MERSEYSIDE YOUNG ANALYSTS COMPETITION

4th March 2017

Central Teaching Laboratory
University of Liverpool

Sponsored by:

RSC Liverpool Section Trust

RSC North West Analytical Division

Agilent Technologies UK Ltd.

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PROGRAMME

9.00 - 9.30	Registration and Coffee
9.30 - 10.00	Introduction and briefing (Lecture Theatre C)
10.00 - 12.45	Practical session (Lab 8)
10.15 – 11.15	ToF Mass Spectrometry – Teacher Session (Lecture Theatre C)
12.45 – 1.30	Buffet lunch (Ground Floor Atrium)
1.30 – 1.45	Prizegiving

SAFETY

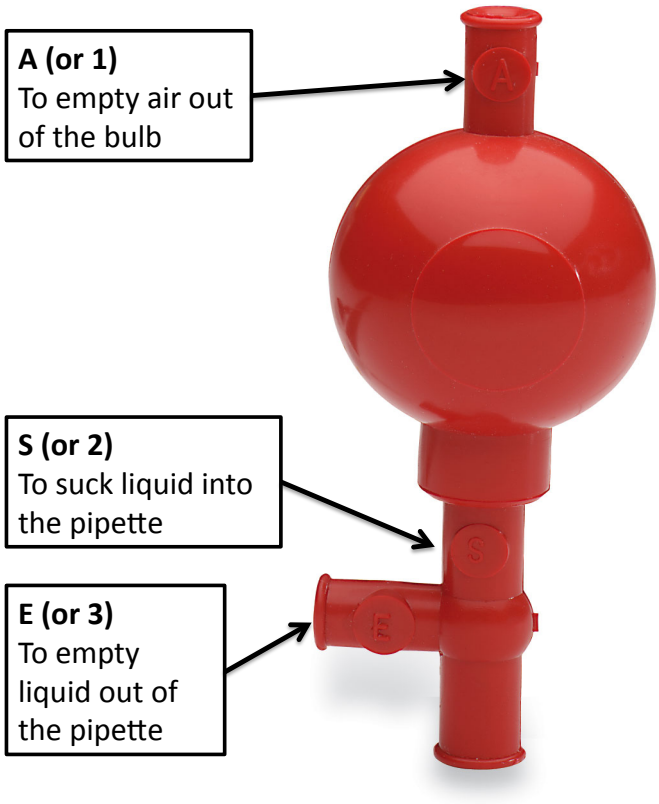
A chemistry laboratory can be a dangerous place and so it is important that the lab safety rules are followed all the time:

- No eating or drinking in the laboratory
- Always wear your lab coat and safety glasses (or overspecs) in the lab.
- Treat all chemicals as potentially dangerous, and avoid any skin contact. If you do get chemicals on your skin, wash them off immediately.
- If you spill any chemicals report the spillage to a Demonstrator at once so that it can be dealt with quickly.
- If you break any 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 next page)

COSHH Assessment for Chemicals

Chemical	**Approx Quantity**	**Hazards**
0.1 M NaOH solution	250 cm^3	Corrosive, irritant
0.1 M HCl	100 cm^3	Corrosive, irritant
Phenolphthalein	Few drops	No hazard
BDH 4.5 indicator	Few drops	No hazard
Analgesic tablets	6 - 8 tablets	No hazard
Caffeine tablet, crushed	1 tablet	No hazard
Dilute solutions (approx 1% by weight) of pure components of analgesic tablets in organic solvent	< 1 cm^3	Irritant, flammable
Absolute ethanol	<100 cm^3	Flammable
Eluent for TLC (EtOAc, EtOH, HOAc)	20 cm^3	Flammable, irritant

USE OF PIPETTE FILLERS



- Take care!!
- Hold the pipette right at the top when fitting the pipette filler
    - Only about 1 cm of the pipette needs to be in the pipette filler
- Press Valve A while squeezing the bulb to expel air form the pipette filler
- To fill the pipette
    - ensure that the tip of the pipette is well below the liquid surface
    - press Valve S
    - take care not to suck liquid into the bulb of the pipette filler
- To empty the pipette press Valve E
- Place your pipette on the pipette rack to prevent it rolling off the bench

ORGANISATION OF THE PRACTICAL EXERCISES

This year's competition consists of 4 parts:

1. Standardization of sodium hydroxide solution

All students will carry out this exercise

2. Determination of the active ingredient in commercially available analgesic tablets

Two students will analyze **one** brand of tablet each

3. Determination of caffeine content of 'Energy Plus' tablets by UV spectrophotometry

One team member will take the main responsibility for this exercise, but all team members will make spectrophotometric measurements and help with calculations. A demonstrator will come and collect your team when a spectrophotometer is free. This may be at any point during the competition.

4. Identification of the contents of an analgesic tablet by thin layer chromatography

You will all work together as a team for the thin layer chromatography (TLC) experiment

	Team member 1	Team member 2	Team member 3
1st task	Exercise 1	Exercise 1	Exercise 3 (Prepare solutions)
2nd task	Exercise 2	Exercise 2	Exercise 1

Reporting Results

Individual results sheets containing tables for burette readings *etc* for each team member and a summary results sheet for each team will be found on the team's lab bench.

The competition will be marked primarily on the content of the team summary results sheet, so please make sure that this is filled in completely and legibly. The individual results sheets may also be used for assessment and will certainly be used in an analysis of the results of the whole competition.

PLEASE FILL IN **ALL** THE RESULTS SHEETS CLEARLY AND LEAVE THEM ON YOUR BENCH FOR COLLECTION AT THE END OF THE PRACTICAL SESSION.

EXERCISE 1 STANDARDIZATION OF SODIUM HYDROXIDE SOLUTION

You will be using sodium hydroxide solution in the volumetric analysis of the unknown carboxylic acids. It is necessary to know the exact concentration of the sodium hydroxide solution and you are going to determine this by titration with standard hydrochloric acid solution.

Method

- Fill the burette with the unknown NaOH solution, and record the initial burette reading.
- Pipette 25.0 cm^3 of standard HCl (approx 0.1 M; you will be told the exact concentration in the lab) into a 250 cm^3 conical flask.
- Add 2-3 drops of BDH 4.5 indicator.
- Titrate to the endpoint with the unknown NaOH solution, and record the burette reading.
- Repeat the titration until you have concordant results.
- Use the results of your titrations to calculate the concentration of the unknown NaOH solution. You will use this value in the volumetric analysis of the unknown carboxylic acids.

Hints on the calculation

1. Calculate the number of moles HCl in 25 cm^3 solution.

2. Calculate the number of moles NaOH which react with 25 cm^3 HCl solution.

3. Calculate the average volume (titre) of $NaOH^-$ which you used.

4. Knowing the number of moles of HCl (from **2**) and the average volume (from **3**) calculate the concentration (in moles dm^{-3}) of the NaOH solution.

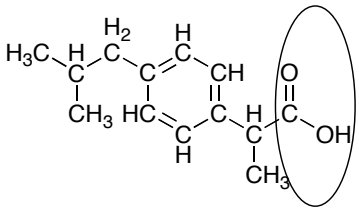
ENTER YOUR RESULTS CLEARLY IN THE SHEETS PROVIDED

EXERCISE 2: DETERMINATION OF THE ACTIVE INGREDIENTS IN ANALGESIC TABLETS

TWO team members will carry out this exercise – one will analyze aspirin tablets; one will analyze ibuprofen tablets

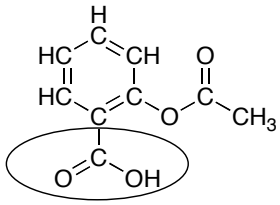
Aim: You are to determine the amount of active ingredient in one of two brands of commercially available analgesic tablets by titration using the NaOH solution that you standardized in Exercise 1.

Both aspirin and ibuprofen are carboxylic acids and so they react with alkalis such as NaOH.



Ibuprofen

Molecular Weight: 206.28



aspirin

Molecular Weight: 180.16

Method: The sample to be analyzed must first be weighed accurately using the 4-figure analytical balance. You are supplied with a sample bottle containing the tablets that you are to analyze, as well as an empty sample bottle to use for weighing. You must weigh each sample accurately using the method known as 'weighing by difference'.

A DEMONSTRATOR WILL BE AVAILABLE TO HELP YOU WITH THIS

- Place the sample bottle *carefully* on the balance pan.
- Close the door of the balance and press the Tare button to obtain a reading of 0.0000g.
- Carefully tip one tablet into a clean 250 cm^3 conical flask (it does not matter if the flask is wet with distilled water)
- Replace the sample bottle on the balance pan. Close the balance door.
- The balance will give a negative reading equivalent to the mass of the tablet. Write down the mass of the tablet (to 4 decimal places) in the table on your results page.

THE BALANCE IS AN EXPENSIVE AND DELICATE INSTRUMENT – PLEASE BE CAREFUL

Method for analysis of aspirin and ibuprofen tablets

- When you have noted the weight of your tablet, add distilled H_2O (approximately 2 cm^3, measured with a measuring cylinder) and swirl the flask to make the tablet disperse. The ibuprofen tablets will take quite some time to disperse. If necessary, use a glass rod to gently crush any remaining lumps, and rinse the glass rod with distilled water before removing it from the flask.
- Add 25 cm^3 ethanol (measured with a measuring cylinder) and swirl the flask to ensure that all of the active ingredient in your tablet has dissolved. The solution will remain slightly cloudy (particularly for the ibuprofen) due to the presence of insoluble filler materials in the tablet.
- Add 2-3 drops of phenolphthalein indicator
- Titrate with the NaOH that you standardized in Exercise 1 until the first permanent pink tinge is observed; the pink colour should persist for at least 30 seconds.
- Repeat the titration with your other tablet.

ENTER YOUR RESULTS CLEARLY ON THE ANSWER SHEET PROVIDED

Hints on the calculation

1. Calculate the number of moles NaOH used in the titration.

2. Remember that 1 mole of either aspirin or ibuprofen reacts with 1 mole of NaOH

3. Calculate the number of moles of aspirin or ibuprofen that was used in the titration

4. Calculate the mass of aspirin or ibuprofen in one tablet (Mr of Ibuprofen = 206.28; Mr of aspirin = 180.16)

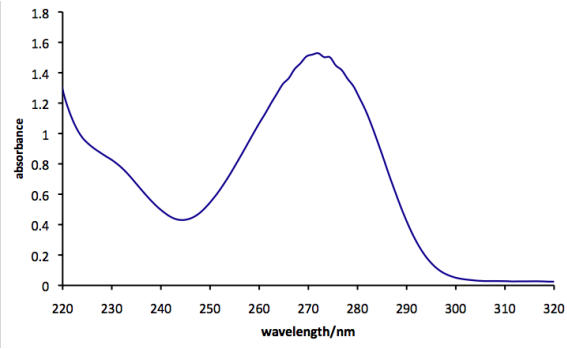
5. Calculate the % by mass of aspirin or ibuprofen in your tablets.

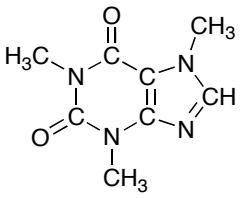
Use your two concordant titrations in the summary results sheet

EXERCISE 3: DETERMINATION OF CAFFEINE IN BOOTS ENERGY-PLUS TABLETS BY UV SPECTROPHOTOMETRY

Theory and background

Caffeine is colourless, but it absorbs radiation in the ulatraviolet (UV) region of the spectrum as shown below. The maximum absorbance is at a wavelength of 273 nm. You are provided with absorbance vaues for a series of standard caffeine solutions with known concentrations, and you will use these to plot a calibration graph. You will then measure the absorbances of solutions of the unknown tablets and use the calibration graph to determine their concentrations.





caffeine

Molecular Weight: 194.19

Method

In order to ease congestion in the lab, a demonstrator will collect you and take you to use the spectrophotometer when it is free.

Accurate weighing of sample - The sample is provided as a powder, made by grinding up the tablets, and must be weighed accurately using the 4-figure analytical balance.

A DEMONSTRATOR WILL HELP YOU WITH THIS

- Place a small filter funnel into the top of your 250 cm^3 graduated flask and take this and the sample of tablet powder to the balance.
- Place the open sample bottle containing the powder onto the balance pan. Close the door of the balance, wait until the reading stabilizes, then press the Tare button to obtain a reading of 0.0000 g.
- Carefully tip the powder into the funnel and replace the sample bottle on the balance pan. Close the balance door.
- The balance will give a negative reading equivalent to the mass of powder transferred. Write down the mass (to 4 decimal places) in the table on your results sheet.

THE BALANCE IS AN EXPENSIVE AND DELICATE INSTRUMENT – PLEASE BE CAREFUL

Preparation of the tablet stock solution –

- Carefully rinse the powder from the filter funnel into the graduated flask.
- Half-fill the flask with distilled water and swirl gently. Caffeine is slow to dissolve and **at least 20 minutes** should be allowed for this.
- Rather than just waiting during this period you should start another exercise, for example the standardization of NaOH.
- Keep swirling the flask in the meantime. **NB** some insoluble filler material will remain after the caffeine has dissolved.
- Make up the solution to 250 cm^3 with distilled H_2O. Stopper the flask and make sure that the solution is well mixed by inverting it at least 10 times.
- The tablet stock solution is now ready to be analyzed.

Preparation of solutions for UV analysis – Three separate samples of the tablet stock solution should be made up as follows: -

- Using a graduated pipette, transfer x cm^3 (x=2, 5 or 8) of tablet stock solution to a 50 cm^3 graduated flask.
- Label each flask 2, 5 or 8 using the labels provided.
- Make up to the mark with distilled H_2O. Stopper the flasks and make sure that the solutions are well mixed by inverting them at least 10 times.

Absorbance measurements – A demonstrator will take you to one of the spectrophotometers where you will measure the absorbance of your solutions at 273 nm.

WRITE DOWN YOUR RESULTS ON THE RESULTS SHEET PROVIDED

See next page for hints on calculations

Calculations

1. Plot a calibration graph using the data on the sheet provided. Plot absorbance on the 'y' (vertical) axis and concentration on the 'x' (horizontal) axis. Remember to use as large a scale as possible.

2. Use your calibration graph to read off the concentration of each of your diluted tablet solutions. Call these concentrations c_2, c_5 and c_8

3. Use the concentrations determined in **2** to work out the concentration, c_{stock}, of your 'stock' tablet solution (i.e. the concentration of the 250 cm^3 tablet solution that you prepared originally). You will get 3 separate values for c_{stock}, and you should use these to work out an average value for c_{stock}.

The example below shows how to work out c_{stock} from c_2. The factor 2/50 arises because you diluted 2 cm^3 of stock solution to 50 cm^3 before measuring the absorbance.

$$c_2 = \frac{2}{50} \times c_{stock}$$

$$\therefore c_{stock} = \frac{50 \times c_2}{2}$$

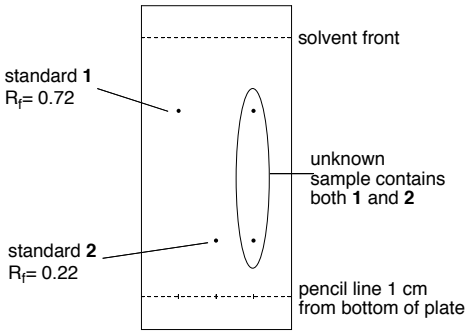
4. From the concentration of the stock tablet solution (this will be in units of mol dm^{-3}), calculate the mass of caffeine in 250 cm^3 of this solution (caffeine = 194.19)

5. Calculate the % by mass of caffeine in the tablet using your value from **4** together with the mass of tablet that you weighed out.

EXERCISE 4: ANALYSIS AN ANALGESIC TABLET BY TLC

Theory:

You are provided with pure solutions of 4 common ingredients of analgesic tablets, and a small sample of an unknown tablet. By measuring the R_f values of all of the standard samples and comparing these with the R_f values for the unknown tablet, you will identify the ingredients of the unknown tablet.



$$R_f = \frac{\text{distance travelled by spot}}{\text{distance travelled by solvent front}}$$

A	B	C	D	X
Aspirin	Caffeine	Paracetamol	Ibuprofen	Unknown tablet

Method

Add to the sample vial X approximately 10 cm^3 of the solvent mixture provided. Replace the top on the vial and shake the mixture to extract the active ingredients from the sample of tablet.

- You are provided with two TLC plates. Draw a pencil line 1 cm from the bottom of the white side of each TLC plate. Carefully draw 3 equally spaced crosses on this line on the first plate, and on the second plate draw 2 equally spaced crosses (take care not to damage the layer of silica on the plate). Carefully label the crosses below the pencil line (A, B, C on the first plate; D and X on the second plate).
- Using a capillary tube, carefully place a small spot of solution A onto the TLC plate on the cross marked 'A'. Repeat this for solutions B, C, D and X. When you have prepared your TLC plates, place the first plate (samples A, B and C) in the screw top jar provided, making sure that the solvent level in the jar is below the pencil line on your TLC plate.
- Leave the TLC plate in the jar until the solvent front is approximately 0.5 cm from the top of the plate, then remove it and mark the solvent front with a pencil line. This will take approximately 8 minutes; make sure that the solvent front doesn't reach the top of the plate.
- Now repeat this with the second TLC plate (samples D and X).
- You will now examine the TLC plate under UV light, and mark with a pencil the spots that you can see. A demonstrator will help you with this.
- Now calculate the R_f value for each of the spots on the plate, and identify the ingredients in the unknown tablet. (The equation is given above)
- By comparing the R_f values of the standards with the R_f values observed for Tablet X you can identify the ingredients of Tablet X

WRITE DOWN YOUR RESULTS ON THE RESULTS SHEET PROVIDED