Schools Analyst Competition 2017

South East Regional Round

19th April 2017

South East Region Analytical Division

Royal Society of Chemistry

Kingston University

School of Pharmacy and Chemistry

Kingston University London

Royal Society of Chemistry
Analytical Division

Programme

9:30	Arrival and Registration
9:45	Welcome and Introduction (PRJG001) Dr Stephen Barton
10:00-14:00	Competition (MB2014/EM1027/EM1028)
12:00-14:00	Lunch Available (“The Diner”)
14:00-15:00	Instrument Demonstrations (Judging)
15:15	Debriefing, Results and Prize giving SERAD Committee Members (PRJG001)
15:30	Depart

Experiments:

1. Estimation of the Mass of Iron in Ferrous Sulphate Tablets

2. Absorption Spectra and the Beer-Lambert Law

3. The Determination of Ethanol in an alcoholic beverage by Gas Chromatography

It is important to plan your time well. Please read through the experiments before starting and work out a rota. Remember you will need to book a slot to use the GC, slots will be issued on a first come first served basis. Divide the tasks among team members. Each team member should have an opportunity to do one titration, measure absorbance and make one injection into the GC. You will also need to allow time for lunch. Safety instructions will be issued in the laboratory. Good Luck.

Experiment 1 - Estimation of the Mass of Iron in Ferrous Sulphate Tablets

Iron is essential to the human body: Its principal role is as a constituent of haemoglobin, the oxygen-carrying agent in the blood.

A satisfactory intake of iron can normally be obtained by eating a suitable diet. Certain foods such as liver, egg yolk or spinach are rich in iron. Some people, such as growing children and pregnant women, may need to supplement the iron taken in the natural diet, with iron tablets bought from a pharmacy.

Iron tablets usually contain iron (II) sulphate which is a cheap, soluble form of iron. In this experiment you are going to analyse these tablets to determine the mass of iron (II) sulphate. Note: the tablets will not be pure iron (II) sulphate, but will probably contain unreactive support materials as well.

Experimental

Weigh accurately one ferrous sulphate tablet. Grind up the tablet with a little 1M sulphuric acid, using a pestle and mortar. Through a funnel, transfer the resulting paste into a 50-cm^3 volumetric flask. Use further small volumes of 1M sulphuric acid to rinse the ground-up tablets into the flask. During this process, you must take great care to ensure that all the particles of tablet get into the flask. When this has been done, add sufficient 1M sulphuric acid to make up the solution to exactly 50-cm^3. Stopper the flask and shake it to make sure that all the contents are thoroughly mixed. The tablet will not completely go into solution but the Fe^{2+} ions which were present in the tablets will be dissolved. Let the solution "settle".

Titrate 10-cm^3 portions of the clear solution with 0.005M potassium manganate(VII). The titration is self-indicating, the end-point being marked by the first permanent purple colour. Brown or red colours should not be allowed to develop; the remedy is to add more 1M sulphuric acid.

Potassium manganate(VII) is a well known oxidising agent, usually used in solutions acidified with a plentiful supply of dilute sulphuric acid. Reference to the following redox potentials shows that manganate(VII) ions should oxidise iron(II) ions:

$$Fe^{3+}_{(aq)} + e^- = Fe^{2+}_{(aq)} \qquad E^o = +0.77V$$

$$MnO^-_{4(aq)} + 8H^+_{(aq)} + 5e^- = Mn^{2+}_{(aq)} + 4H_2O_{(l)} \qquad E^o = +1.51V$$

Combining these two equations gives the overall equation for the reaction:

$$MnO^-_{4(aq)} + 8H^+_{(aq)} + 5Fe^{2+}_{(aq)} = Mn^{2+}_{(aq)} + 5Fe^{3+}_{(aq)} + 4H_2O_{(l)}$$

so that in acid solution 1 mole of MnO_4^- reacts with 5 moles of Fe^{2+} (aq).

Solutions containing MnO_4^- ions have an intense purple colour, whereas those containing Mn^{2+}(aq) ions are virtually colourless. Solutions containing Fe^{2+}(aq) ions

can be titrated against potassium manganate(VII) solution. The colour of the manganate(VII) is discharged, the end-point of the titration being the point at which the addition of one more drop of potassium manganate(VII) gives a permanent purple colour. This titration forms the basis of an analytical technique for the estimation of iron.

Results

Calculate the mass of iron sulphate in the tablets from your results. Record this result on the data sheet provided at the back of this manual.

Experiment 2 - Absorption Spectra and the Beer-Lambert Law

All coloured compounds have their own characteristic absorption spectrum. At any particular wavelength the amount of absorption of light energy occurring when visible light passes through a solution of a coloured compound depends on:

(i) the length of the light path through the solution
(ii) the concentration of the coloured compound

This is expressed in the Beer-Lambert law, which states that:

$$A = \varepsilon Cl$$

Where A is absorbance, ε is a constant (whose value depends on the units of concentration) C is the concentration of the coloured compound (usually expressed as molarity) and l is the length of the light path through the sample cuvette (usually 1cm).

When 1, the length of the light path is constant, absorbance is proportional to concentration if the Beer-Lambert law is obeyed.

When C is expressed as a molarity, ε, molar absorptivity, is described as the absorbance that would be measured when light of a specified wavelength passes through 1cm of a 1M solution of a coloured compound.

If ε is known for a particular compound at a specific wavelength, the relationship can be rearranged to determine the concentration of a solution of that compound.

$$\text{Concentration} = A/\varepsilon$$

Aim

The aim of this experiment is to prepare TWO Beer plots, and determine the slope, which is equivalent to ε. Then to calculate the concentration of the TWO unknown solutions using the relationship $C = A/\varepsilon$

Reagents

Bromophenol blue (BB) (1.5×10^{-5}M)
Methyl orange (MO) (3×10^{-5} M)

Two solutions of unknown concentration.

Experimental

1 Prepare a series of dilutions in test tubes, of <u>both</u> bromophenol blue and methyl orange, using the protocol set out below, to cover the concentration range shown in the table.

Tube No	2	4	6	8	10
Stock solution (cm^3)	0.6	1.2	1.8	2.4	3.0
Distilled water (cm^3)	2.4	1.8	1.2	0.6	0
Dilution Factor	*0.2*	*0.4*	*0.6*	*0.8*	*1*
Concentration BB (M)					
Concentration MO (M)					

Work out the concentration of each solution by multiplying standard concentration by the dilution factor.
Note that this protocol gives a series of 5 equally spaced dilutions. A total of 3cm³ has been chosen to give a sufficient volume of each solution to fill a colorimeter cuvette.

2 Set the colorimeter to the wavelength of maximum absorbance for either bromophenol blue or methyl orange (given), zero against distilled water and measure the absorbance of each dilution of that particular compound.

3 Repeat the above procedure for the other dilution series.

4 Plot absorbance (Y axis) against concentration (X axis) for each dilution series on the same graph.

Report

From the slope of each line obtained determine the **absorption coefficient ε** for each compound.
Use the value of ε to calculate the concentration of the TWO unknowns.

(λ_{max} bromophenol blue = 590 nm ; λ_{max} methyl orange = 460 nm)

Experiment 3 - The Determination of Ethanol in an alcoholic beverage by Gas Chromatography

Gas chromatography (GC) is used for the separation and quantitative analysis of mixtures where the components are sufficiently volatile and thermally stable to be passed through a chromatographic column in the vapour state. This normally requires elevated temperatures of 100 to 400^{o}C. It is used in the analysis of petrochemicals and many products based on them, solvents, volatile natural products, pesticide and herbicide residues, and paints and polymers after pyrolysis. The components of a mixture are carried through the column by an inert carrier gas and are generally eluted in order of increasing boiling points, although differing affinities for the stationary phase may affect the order of elution.

In this experiment the GC uses a capillary columns, 25 meters long and the carrier gas is helium. A calibration graph and sample chromatogram has been supplied.

Experimental

Use a pipette to transfer 2 cm^3 of the sample beverage into a 250 cm^3 volumetric flask. Using a pipette add 10 cm^3 of the standard propanol solution (4%) and make up to volume with distilled water.

Inject 0.5 μl of this solution into the GC. When the peaks have emerged, identify them from the trace shown and calculate the ratio of the peak areas ethanol/propanol. Repeat the injection until the ratios are constant.

Results

On the data sheet provided tabulate the values for the peak areas for propanol and ethanol and the corresponding ratios. Use the calibration graph provided to work out the percentage of ethanol in the beverage. Attach the calibration graph for your instrument, include a sample chromatogram and do not forget to record your experimental conditions.

DATA SHEET School_____ Team _____

Names_____ _____ _____

Experiment 1

Mass of Iron tablet _____ mg

Titration values 1 _____ cm^3
2 _____ cm^3
3 _____ cm^3

Mass of iron (II) sulphate per tablet _____ mg

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Experiment 2

Absorption coefficient, ε for bromophenol blue =_____

Absorption coefficient ε for methyl orange =_____

Concentration of bromophenol blue unknown =_____ (M)

Concentration of methyl orange unknown = _____ (M)

Please attach graphs.

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Experiment 3:

Instrument used:

Table:

The % ethanol in the beverage was found to be _____ %

Remember to attach the calibration graph, chromatogram and experimental conditions.

Technicians Sheet

Experiment 1

One Ferrous Sulphate Tablet
Mortar and Pestle
Funnel
Pasteur Pipettes
1M H_2SO_4
1 x 50 ml vol flask
1 x 10ml pipette
Burette, stand, flasks
0.005M potassium permanganate (VII)

Experiment 2

Bromophenol blue (10.4 mg/l) minimun10ml each [MW = 691.9]
Methyl orange (9.8 mg/l) minimum10 ml each [MW = 327.3]
Unknown Solution BB (4 mg/l) minimum 6 ml each
Unknown Solution MO (6 mg/l) minimum 6 ml each
Distilled water
5 test tubes
Test tube rack
2 x graduated 5ml pipette
Pipette bulb
Colorimeter and cuvettes (plastic)

Experiment 3

Alcoholic beverage
2ml pipette
10 ml pipette
250 ml vol flask
4% propan-1-ol solution (20 ml per group)
GC