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Aim: To become familiar with the principles of calorimetric analysis and spectrometric determination of Iron.

Requirements:

1) Spectronic 301 spectrophotometer
2) ~~o-phenanthroline~~. O- phenanthroline.
3) Fe(II)

Theory

Calorimetric analysis is based on the change in the intensity of the colour of a solution with variations in concentrations.

An increase in sensitivity and accuracy results when a spectrophotometer is used to measure the colour intensity. Basically, it measures the fraction of an incident beam of light which is transmitted at a specific wavelength.

Measure the difference in intensity of light beam by percent transmittance.

$$\% T = \frac{I_0}{I}, \quad \log \frac{1}{T} = -\log T$$

Beer - Lambert Law $A = \varepsilon b c$

A → Absorbance
ε → extinction coefficient ($M^{-1} cm^{-1}$)
b → solution path (cm)
c → concentration (moles/litre)

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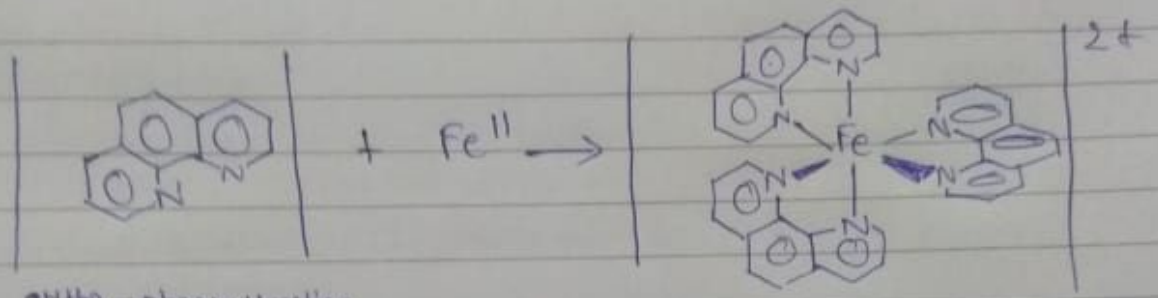
Not all analysis follow Beer - Lambert Law, hence you can construct calibration curve that will provide the relationship instead.



Ortho - phenanthroline

Ferrous tris - o - phenanthroline.

Because we are starting with an Fe^{3+} solution and in order to be quantitative all of the iron must be reduced to Fe^{2+} by using excess of hydroxylamine hydrochloride.

$$4Fe^{3+} + 2NH_2OH \cdot HCl \rightarrow 4Fe^{2+} + N_2O + 4H^+ + H_2O$$

PROCEDURE

1) The STANDARD IRON solution contains 0.2500 g/L of pure iron. Pipette 25.00 mL of this standard iron solution into a 500mL volumetric flask and dilute up to the mark with distilled water.

2) Pipette 10.00 mL of an unknown sample solution into a 250mL volumetric flask and dilute to the mark with distilled water. Invert and shake the flask several times.

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3) Pipette two 25.00 mL aliquots of this solution into two 50 mL volumetric flasks labeled unknown.

4) Using a 10 mL graduated cylinder, add 4.0 mL of 10% hydroxylamine hydrochloride solution and 4.0 mL of 0.3% o-phenanthroline solution to each volumetric flask.

5) Swirl and allow the mixture to stand for 10 minutes.

6) Dilute each flask to the mark with distilled water and mix well by inverting and shaking the capped volumetric flasks several times.

7) Using the Spectronic 301 spectrophotometer, carefully measure the various solutions including the unknown.

Table:

Solution	Absorbance
0.00	0.000
0.05	0.201
0.10	0.389
0.15	0.587
0.20	0.787
0.25	1.023
Unknown 1	0.674
Unknown 2	0.677

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Calculations & Discussions

1) Prepare a lot of absorbance versus concentration of the known solutions (express the conc^n in mg Fe/50mL) Draw the Beer-Lambert law.

2) Place the best Absorbance value of each unknown solution onto this plot and determine their concentration.

3) Calculate the amount of iron in the unknown sample. Unit used (mg/L Fe)

Original Concentration

$$0.10 \frac{\text{mg Fe}}{50\text{ mL}} \times 50 \text{ (dilution factor)} \times \frac{1000\text{mL}}{\text{L}} = \frac{100\text{mg Fe}}{\text{L}}$$

$$\text{Relative Error} = \frac{\text{Experimental value} - \text{accepted value}}{\text{accepted value}} \times 100\%$$

Safety :-

The wearing of safety glasses is mandatory all times. Those wearing prescription glasses must wear goggles over their glasses. Students without prescription must wear the safety glasses provided. NO use of contact lenses.

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