Spectral study for Determination of the composition of iron complex

Materials:

1- standard solution of ferric ammonium sulphate 0.0005 M
2- spectrophotometricaly reagent 1,10-phenanothroline 0.0005 M
3- Sodium acetate 10g/100 ml.
4- Hydroxyl amine hydrochloride 10g/100 ml.

Theory:

Two different analytical methods are used spectrophotometrically to study the composition of complexes:

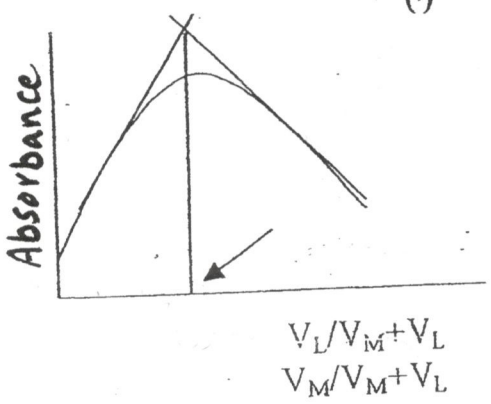
1- continuous variations method:

In this method the sum of the molar concentrations of the two reactants is kept constant as their ratio is varied. The abscissa of the extrapolated peak will correspond to the ratio present in the complex.

Procedure:

1-. transfer 0,1,2,3,4,5,6,7,8 ml of iron solution (V_m) to 9 volumetric flasks 25 ml , then add to each 0.5 ml of hydroxyl amine hydrochloride solution.
2- Add to all flasks in order 10, 9, 8,7,6,5,4,3,2 ml of spectrophotometricaly reagent (V_L) (always total =10), then wait for 10 min.
3- Add to all flasks 4 ml of sodium acetate solution and complete to the mark by distilled water.
4- Measure the absorbance of all solutions at 508 nm - * blank??
5- Plot the relationship between Absorbance and the ratio [L/M] as shown in figure (1), and then calculate the ratio [L/M].

(1)



طريقة النسبة المولية

2- molar ratio method:

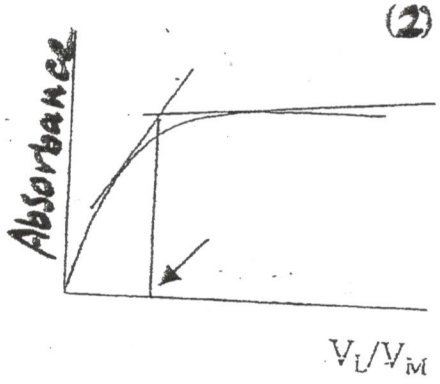
تثبيت تركيز M وتغيير تركيز L باستمرار

In this method the concentration of metal ion is held fixed and the concentration of the reagent is increased stepwise. On the graph the absorbance versus moles of reagent added, the intersection of the extrapolated linear segments determines the ratio: moles of reagent / moles of metal.

Procedure:

1- Transfer 2 ml of iron solution (V_m) to 8 volumetric flasks 25 ml, and then add to each 0.5 ml of hydroxyl amine hydrochloride solution.
2- Add to all flasks in order 2, 3 4, 5, 6, 7, 8 ml of spectrophotometricaly reagent (V_L) , then wait for 10 min.
3- Add to all flasks 4 ml of sodium acetate solution and complete to the mark by distilled water.
4- Blank is 0.5 ml of hydroxyl amine hydrochloride, 5 ml of spectrophotometricaly reagent and 4 ml of sodium acetate, then complete to the mark by distilled water.
5- Measure the absorbance of all solutions at 508 nm.
6- Plot the relationship between Absorbance and the percentage of reagent to metal as shown in figure (2), and then calculate the percentage of reagent to metal.

(2)



PHOTOMETRIC TITRATION OF COPPER (II) WITH EDTA

Experiment

In photometric titration, a spectrophotometer is used so that we can detect end points graphically. The absorbance of a titrated species is measured after each addition of titrant until well past the end point. A plot of absorbance vs. milliliters of titrant consists of two straight lines that intersect at the end point of the titration.

. The reaction for this experiment is:

$$H_2Y^{2-} + Cu^{2+} \text{ === } CuY^{2-} + 2H^+$$

Where H_2Y^{2-} is Na_2H_2Y

The titration is performed at 625 nm; both the copper-EDTA chelate and the copper (I) ion absorb at this wavelength.

The pH is critical for this titration, because a large change in pH changes the effective binding constant. An acetate buffer is used to maintain the pH between 2.4 and 2.8 to avoid this problem. This low pH also permits the copper to be titrated in the presence of metal ions that form weaker complexes with EDTA.

Procedure:

Preparation of Samples

1. Prepare an acetate buffer solution by adding 4.1 g of anhydrous sodium acetate (or the equivalent amount of the hydrate) to 50.0 mL of water. Add 1 M hydrochloric acid to the buffer solution until the pH of the mixture is 2.2, as indicted by a pH meter.

2. Prepare 0.2M of EDTA in 50 ml volumetric flask.

3. Prepare unknown solution of COPPER (II) by adding 1.431gm of CuSO4 to 100 ml volumetric flask and 1 ml of H_2SO_4 , then diluted to the mark by distilled water.

4. In nine conical flasks, transfer 10 ml of unknown solution of copper by using pipette.

5. Add in each flask 10 ml of buffer solution

6. Transfer 0, 2, 3, 4, 4.5, 5, 5.5, 6, 7 ml of EDTA to every one of conical flask. The first one is blank .

7. Measure the absorbance at 625 nm.

8. Plot the absorbance of the solution against the volume of the EDTA, determine the endpoint by extrapolating the two linear portions of the curve to an intersection point. . Determine the corresponding volume of EDTA and calculate the percentage of copper.