Practical 1 Procedure – Iron by Titrimetry

Introduction

This experiment quantifies Fe^{2+}, ferrous iron, by reacting it with ceric sulphate in a 1:1 reaction, i.e. one mole of ferrous iron reacts with one mole of ceric sulphate.

Procedure

All results should be recorded on the Results Sheet.

Ferrous Sulphate Tablets

Count out five tablets and weigh them to find the average tablet weight. Crush the tablets finely with a mortar and pestle. It is essential that the tablets are finely ground. Weight out approximately 0.4000g of powdered tablet and record the weight accurately. Using $15cm^3$ of 5M sulphuric acid transfer the powder quantitatively into a conical flask. Add two drops of ferroin indicator, swirl, and leave for five minutes. Titrate using 0.1M cerium sulphate. The end point is indicated by a colour change from red to milky yellow.

Leave the conical flask to stand for five minutes. If the red colour returns, swirl and titrate to the end point again. Repeat the five minute wait and titration till the conical flask contents remain blue after the five minute waiting period. Record the total titre.

Each $1cm^3$ of 0.1M ceric sulphate is equivalent to 0.01519g of $FeSO_4$. Use this to calculate the weight of ferrous sulphate in the powdered sample you took and hence the percentage of ferrous sulphate in the powder.

Repeat the experiment with a further 0.4000g sample of powdered tablets.

Find the average percentage of ferrous sulphate and use this and the average tablet weight to calculate the weight in milligrams of ferrous sulphate per tablet.

Unknown Ferrous Iron Compound

Weight out accurately approximately 2.0000g of the unknown ferrous compound. Note the weight. Transfer quantitatively to a $100cm^3$ volumetric flask with $50cm^3$ 5M sulphuric acid. Mix well. Leave to stand for five minutes. Make up to the mark with 5M sulphuric acid and mix again. Label as *Unknown Fe^{2+} Solution*.

Pipette $20cm^3$ of the *Unknown Fe^{2+} Solution* into a conical flask, add two drops of ferroin indicator and titrate as for the ferrous sulphate tablets. Record the titre.

Repeat the experiment with a further $20cm^3$ aliquot of the *Unknown Fe^{2+} Solution*.

Find the average titre.

One mole of the unknown ferrous compound reacts with one mole of ceric sulphate. Using this and the relationships below calculate the molecular weight of the unknown ferrous compound.

$$number\ of\ moles = \frac{molarity \times volume\ in\ cm^3}{1000}$$

$$number\ of\ moles = \frac{weight\ \ in\ grams}{Molecular\ Weight}$$

Note:- A milligram, mg, is one thousandth of a gram, i.e. 1g is 1000mg.

Molecular weight is also known as formula weight and as molecular mass and molar mass

Practical 2 Procedure – Iron in IrnBru by Colorimetry

Introduction:
The measurement of small concentrations of Fe^{3+} ions can be achieved by reaction with thiocyanate (SCN^-) ions to form an orange/red coloured complex. Nitric acid is added to the solution to stabilise the complex.

$$Fe^{3+} + SCN^- \longrightarrow [Fe(SCN)]^{2+}$$

Colourless orange/red

This complex has a characteristic absorption in the visible region of the electromagnetic spectrum which shows maximum absorbance at a wavelength of 460 nm. This absorbance is directly related to concentration and can be measured by means of a spectrophotometer.

By using a series of calibration standards containing known concentrations of Fe^{3+} ions, a calibration graph of Absorbance (y-axis) against Concentration (x-axis) should give a straight line through the origin.

By measuring the absorbance of an Irn-Bru sample reacted with excess thiocyanate, the concentration of Fe^{3+} can therefore be obtained from the calibration graph.

Irn-Bru has an added food colouring that also absorbs light at 460 nm. This is corrected for by measuring the absorbance of an Irn-Bru blank solution which contains Irn-Bru but none of the complexing agent so that its absorbance is due solely to the added food colour.

Procedure:
All results should be recorded on the Results Sheet.

Thiocyanate Solution
Weigh out approximately 10g of potassium thiocyanate into a 100cm^3 beaker. Note the weight taken. Add 50cm^3 of water and stir to dissolve the solid. Transfer the contents of the beaker to a 100cm^3 volumetric flask, make up to the mark with deionised water and mix well. Label as "10% Thiocyanate".

Fe^{3+} Calibration Standard Solutions
Pipette 10 cm^3 of the provided stock Fe^{3+} solution which contains 100mg of Fe^{3+}/dm^3 (i.e. 100mg/litre) into a 100 cm^3 volumetric flask and dilute to the mark with deionised water. Mix well. Label as working Fe^{3+} solution.

Using a burette, add 0, 1, 3, 5, 7, 9 and 11 cm^3 of the working Fe^{3+} solution into each of seven 25 cm^3 volumetric flasks.

Add by measuring cylinder, 5 cm^3 of 4M nitric acid and 5 cm^3 of the 10% potassium thiocyanate solution to each flask. Dilute to the mark with deionised water and mix well. Label each flask as 0, 1, 3, 5 etc.

The flask labelled "0" is the Standard Blank as it contains reagents but no Fe^{3+}. This solution is used to zero the spectrophotometer before making any absorbance measurements.

Irn-Bru

Pipette 10cm^3 of degassed Irn-Bru into each of two 25cm^3 volumetric flasks.

Add by measuring cylinder 5cm^3 of 4M nitric acid to both flasks.

To only ONE of the flasks add by measuring cylinder 5cm^3 of 10% thiocyanate.

Make both flasks up to the mark with water and mix well.

The flask with no thiocyanate is labelled as "Irn-Bru Blank" while the other is labelled "Irn-Bru Sample".

Measurements Using the Spectrophotometer

Leave all the solutions, both the Fe^{3+} standards and the Irn-Bru samples to stand for 10 minutes to allow the complex ion to form and stabilise.

Use the same 1 cm^3 cell and spectrophotometer for all the measurements. The cell should be carefully rinsed with the test solution each time and filled almost to the top. Carefully dry the outside of the cell and place in the spectrophotometer with the smooth sides in line with the light beam, i.e. left - right.

Set the wavelength on the spectrophotometer to 460 nm. Fill the cell with the Standard Blank, i.e. the flask labelled "0", and zero the absorbance.

Starting with the most dilute solution, place each of the remaining six Fe^{3+} standard solutions in the cell and record the absorbance to three decimal places. Note the result in Table 1 of the Results Sheet. Remove the cell then replace it and make a second reading of the same sample. Record this in Table 1 as well.

Fill the cell with the "Irn-Bru Blank" and make two measurements of the absorbance as for the Fe^{3+} standards. Note these in Table 2 of the Results Sheet.

Fill the cell with the "Irn-Bru Sample" and make two measurements of the absorbance as for the Fe^{3+} standards. Note these in Table 2 of the Results Sheet.

Calculate the absorbance of Fe^{3+} in Irn-Bru by subtracting the average absorbance of the Irn-Bru Blank from the average absorbance of the Irn-Bru Sample. Note this in Table 2 of the Results sheet.

Practical 3 - What Did the Victim Overdose On?

Introduction

It is an unfortunate truth that some people use commonly available over-the-counter pharmaceuticals to attempt to take their own life. It is essential that the Hospital can be advised as quickly as possible what the victim has taken. You will be supplied with a crushed tablet found by the victim's bed and it will be your job to identify what it is rapidly enough to help save the victim's life.

TLC

The technique we will use to determine which drug was used is called Thin Layer Chromatography or or TLC, a simple and versatile technique that can provide useful information about drugs and drug products. Like other chromatographic processes, the separation of analytes in a TLC analysis depends on the relative interaction of the analyte between a solid stationary phase and a liquid mobile phase. The two most common types of TLC modes are Adsorption, sometimes called Forward Phase TLC, and Partition, sometimes called Reverse Phase TLC. Forward phase TLC, the most popular type and the type used in this experiment, acts through an adsorption, or hydrogen bonding interaction between the analyte and a polar stationary phase such as silica gel.

Rf Values

In both forward and reverse phase TLC analyses, the analyte is observed as a spot on the TLC plate. The extent of interaction is quantified with an Rf or retardation factor value. Rf values are calculated as the ratio of the distance travelled by the analyte to that travelled by the solvent. We will try to match the Rf value of the unknown sample, the tablet, to that of a series of known standards to identify which tablet the victim took. Implicit in this is that we have a limited range of possible tablets to test the unknown against, and that all of these possibilities have different Rf values.

Procedure

Preparation of Mixed Solvents

Extracting Solvent:- $10cm^3$ of 60-80 pet ether and $10cm^3$ of methylated spirits.
Developing Solvent:- $10cm^3$ ethyl acetate, $2cm^3$ 68-80 pet ether, $0.2cm^3$ glacial acetic acid

Preparation of Standards

There are three standards; aspirin, paracetemol and ibuprofen. For each of the three standards weigh out about 50mg into a sample tube, add $3cm^3$ of extraction solvent, and shake for three minutes. Label the sample tubes.

Preparation of Unknown Tablet

Transfer the crushed tablet white powder to a sample tube. Add about $5cm^3$ of the extraction solvent and shake gently for three minutes. Allow the sample tube to stand for five minutes to allow the powder to settle. Pack a little glass wool into a Pasteur pipette and mount it on a clamp stand with a clean empty sample bottle directly below the pipette. Filter the crushed tablet solution through the cotton wool plug. This solution is too concentrated to be run directly. Dilute it by a factor of 3 by adding 5 drops of the tablet solution to 15 drops of extracting solvent.

Loading the Plates

Take a careful note of how the demonstrator spots the sample plates. (See Diagram of TLC Plate). Load four spots, one each of the three standards and one of the unknown tablet solution.

Helpful Hints:

- Do not spot too heavily. The spots will run and give poor results.
- Make sure your spotting capillaries and developing jar are free from contaminants.
- Use a new capillary for each standard and for the sample.
- Only label your TLC plates with pencil, as ink is organic and will run along with your spots. Do not touch the absorbent side of the plate. The oils from your fingers will also give spots and confuse your results.

Visualising the Plate
You cannot see the spots in normal light. Use Ultra Violet (UV) light to visualise the spots on the plate. You must take care and DO NOT LOOK AT THE UV LIGHT DIRECTLY. Ask the instructor to check your plate before you run it.

Developing the Plate
Add $5cm^3$ developing solvent to the TLC development jar. Carefully lower the TLC plate into the jar. Screw on the lid. The solvent will start to run up the TLC plate. YOU MUST NOT MOVE THE JAR WHILE THE SOLVENT IS RUNNING. When the solvent has run to within 1cm of the top of the plate, unscrew the lid from the TLC development jar and carefully remove the plate. Mark with a pencil the position of the solvent front. Let the plate dry.

Visualising the Developed Plate
View your plate under UV light. Outline all the spots with a pencil. Mark the centre of each spot with a dot. Measure the distance from the starting line to the solvent front. This is the *solvent front distance*, $Solv_{dist}$

For each spot :-
measure the distance from the starting line to the dot in the middle of the spot. This is the *spot distance*, $Spot_{dist}$.
Calculate the R_f value as $Spot_{dist}$ / $Solv_{dist}$

<u>Conclusions</u>
By comparing the R_f values from the tablet and the standards determine which type of tablet the victim has taken

<u>Diagram of TLC Plate</u>