Schools' Analyst Competition

Manchester Heat

Wednesday 19th April 2017

School of Chemistry

The University of Manchester

Sponsored by

RSC North West Division Analytical Trust

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PROGRAMME OF EVENTS

09.30 – 09.45	**Arrival, registration & refreshments (Concourse – Chemistry Building)**
09:45 – 10.15	**Welcome, Instructions & Health and Safety Briefing (Lecture Theatre G.53)**
10.15 – 10.45	**RSC talk – Teachers only (G.53)**
10.30 – 12.45	**Lab Analytical Challenges (Measurement Lab)**
11.00 – 12.30	**Refreshments & CPD (ToF MS) - Teachers only (GE.005)**
12.45 – 13.30	**LUNCH for all attendees (GE.005)**
13.30 – 14.15	**Tour of the School of Chemistry**
14.15 – 15.00	**Demonstration lecture – "A Quantum of Science" (G.53)**
15.00 – 15.30	**Prize-giving, photos and close (G.53)**

Safety

A chemistry laboratory can be a dangerous place, and so it is important that the lab safety rules are followed all the time:

- *No eating or drinking in the laboratory*
- *Always wear your lab coat and safety glasses (or over specs) in the lab*
- *Long hair should be tied back*
- *Treat all chemicals as potentially dangerous and avoid any skin contact. If you do get chemicals on your skin, wash them off immediately.*
- ***Gloves:** These can protect your skin from exposure, but **ONLY** if they are used properly: Use only when risk of skin exposure is serious (e.g. strong NaOH solution) and **discard immediately once contaminated**. NEVER exit a lab with gloves. NEVER hold a pen with gloves, or operate a calculator or computer with gloves. These rules ensure that contamination is not transferred to items which can leave the lab.*
- *If you spill any chemicals report the spillage to a Demonstrator at once so that it can be dealt with quickly*
- *If you break glassware, report it to a Demonstrator so that it can be cleared up safely and replaced*
- *Take great care when putting pipette fillers onto pipettes (see page 4 of this booklet)*

COSHH Assessment for Chemicals

Chemical	*Approximate Quantity*	*Hazards*
Approx. 0.02 M EDTA solution	***250 cm^3***	***Irritant***
Approx. 0.02 M Ca^{2+} solution	***100 cm^3***	***No hazard***
NaOH solution dilute and 8M	***20 cm^3***	***Corrosive irritant***
Patton Reed Indicator Solution	***3 cm^3***	***No hazard***
Milk	***100 cm^3***	***No hazard***
Lucozade	***100 cm^3***	***No hazard***

SAFE LAB PROCEDURE: Pipettes

There is a significant risk of serious injury when attaching a pipette filler to a pipette. To minimise this risk:

- ALWAYS take hold of a pipette near the top, NOT round the expanded portion in the centre.
- When attaching a pipette filler to a pipette, hold the pipette near the top, so that there is a maximum of 5 cm between the thumbs of each hand. See diagram:

Adhering to this rule will avoid any trips to A and E.

Operation of pipette:

General aspects of correct pipette use should be covered in your school curriculum, but since not all schools use the same type of pipette filler, brief instructions on the use of the rubber 'bulb' type are presented below.

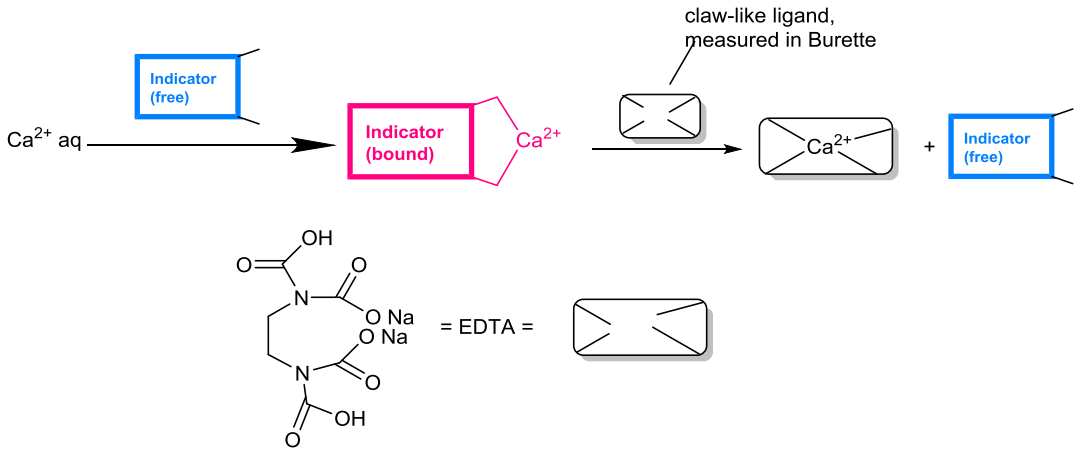
1. Attach a pipette filler as above.
2. Press valve 'A' and expel air from the bulb by squeezing firmly, then release valve 'A'.
3. Insert the pipette tip into a portion of the solution, so that the tip is fully immersed, but not touching the bottom of the container.
4. Press valve 'S' to 'Suck' solution into the pipette.
5. Press valve 'E' to 'Empty' solution to waste until the level of the meniscus matches the calibration line on the pipette. Very gentle pressure is required for fine control.
6. Discharge the measured quantity to a flask by firm pressure on valve 'E'.

ASSESSMENT

Your main task is to perform a 'complexometric' titration. You may have studied acid-base titrations, but are unlikely to have encountered complexometric titration. It is very similar to acid-base. An indicator is used to show the progress of the reaction. The indicator is a strong binder to metal ions. When it binds to a metal ion, it changes its colour. When freed from the metal ion, it shows its inherent free colour. We can use a compound which is even better at binding to metals, which we term a 'ligand' to strip the indicator molecule from the metal. The ligand we will use is called ethylene diamine tetra-acetic acid, or EDTA, for short. It grabs the ion in a claw-like grip. If we measure

how much EDTA we need to free all of the indicator, since we know that one EDTA wraps up one metal ion, we can determine the amount of metal ion present.

The process is illustrated schematically below:



Winners will be determined by the quality of the results using a statistical algorithm, and by adherence to correct selection and use of apparatus, and safe laboratory procedure, as determined by the judges.

INSTRUCTIONS

For each sample, determine the amount of calcium present in solution. Express your result in the form XXX mg Ca L^{-1}, using the degree of precision you consider most appropriate.

You are provided with

- **0.025 M EDTA solution**, a chelating agent. One mole of EDTA binds to one mole of Ca^{2+} ions.
- **Patton-Reeder Indicator**. This is **blue** when free, but **pink** when bound to Ca^{2+} ions. The end point is when the last traces of purple (a mix of pink and blue) disappear, before a pure blue colour is apparent.
- **Concentrated NaOH aq**. (CAUTION: GLOVES!)
- A Burette
- Pipettes, of 10 mL and 25 mL size
- Pipette filler bulbs
- Volumetric Flasks, 250 mL
- Conical Flasks, 250 mL
- Measuring cylinders
- Plastic Pasteur pipettes
- Your samples for Analysis
- A calculator

PROCEDURE

1. Take a precisely measured volume of sample, either 10 or 25 mL, of one of the five provided 'analyte' samples and place in a clean 250 mL conical flask.
2. Dilute this to a volume of 50 mL, i.e with 40 mL water (for a 10 mL sample), or 25 mL water (for a 25 mL sample).
3. Add 4 mL of NaOH (aq) 8M. CAUTION: strongly caustic. This is to precipitate any Mg^{2+} ions present as insoluble $Mg(OH)_2$, thereby removing them as an interference in the Ca^{2+} analysis. It also keeps the indicator in the deprotonated form required to bind to the metal.
4. Add a spatula tip of the solid indicator powder, then shake vigorously to disperse.
5. Titrate the sample vs EDTA.
6. Record your results in the tables overleaf.
7. Repeat 1 - 6 for concordant results.

WORK AS A TEAM. GET SEVERAL RESULTS ON ALL 5 SAMPLES, then COMBINE your results.

ONE report sheet per team is submitted to the judges at the end.

NOTE THAT: Good laboratory practice requires that volumes measured by Burette lie in the range 10-30 mL, and that the Relative Molar Mass of Calcium is 40 g mol^{-1}.

RESULTS: Use these sheets for your individual results, then compare and fill in the separate TEAM results sheet, remembering to add the name of your School/College.

SAMPLE 1. Milk						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample 1:						

SAMPLE 2. Mineral Water A.						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample 2:						

SAMPLE 3. Mineral Water B.						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample 3:						

SAMPLE 4. Manchester Tap Water						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample 4:						

SAMPLE 5. Lucozade						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample 5:						

SPARE DATA GRID (in case of errors).

SAMPLE ___. _____.						
Volume of sample /mL	Initial titrant (EDTA) volume /mL	Final titrant (EDTA) Volume /mL	Titre of EDTA /mL	Molarity of titrant (EDTA) /mol L^{-1}	Number of moles of calcium in 1 sample volume /mol	Ca content /mg Ca L^{-1}
Mean Result: Ca content of Sample __:						

At some point in the session, your demonstrators will take you to do a Caffeine analysis (see over).

Use of LC-MS and GC-MS to quantify and identify components of sports drinks

Background

The consumption of energy drinks has dramatically increased in recent years, with high popularity among university students, especially at exam time. The European Food Safety Authority (EFSA) published a study on energy drinks consumption which reports that 13.3% of young adults (in the age range 18–29 years) consume energy drinks 4 or 5 times a week or more, yielding an estimated intake of 4.5 L per month per person. Lack of consistent labelling, (which currently is under-legislated) and variations in allowances in sale of these drinks coupled with aggressive marketing campaigns, primarily targeted to young adults, promoting the stimulant effects of these beverages have not provided enough warning about the possible negative consequences that some of their ingredients may have on their health. The increasingly excessive consumption of these products has led to a marked increase in reported medical treatment. Chronic consumption of these products is a public health concern since the long-term health effects are uncertain and inadequately studied.

Caffeine is the major active component of these drinks, and the risks derived from its consumption have been extensively studied. Nevertheless, besides caffeine, energy drinks contain high amounts of B vitamins, particularly pyridoxine (vitamin B6) and riboflavin (vitamin B2). Pyridoxine plays an essential role in the interaction of amino acid, carbohydrate, and fatty acid metabolism through the citric acid cycle by means of B6 coenzymes. Riboflavin is widely distributed in plant and animal cells. Flavoproteins (proteins that contain a nucleic acid derivative of riboflavin) act as catalysts in biological redox systems, and are essential for carbohydrate, lipid and amino acid biosynthesis and metabolism, as well as for the activation of other vitamins.

Riboflavin is absorbed quickly; however, the dose per can in some of these beverages exceeds the recommended intake (1.6 mg/day). The amounts of pyridoxine contained in these drinks are more concerning since the compound can induce a severe sensory neuropathy, even leaving patients unable to walk. Photo-sensitivity and dermatitis have also been described due to pyridoxine toxicity. The U.S. Recommended Dietary Allowance for a healthy adult is 1.3 mg/day of pyridoxine. Monster beverages contain **declared** values of 2 mg of pyridoxine per can, whereas Red Bull contains about 5 mg per can. Some other drinks contain amounts as high as 40 mg per can.

Caffeine has been established as a target analyte in many undergraduate analytical chemistry courses, and several methods for its determination in different samples have been described. Pyridoxine has also been determined by high-performance liquid chromatography (HPLC), using UV detection, in undergraduate analytical chemistry courses. In a previous research paper, HPLC with fluorescence detection was proposed for determining riboflavin and pyridoxine together with other vitamins in various beverages, including energy drinks.

Mass spectrometry (MS) is a powerful technique for the detection, identification, and quantification of organic compounds, frequently coupled with chromatographic separation either with **gas chromatography** or **liquid chromatography**, i.e. **GC-MS** or **LC-MS**, so-called hyphenated mass analysis. As mass spectrometers have become more user-friendly and affordable, many research projects incorporate mass spectrometry into their research.

In this part of the Schools' Analyst competition you will take a sample of Lucozade to a demonstrator and observe while they use LC-MS to analyse the contents.

You will receive a mass selected ion count from the caffeine signal. Use a supplied standard curve to estimate how much caffeine is in Lucozade.