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# *Chapter – 7*

## **SPECTROPHOTOMETRIC DETERMINATION OF IRON(III)**

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## Introduction

Iron<sup>1</sup> is an abundant element, the fourth largest among the elements, and it is an essential component of almost every organism in the biosphere. It occurs in a variety of rock and soil minerals of oxidation states 2 and 3; but it is only a trace element in biological system. Iron plays a central role in the biosphere. It is an essential component (or) cofactor of numerous metabolic reactions, every living cell, both plants and animals contains iron. It is involved in oxygen transport and electron transfer and in enzymes including hydroxylases, peroxidases and dismutases<sup>2</sup>. Iron deficiency anemia is one of the world's most common nutritional deficiency diseases. Evidence has been presented that at low levels iron is an essential element in the diet, whereas at higher concentrations it is toxic<sup>3</sup>. Excess of iron in body causes "harmachromatosis". The toxicity of iron and in particular iron overload has been used considerable interest in recent years<sup>4</sup>.

Traister and Schitt<sup>5</sup> have reviewed the spectrophotometric methods proposed for the determination of iron employing a variety of organic reagents. Most of the spectrophotometric methods proposed for the determination of iron are based on the formation of intensely coloured complexes between the metal ion and the selected reagent<sup>6-11</sup>. Black<sup>12</sup> has stated that numerous methods have been employed to assess the available states of iron in soils. The analytical results evaluated in some of the recently proposed spectrophotometric methods for iron are reviewed and presented in Table 7.1.

A overall study of the data in Table 7.1 reveals that though a plethora of spectrophotometric methods are available for the determination of iron, every method has its own limitations either in its sensitivity or selectivity or stringent experimental

conditions. Hence, there is a need to propose simple sensitive and selective methods for the determination of iron in various complex materials.

### **Present investigations**

Iron (III) forms a dark brown coloured water soluble complex with 2-Amino acetophenone isonicotinoyl hydrazone 2-AAINH in acidic medium. The colour formation was instantaneous and was stable for more than 48 hours. Hence, a detailed study of the system is undertaken with a view to develop a new spectrophotometric method for the determination of iron (III).

**Table 7.i.1. (LS table 1–6 Pages)**

**Analytical results of various**

**Table 7.i.1. (LS table 2–6 Pages)**

**Analytical results of various**

**Table 7.i.1. (LS table 3–6 Pages)**

**Analytical results of various**

**Table 7.i.1. (LS table 4–6 Pages)**

**Analytical results of various**

**Table 7.i.1. (LS table 5–6 Pages)**

**Analytical results of various**



**Table 7.i.1. (LS table 6–6 Pages)**

**Analytical results of various**

### **Section i : Zero order spectrophotometric determination of iron (III)**

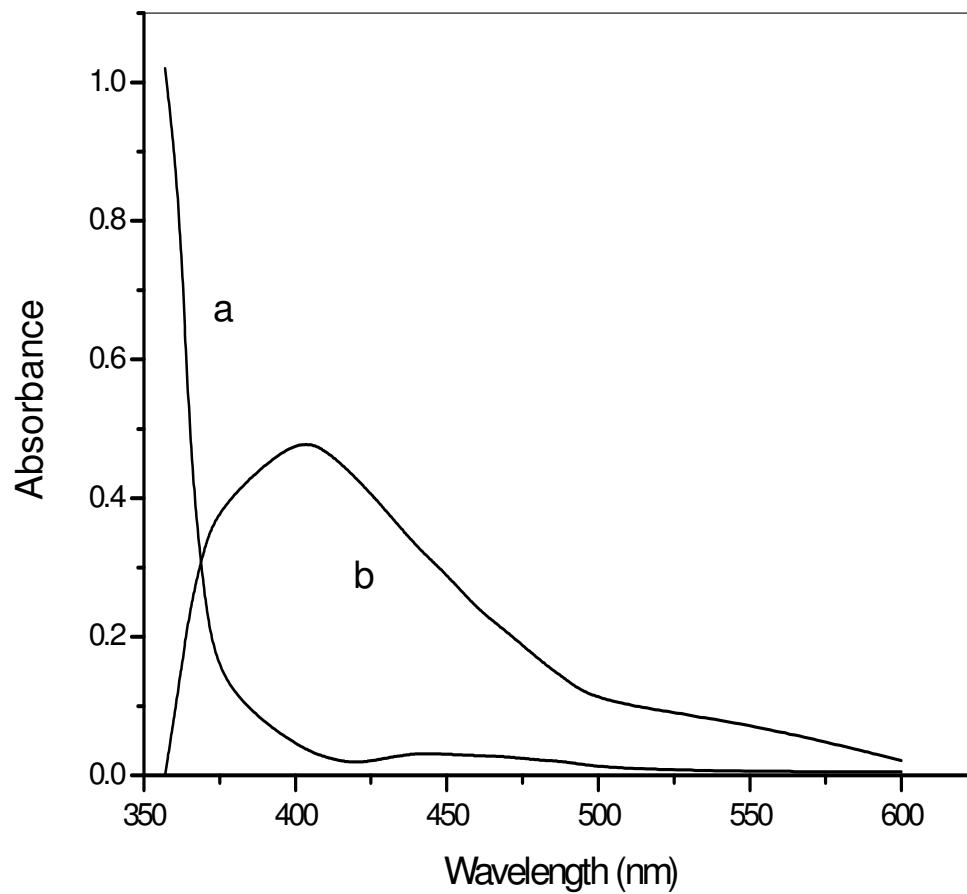
The reaction between iron (III) and 2-Amino acetophenone isonicotinoyl hydrazone (2-AAINH) in the pH range 1.0–6.0 resulted in dark brown coloured water soluble complex. The coloured formation is instantaneous and remained constant in the pH range 3.5 to 6.0. The colour was stable for more than 48 hours.

#### **a) Absorption Spectra**

The absorption spectra of dark brown coloured [Fe (III) –2-AAINH] water soluble complex were measured in the wavelength region 350-600 nm using the general procedure 3a and are presented in Fig. 7.i.1. The metal complex has absorption maximum at 400 nm, where the reagent has considerably negligible absorbance. Therefore, Fe (III) was determined by measuring the absorbance of experimental solutions at 400 nm using reagent blank as reference solution.

#### **b) Effect of pH**

The dark brown colour formation between Fe (III) and 2-AAINH was pH dependent and occurs only in acidic buffer medium. Hence, optimum pH range in which the colour intensity was maximum and constant was determined by measuring the absorbance of metal complex solution at different pH values employing the procedure given in 3b. The results presented in the form of a plot in Fig 7.i.2 reveal that maximum sensitivity can be obtained in the pH range 3.5 to 6.0. Therefore, pH 4.0 was taken as the optimum pH to get maximum sensitivity for the determination of Fe (III).



**Fig. 7.i.1** Absorption spectra of  
 (a) 2-AAINH Vs buffer blank  
 (b) [Fe(III)] – [2-AAINH] Vs reagent blank  
 [Fe(III)] =  $2 \times 10^{-6}$ M; [AAINH] =  $1.0 \times 10^{-2}$ M  
 pH = 4.0

#### **c) Effect of 2-AAINH concentration**

The studies using the procedure 3c revealed that 10 fold molar excess of 2-AAINH was essential for complete and constant colour development. The results presented in Table 7.i.1. reveals that excess of reagent decreases the absorbance.

#### **d) Stability of the colour reaction**

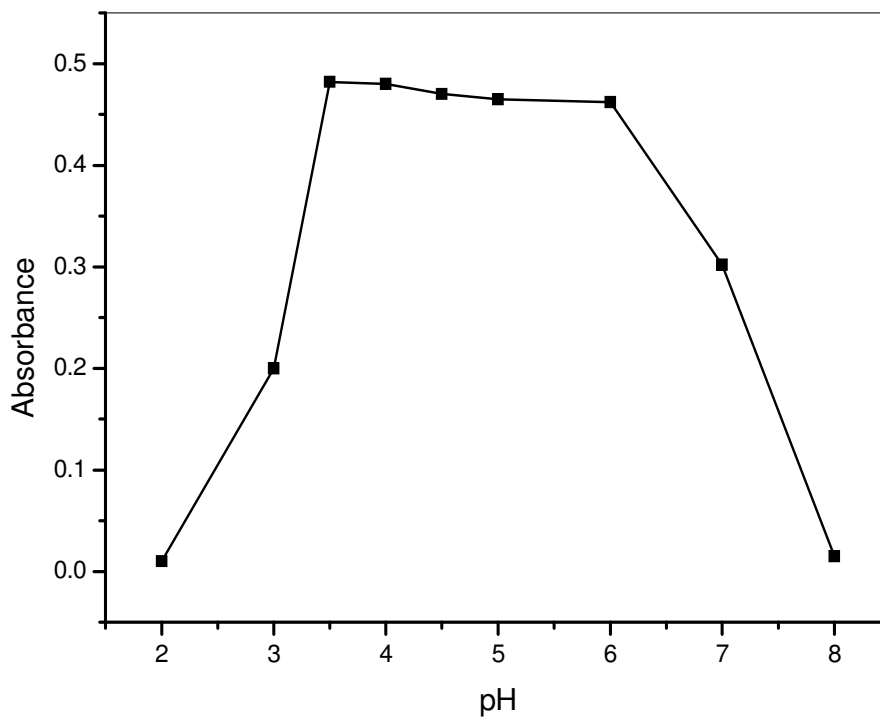
The absorbance of the solution was measured at different time intervals to ascertain the time stability of the colour of the complex as described in 3d. The colour reaction between Fe (III) and 2-AAINH was found to be instantaneous at room temperature and the colour remained stable for more than 48 hours.

#### **e) Applicability of Beer's law**

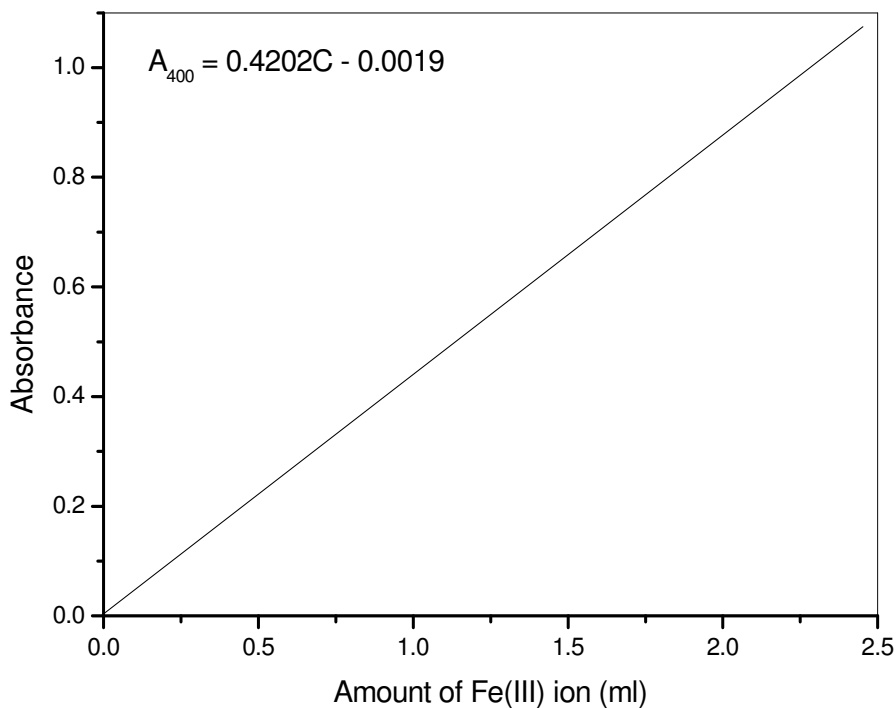
For the possible determination of iron (III) at micro levels, the absorbance of the solutions containing different amounts of metal ion was measured by adopting the procedure 3e. Calibration plot drawn between absorbance and amount of Fe (III) (Fig7.i.3), showed that Beer's law was obeyed in the concentration range 0.25-2.25  $\mu\text{g mL}^{-1}$  of Fe (III). The straight line obeyed the equation  $A_{400} = 0.4202C - 0.0019$ . The Molar absorptivity and Sandell's sensitivity were  $8.82 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$  and  $6.32 \times 10^{-4} \mu\text{g cm}^{-2}$  respectively.

#### **Statistical analysis of results**

The experimental data was subjected with different statistical methods and the results are presented in Table 7.i.2.



**Fig. 7.i.2** Effect of pH on absorbance of [Fe(III)] – 2-AAINH System  
 [Fe(III)] =  $2 \times 10^{-5}$ M; [2-AAINH] =  $5.0 \times 10^{-4}$ M  
 Wavelength = 400 nm



**Fig. 7.i.3** Absorbance Vs Amount of Fe(III) ( $\mu\text{g mL}^{-1}$ )  
 [2-AAINH] =  $5.0 \times 10^{-4}$ M  
 pH = 4.0; Wavelength = 400 nm

**Table 7.i.1: Effect of 2-AAINH concentration**

$$\text{Fe (III)} = 4 \times 10^{-5} \text{ M}$$

$$\text{pH} = 4.0$$

$$\text{Wave length} = 400\text{nm}$$

[Fe(III)] : [2-AAINH]	Absorbance
1:5	590
1:10	615
1:15	600
1:20	589
1:25	578
1:30	564

**Table 7.i.2****Statistical analysis for the experimental data**

Parameter	Fe(III) – 2-AAINH
Angular coefficient (m)	0.42018
Y-Intercept (b)	-0.001
Correlation coefficient (r)	0.9966
Standard deviation (s)	±0.00623

**f. Effect of foreign ions**

The effect of several diverse ions on the determination of Fe (III) was examined under the optimum conditions 3f. The extent of interference by various anions and cations was determined by measuring the absorbance of solutions containing a constant amount of Fe (III) and varying amounts of diverse ions. The results are presented in Table 7.i.3. The results in the Table 7.i.3 reveal that majority of metal and non-metal ions do not interfere in the present method even in more than 100-fold excess. U (VI) and V(V) present were tolerable up to 20 folds. Mo(VI) and Ru (III) were tolerable up to 10 folds.

**Table 7.i.3****Tolerance limits of diverse ions**Amount of Fe (III) = 1.95  $\mu\text{g mL}^{-1}$ 

<b>Diverse ion</b>	<b>Tolerance limit (<math>\mu\text{g mL}^{-1}</math>)</b>	<b>Diverse ion</b>	<b>Tolerance limit (<math>\mu\text{g mL}^{-1}</math>)</b>
Sulphate	989	Ba(II)	1550
Iodide	900	Na(I)	1480
Bromide	880	Mg(II)	1266
Thiourea	844	Zn(II)	125
EDTA	798	Te(VI)	747
Phosphate	740	Al(III)	888
Thiosulphate	555	Cu(II)	802
Chloride	500	Co(II)	789
Carbonate	470	Ce(IV)	714
Oxalate	430	W(VI)	666
Flouride	395	Zn(II)	632
Tartrate	310	La(III)	575
Citrate	250	Sr(II)	521
Nitrate	144	Sn(II)	464
		Cr(VI)	422
		Ga(III)	312
		Ni(II)	211
		Pb(II)	174
		Au(III)	121
		Ag(I)	132
		Pd(II)	98
		Hg(II)	58
		U(VI)	25
		V(V)	20
		Mo(VI)	10
		Ru(III)	10

### g. Composition and stability of the complex

The composition of the brown coloured [Fe(III)- 2-AAINH] complex solution was determined by Job's continuous variation method as described in 3g and the results are presented in Fig.7.i.4. The composition of the complex was also evaluated by molar ratio method as described in procedures 3h the results are obtained are shown in Fig. 7.i.5. All the methods gave 1:1 stoichiometry to the complex. The stability constant of the complex was calculated from Job's curve and obtained as  $7.5 \times 10^6$ .

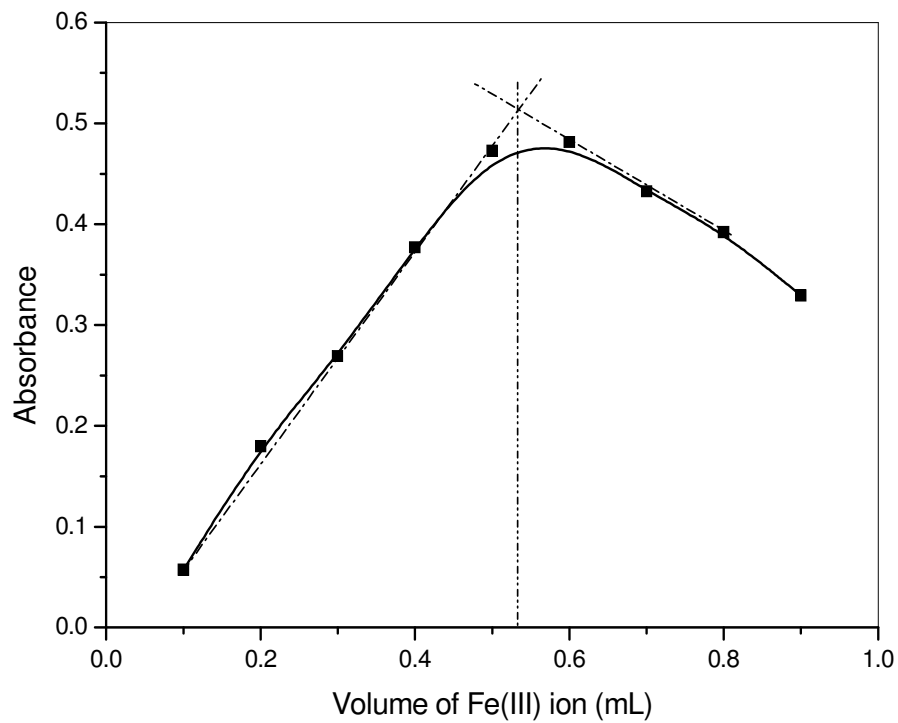
### Applications

The present method was employed for the determination of the amount of iron present in alloy and pharmaceutical samples and surface soil samples. The sample solutions were prepared according to the procedure given in 3(l), 3p and 3q respectively. Different aliquots of sample solutions were treated with known and required volume of 2-AAINH at pH 4.0, and diluted to 10ml with distilled water. The absorbance of the resultant solutions was measured at 400 nm and the amount of iron present was computed from the predetermined calibration plot. The results are presented in Table 7.i.4 and 7.i.5.

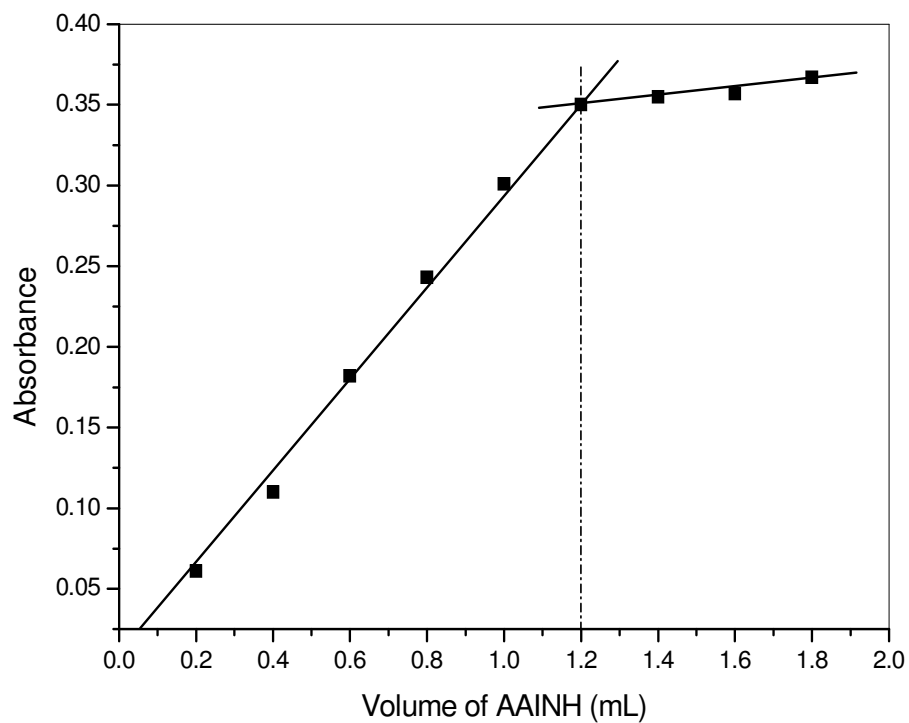
**Table 7.i.4 : Determination of iron in alloy and Pharmaceutical samples**

S.No	Sample	Amount of Fe Found(%), (mg/tablet)	
		Standard method	Present method
<b>I</b>	<b>Alloy/ ore</b>		
1.	Hematite	35.0%	26.8%
2.	Steel	67.5%	60.1%
<b>II</b>	<b>Capsule/ Tablet</b>		
1.	Autrin	32.8mg	30.22mg
2.	Fetol	150mg	142.3mg





**Fig. 7.i.4** Job's curve  
 $[\text{Fe(III)}] = [\text{2-AAINH}] = 2.0 \times 10^{-4}\text{M}$   
 pH = 4.0 ; Wavelength = 400 nm



**Fig. 7.i.5** Mole ratio plot  
 $[\text{Fe(III)}] = [\text{2-AAINH}] = 2.0 \times 10^{-4}\text{M}$   
 pH = 4.0 ; Wavelength = 400 nm

**Table 7.i.5 : Determination of iron in surface soils**

Sample	Source of the Sample	Iron found (mg Kg <sup>-1</sup> )	
		Present method ± S.D (n=5)	AAS method ± S.D (n=5)
S1	Sweet lemon cultivation soil, Garladinne, Anantapuramu district	40.32 ± 0.62	42.43 ± 0.71
S2	Paddy cultivation soil Garladinne, Anantapuramu district	34.76 ± 1.76	35.82 ± 0.44
S3	Groundnut cultivation soil Akuthotapalli, Anantapuramu district	47.62 ± 0.56	45.33 ± 0.67
S4	Cotton cultivation soil Singanamala, Anantapuramu district	25.37 ± 0.64	23.47 ± 0.81

## **Section ii: Derivative spectrophotometric determination of iron (III)**

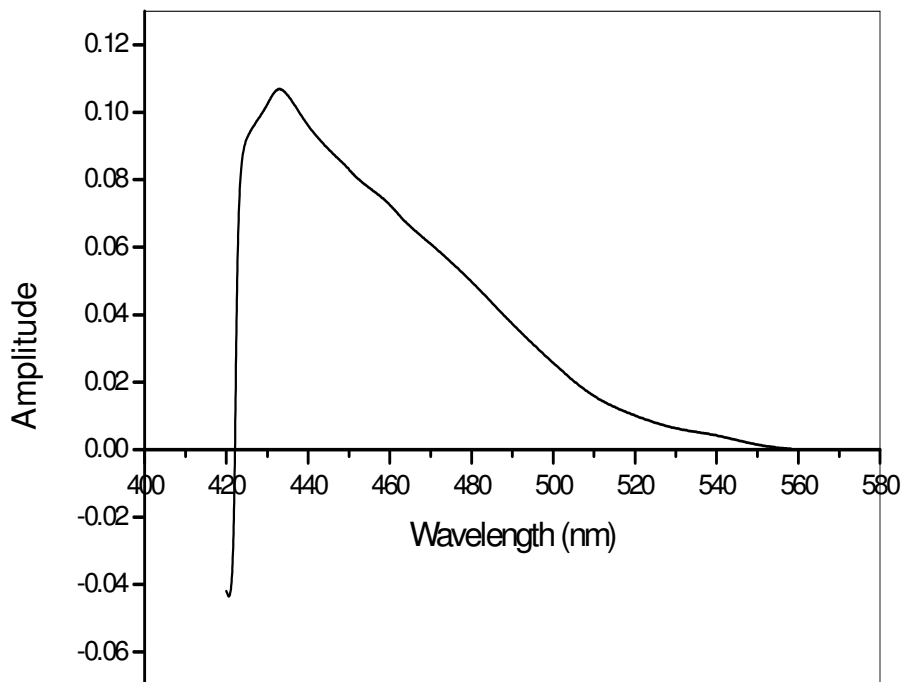
Iron (III) forms dark brown coloured complex with 2-Amino acetophenone isonicotinoyl hydrazone. An attempt was made to develop derivative spectrophotometric methods for the determination of iron (III) in micro amounts. The results are presented in this section (according to general procedure 3i).

### **First derivative method**

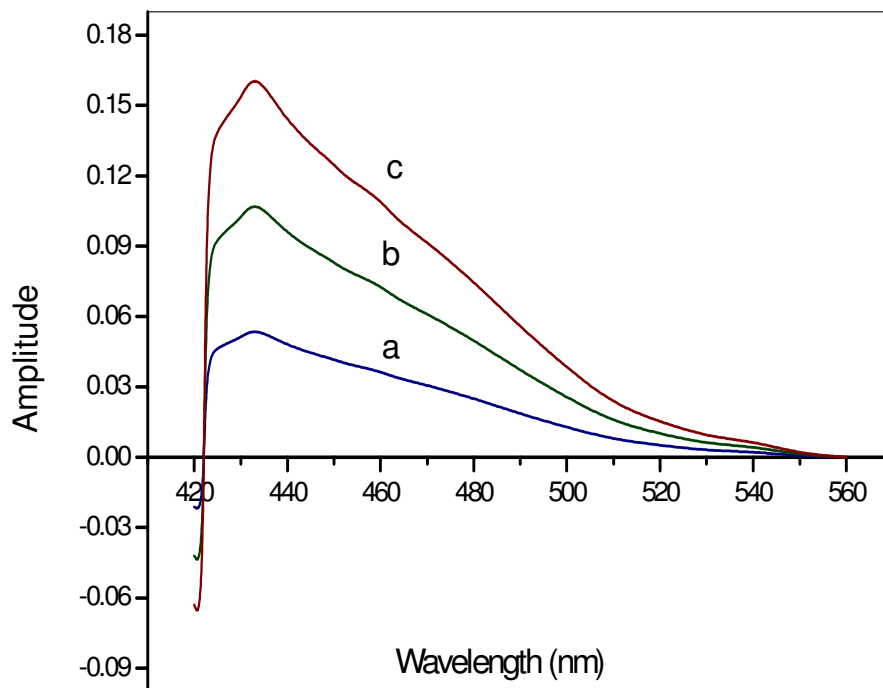
The first derivative spectra of the Fe (III) - 2-AAINH complex at different concentrations of Fe (III) recorded and are presented in Fig. 7.ii.1 & 7.ii.2. To determine the amount of Fe (III) present, a plot was made between the amount of Fe(III) and the derivative amplitude at 433nm are shown in Fig.7.ii.3. The plot was linear and obeyed the equation  $A = 0.0848C - 0.0013$ . The first derivative method allowed the determination of Fe (III) in the range of  $0.134\text{--}2.789\mu\text{g mL}^{-1}$ . The standard deviation of the method for ten determinations of  $1.226\mu\text{g mL}^{-1}$  of Fe (III) was  $\pm 0.0044$ .

### **Second derivative method**

The second derivative spectra (Fig.7.ii.4) show maximum amplitude at 450nm. The derivative amplitudes at 450 nm were proportional to the concentration of Fe (III) Fig. 7.ii.5 (procedure adopted in 3j). A plot was made between the amount of iron (III) and derivative amplitude and shown in Fig. 7.ii.6. The plot was linear in the range of  $0.55\text{--}1.669\mu\text{g mL}^{-1}$  of Fe (III). The standard deviation of the method for ten determinations of  $0.8376\mu\text{g mL}^{-1}$  of Fe (III) was  $\pm 0.0049$ .



**Fig. 7.ii.1** First derivative spectra of  
 Fe (III) – 2-AAINH Vs reagent blank  
 Fe (III) =  $1.106 \mu\text{g mL}^{-1}$  ; pH = 4.0



**Fig. 7.ii.2** First derivative spectra of  
 Fe (III) – 2-AAINH Vs reagent blank  
 Fe (III) ( $\mu\text{g mL}^{-1}$ ) = a) 0.554 ; b) 1.106; c) 1.669;  
 pH = 4.0

### Effect of foreign ions

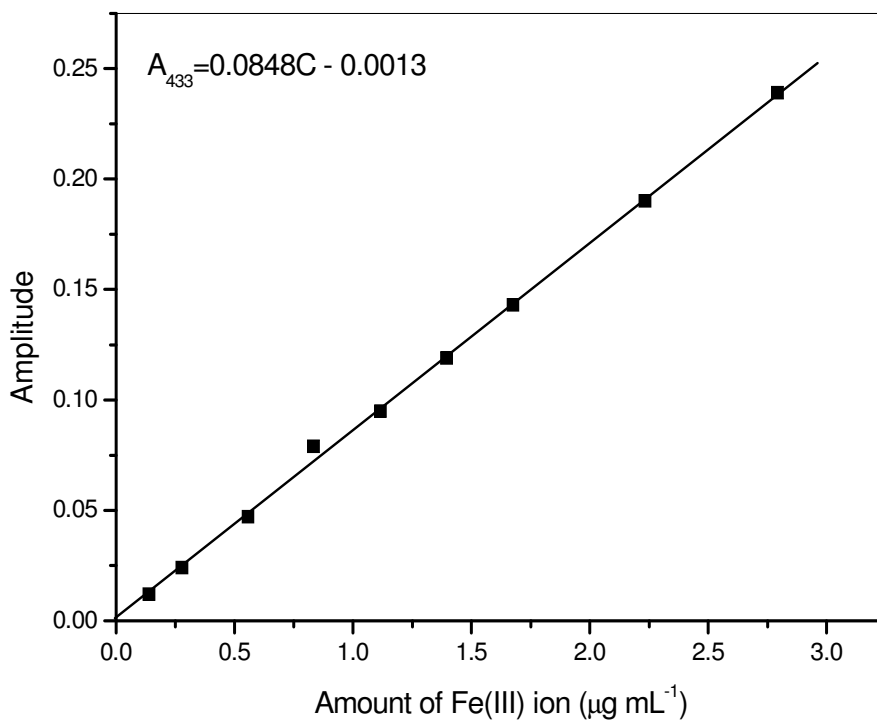
When compared to the zero order method, the tolerance limits of large number of ions which were very low in the zero order method, were found to be improved in the derivative method (procedure adopted in 3k). This shows the greater selectivity of the derivative methods, when compared to zero order method. The results obtained in the studies are presented in Table 7.ii.1.

**Table 7.ii.1 : Effect of foreign ions**

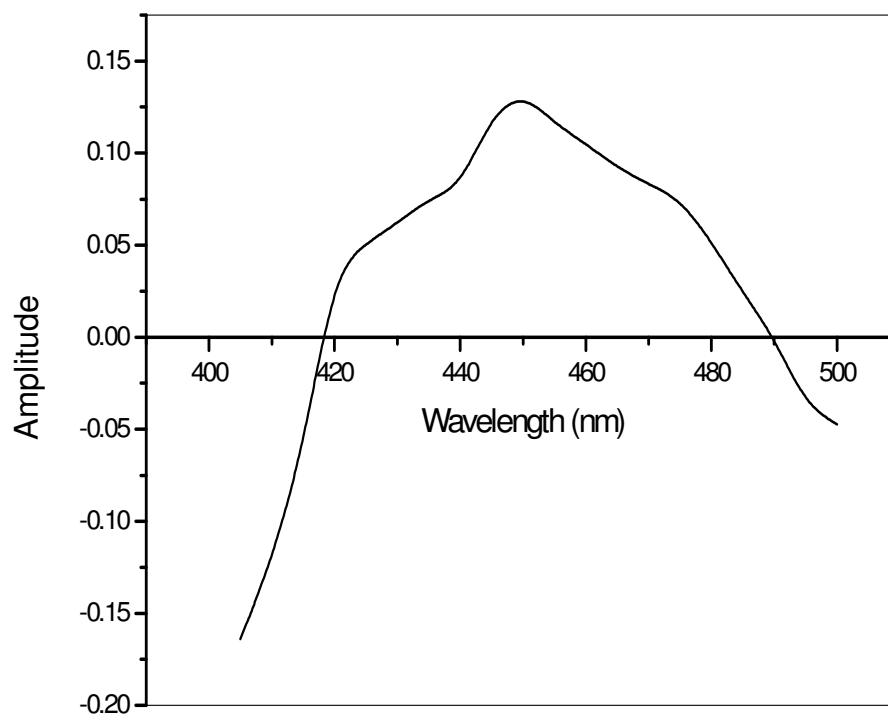
S. No	Diverse Ion	Zero Order	First Derivative	Second Derivative
1.	U(VI)	25	80	> 100
2.	V(V)	20	65	90
3.	Mo(VI)	10	40	>100
4.	Ru(III)	10	60	80

### Analytical results of Fe (III)- 2-AAINH

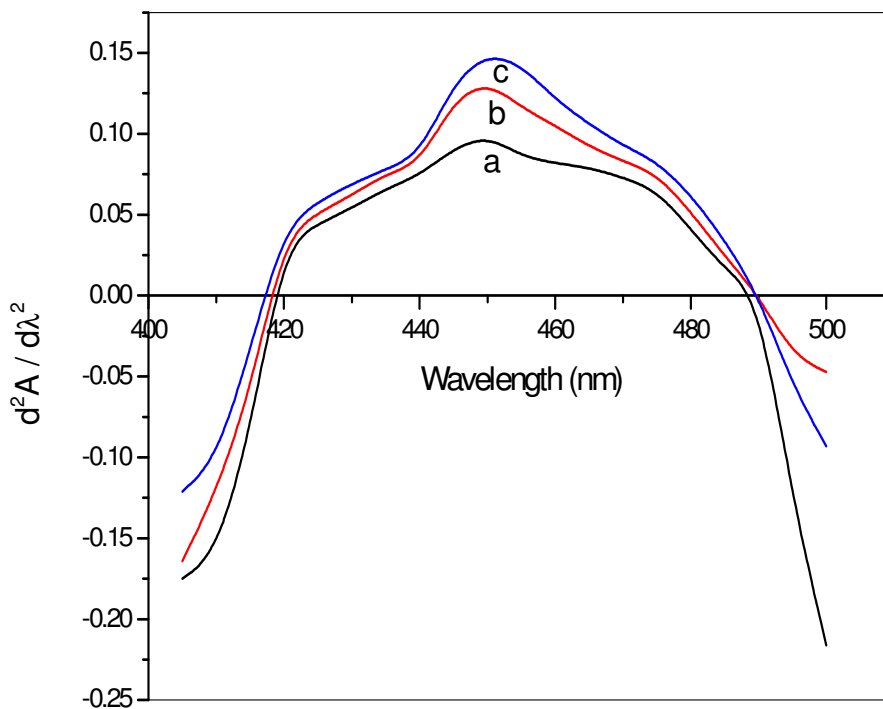
The results obtained in zero order and derivative spectrophotometric methods for [Fe(III)- 2-AAINH] complex were compared and presented in Table 7.ii.2. From this it was noticed that in derivative spectra, the peak position shift towards higher wavelengths and Beer's law range was also improved compared to zero order method.



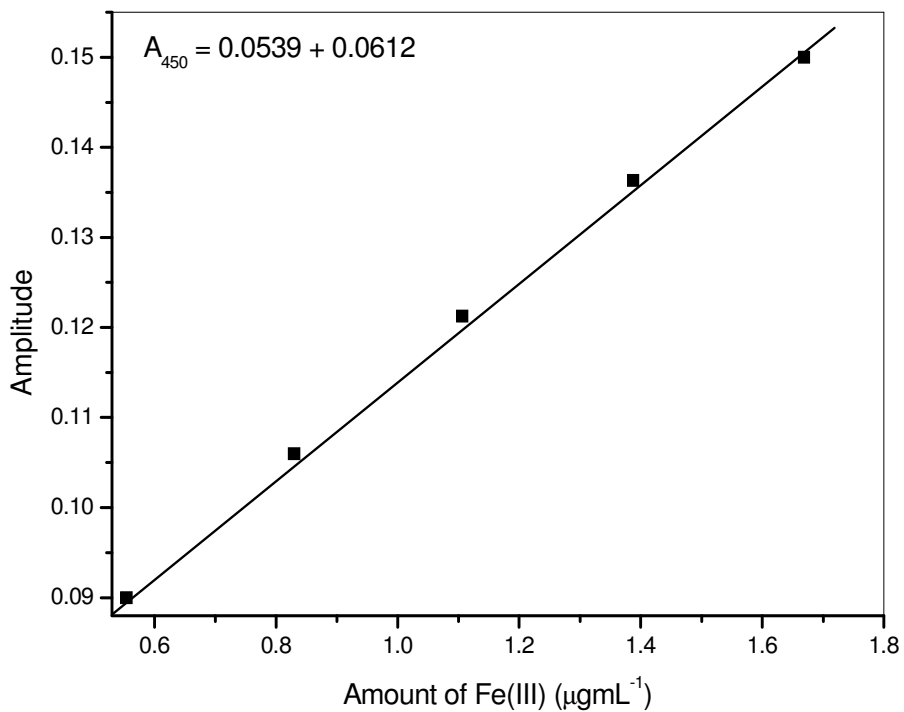
**Fig. 7.ii.3 First derivative amplitude Vs amount of Fe (III)**  
 $\lambda = 433$  ; pH = 4.0  
 $[2\text{-AAINH}] = 5.0 \times 10^{-4}\text{M}$



**Fig. 7.ii.4 Second derivative spectra of**  
 Fe (III) – 2-AAINH Vs reagent blank  
 Fe (III) =  $1.106 (\mu\text{g mL}^{-1})$  ; pH = 4.0



**Fig. 7.ii.5** Second derivative spectra of Fe (III) – 2-AAINH Vs reagent blank  
 Fe (III) ( $\mu\text{g mL}^{-1}$ ) = a) 0.554; b) 1.106; c) 1.669;  
 pH = 4.0



**Fig. 7.ii.6** Second derivative amplitude Vs amount of Fe (III)  
 $\lambda = 450$  ; pH = 4.0  
 $[2\text{-AAINH}] = 5.0 \times 10^{-4}\text{M}$

**Table 7.ii.2**  
**Analytical results of Fe (III)- 2-AAINH**

Parameter	Zero order	First derivative	Second derivative
Analytical wave length (nm)	400	433	450
Beer's law range ( $\mu\text{g mL}^{-1}$ )	0.25-2.25	0.134-2.789	0.554-1.669
Angular coefficient (m)	0.42018	0.0848	0.0539
Y-intercept (b)	-0.0019	-0.0013	0.0612
Correlation coefficient (r)	0.9996	0.9997	0.9997
Standard derivation (s)	$\pm 0.00623$	$\pm 0.0044$	$\pm 0.0049$
Molar absorptivity ( $\text{L mol}^{-1} \text{cm}^{-1}$ )	$8.82 \times 10^4$	-	-
Stability constant	$7.5 \times 10^6$	-	-
Sandell's sensitivity ( $\mu\text{g cm}^{-2}$ )	$6.32 \times 10^{-4}$		
Detection limit	0.0162	0.0272	0.0194
Determination limit	0.0486	0.0813	0.0572

### Discussion

2-Amino acetophenone isonicotinoyl hydrazone reacts with Fe (III) forming a brown coloured water soluble complex [Fe(III)- 2-AAINH] in acidic medium. The complex colour was stable for more than 48 hours. The absorption spectrum of the complex showed maximum absorbance at 400 nm while the reagent absorbance was minimum at this wavelength. The complex solution colour was maximum and constant in the pH range of 3.5-6.0.

The complex solution obeyed Beer's law in the range of 0.25-2.25  $\mu\text{g mL}^{-1}$  with Molar absorptivity  $8.82 \times 10^4 \text{ Lmol}^{-1} \text{cm}^{-1}$ , Sandell's Sensitivity  $6.32 \times 10^{-4} (\mu\text{g cm}^{-2})$ . The experimental data fitted in to a regression equation  $Y = 0.42018x - 0.0019$ . The correlation coefficient ( $r = 0.9996$ ) shows the good linearity for the regression equation. The LOD ( $0.0162 \mu\text{g mL}^{-1}$ ) and LOQ ( $0.0486 \mu\text{g mL}^{-1}$ ) values indicate the sensitivity of the proposed method. Good number of diverse ions are tolerable in reasonably large amounts. All these results indicate that the developed method is



simple, sensitive and selective. To establish the applicability of the proposed method, it was applied for the determination of iron in surface soil samples and commercial samples. In order to improve the sensitivity and selectivity of the analytical method proposed, an attempt has been made to develop derivative spectrophotometric methods for the determination of iron (III), in which the Beer's law range is improved. The results of the present method were compared with those of some of the reported spectrophotometric methods and are presented in Table 7.ii.3.

The results reveal that the present method is non-extractive, or any other stringent reaction conditions and offers the advantage of high color stability (48h). The intensity of the coloured species will not be affected by slight variation of the experimental parameters such as concentration of the reagent. when compared its sensitivity and selectivity with the reported methods, iron (III) establishes its place for the spectrophotometric determination.

**Table 7.ii.3**

**Comparison of the analytical results of the proposed method with already reported methods for iron (1 of 2 Pages)**

**Table 7.ii.3**

**Comparison of the analytical results of the proposed method with already reported methods for iron (2 of 2 Pages)**

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**Table 7.1**  
**Analytical results of various spectrophotometric methods for iron(III)**

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ ( $\text{Lmol}^{-1}$ $\text{cm}^{-1}$ )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/ extraction	Remarks/ Interference	Ref. No
3-methoxy Salicylaldehyde thiosemicarbazone	590	2.0-5.1	0.4	-	Aqueous	Heating is necessary	13
3-amino-5,6-di(2-pyridyl),1,2,4-triazine	540	-	-	-	Extraction	-	14
Ferron	465	-	-	-	Extraction	-	15
Triton and cetyl pyridium chloride	520	5.6	1.58	1-14	Extraction	-	16
3-hydroxy picolic acid	440	5.5	1.50	-	Aqueous	-	17
3-hydroxy-2-picolinamide	425	-	2.24	0.2-0.5	Aqueous	-	18
2-hydroxy nicotinic acid	360	5.0	4.88	-	Aqueous	-	19
5-methyl Salicylaldehyde guanyl hydrazone	430	8.8	0.32	2-75	Aqueous	-	20
	370	4.5-5.5	1.20	0.3-4.5	Aqueous	-	21
	375	5.0-6.0	1.10	0.3-3.0	Extraction(n-butanol)	Co, Cu & Ni interfere	22



Table 7.1 continued

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ ( $\text{Lmol}^{-1}$ $\text{cm}^{-1}$ )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/ extraction	Remarks/ Interference	Ref. No
Cyclohexanone-1,2,3-trione,1,3-dioxime-2-thiosemicarbazone	570	HCl	1.65	0-4.0	Aqueous	Applied to the determination of Fe in vinyl foods	23
2,4-Diacetophenone thiosemicarbazone	360	4.6	0.50	1.1-7.8	Aqueous	-	24
2,4'-Dihydroxy benzophenone thiosemicarbazone	360	3.0-5.5	0.75	0.67-4.7	Aqueous	-	25
5,5'-Dimethyl-1,2,3-cyclohexanetrione-1,2-dioxime-3, thio semicarbazone	550	Strongly acidic medium	0.89	-	Aqueous	Applied to the determination of Fe in wines, minerals and in foods	26
2-hydroximino cyclohexanone thiosemicarbazone	516	1.0-1.5	0.60	-	Aqueous	-	27
5-bromosalicylaldehyde thiosemicarbazone	385	5.0-6.0	1.72	0.28-5.60	Aqueous	The method has been employed successfully for the determination of Fe(II) in grape leaves, multivitamin capsules and human blood	28

Table 7.1 continued

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ (Lmol <sup>-1</sup> cm <sup>-1</sup> )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/ extraction	Remarks/ Interference	Ref. No
1-Phenyl-1,2-Propanedione-2-oxime thiosemicarbazone	395	-	-	-	Aqueous	The method is applied for the determination of Cu(II) and Ni(II) in synthetic mixtures and also in edible oils and seeds	29
Pyridine-2-,6-diphydrazide	524	10	19,500	0.3-6.0	Aqueous	-	30
Di-2-pyridylmethanone 2-(5-nitro) pyridylhydrazone	505	2.0-7.5	5.38	0-8.4	Tolerance	Extremely sensitive	31
3-(4-phenyl-2-pyridyl)-5,6-diphenyl-1,2,4-triazine and tetraphenylborate	567	5.1-7.4	$2.9 \times 10^4$	-	Naphthalene	-	32
Bromopyrogallol red and pyrogallol red	635	-	$5.6 \times 10^4$	0.08-0.5	Quaternary ammonium	Interferences of metal ions supposed by using masking agents	33
2-(3,5-Dibromo-2-pyridylazo)-5-[N-ethyl-N-(3-sulfopropyl) amino] phenol	750	-	39000	20-440	Aqueous	Interferences of other metals eliminate by using N-(dithiocarboxy) sarcosine and EDTA	34
4-hydroxy-1,10-phenanthroline	750	10	$1.19 \times 10^4$	$1 \times 10^{-5}$ - $8 \times 10^{-5}$	Alkaline	-	35
Pyridine-2-aldehyde guanylhydrazone	380	8.5	$1.3 \times 10^4$	0.5-4.7	Aqueous	-	36

Table 7.1 continued

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ (Lmol <sup>-1</sup> cm <sup>-1</sup> )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/extraction	Remarks/ Interference	Ref. No
2,3 dihydroxy benzaldehyde isonicotinoyl hydrazone	-	-	-	-	-	-	37
Di-2-pyridyl ketone benzylhydrazone	-	-	-	-	-	-	38
2-hydroxy1-naphthal dehyde p-hydroxy benzoic hydrazoe	405	-	5.6	0.118-3.534	Aqueous	-	39
4-hydroxy3,5 dimethoxy benzaldehyde 4 hydroxy beozoyl hydrazone	380	5.0	1.71	0.279-2..79	C-TAB(5%)	-	40
Picolinaldehyde thiosemicarbazone	610	9.4- 12.3	0.58	-	Extraction	-	41
Quinoline -2-aldehyde thiosemicarbazone	600	7.2-8.0	0.39	0.5-6.0	Extraction	-	42
Salicylaldehyde thiosemicarbazone	590	10.0	0.17	1-28	Aqueous	Fe and Mo can be determined in presence of each other	43
2-Acetyl Pyridine thiosemicarbazone	550	2.0-3.0	0.53	0-8.0	Aqueous	-	44

Table 7.1 continued

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ (Lmol <sup>-1</sup> cm <sup>-1</sup> )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/extraction	Remarks/ Interference	Ref. No
Biacetyl-2(pyridyl) hydrazone thiosemicarbazone	570	Acidic medium	0.40	0.8-11.2	Aqueous	Cu and Fe determined in alloys, ores and pharmaceutical preparations	45
2,2'-Dihydroxy benzophenone thiosemicarbazone	365	3.0-3.5	0.59	1.0-2.0	Aqueous	-	46
2,4-Diacetophenone thiosemicarbazone	360	4.6	0.50	1.1-7.8	Aqueous	-	47
2-2'Dipyridyl-2- furancarbothiohydrazone	738	3.0-8.0	1.17	0-40	C <sub>6</sub> H <sub>6</sub>	-	48
1,3 diphenyl- 4- carboethoxy pyrazole -5- one	525	3.5-4.0	Aqueous	0.5-10	1.156	Cu(II), Co(II), Zn(II), Mo(VI) and EDTA	49
Di formyl hydrazide	470	7.3-9.3	Aqueous	0.25-13	.3258	-	50
4,7-di phenyl 1,10 phenanthroline and tetraphnyl borate	534	-	Extraction	0-20.0	2	-	51

Thio cyanate phenanthroline	520	-	Aqueous	0-24	1.87	-	52
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Table 7.1 continued

Reagent used	$\lambda_{\max}$ (nm)	pH	$\epsilon \times 10^4$ (Lmol <sup>-1</sup> cm <sup>-1</sup> )	Beer's law range ( $\mu\text{g mL}^{-1}$ )	Aqueous/extraction	Remarks/ Interference	Ref. No
Thiocyanate acetone	480	HClO <sub>4</sub>	Aqueous	-	2.1	NO <sub>2</sub> <sup>-</sup> , S <sub>2</sub> O <sub>3</sub> <sup>2-</sup> , H <sub>2</sub> PO <sub>4</sub> <sup>2-</sup> and C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	53
Salicylaldehyde acetic acid hydrazone	395	3.0	-	0.027-0.27	61.2x10 <sup>-3</sup>	-	54
2,4 di hydroxy benzaldehyde Isonicotinyl {(E)-N <sup>1</sup> -(2,4 di hydroxy benzeledine) hydrazone	395	7.0	-	0.1-1.5	3.5	Co(II), V(V), Al(III) and Pb(II)	55
4-hydroxy 3,5-di methoxy benzaldehyde 4hydroxy benzoyl hydrazone	380	1.0-6.0	Aqueous	0.279-2.79	1.71	-	56
2 hydroxy-1 naphthaldehyde-p-hydroxy benzoic hydrazone	405	5.0	Aqueous	0.05-1.37	5.6	Sn(II), Co(II), Ni(II), Zn(II), Al(III) and Cu(II)	57
2-Amino Acetophenone Isonicotinoyl Hydrazone	400	4.0	8.82	0.25-2.25	-	-	Present Method

**Table 7.ii.3**  
**Comparison of the analytical results of the proposed method with already reported methods for iron**

Reagent	$\lambda_{\max}$	pH/ medium	Aqueous/ extraction	Beer's law range ( $\mu\text{g mL}^{-1}$ )	$\epsilon \times 10^4$ $\text{L mol}^{-1}\text{cm}^{-1}$	Interference	Ref. No
3-methoxy Salicylaldehyde thiosemicarbazone	590	2.0-5.1	0.4	-	Aqueous	Heating is necessary	13
5-methyl Salicylaldehyde guanyl hydrazone	430	8.8	0.32	2-75	Aqueous	-	20
Picolinaldehyde thiosemicarbazone	610	9.4-12.3	0.58	-	Extraction	-	41
Quinoline -2-aldehyde thiosemicarbazone	600	7.2-8.0	0.39	0.5-6.0	Extraction	-	42
Salicylaldehyde thiosemicarbazone	590	10.0	0.17	1-28	Aqueous	Fe and Mo can be determined in presence of each other	43
2-Acetyl Pyridine thiosemicarbazone	550	2.0-3.0	0.53	0-8.0	Aqueous	-	44
Biacetyl-2(pyridyl) hydrazone thiosemicarbazone	570	Acidic medium	0.40	0.8-11.2	Aqueous	Cu and Fe determined in alloys, ores and pharmaceutical	45

						preparations	
2,2'-Dihydroxy benzophenone thiosemicarbazone	365	3.0-3.5	0.59	1.0-2.0	Aqueous	-	46
2,4-Diacetophenone thiosemicarbazone	360	4.6	0.50	1.1-7.8	Aqueous	-	47

Table 7.ii.3 continued

Reagent	$\lambda_{\max}$	pH/ medium	Aqueous/ extraction	Beer's law range ( $\mu\text{g mL}^{-1}$ )	$\epsilon \times 10^4$ $\text{L mol}^{-1}\text{cm}^{-1}$	Interference	Ref. No
2-2'Dipyridyl-2-furancarbothiohydrazone	738	3.0-8.0	1.17	0-40	$\text{C}_6\text{H}_6$	-	48
1,3 diphenyl- 4- carboethoxy pyrazole -5- one	525	3.5-4.0	Aqueous	0.5-10	1.16	Cu(II), Co(II), Zn(II), Mo(VI) and EDTA	49
Di formyl hydrazide	470	7.3-9.3	Aqueous	0.25-13	0.3258	-	50
Thio cynate phenanthroline	520	-	Aqueous	0-24	1.87	-	52
Thiocyanate acetone	480	$\text{HClO}_4$	Aqueous	-	2.1	$\text{NO}_2^-$ , $\text{S}_2\text{O}_3^{2-}$ , $\text{H}_2\text{PO}_4^{2-}$ and $\text{C}_2\text{O}_4^{2-}$	53
2,4 di hydroxy benzaldehyde Isonicotinyl {(E)-N <sup>1</sup> -(2,4 di hydroxy benzeledine) hydrazone	395	7.0	-	0.1-1.5	3.5	Co(II), V(V), Al(II) and Pb(II)	55
4-hydroxy 3,5-di methoxy benzaldehyde 4hydroxy benzoyl hydrazone	380	1.0-6.0	Aqueous	0.279-2.79	1.71	-	56
2 hydroxy-1 naphthaldehyde-p-hydroxy benzoic hydrazone	405	5.0	Aqueous	0.05-1.37	5.6	Sn(II), Co(II), Ni(II), Zn(II), Al(III) and Cu(II)	57
2-Amino Acetophenone isonicotinoyl hydrazone	400	4.0	Aqueous	0.25-2.25	1.245	-	Present method



