RPS DEGREE COLLEGE

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Lab Manual

Chemistry (HC 5th & HC 6th Semester)

Department of Chemistry

Experiment 1

Aim:-To prepare and analysepure sample of Potassium Trisoxalatoferrate(III) Trihydrate, K3[$Fe(C_2O_4)_3$].3H_2O

APPARATUS AND CHEMICALS:

$K_2C_2O_4.H_2O$, funnel, FeCl3.6H2O, filter paper, $K_3Fe(CN)_6$ solution, 100-mL beaker, H_2SO_4 solution, test tubes, distilled water, opaque objects

THEORY: Potassium trisoxalatoferrate(III) trihydrate, $K_3[Fe(C_2O_4)_3].H_2O$ is a green crystalline salt, soluble in hot water but rather insoluble when cold. It can be prepared by the reaction of $K_2C_2O_4.H_2O$ with $FeCl_3.6H_2O$.

$3K_2C_2O_4.H_2O(aq) + FeCl_3.6H_2O(aq) \rightarrow K_3Fe(C_2O_4)_3].3H_2O(aq) + 3KCl(aq)$

The complex anion is photo-sensitive.This means that upon exposure to light of anappropriate wavelength (<450 rim in this case) the $Fe(C_2O_4)_3^{-3}$undergoes an intramolecularredox reaction in which the Fe(III) anion is reduced to Fe(II) while one of the oxalate groupsis oxidized to CO_2.

$[Fe(C_2O_4)_3]^{3-} \rightarrow Fe^{2+} + 5/2\ C_2O_4^{2-} + CO_2(g)$

As mentioned above, light causes an internal electron-transfer reaction to occur in $[Fe(C_2O_4)_2]^{3-}$ ion, producing CO_2 and Fe^{2+} ions. The Fe^{2+} that is produced can readily be detected by adding a solution of potassium ferricyanide $K_3Fe(CN)_6$. A deep blue colored ferroferri cyanide complex is formed.

$Fe^{2+} + Fe(CN)_6^{3-} \rightarrow Fe[Fe(CN)_6]^-$

ferroferricyanide deep blue.

PROCEDURE: A. Preparation of $K_3[Fe(C_2O_4)_3].3H_2O$

1. Weigh approximately 9.0 g of hydrated potassium oxalate, $K_2C_2O_4.H_2O$ into a 250 mL beaker.

2. Add 30 mL of distilled water and heat to dissolve (do not boil).

3. In a second small beaker dissolve 4.4 g of $FeCl_3.6H_2O$ in a minimum amount of cold water (10-15 mL). Add the $FeCl_3.6H_2O$ solution to the warm

oxalate solution and stir with a glass rod. Allow the product to crystallize (away from strong sunlight) by cooling the solution in an ice-water mixture.

4. Collect the crystalline product by filtration. The product is $K_3[Fe(C_2O_4)_3].3H_2O$. B.

Blueprinting

1. Wet a piece of filter paper with $[Fe(C_2O_4)_2]^{3-}$ solution.

2. Leave it to dry. (Meanwhile you can follow part C)

3. Place small opaque objects (coins, keys, etc.) on the paper.

4. Irradiate for few minutes using a light source (If not available you may use bright sunlight)

5. Dip the paper into potassium ferricyanide solution (CAUTION potassium ferricyanide is poisonous. Avoid contact with your skin. If it happens immediately wash your skin with plenty of water.)

6. Remove the developed blueprint and dip in a beaker of distilled water to wash off excess ferricyanide solution. Explain your observations.

C. Photochemical Reaction of $[Fe(C_2O_4)_2]^{3"}$

1. Dissolve 0.7 g of your complex in 100 mL of distilled water in a flask. Add 3 mL of 2 M H_2SO_4 and swirl the mixture. To each three labeled test tubes add 10 mL of this solution.

2. Keep one tube away from the light source as the control and irradiate the remaining two tubes with the light source for 1 and 5 minutes respectively.

3. To all three tubes add 1 mL of 0. 1 M potassium ferricyanide solution $K_3Fe(CN)_6$.

4. Record and explain your observations.

Experiment:2

Aim:-To verify Beer-Lambert law for KMnO4 and determine the concentration of the given KMnO4 solution.

Chemical Required:- solid KMn04.

Apparatus Required:- Spectrophotometer or Elico colorimeter, measuring flasks (100ml and 1000ml), weight box, fractional weights, graph papers.

OBJECTIVE:-

In it we used Beer Lamberts law, this law was dependent on absorbance phenomena.For it number of standard solutions of different concentrations are prepared. Their absorbance is determined. Then a plot of *A* vs c is drawn. It is a straight line passing through the origin. This proves the validity of Beer-Lambert law. Then the absorbance of the unknown solution is determined under the same experimental conditions. The concentration corresponding to this absorbance is read from the calibration graph.

PROCEDURE:-

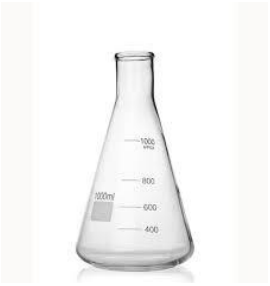
(i) Prepare a stock solution of 10^{-3}M KMn04 by dissolving 0.0316g solid KMn04 in one liter distilled water. .

(ii) Took four 100ml flat-bottomed measuring flasks and name them as A, B, C and D respectively. (iii)Now pipette out 20, 40, 60 and 80ml of stock solution of KMn04 into flask A, B, C and D respectively. Make the solution up to the given mark in Conical flask by dilution with distilled water in every 100ml flask.

20ml stock solution+	40ml stock sol.+	60ml stock sol+	80ml stock sol+
80ml distilled	60ml water	40ml	

water 20ml water



A **B** **C**

D

(iv) Fill one optical cell with the stock solution and 2nd with distilled water. Insert them in colorimeter. Insert different filters one by one and find the filter that gives maximum absorbance. This filter is not to be changed throughout the experiment.

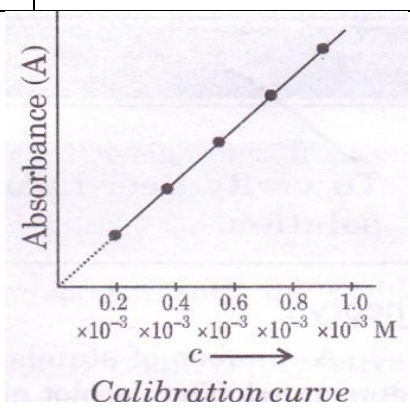
(v) Remove the stock solution and fill that optical cell with the solution (minimum 4ml) from flask A. Note the absorbance. Repeat the experiment with solution from flask B, C and D and note the absorbance in each case. Plot the calibration curve between *A* vs c.

(vi) Now fill the unknown solution and note the absorbance.

OBSERVATION AND CALCULATION:-

Solution	**Concentration**	**Absorbance**
Stock solution	10^{-3} M	.....
Flask A	0.2 x 10^{-3} M	.....
Flask B	10^{-3} M	.....
Flask C	0.6 x 10^{-3} M	.....
Flask D	10^{-3} M	.....

From the calibration curve, read the concentration of the unknown solution corresponding to the absorbance.



Further a straight line verified the Beer-Lambert's law.

RESULT:

The concentration of given KMnO4 solution is……
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EXPERIMENT 3

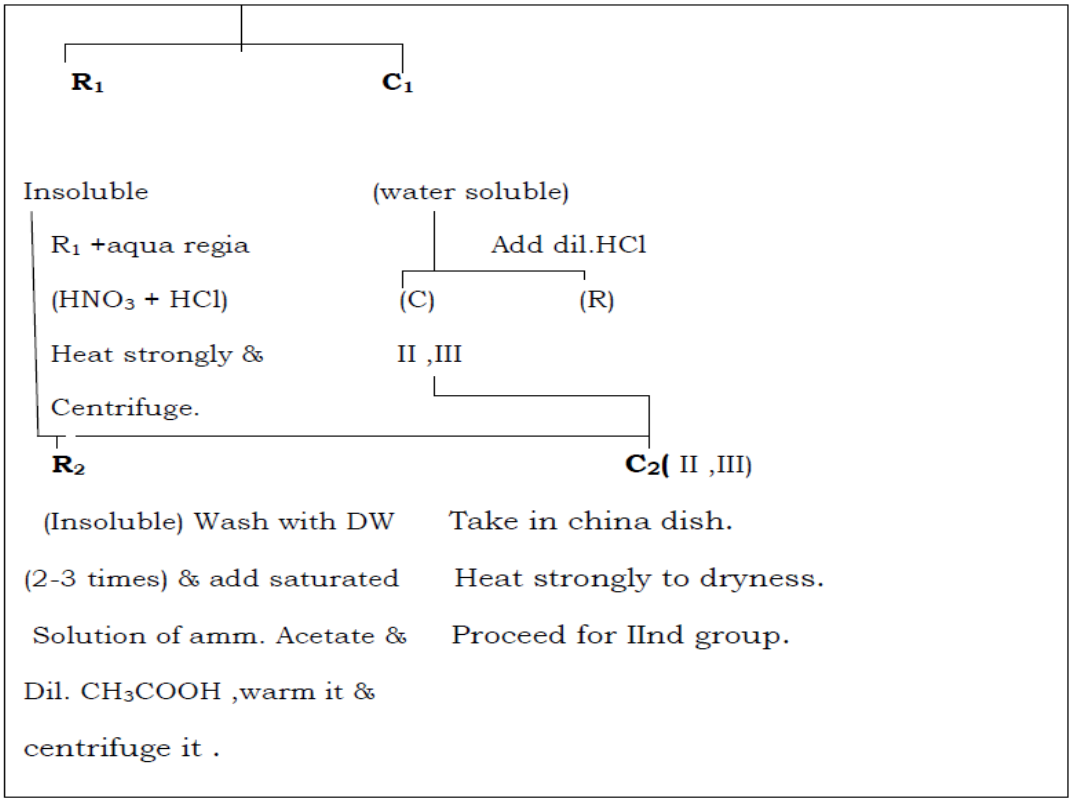
Aim: Analysis of Insoluble Insoluble salt.

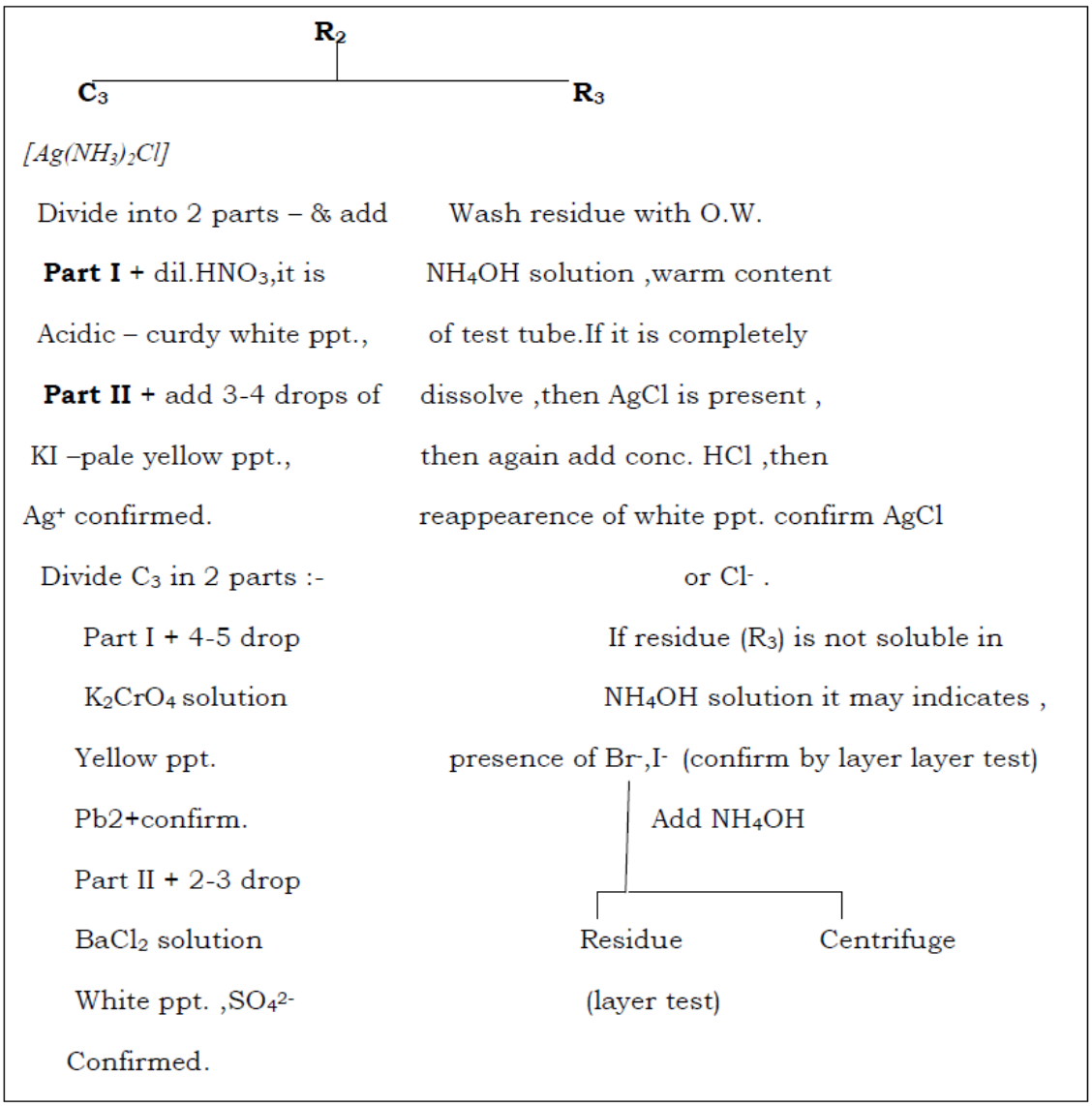
Sulphates - $PbSO_4$, $BaSO_4$

Oxides -AlO_3 , Cr_2O_3 , SnO_2 , SiO_2 , TiO_2

Confirmation Test :-

To given mixture, add distilled water, heat it & cool, then centrifuge it.





-If all test for halides are –ve , then proceed for another salts .

-then , do fusion with residue R_3 :- [Done in Ni crucible]

Two Methods :-

1. At the bottom of Ni crucible make a layer of NaOH pallets + few crystal of KNO_3+ add residue part.
2. Ni crucible + (K_2CO_3 + Na_2CO_3) + KNO_3& then add residue ,then strongly heat the Ni crucible upto red hot ,cool it at room temp. & add saturated solution of Na_2CO_3 & then centrifuge it .

R_4 | **C_4**

(may contain $BaSO_4$, TiO_2)
Add dil. CH_3COOH till all the CO_2 expels & again centrifuge.

If centrifuge is of orange colour then add dil. CH_3COOH to neutralize it ,then add 4-5 drops of Pb acetate solution.
–Appearance of yellow ppt. Cr_2O_3
- If centrifuge is colourless , ,add conc. HCl to neutralize .After that boil & centrifuge

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R_4

R_5 | **C_5** – Ba^{2+}

Add conc. H_2SO_4,boil for 2-3 min. & cool & appearance of orange colour centrifuge.
confirm TiO_2.

- To C_5 ,add K_2CrO_4 it gives yellow ppt.
-Appearance of yellow ppt. confirms Ba^{2+}.

C_4

R_5 | **C_5**

-May contains SiO_2 .To residue add any fluoride & conc. H_2SO_4 ,heat & during heating take a glass rod dipped in DW & deposition of waxy solid confirm SiO_2

-Divide it in 2 parts.

-To 1st ,add 2-3 ml OS & then pass H_2S gas formation of brown or Yellow ppt. confirm SnO_2.

-To 2nd part ,add NH_4Cl, then heat cool it & add excess of NH_4OH solution .Appearance gelatinous white ppt. confirms Al_2O_3.

Semester 6th

Experiment: 1

Aim:-To prepare pure crystals of tetra ammine copper (II) sulphate.

Objective:-

Tetra ammine copper sulphate is a complex of Cu(II) with co-ordination no. 4. When NH_40H i: gradually added to an aqueous solution of$CuS0_4$, CU(OH)2 is first precipitated and the precipitate ther dissolves, yielding a solution of a bright blue colour which is due to formation of tetra ammine copper (II) ion, $[Cu(NH_3)4]$

$$CuSO_4 + 2NH_4OH \rightarrow Cu(OH)_2 + (NH_2)_2 SO_4$$

$$\underset{\text{(ppt.)}}{Cu(OH)_2} \rightleftharpoons \underset{\text{(dissolved)}}{Cu(OH)_2} \rightleftharpoons Cu^{2+} + 2OH^-$$

$$Cu^{2+} + 4NH_3 \rightleftharpoons [Cu(NH_3)_4]^{2+}$$

The removal of practically the whole cupric ion from the solution shifts the equilibrium of equation (2) towards the right, until the cupric hydroxide has completely passed into solution. The deep blue solution containing tetraamminecopper (II) ion is known as **Schweitzer's reagent.**

It has an ammonicalodour, and crystals ofthe complex are obtained by adding ethyl alcohol to the above solution.

CHEMICAL REQUIREMENTS:-

Copper sulphate	5gm
1 : 1 Ammonia	20ml
Ethyl Alcohol	20-25ml
Conc. H_2S0_4	1-2ml

APPARTUS REQUIRED:-Beaker,Burner,Measuring Cylinder.

PROCEDURE:-

(i) Take 5gm powdered copper sulphate in a 250ml clean beaker and dissolve it **in** a minimum amount of water. Add 1-2ml cone. H_2S0_4 to make the solution clear.

(ii) Now pour 1 : 1 NH_40H very slowly into the beaker with constant stirring till a ppt. of Cu(OH)2 first formed is redissolved yielding a deep blue solution due to formation of $[Cu(NH_3)4j2+$ and smell of ammonia is present due to slight excess of NH_40H.

(iii) Now to the blue solution, add 20-25ml of ethanol dropwise with constant stirring. Add about 2ml of NH_40H and heat the beaker in a water bath at 60-70°C for about 15-20 minutes.

(iv) Stop heating, cover the beaker with clock glass and allow it to stand undisturbed for 2-3 hours (preferably overnight)

(v) Long needle shaped blue crystals of complex are formed. Filter, wash with little alcohol and dry the crystals gently by pressing in between the folds of the filter paper or by placing in a desiccator.

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OBSERVATIONS:-

(i) Yield= about5gm

(ii) Colour= Blue

(iii) Shape = Needle-shaped crystals

Experiment : 2

Aim: Determination of iron by thiocyanate colorimetry

CHEMICAL REQUIREMENTS:-

Ferric ammonium sulfate $FeNH_4(SO_4)_2 \cdot 12H_2O$ standard solutions: 2, 4, 6, 8 and 10×10^{-5} mol L^{-1} (see below for preparation) •
Iron tablet
1 mol L^{-1} ammonium thiocyanate solution
1 mol L^{-1} sulfuric acid
0.15 mol L^{-1} potassium permanganate solution
100 mL beaker
100, 200, 250 and 500 mL volumetric flasks
5 mL pipette
10 mL measuring cylinder
100 mL conical flask
at least 6 boiling tubes (or large test tubes)
distilled water

Theory:

Introduction Iron is one of the many minerals required by the human body. It is used in the manufacture of the oxygen-carrying proteins haemoglobin and myoglobin. A deficiency of iron in the body can leave a person feeling tired and listless, and can lead to a disorder called anemia. Many of the foods we eat contain small quantities of iron. In this analysis the iron present in an iron tablet (dietary supplement) or a sample of food is extracted to form a solution containing Fe^{3+} (ferric) ions. To make the presence of these ions in solution visible, thiocyanate ions (SCN^-) are added. These react with the Fe3+ ions to form a blood-red coloured complex: Fe^{3+} (aq) + SCN^- (aq) → $[FeSCN]_{2+}$ (aq) By comparing the intensity of the colour of this solution with the colours of a series of standard solutions, with known Fe^{3+} concentrations, the concentration of iron in the tablet or food sample may be determined. This technique is called colorimetry.

Method:

Preparation of Fe3+ standard solutions:

It may take several days to dissolve the Fe3+ salt used here, so carry out this preparation well in advance of the rest of the experiment. Weigh out about 3.0 g of ferric ammonium sulfate (FeNH4 (SO4) 2 •12H2 O). Use a mortar and pestle to grind the salt to a fine powder. Accurately weigh 2.41 g of the

powder into a 100 mL beaker and add 20 mL of concentrated sulfuric acid (see safety notes). Leave powder to soak in acid overnight. The next day, carefully pour the acid/ powder slurry into a 500 mL volumetric flask, rinsing the beaker into the flask a few times with water, then make up to the mark with distilled water. Let this solution stand for several days until the ferric ammonium sulfate powder has fully dissolved. If possible, insert a magnetic stirrer bar and stir the solution to speed up this dissolving process. Use a pipette to transfer 20 mL of ferric ion solution to a 200 mL volumetric flask and make up to the mark with distilled water. This gives a solution with [Fe3+] = 0.001 mol L−1. To prepare a 2 × 10−5 mol L−1 standard solution pipette 10 mL of the 0.001 mol L−1 solution into a 500 mL volumetric flask, add 10 mL of 1 mol L−1 sulphuric acid, and then make up to the mark with distilled water. Repeat this procedure inseparate 500 mL volumetric flasks, pipetting in 20, 30, 40 and 50 mL of 0.001 mol L−1 Fe3+ solution in turn, to obtain 4, 6, 8 and 10 × 10−5 mol L−1 solutions respectively.

Preparation of 1 mol L−1 ammonium thiocyanate solution:

Weigh 38 g of solid ammonium thiocyanate into a 500 mL volumetric flask and make up to the mark with distilled water. 3. Preparation of 0.15 mol L−1 potassium permanganate solution (only required for analysis of iron tablet): Weigh 2.4 g of solid potassium permanganate into a 100 mL volumetric flask and make up to the mark with distilled water.

Preparation of iron tablet for analysis:

1. Place iron tablet in a 100 mL beaker and use a measuring cylinder to add 20 mL of 1 mol L−1 sulfuric acid. Allow the tablet's coating to break down and its contents to dissolve. You may help this process by using a stirring rod to carefully crush the tablet and stir the solution. (NB: iron tablets sometimes contain filler materials that may not fully dissolve in acid)

2. Once the iron tablet is dissolved, add 0.15 mol L−1 potassium permanganate solution dropwise, swirling the beaker after each addition. Iron tablets usually contain ferrous sulfate, with iron present as Fe2+ ions. Since Fe2+ does not form a coloured complex with thiocyanate, permanganate ions are added to oxidise all the Fe2+ to form Fe3+ ions. For the first few drops of permanganate, the purple colour will disappear

immediately upon addition to the iron solution; however, as further drops are added the colour will begin to linger for a little longer. Stop adding potassium permanganate drops when the purple colour persists for several seconds after addition − usually no more than about 2 mL of 0.15 mol L−1 permanganate solution will be required.

3. Transfer the iron solution to a 250 mL volumetric flask, rinsing the beaker with distilled water a few times and transferring the washings to the volumetric flask. Make up to the mark with distilled water, stopper the flask and mix well.

4. Use a pipette to transfer 5 mL of iron solution to a 100mL volumetric flask and make up to the markwith distilled water. This diluted solution will be used for colorimetric analysis.

Preparation of food sample for analysis:

1. Accurately weigh a few grams (typically 2 − 5 g is required, depending on iron content of sample) of your food sample into a crucible.

2. Heat the crucible over a bunsen burner (see Figure 1) until the sample is reduced completely to ash, or (preferably) combust the sample directly in the bunsen flame (as shown in Figure 2), reducing it to ash. NB: be very careful with the bunsen flame while heating/combusting your sample. Also beware that the crucible will become very hot during this process, so handle it only with crucible tongs − or preferably not at all − until it has cooled.

3. When the sample and crucible have cooled, use a stirring rod to crush the ash to a fine powder (see Figure 3). Use a measuring cylinder to add 10 mL of 1 mol L−1 hydrochloric acid and stir for 5 minutes, making sure that all the ash is soaked.

4. Add 5 mL of distilled water and filter the solution into a 100 mL conical flask to remove the ash. This filtered solution will be used for colorimetric analysis.

Colorimetric analysis: this analysis method applies to samples prepared using either of the two methods above (iron tablets or food samples).

1. Accurately measure 10 mL of your sample solution into a clean, dry boiling tube/test tube. NB: this is most accurately done using a 10 mL

pipette; however, it is possible to do this accurately enough (and with less hassle) using a clean 10 mL measuring cylinder if you measure carefully.

2. Next, measure 10 mL of each Fe^{3+} standard solution into separate boiling tubes (one standard per tube) in order of increasing concentration, beginning with the 2×10^{-5} mol L^{-1} standard. It is a good idea to first rinse your pipette or measuring cylinder with a few mL of the 2×10^{-5} mol L^{-1} standard). NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contain. A test tube rack is very useful for holding and transporting your tubes (see Figure 4). Alternatively you can use a large beaker to hold them.

3. Using a 10 mL measuring cylinder, measure 10 mL of 1 mol L^{-1} ammonium thiocyanate solution into each of six small clean vessels − six boiling tubes is ideal. You should now have one measured portion of thiocyanate solution for each of your iron solutions.

4. As quickly as possible, pour 10 mL of thiocyanate solution (the portions measured out above) into each of your iron solutions.

5. Mix the solutions by swirling. A stable red colour will appear over the next few minutes.

6. Allow the red colour to develop for 15 minutes. Then estimate the concentration of Fe^{3+} ions in your iron sample by identifying which of your Fe^{3+} standards matches its colourmost closely. Figure 4 illustrates the range of colour intensities that you can expect from your set of Fe^{3+} standards. Tip: If you are using boiling/test tubes all of identical sizes, the best way to compare colours is by looking at your solutions from above − looking down into the tubes (see Figure 5).

7. If the colour of your unknown iron solution is stronger than the colour of your highest concentration Fe^{3+} standard you will need to modify the above procedure. In the case of an iron tablet, you should repeat the analysis with a more dilute solution of the dissolved iron tablet. In the case of a food sample, you should repeat the analysis using a smaller mass of your food.

Calculations

1. Assume that the concentration of Fe^{3+} in your unknown iron solution is approximately equal to that of the Fe^{3+} standard whose colour was the closest match.

2. Use this concentration to calculate the mass of iron (in mg) in your original tablet or food sample