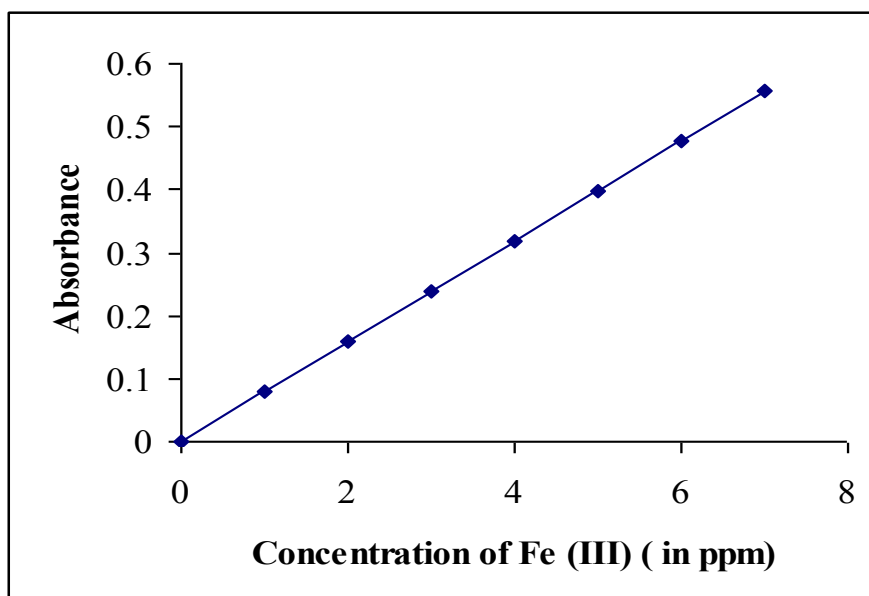


Fig. 1. Calibration plot of Fe (III): HNPH complex.

### 3. 8. Nature of extracted species

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

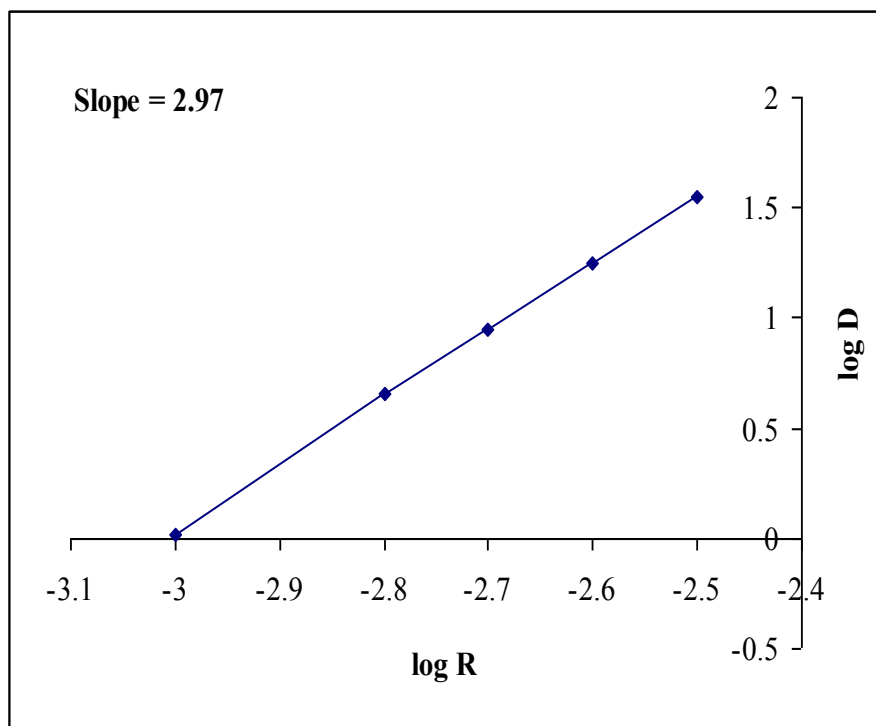


Fig. 2. Slope ratio method for Fe(III) : HNPH complex.

### 3. 9. Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 50  $\mu\text{g}$  of Iron. The ions which show interference in the spectrophotometric determination of Iron were overcome by using appropriate masking agents.

### 3. 10. Precision and Accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing five solutions each containing 30  $\mu\text{g}$  of Iron in aqueous phase. The average of five determinations was 29.99, variance 0.03 and variation from mean at 95 % confidence limit was  $\pm 0.23$ .

### 3. 11. Applications

Various commercial samples and synthetic mixtures containing Fe(III) were prepared and analyzed according to the recommended procedure and the results were compared to those obtained by standard method. The proposed method facilitates separation of Iron (III) from synthetic mixtures.

These metal ions do not extract and remain quantitatively in the aqueous phase under the optimum extraction conditions of Iron (III) with HNPH system facilitating separation of bivalent Iron quantitatively by the proposed method. The results found to be in good agreement with those obtained by the standard known method [10] (Table 1).

**Table 1.** Determination of Fe(III) using HNPH from different samples.

Sr. No.	Sample	Amount of Fe	
		Standard method	Present method
I	Alloy / Ore		
1	Hematite	35.0 %	34.9 %
2	Steel	67.5 %	67.4 %
II	Capsule / Tablet		
1	Autrin	32.8 mg	32.77 mg
2	Fefol	148 mg	148 mg
III	Synthetic mixture		
1	Fe (5) + Zn (5)	4.98 ppm	4.96 ppm
2	Fe (5) + Mg (5)	4.98 ppm	4.97 ppm

#### 4. CONCLUSION

The results obtained show that HNPH in ethyl acetate can be effectively used for quantitative extraction from aqueous media. The proposed method is found to be quantitative as compared to other standard methods. The equilibrium time required is very little, i.e. only 1 min and the complex is stable for 30 hrs. The results show good agreement with the standard method. The method is very fast, accurate and precise. The 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone was used for the first time for extraction of Fe(III) from various binary mixtures.

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