RSC | Advancing the Chemical Sciences

WITH PLYMOUTH UNIVERSITY

Royal Society of Chemistry
Analytical Division

2013 Schools' Analyst Competition

27 March 2013

Centre for Chemical Sciences
University of Plymouth

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SAFETY INFORMATION: Experiment 1: Assay of Citric Acid B.P.

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a standard procedure you also need to know the potential risks of the chemicals being used in this experiment.

Each group member should read these before beginning the practical. Each member of the team should sign to say they have read and understood this.

Sodium hydroxide

Principal hazards: Irritating to eyes, respiratory system, skin. Chronic exposure may cause allergic reaction in sensitive individuals

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

Potassium hydrogen phthalate

Principal hazards: Irritating to eyes, respiratory system, skin. Chronic exposure may cause allergic reaction in sensitive individuals

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

Citric Acid

Principal hazards: Citric acid can cause serious irritation if it gets into your eyes
Repeated exposure can cause skin irritation or, eventually, allergic reaction, in some susceptible individuals

Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for medical help.

Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin appears damaged, call for medical aid.

If swallowed: Call for immediate medical help.

Disposal: Weak solutions may be poured down the sink and washed away with plenty of water. Larger amounts should be neutralised before disposal

School Name:

Team Names	Signature

THIS SHEET TO BE HANDED IN WITH RESULTS

SAFETY INFORMATION: Experiment 2: Estimation of Paracetamol in Paracetamol Tablets

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a standard procedure you also need to know the potential risks of the chemicals being used in this experiment.

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Ethanoic Acid

Principal hazards: Contact with the eyes can cause serious long-term damage.
The pure material and its solutions are corrosive; concentrated solutions can cause serious burns.

Safe handling: Always wear safety glasses. Do not allow solid or solution to come into contact with your skin.

Paracetamol

Principal hazards: Harmful if swallowed. Skin, eye and respiratory irritant. Human mutagen

Safe handling: Always wear safety glasses. Do not allow the powder to come into contact with your skin.

Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for medical help.

Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin appears damaged, call for medical aid.

If swallowed: Call for immediate medical help.

Disposal: Weak solutions may be poured down the sink and washed away with plenty of water. Larger amounts should be neutralised before disposal

School Name:

Team Names	Signature

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SAFETY INFORMATION: Experiment 3: Analysis of an Unknown Compound Q

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a standard procedure you also need to know the potential risks of the chemicals being used in this experiment.

Each group member should read these before beginning the practical. Each member of the team should sign to say they have read and understood this.

Compound Q

Principal hazards: Not classified as dangerous according to EC Directives.
Not combustible. May evolve toxic fumes in fire.

Safe handling: Take up dry. Forward for disposal. Clean up affected area. Avoid generation of dusts..

Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for medical help.

Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin appears damaged, call for medical aid.

If swallowed: Call for immediate medical help.

Disposal: Weak solutions may be poured down the sink and washed away with plenty of water. Larger amounts should be neutralised before disposal

School Name:

Team Names	Signature

THIS SHEET TO BE HANDED IN WITH RESULTS

Laboratory Experiments

Experiment 1: Assay of Citric Acid B.P.

A. Preparation of standard ~0.1 *mol dm^{-3}* potassium hydrogen phthalate solution

Accurately weigh (analytical balance) approximately 2g of potassium hydrogen phthalate and add it to a 100 cm^3 volumetric flask. Dissolve the solid in distilled water and make up to volume.

B. Standardisation of the ~0.1 *mol dm^{-3}* sodium hydroxide solution

Measure accurately 25 cm^3 of the potassium hydrogen phthalate solution into a 250 cm^3 conical flask. Add a few drops of phenolphthalein as indicator. Fill a 50 cm^3 burette with the ~0.1M NaOH solution provided. Ensure that no air bubbles are trapped below the tap by fully opening the tap and running a little of your solution out again. Take an initial burette reading. Titrate the potassium hydrogen phthalate solution by adding small aliquots of NaOH solution to the flask and swirling it until a pale pink colour is obtained that persists for at least 30 seconds. Take a new burette reading. This is the first rough titration to get an idea of the volume. Repeat the titration accurately a further two times, refilling the burette as necessary. Remember to go slowly near the end, adding no more than 1 drop at a time. Calculate the average volume used from the two accurate titrations and then use your average volume to calculate the exact concentration of the sodium hydroxide solution (see results/calculation sheet).

C. Preparation of an accurate ~0.05 *mol dm^{-3}* citric acid solution

Accurately weigh approximately 2.4g of citric acid. Add to a 250 cm^3 volumetric flask. Dissolve the solid in distilled water and make up to volume.

D. Titration of Citric Acid Solution

Pipette 25 cm^3 of citric acid solution into a conical flask. Add a few drops of *phenolphthalein* indicator to the conical flask. Place ~0.1 mol dm^{-3} NaOH solution into a 50 cm^3 burette and take an initial volume reading. Titrate the citric acid by adding small aliquots of 0.1 mol dm^{-3} NaOH solution to the flask and swirling it until a permanent pink colour is obtained that persists for at least 30 seconds. Note the volume of 0.1 mol dm^{-3}

NaOH solution added. Repeat the titration accurately two more times. Use the mean volume of the two accurate titrations to calculate the exact amount of citric acid present, and from this the purity of the citric acid used (see results/calculation sheet) Does it conform to the specification?

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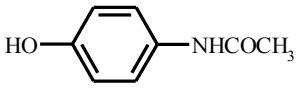
Experiment 2: Estimation of Paracetamol in Paracetamol Tablets

A. Calibration curve for *paracetamol*



You are provided with a standard paracetamol solution that contains approximately 5000 milligrams per cubic decimeter (5000 mg dm^{-3}) in 0.05 M ethanoic acid. From this solution you will need to prepare, accurately, an approximately 500 mg dm^{-3} STOCK solution in the 100 cm^3 flask provided. Using this STOCK solution you must prepare standard solutions of approximately 0, 2.5, 5, 10 and 20 mg dm^{-3} in the 100 cm^3 volumetric flasks provided with 0.05 M ethanoic acid. Label these flasks A, B, C, D and E respectively.

B. Assay of Paracetamol in Paracetamol Tablets B.P.

Press two tablets out of the packaging and weigh them accurately (analytical balance - note exact weight). Powder the two tablets with a pestle and mortar. Weigh accurately (analytical balance) about 140 mg of the powder and add this sample to a 500 cm^3 volumetric flask. About half fill the flask with 0.05 M acetic acid. Shake for 10 minutes then adjust the volume to 500 cm^3 with more 0.05 M acetic acid and mix thoroughly. Filter ~50 cm^3 of the solution through a filter paper into a 100 cm^3 conical flask. Transfer 3 separate 5 cm^3 aliquots (accurately measured – use a bulb pipette) of the filtrate to 100 cm^3 volumetric flasks and adjust the volumes to 100 cm^3 with 0.05 M acetic acid. Label these flasks F, G and H respectively.

Data treatment

Take your calibration and sample solutions to the spectrophotometer and measure the absorbance of each (you will be show how to do this) at 243 nm.

Record your data using the results sheets provided.

Plot a graph of absorbance (y-axis) versus paracetamol concentration (x-axis) using the graph paper provided. This is the calibration curve/line.

Using this graph and the sample absorbance data obtained, deduce the concentration of paracetamol in each of the samples.

Calculate the percentage of paracetamol present in the tablets.

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Experiment 3: Analysis of an Unknown Compound Q

You are provided with a sample of an unknown compound Q. Q contains two cations and an anion. Carry out the tests below and record your observations and conclusions on the results sheet provided.

1. Heat, in a test-tube, a spatula full of the solid Q.

2. Mix a spatula load of Q with a spatula load of soda lime. Heat the mixture strongly in a test-tube for 30 seconds. **Cautiously** smell the vapour produced. Test the vapour with moist red litmus.

Make a solution of Q by dissolving 2 g of Q in 40 cm^3 of water

3. Add 1 cm^3 of silver nitrate solution to a 2 cm depth of solution Q in a test tube.

4. Add 1 cm^3 of silver nitrate solution to a 2 cm depth of dilute HCl in a test tube.

5. Add 1 cm^3 of barium nitrate solution to a 2 cm depth of solution Q in a test tube.

6. Add 1 cm^3 of barium nitrate solution to a 2 cm depth of dilute sulphuric acid in a test tube.

7. Add a few drops of sodium hydroxide to a 2 cm depth of solution Q in a test tube. The colour of the product should indicate one of the cations.

From these observations and deductions you should be able to give a suggestion for the identity of compound Q.