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A comparative study of determination of Iron (II) by analytical methods

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Abstract:

Iron is an essential trace element. In human body, iron is used for the formation of haemoglobin and oxidative process of living tissues. Iron is used in the prevention and treatment of iron-deficiency anaemia. A large number of analytical methods have been employed for the determination of Fe(II) at trace level. The present study deals with the comparison of two analytical methods for the determination of Fe(II). Spectrophotometric determination of Fe(II) with o-phenanthroline and potentiometric titration of Fe(II) with potassium dichromate are the analytical methods selected for the comparison study. The standard solution of Fe(II) has been prepared. The concentration of Fe(II) in standard solution has been found out by known method. Further investigation of Fe content in Fe-Tablets has been carried out by the analytical methods. The results obtained from these analytical methods were compared and reported in this paper.

(Keywords: Iron, Fe(II), analytical, spectrophotometric, potentiometric, Tablet.)

1.0 Introduction:

Iron is the fourth most abundant element on the earth. It is an essential biological trace element for all living organisms. The human body contains on an average of 3-4 g of iron [1,2]. Most of this iron exists in the complex forms bound to proteins such as haemoglobin and myoglobin. Iron is used in the prevention and treatment of iron-deficiency anaemia. Iron is a mineral that is available as a dietary supplement. It works by helping the body to produce red blood cells. In pharmaceutical industries, iron is used in various forms which are consumed as tablets, capsules, injectables, syrups and drinking ampules. Iron exists in two oxidation states Fe(II) and Fe(III). The balance between these two forms is also important for the metabolism of iron in the biological systems [3]. The oxides of iron are used as inorganic dyes, pigments for cosmetics and food additives [4,5].

A large number of analytical methods have been employed for the quantitative determination of iron at trace levels. These methods include spectrophotometry [6-11], fluorimetry [12,13], voltametric methods [14-16], atomic emission and absorption spectrometry [17,18], capillary electrophoresis [19-20]and chromatographic techniques [21,22]. The present paper reports the comparison study of the different analytical methods for the quantitative estimation of Fe(II) in an iron tablet. Spectrophotometric determination of Fe (II) with 1,10-phenanthroline, redox titration of Fe (II) with Ceric ammonium sulphate and potentiometric titration of Fe (II) with potassium dichromate are the analytical methods selected for the comparison study. These methods have been used to analyse Fe content in Ferrous Ascorbate tablets. Ferrous Ascorbate is an antianemic agent. It is a synthetic molecule of ascorbic acid and iron. [23] This drug is used as a vital vitamin that help pregnant women to attain a good health during the entire pregnancy.

2.0 Experimental:

2.1 Instruments and Apparatus:

Equiptronics Digital spectrophotometer EQ820D with 1 cm quartz cells was used to measure the absorbance of solution. Equiptronics Potentiometer (Model no.EQ 602) was used. Saturated calomel electrode and Platinum electrode were used. Electric hot plate was also used.

2.2 Chemicals and Reagents:

Analytical grade (AR) chemicals were used for the experimental work. Ferrous ammonium Sulphate hexahydrate (BDH), 0.1 % of 1,10-phanathroline (Aldrich 99%), 6 M hydrochloric acid (Aldrich 37%), 10 % sodium acetate (BDH, AnalaR), 10 % hydroxylamine hydrochloride (Merck), 0.1 N potassium dichromate (Merck), Saturated potassium chloride, 4 N sulphuric acid, diphenylamine indicator, 0.1 N ceric ammonium sulphate (Aldrich) and ferroin indicator were prepared by dissolving the suitable amount of respective reagent in distilled water.

Ferrous Ascorbate tablets (Orofer XT- Emcure pharmaceuticals ltd.) were used for quantitative estimation of Fe(II) .

2.3 Preparation of Standard Fe(II) solution :

Standard ferrous ion solution (1000 mg/ dm^3) was prepared as follows: 7.02 g of analytical grade ferrous ammonium sulphate hexahydrate

[Fe $(NH4)_2(SO4)_2.6H_2O$] was accurately weighed and quantitatively dissolved into distilled water. 5 cm^3 of concentrated sulphuric acid was added. Then the solution was diluted to 1000cm3 with distilled water. The amount of Fe(II) in the standard solution was determined by a known method.

2.4 Preparation of Fe-Tablet sample solution:

One Fe- tablet (Ferrous Ascorbate tablet) equivalent to 100 mg of iron was crushed into fine powder in mortar and pestle. The powder was then transferred into a beaker and boiled with 25 cm^3 of 6M HCl for 15 minutes. The solution is cooled and filtered into a volumetric flask of 100 cm^3. It was further diluted with distilled water to the mark. This solution was labelled as Fe-Tablet Sample solution.

3.0 Analysis of Fe (II) in standard solution:

The stock solution of Fe (II) was standardized by titration with potassium dichromate using diphenylamine as an indicator.

10 cm^3 of stock solution was taken in a conical flask. To this solution, 30 cm^3 of dilute sulphuric acid, 25 cm^3 of water, 7ml of 85% phosphoric acid and 5 drops of diphenylamine indictor were added. The solution was titrated with dichromate till color changes to blue. The amount of Fe(II) in the standard solution was calculated. The calculated amount was considered as content of Fe (II) in the standard solution.

The Fe content in Fe(II) standard solution was also determined by potentiometric titration with potassium dichromate and Direct titration with Ceric ions using ferroin indicator. The purpose was to compare the Fe content in standard and tablet sample estimated by these analytical methods. The amount of Fe(II) in standard solution is reported in Table 1.

4.0 Analytical Methods for quantitative estimation of Fe (II) in Fe-Tablet:

4.1 Spectrophotometric Determination of Fe in Fe-tablet sample solution:

Ferrous ions (Fe^{2+}) react with 1,10 phenanthroline in a ratio of 1:3 to form an orange red coloured complex $[(C_{12}H_8N_2)3Fe]^{2+}$ in aqueous medium.

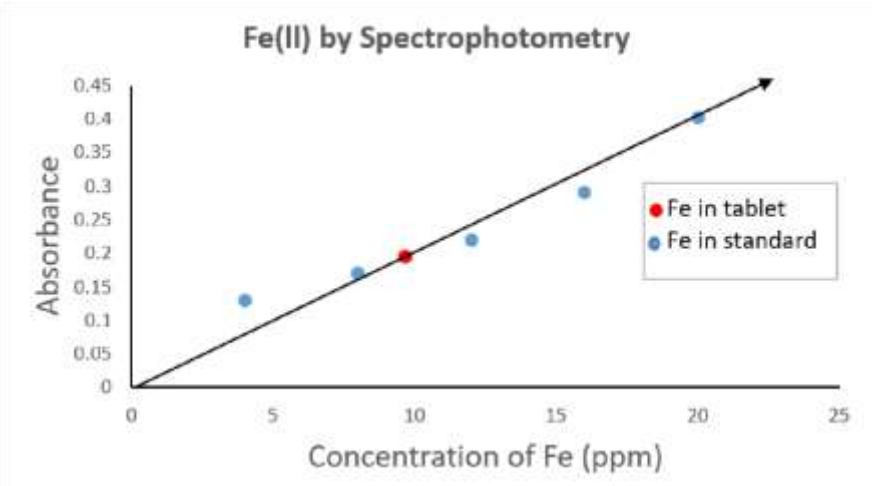
$$Fe^{2+} + 3PhenH^{+} \leftrightarrow Fe\ (Phen)_3\ ^{2+} + 3H^{+}$$

The absorbance of the coloured complex is measured at 508 nm using a spectrophotometer.

. Five standard solutions of Fe(II) were prepared in the range of 4 to 20 ppm using the standard solution of 1000 ppm Fe(II) solution. To these standard solutions of Fe(II) , 1 cm^3 of hydroxylamine hydrochloride and 5 cm^3 of 1, 10phenanthroline and 8 cm^3 of Acetic acid-sodium acetate were added. After 15 minutes, when the colour of complex is developed in all flasks then the dilutions were made to 100 cm^3 with distilled water. 1 cm^3 of Fe-Tablet Sample solution was diluted to 100cm^3 with distilled water in a volumetric flask. 10 cm^3 of

this solution was taken in a volumetric flask labelled as Sample for the spectrophotometric method. This sample solution was treated in the same manner as that of standard solutions. The absorbance of the standard solutions and sample solution were read at 508 nm by using a reagent blank solution. The calibration curve was plotted for Fe (II) and the concentration of Fe (II) in the Tablet Sample was obtained from the calibration curve (Graph 1). The amount of Fe in tablet sample was further calculated and results are recorded in Table 1.

Graph 1



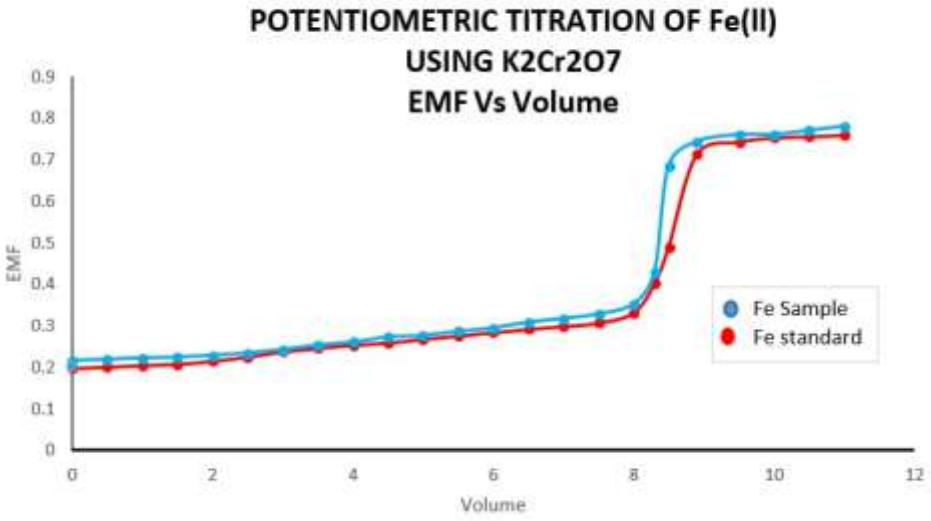
4.2 Determination of Fe(II) in Fe-Tablet sample by Potentiometric titration of Fe(II) with Potassium dichromate :

This method is based on the redox reaction between ferrous ions and dichromate ions using potentiometer. During this potentiometric titration, the solution potential changes due to the change in the concentration of oxidised/reduced form. At the end point, there is a sharp change in potential.

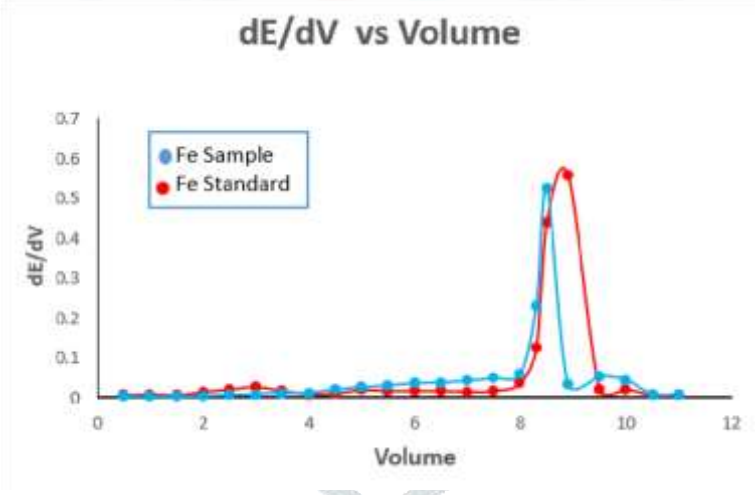
$$Cr_2O2^{-7} + 6\ Fe^{2+} + 14H^{+} \rightleftharpoons 6\ Fe^{3+} + 2Cr^{3+} + 7\ H_2O$$

To 25 cm^3 of Fe-Tablet sample solution, 25 cm^3 dilute H_2SO_4 and 50 cm^3 of distilled water were added in a 250 cm^3 beaker. Platinum electrode is dipped into this solution as the indicator electrode. Saturated Calomel Electrode is used as the Reference electrode and. KCl salt bridge was used to connect the two half cells. To the Fesolution, 0.5 cm3 of 0.1 N Potassium dichromate solution was added from the burette and emf was measured for the cell. The addition of dichromate solution was continued to observe the changes in emf measurements. The readings were recorded. The change in emf readings was also calculated against the volume of dichromate added. Two graphs were plotted to locate the equivalence point (Graph 2 and 3). The amount of Fe (II) ions in Fe-Tablet sample solution was calculated and recorded in Table 1.

Graph 2



Graph 3



4.3 Analysis of Fe(II) in Fe-Tablet sample solution by Redox titration with ceric ammonium sulphate :

In this method, ferrous ions react with ceric ammonium sulphate by a redox reaction. The end point of this redox titration is obtained by using ferroin as redox indicator. Ceric ions act as oxidising agent and reduces ferrous to ferric ions. The redox reaction is represented as

$$Ce^{4+} + Fe^{2+} \rightarrow Ce^{3+} + Fe^{3+}$$

The burette is filled up with 0.1 N Ceric ammonium sulphate solution. In a conical flask 25 cm^3 of Fe-Tablet sample solution was taken. 25 cm^3 of dil. Sulphuric acid was added. 2-3 drops of ferroin indicator were added. The solution was titrated against the ceric solution in burette till the disappearance of red colour. The procedure is repeated three times and the burette readings were recorded. The amount of Fe(II) in Fe-Tablet sample solution was calculated and reported in Table 1.

Table 1

Analytical Method	Fe content (Amount in mg)				
	Standard solution		Fe-Tablet sample		
	Label	Observed	Label	Observed	Relative error
Spectrophotometry	100	----	100	97.8	0.022
Potentiometry With dichromate	100	98.9	100	96.8	0.032
Redox titration with ceric ions	100	98.4	100	95.9	0.041

5.0 Conclusion and Discussion:

Iron content in Fe- Tablet was found out by three different analytical methods. The results obtained were compared with amount of Fe(II) present in the standard solution. Relative error for these methods were calculated. Spectrophotometric determination of Fe(II) with 1,10 phenanthroline was found to give better results in comparison with the other two methods. Direct titration method of Fe(II) with Ce(IV) is very simple and less time consuming method. But the accuracy is less as compared to other two methods. Potentiometric titration of Fe(II) with dichromate ions requires long time to carry out. But in this method emf changes during redox reaction can be observed simultaneously. Equivalence point was located by graphical methods. Estimation of Fe(II) in biological and environmental samples by these analytical methods will be done in future.

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