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Iron Determination by Colorimetric Method Using O-Phenanthroline

Abstract

Fe^{2+} reaction with 1, 10-Phenanthroline determined the quantity of soluble iron (II) in the sample to transform a weakly colored iron into an intensely colored complex which could be used in the analysis. The substance absorbed certain wavelengths when a light from the source with a certain intensity and frequency range was passed to this intensely colored complex. The intensity of a solution's color is proportional to the absorbing species concentration and the absorption is proportional to the substance concentration. A separate concentration of standards was prepared and absorbance in 511 nm, the largest wavelength, was determined using Colorimeter. Using the same standard technique and reagents, a blank and three unknown samples were also prepared. A calibration curve was built following Beer's Law. The iron concentration was verified using the equation of the calibration curve and the absorption under the same experimental conditions of three unknowns.

Keywords: Colorimeter, 1,10 phenanthroline, standard solutions, calibration, wavelength

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1. Introduction

The variation of the colour of the system with concentration is the basis of colorimetry. Colorimetric analysis is useful for the systems in which substances or their solutions are colored. Colorimeters are provided with the arrangements to select appropriate wavelength of light with the help of filter and grating. A light of proper wavelength is allowed to pass through a coloured test solution. The light transmitted from this solution is made to pass through photocell and proportional to the amount of light the solution transmits. In the photocell, a current is produced. A meter is calibrated to demonstrate the absorbed light fraction proportional to the colored substance concentration in the sample solution. From the measurements concentrations of coloured solution can be obtained by using Lambert-Beer law. When a substance is colorless, then a suitable complexing agent is added to the solution so that a colored complex is obtained.

There has now been an increasing awareness of the significance of lot of metals in biological systems. For instance, iron is needed in transport of oxygen and in many enzymes for energy storage, copper and zinc are present and molybdenum is essential in bacteria and plants fixing nitrogen and catalyzing multiple oxidation responses. It may lead in severe illness or death if any of these vital components are missing from diet [2].

Many individuals bring "mineral additives" pills to guarantee that these metals are adequate. Iron-added vitamins are prevalent. Soluble iron (II) is the iron present in the vitamins. For living systems,

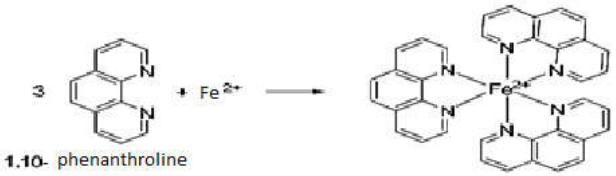
the other type of iron, insoluble iron (III) is usually ineffective. Despite their elevated iron content, soil regions rich in insoluble iron (III) do not support plant life [3].

In the experiment, the amount of iron in a water sample was determined. Since the iron in the strongly acid solution is in the ferric state, it is necessary to reduce to ferrous state before adding o-phenanthroline. Most materials could be transformed to a colored derivative and easy spectrophotometry could thus be determined.

Hydroxylamine hydrochloride ($NH_2OH.HCl$) is an additional reducing agent to ensure that all dissolved iron was Fe^{2+}. It decreased any iron (III) existing in iron (II). Although there are many other techniques to decrease Fe^{3+} to Fe^{2+}, hydroxylamine hydrochloride is best because it does not interfere with absorption measurement:

$$2\ Fe^{3+}_{(aq)} + 2\ NH_3OH^+_{(aq)} \xrightarrow{\text{acid}} 2\ Fe^{2+}_{(aq)} + N_{2(g)} + 4\ H^+_{(aq)} + 2\ H_2O_{(l)}$$

In the estimation, the iron (II) could not be used directly since Fe^{2+} is only slightly colored (pale blue green). Before analyzing it was reacted to create an intense red color with 1, 10-phenanthroline.[6] The stoichiometric reaction of Fe^{2+} is shown below with three ligand molecules:



In order to regulate the pH of the components, acetic acid – sodium acetate has been introduced as the complex created is very susceptible to pH in order to prevent mistakes in the assessment owing to deviation from the Beer Law (nonlinearity). Nonlinearity of chemical causes occurs when there is a non-symmetric equilibrium. An instance is a weak acid that absorbs at a specific wavelength but does not have an anion. The proportion of acid to salt depends on the pH. This ratio will stay continuous at all acid levels as long as the solution is buffered or very acid. However, the degree of ionization will improve in the unbuffered solution as the acid is diluted. Thus, there is a lower fraction of the species being absorbed in the acid type available for dilute acid solution [1]. The result will be a positive deviation from linearity at higher concentrations. If the species were to be absorbed by the anion type, the deviation would be negative.

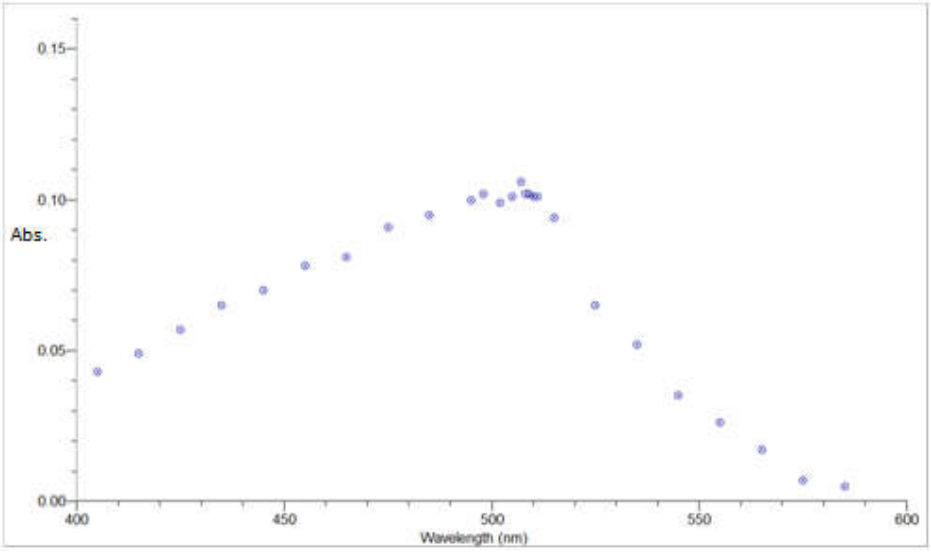


Figure 1: Determination of maximum wavelength

2. Methodology

The first part of the experiment was the preparation of reagents needed that includes standard ferrous solution (10 ppm) that was prepared from ferrous ammonium sulphate hexahydrate. Other necessary reagents like 1, 10-Phenanthroline, hydroxylamine hydrochloride, and acetic acid -sodium acetate buffer was also prepared. The **λ**max was determined by measuring the absorbance of standard solutions of different concentrations using the Colorimeter (Figure 1). A graph is plotted between absorbance and concentration, a straight line for solutions obeying Lambert-Beer law was obtained. The absorption range is directly proportional to the absorbing medium concentration. This calibration curve is then used for finding the unknown concentration of same solute in given solution by measuring the absorbance.

For the determination of ferrous ions in the given sample, first Iron is reduced from Fe^{+3} state to Fe^{+2} state. This is done by boiling water sample with HCl and hydroxyl amine hydrochloride. Ferrous ions react with o-phenanthroline to give stable, orange coloured, chelated complex ion $[(C_{12}H_8N_2)_3Fe]^{2+}$. The intensity of colour does not depend on pH in the range 3 to 9.

Preparation of Stock solution: For making stock solutions of Fe^{+2} ions dissolved 1.404 gm of Mohr's salt $[FeSO_4(NH_4)_2SO_4.6H_2O]$ in 50 mL of distilled water and added 20 mL of conc. H_2SO_4 . Added 0.1N KMnO4 solution drop by drop till a pink colour persists. Diluted the solution upto one litre. From this stock solution, prepare five standard solutions of different concentrations.

Take 1mL solution of first standard stock solution, in a 50 mL measuring flask. To it added 5 mL each of hydroxyl amine hydrochloride solution, sodium acetate and o-phenanthroline solutions. Now made up the solution up to 50 mL mark with distilled water and mixed properly. Similarly prepared the other four solutions by repeating the same procedure for different concentrations by taking 2, 3,4 and 5 mL stock solutions .

Sample and blank were diluted to mark after all the standard solutions. The absorbance of each solution in 511 nm was identified as shown in Figure 2. A calibration curve that obeys Beer's law was constructed. From the curve the concentration of iron as in water was calculated.

3. Results and Discussions

The primary goal of the experiment is to analyze the quantity of iron in the water by using the UV-VIS spectrophotometer. The sample solution absorbs electromagnetic radiation from the suitable source in spectrophotometric techniques such as UV-VIS, and the quantity produced is linked to the analyte concentration in the solution. In addition, the intensity of a solution's color is proportional to the absorbing species concentration. However, more light is absorbed in more focused solutions; the wavelength of the absorbed light is not changing. A tool could be a comparison of the color intensity of known concentration solutions with the intensity of an unknown solution. [1, 4]

Considering the absorption of light in the visible region, a qualitative image of the absorption of radiated could be acquired. An object could be seen because only a portion of the region is transmitted or reflected. The object will absorb certain wavelengths when a polychromatic light (white light) is transferred to an object, leaving the unabsorbed wavelength to be transferred. The color transferred is in addition to the colors consumed. Beer-Bouguer-Lambert law, frequently referred to as Beer's Law, describes the quantity of light absorbed by the sample. The quantity of light absorbed by a solution relies on its characteristics, the concentrations of the absorbing species, and the inside diameter of the sample holding pipe. The Beer-Lambert Law establishes the relationship between these factors:

$$A = \varepsilon bc \qquad \text{(eq. 1)}$$

where A is absorbent, b is the length of the solution path (1 cm in the experiment), c is the concentration of the absorbent substance in moles per liter, and ε is the analyte's molar absorption.

For a specified wavelength, absorptivity or molar absorption is a constant. Therefore it can be useful in qualitative identification of the substance. But in the experiment conducted the beer's law was used in quantitative analysis because of the broad band of the spectra. [5]

The instrument used was a spectrophotometer that resolved polychromatic light into different wavelengths and measures the absorbance of the particular wavelength by a sample. All spectrophotometers involve (a) a source of constant radiation over wavelengths of interest, [b] a monochromator to select a small wavelength band from the source spectrum,[c] a detector or transducer to convert radiant energy into electrical energy, and [d] a device to read out the detector reaction. Each of these will differ with the exception of the read-out device, based on the region of the wavelength [1]. The results obtained have been given in Table1.

Table 1: Absorbance obtained from the different concentrations of iron (II)

S. No.	Concentration (ppm)	Absorbance
1	3.56	0.14
2	7.12	0.44
3	10.68	0.73
4	14.28	1.00
5	17.8	1.25

Calculation of Concentration

$\frac{1.404g}{392x1l} = \frac{1}{280} mol/l = 0.00356 mol/l = 3.56X10^{-3} mol/l$

$= \frac{3.56X10^{-3}}{10^3} = 3.56X10^{-6}$mol in 1 mL

$7.12X10^{-6}$mol in 2 mL

$10.68X10^{-6}$mol in 3 mL

$14.28X10^{-6}$mol in 4 mL

$17.80X10^{-6}$mol in 5 mL

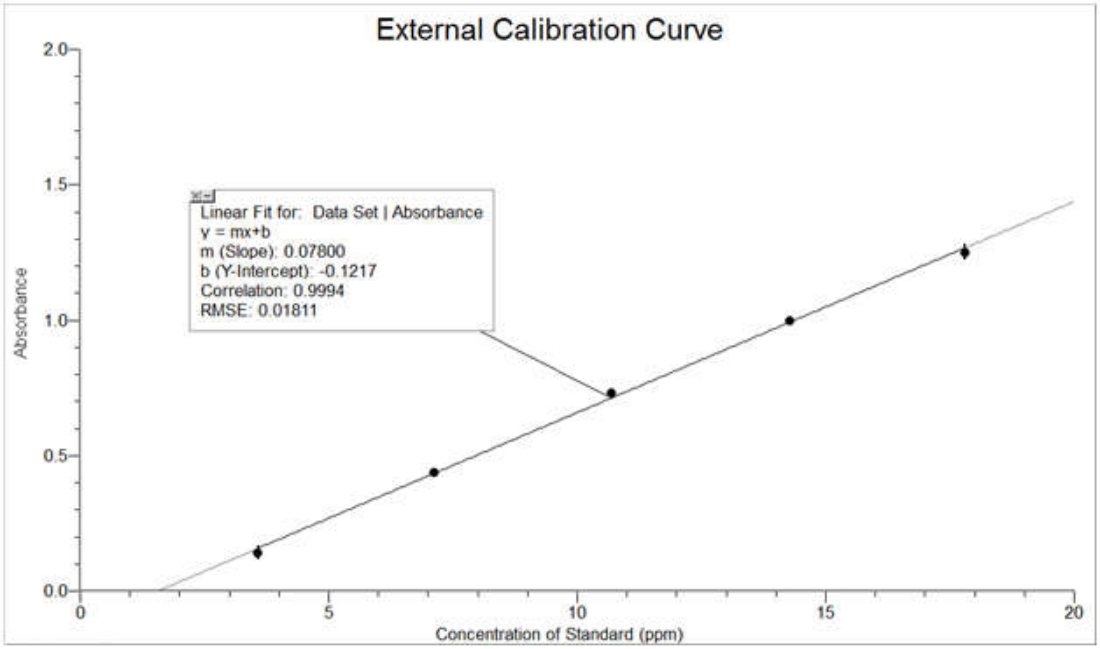


Figure 2: Relationship between Absorbance and Concentration

Figure 2 indicates a linear feature, as expected by Beer's Law, upon plotting absorbance versus normal concentration at 511 nm (ɪɪmax), and using the calibration curve equation and absorption of the unknown sample treated under the same experimental circumstances, the unknown sample concentration can be calculated.

The calibration curve equation y = 0.078x-0.1217 where "y" is the absorbance at the specified wavelength, "x" is the concentration of the unknown, "m" which is the slope relates to the molar absorptivity, and "b" is the length of the path light traveled to the sample. A regression of 0.9994 signifies that the reliability of the data points is linearly fit.

The concentration of the three unknowns was calculated using the formula from the calibration curve shown in the figure: Cunk= (Aunk + yint.)/slope, by accounting for the dilution factor. The findings achieved are as follows:
Sample 1: A=0.55, Concentration =8.61ppm
Sample 2; A=0.83, Concentration= 12.20 ppm
Sample 3; A=1.08, Concentration= 15.41 ppm

4. **Acknowledgement**

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