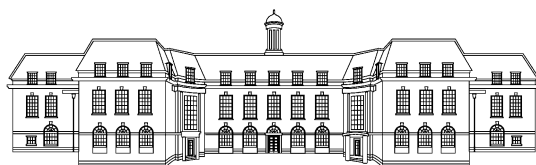# The Royal Society of Chemistry Schools' Analyst Competition

Northern Ireland Regional Heat

at

STRANMILLIS UNIVERSITY COLLEGE

A College of Queen's University Belfast

25th March 2017

Introduction

Welcome to the Northern Ireland Regional Heat of the Royal Society of Chemistry Schools' Analyst Competition organised by the Society's Analytical Division (Northern Ireland sub-region) at Stranmillis University College Belfast.

The schools taking part in the competition are:

Thornhill College Derry

St Dominic's High School Belfast

Down High School Downpatrick

Friends' School Lisburn

Royal Belfast Academical Institution

Lumen Christi College Derry

Cookstown High School

Belfast Royal Academy

Regent House N'Ards

South Eastern Regional College Lisburn

Rainey Endowed Magherafelt

Loreto College Coleraine

The aim of the competition is to raise standards of practical analytical chemistry amongst sixth-form students and is based on problems relevant to industrial and social needs. Students are assessed in their practical skills as well as in their understanding of chemical analysis and their ability to work safely as a team.
This year the experiments relate to the role of analytical chemistry in the analysis of household and pharmaceutical substances. Each team will carry out three experiments which all carry equal marks.

Safety

The experiments have been well tried and tested and the hazards involved are relatively low. A full Risk Assessment has been carried out. Nevertheless, accidents can be caused by carelessness, thoughtlessness or improper procedures. Students are required to take due care and attention at all times during the practical work and to observe the instructions of the demonstrators and the manual. Some of the chemicals you will be using can damage or stain clothing or skin, and therefore safety gloves, laboratory coats and safety glasses should be worn at all times. In the unlikely event of an accident or fire occurring students should seek help and advice from a demonstrator.

Note: Safety glasses and lab coats must be worn at all times in the laboratory.

Timetable:

9.30am-9.45am   Coffee/Tea and scones on arrival Coffee bar (Central building)

9.45am-10.00am   Welcome by Dr Irene Bell (Head of STEM at Stranmillis
                 University College Belfast)
                 Details of laboratory allocation (Dr McCullagh)

10.00am-1.00pm   Students carry out experiments in the allocated laboratories.
                 Teachers are free until 1.00pm.

1.15pm-2.15pm   Lunch will be provided courtesy of the Royal Society in the
                 College's 'Chatz' restaurant.

2.15pm-2.30pm   Presentation of prizes to participants.

The Royal Society of Chemistry (RSC)

The RSC is the principal body serving the interests of chemists in the British Isles and further afield. It provides a qualification structure with designations such as FRSC, MRSC and CChem. It is organised in six divisions, which reflects the traditional

interest of organic, inorganic and physical chemistry and also the major areas of analytical chemistry, industry and education. These divisions are all organised into regions of which Ireland is one and Northern Ireland is a sub-region of the Analytical Division. The society has around 45,000 members of which 10,000 are members of the Analytical Division, which includes around 100 in Northern Ireland.

The RSC provides hundreds of lectures each year and organises conferences of high international reputation. The Society directs much of its activity towards schools. It produces the Journal of Education in Chemistry and numerous posters, career leaflets and other materials. Schools are able to subscribe to the RSC Schools' publications and material from fellow societies around the world is also available to subscribers.

To find out more visit the website www.rsc.org

Acknowledgements.

The Analytical Division in Northern Ireland would like to thank Stranmillis University College for once again hosting the competition.

Health and Safety in the Laboratory

To ensure your safety and the safety of others you must at all times wear safety glasses.
It is also recommended that you

- **Exercise caution when handling delicate glassware such as pipettes and burettes.**

- **Keep your bench orderly and tidy.**

- **If you are not sure of what you are doing please ask a demonstrator.**

Protective gloves are provided

Experiment 1. The determination of the citric acid content of lemon juice

Introduction

Lemons are a healthy citrus fruit full of vitamins, minerals, citric acid and antioxidants. Because of their sour taste and low sugar content, lemons are usually added to drinks or foods, especially baked desserts. Lemon juice is also commonly used in cooking to prevent discolouring of sliced fruits such as apples and bananas. Lemons contain citric acid as well as smaller amounts of ascorbic acid (Vitamin C) and malic acid. In this experiment you will assume that all the acidity is due to citric acid.

Citric acid is a weak organic acid found in many fruit and vegetables. A molecule of citric acid contains three acidic groups.
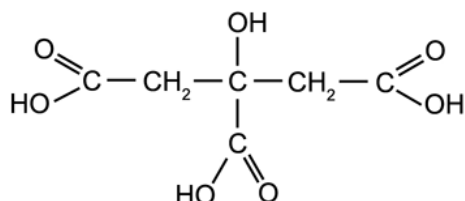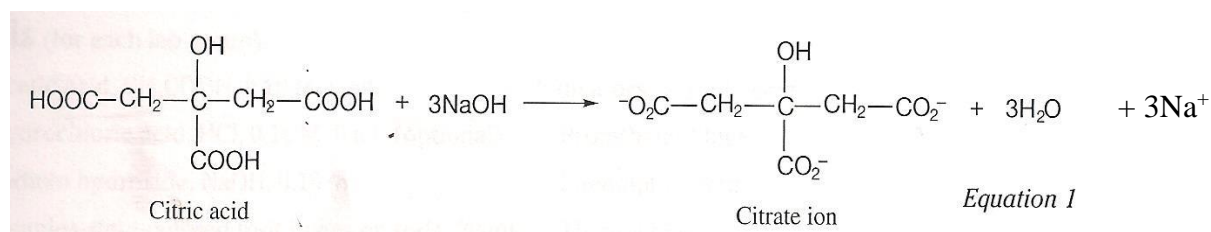

Theory

Citric acid is a weak acid and its concentration can be determined by titration with a standard solution of an alkali such as sodium hydroxide solution, in the presence of an acid-base indicator. As the titration is between a weak acid (citric acid) and a strong base (sodium hydroxide) the most suitable indicator is phenolphthalein. Phenolphthalein indicator is colourless in solutions which have a pH below 10 and is pink in solutions above 10. The endpoint is determined when the solution first takes on a permanent hint of pink. Each oxalic acid molecule contains three acid groups and

therefore requires three sodium hydroxides for neutralisation. The equation for the reaction is given below.

$$HOOC-CH_2-\underset{\underset{COOH}{|}}{\overset{\overset{OH}{|}}{C}}-CH_2-COOH \ + \ 3NaOH \longrightarrow \ {}^-O_2C-CH_2-\underset{\underset{CO_2^-}{|}}{\overset{\overset{OH}{|}}{C}}-CH_2-CO_2^- \ + \ 3H_2O \ + 3Na^+$$

Citric acid   Citrate ion   *Equation 1*

Method.

Safety Glasses must be worn at all times
**Handle all glassware with care**
Ensure your bench is tidy

1. Using a 25.0cm^3 pipette transfer 25.0cm^3 of the sample of lemon juice provided into a 250cm^3 volumetric flask and make to volume with distilled water. Invert several times to ensure mixing.
2. Using a 25.0cm^3 pipette transfer 25.0cm^3 of this diluted solution of lemon juice into a conical flask.
3. Add three drops of phenolphthalein indicator and gently swirl the flask.
4. Titrate the contents of the conical flask with 0.100M NaOH. The endpoint corresponds to the first permanent hint of pink in the solution.
5. Repeat the above procedure to obtain three titre values and enter results in Mark Sheet 1 provided.
6. Use your results, along with the balanced equation, and the relative atomic masses (C=12.01. H=1.01, O=16.00) , to determine the mass of citric acid present in 100cm^3 of the sample of lemon juice (the percentage mass).

Experiment 2. The determination of the formula of oxalic acid crystals by redox titration

Introduction

Instrumental methods of analysis, such as infra-red spectroscopy and spectrophotometry are routinely used by analytical chemists to determine the exact molecular formulae and structure of compounds. Bench techniques can also be very useful. In this experiment you will use a redox titration to determine the formula of oxalic acid. This compound contains within its structure molecules of water, known as water of crystallisation. This phenomenon is also found in other compounds. (You may be familiar with how the water of crystallisation present in the blue crystals of copper sulphate can be driven off by heating, to leave behind white anhydrous copper sulphate.) In this experiment you will prepare an aqueous solution of oxalic acid and use a redox titration to determine the exact number of molecules of water present, and therefore the value of X in the formula, $(COOH)_2.XH_2O$.
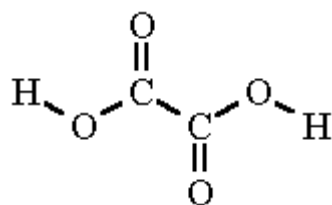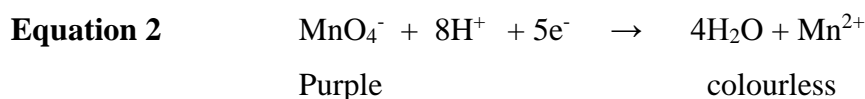

Molecular structure of oxalic acid $(COOH)_2$

Theory

Oxalate ions, produced when oxalic acid is dissolved in water, undergo a **redox** reaction with a solution of potassium permanganate under acidic conditions. In this reaction electrons are transferred from the oxalate ion to the permanganate ion. Each oxalate ion **loses** two electrons to form carbon dioxide. The oxalate ion is said to be **oxidised** as it has **lost** electrons. This can be represented by Equation 1 below.

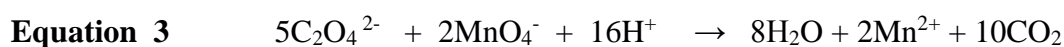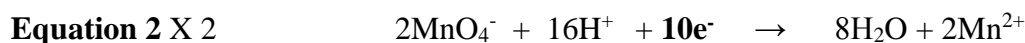**Equation 1** $\qquad C_2O_4{}^{2-} \quad \rightarrow \quad 2CO_2 + 2e^-$

These electrons are **gained** by the permanganate ions (manganese (VII)) which become manganese (II) ions. The permanganate ion is said to be **reduced.** This can be represented by Equation 2 below.

Equation 2 $\qquad MnO_4{}^- + 8H^+ + 5e^- \quad \rightarrow \quad 4H_2O + Mn^{2+}$

$\qquad\qquad\qquad$ Purple $\qquad\qquad\qquad\qquad\qquad$ colourless

An overall balanced equation can be obtained by ensuring that the number of electrons produced in Equation 1 is equal to the number of electrons used up in Equation 2. This can be done by multiplying Equation 1 by five and combining it with Equation 2 multiplied by two. When we do this we can see that the number of electrons on each side of the equation is equal and so they cancel each other out and therefore do not appear in the final overall balanced equation. (Equation 3)

Equation 1 X 5 $\qquad\qquad$ $5C_2O_4{}^{2-} \rightarrow 10CO_2 + \mathbf{10e^-}$

Equation 2 X 2 $\qquad\qquad$ $2MnO_4{}^- + 16H^+ + \mathbf{10e^-} \rightarrow 8H_2O + 2Mn^{2+}$

_____

Equation 3 $\qquad$ $5C_2O_4{}^{2-} + 2MnO_4{}^- + 16H^+ \rightarrow 8H_2O + 2Mn^{2+} + 10CO_2$

Note that each oxalate ion produces **two** electrons, but each permanganate ion requires **five** electrons. Therefore we require 2.5 times as many oxalate ions to provide enough electrons for each permanganate ion.

The oxalate and permanganate ions react in the ratio 2:5 as shown in Equation 3.

The reaction is very slow at room temperature so the solution of oxalic acid and dilute sulphuric acid must be heated. As the purple permanganate ions are added from the burette, they react with the oxalate ions present in the conical flask and are converted to colourless manganese (II) ions, preventing the purple colour of permanganate appearing in the conical flask. At the endpoint all the oxalate ions have now been destroyed by the permanganate. Any permanganate added beyond the endpoint will no longer have oxalate ions to react with and so will remain to produce a faint purple colour in the conical flask. The endpoint for the titration occurs at the point when the solution takes on the first hint of pink.

Safety Glasses and protective gloves must be worn at all times during this experiment.
Exercise caution when handling hot apparatus and hot glassware.
Method
  1. Set the balance to zero by pressing 'Tare'.
  2. Weigh out approximately 1.5g of oxalic acid using the weighing boat provided and record the exact mass of weighing boat and sample to 2 decimal places, and record in Mark Sheet 2.

3. Carefully transfer all of this sample of oxalic acid into a 250cm^3 beaker

4. Set the empty boat back on the balance and record its mass.

5. By difference calculate the exact mass of oxalic acid used.

6. Using a measuring cylinder, add approximately 200cm^3 of distilled water to the beaker. Ensure that the volume of water present in the beaker does not exceed 250cm^3.

7. Using a glass rod, stir the contents of the beaker in order to dissolve all of the oxalic acid. Wash down the glass rod with distilled water to ensure none of the solution is lost from the beaker when the glass rod is removed.

8. Using a glass funnel, transfer the solution into the 250 cm^3 volumetric flask and make to volume with distilled water. Use a plastic dropper to add the final few cm^3 of water for accuracy. Invert several times to ensure thorough mixing of the contents of the flask.

9. Using a 25.0cm^3 pipette, transfer 25.0 cm^3 of this solution of oxalic acid into a conical flask and, using a measuring cylinder, add 25 cm^3 of 1M sulphuric acid.

10. Carefully set the conical flask onto the tripod and wire gauze and heat the solution to 80 degrees Celsius. **(Do not heat until boiling).**

11. **When the conical flask has cooled sufficiently to hold, carefully** lift the **hot** conical flask off the wire gauze and set it on the bench. Before removing the thermometer from the conical flask, use the wash bottle to wash any traces of solution back into the flask.

12. Titrate the hot solution with the standard solution of 0.020M potassium permanganate solution and record your endpoint in the table provided in Mark Sheet 2.

13. Repeat the titration on another two 25.0cm^3 samples of solution, recording your tire values in Mark Sheet 2.

14. Calculate the mean titre value and the number of molecules of water of crystallisation as directed in Mark Sheet 2.

**Experiment 3.   Colorimetric analysis of a health supplements.**

Introduction

Treatment for iron deficiency anaemia usually involves taking iron supplements and changing your diet to increase your iron intake levels, as well as treating the underlying cause. In this experiment you will determine the amount of iron in two supplements which contain iron (II) ions. The concentration of iron (II) is determined by a technique called colorimetry.

Theory

Colorimetry is a quantitative analytical technique suitable for determining species present in solution in very low concentrations. For ionic species to be determined they are usually reacted with a complexing agent with which they form a stable and brightly coloured product. The basic principle behind this technique is that the amount of light absorbed depends on the concentration of the species being determined.
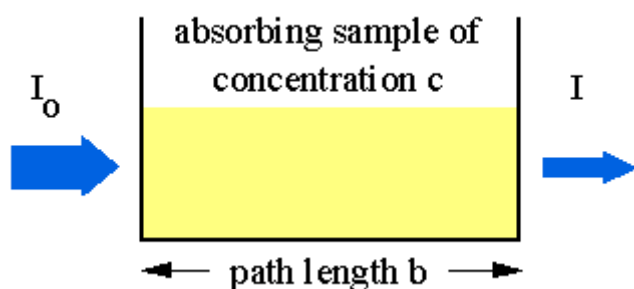
If the intensity of light incident on the sample is represented by I_o and I represents the intensity of the same light beam after passing through the sample, the absorbance is defined as,

**Absorbance, A = $\log_{10} I_o/I$**

The Absorbance depends on, the length of the cell ('b' or often denoted as 'l'), the concentration of the solution ('c') and the "tendancy" of the particular species present in solution to absorb light (its extinction coefficient, 'ε'). Thus,

$$A = \varepsilon cb$$

Therefore if we keep the cell length constant (by using the same cuvette), the absorbance is directly proportional to the concentration. This relationship is called the Beer-Lambert law and allows us to plot a calibration graph that can be used to determine the concentration of an unknown solution. Note that at higher concentrations the plot is no longer linear and begins to curve. It is therefore best to work at concentrations where the linear relationship exists.
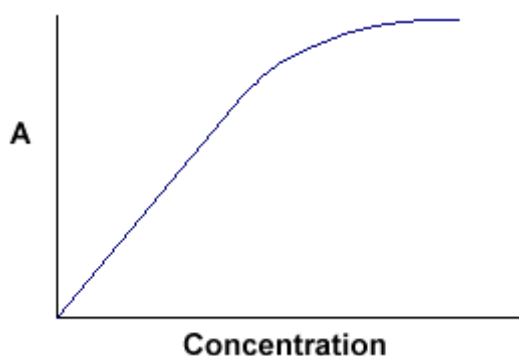
In this experiment the iron (II) ions will be reacted with an aqueous solution of the complexing agent 1,10-phenanthroline. Each 1,10-phenanthroline molecule has two nitrogen atoms available for coordination to the iron ion. Each iron ion coordinates to 3 molecules of 1, 10-phenanthroline to form the brightly coloured complex.
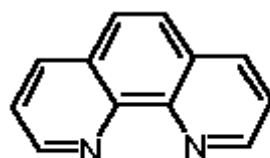
1,10-phenanthroline

If we represent the 1,10-phenanthroline as L we can represent the reaction as,

$Fe^{2+} + 3 L \rightarrow [FeL_3]^{2+}$

The complex only forms with divalent iron ions and at a pH between 2.5 and 4. Therefore it is necessary to add the reducing agent ascorbic acid (this converts any iron (III) present into iron(II)) and sodium acetate (to buffer the solution). This method consists of measuring the absorbance at a fixed wavelength of a range of solutions of iron(II) and the absorbance of the sample of health supplement after formation of the coloured iron(II) complex. The effect of any background colour is allowed for by measurement of a blank. A calibration graph (Absorbance vs Concentration) is plotted which allow us to determine the concentration of Fe^{2+} in the tablet.

Method

Calibration Graph

1. Into a series of $100cm^3$ volumetric flasks add from a burette, 5.0, 10.0, 15.0, 20.0 and 25.0 cm^3 of the standard 10mg / litre solution of Fe^{2+}.
2. To each flask add the following:

. $10cm^3$ (using a measuring cylinder) of 0.1M sodium acetate

. $1cm^3$ (use the plastic dropper) of 10% ascorbic acid

. $5cm^3$ (using a measuring cylinder) of the 0.25% 1, 10-phenanthroline solution
   Dilute each flask to the mark with distilled water and **mix thoroughly.**

Preparation of 'Blank'.

Into a $100cm^3$ volumetric flask add

- $10cm^3$ (using a measuring cylinder) of 0.1M sodium acetate

- $1cm^3$ (use the plastic dropper) of 10% ascorbic acid

- $5cm^3$ (using a measuring cylinder) of the 0.25% 1, 10-phenanthroline solution
   Dilute the flask to the mark with distilled water and **mix thoroughly.**

Sample Analysis

Feroglobin

1. Place one tablet in a $250cm^3$ beaker. Three quarters fill the beaker with distilled water and wait for all the tablet to dissolve.
2. Using a funnel, carefully transfer the solution into a IL volumetric flask. Rinse the beaker with distilled water and add this to the flask and make to volume with distilled water. Invert several times to ensure thorough mixing.
3. Pipette $10.0cm^3$ of this diluted solution into a $100cm^3$ volumetric flask and add ascorbic acid, sodium acetate and the 1,10-phenanthroline solution as above.
4. Repeat on a duplicate sample of the solution of Feroglobin.
5. Empty out the IL flask and rinse with deionized water for the analysis of Floravital.

Floravital

1. Using a 20.0cm^3 pipette, transfer 20.0cm^3 of your sample of Floradix into a 1L volumetric flask and make to volume with distilled water.

2. Pipette 10.0cm^3 of this diluted solution into a 100cm^3 volumetric flask and add ascorbic acid, sodium acetate and the 1,10-phenanthroline solution as above. Repeat on a duplicate sample of the diluted solution.

Absorbance Measurement

1. Select the wavelength range 470-520nm of the colorimeter by selecting Filter Number 3.

2. Three quarters fill a cuvette with the Blank sample and place it into the colorimeter. Set the colorimeter to zero by pressing the zero button.

3. **Using the same cuvette** measure the absorbance of each of your solutions and record the results in Mark Sheet 3. In the same way measure and record the absorbance of the duplicate samples of each of the health supplements.

3. Plot a graph of the Absorbance (y axis) against the Concentration of the iron^{2+} (mg/ litre) (x axis).

4. Draw a 'line of best fit ' on your graph and use it to determine the concentration of iron^{2+} present in each of the health supplements as directed on the Mark Sheet.