Royal Society of Chemistry
Analytical Division
East Anglia Region

2017 National Schools' Analyst Competition

East Anglia Region Heat
Thursday 20th April, 2017

School of Chemistry
University of East Anglia
Norwich

Laboratory Experiments

Experiment 1: Assay of Citric Acid B.P.

A. Preparation of *0.1 M sodium hydroxide* solution

Weigh out approximately 2g of sodium hydroxide pellets using a top pan balance. Note the weight used (though this figure is not actually required for calculations). Add the pellets to a 500mL volumetric flask and dissolve in about 300mL distilled water. Make up to volume using a Pasteur pipette for the last bit, to ensure you do not overshoot. Mix thoroughly. Keep the solution stoppered when not in use to reduce absorption of carbon dioxide from the air

B. Preparation of standard ~0.1M potassium hydrogen phthalate solution

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

C. Standardisation of the 0.1 M sodium hydroxide solution

Measure accurately 25 mL of the potassium hydrogen phthalate solution into a 100 mL conical flask. Add a few drops of phenolphthalein as indicator. Fill a 50 mL burette with your ~0.1M NaOH solution. 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. Remember to go slowly near the end, adding no more than 1 drop at a time. Take a new burette reading. Repeat the titration a further two times, refilling the burette as necessary. Calculate the average volume used and then use your average volume to calculate the exact concentration of the sodium hydroxide solution (see results/calculation sheet).

D. Preparation of an accurate ~0.05M citric acid solution

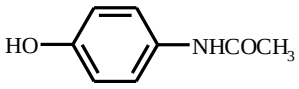
Accurately weigh (to 4 decimal places) approximately 2.4g of citric acid. Add to a 250 mL volumetric flask. Dissolve the solid in distilled water and make up to volume.

E. Titration of Citric Acid Solution

Pipette 25mL of citric acid solution into a conical flask. Add a few drops of *phenolphthalein* indicator to the conical flask. Place ~0.1 M NaOH solution into a 50 mL burette and take an initial volume reading. Titrate the citric acid by adding small aliquots of 0.1 M NaOH solution to the flask and swirling it until a permanent pale pink colour is obtained that persists for at least 30 seconds. Note the volume of 0.1M NaOH solution added. Repeat the titration two more times. Use the mean volume 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 B.P. specification?

Experiment 2: Estimation of Paracetamol in Paracetamol Tablets

A. Calibration curve for *paracetamol*



Weigh accurately (analytical balance) about 60 mg (note exact weight) of paracetamol. Place into a 100 mL volumetric flask and add about 70 mL of 0.05 M acetic acid. Mix until dissolved (an ultrasonic bath may help if you have difficulty getting it to dissolve – please ask a demonstrator) and then adjust the volume to 100 mL with more 0.05 M acetic acid, ensuring you mix the flask thoroughly. Using graduated pipettes, prepare dilutions as follows: take 0.5 mL, 1 mL, 2 mL, 3 mL and 4 mL of the stock paracetamol solution in 5 separate 100 mL volumetric flasks and adjust the volumes to 100 mL with 0.05 M acetic acid.

Measure the absorbance of each solution at 243 nm using a spectrophotometer (the demonstrators will show you how to do this). Take two readings of each dilution. Draw a suitable calibration graph (see results/calculation sheet for instructions on how to do this).

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 140mg of the powder and add this sample to a 500 ml volumetric flask. About half fill the flask with 0.05 M acetic acid. Shake for 10 minutes then adjust the volume to 500 mL with more 0.05 M acetic acid and mix thoroughly. Filter ~50 mL of the solution through a filter paper into a 100 mL conical flask. Transfer 3 separate 5 ml aliquots (accurately measured – use a bulb pipette) of the filtrate to 100 ml volumetric flasks and adjust the volumes to 100 ml with 0.05 M acetic acid. Take your solutions to the spectrophotometer and make two absorbance

readings of each dilution at 243 nm. Use these values to calculate the % w/w paracetamol in the tablets (see results/calculation sheet for instructions on how to do this). Compare the value with the stated tablet content on the packaging. Is it correct?

Experiment 3: Estimation of Caffeine in Filter Coffee by High Performance Liquid Chromatography (HPLC)

A. Making up the Caffeine Standard Solution

In a weighing boat, accurately weigh about 100mg of caffeine (you will need to use the four figure analytical balances for this). Record the exact weight of the caffeine.

Transfer the caffeine into a 250 mL volumetric flask, using distilled water to wash any residues in the weighing boat into the flask. Make up to just below the line and shake the flask until all the caffeine has dissolved. Make up to exact volume using distilled water and a Pasteur pipette. Mix thoroughly again.

Using a 25 mL pipette, transfer 25 mL of the caffeine solution into a 250 mL volumetric flask, make up to volume with distilled water and mix thoroughly. This solution is your CAFFEINE STANDARD SOLUTION.

B. Making up the solutions for HPLC analysis

Using a 5 mL bulb pipette, transfer 5 mL of coffee extract provided into a 50 mL volumetric flask. Make up to exact volume with distilled water (add it carefully to avoid foaming) and mix thoroughly. This is your coffee sample for analysis. Label the flask

Using a 5 mL pipette, transfer 5 mL of coffee extract provided into a second 50 mL volumetric flask Using a 25 mL pipette, transfer 25 mL of caffeine standard solution (see above) into the flask. Make up to exact volume with distilled water (add it carefully) using a Pasteur pipette and mix thoroughly. This is your coffee sample with added standard. Label the flask.

C. HPLC Analysis of the Samples

Take your samples to the instrument lab, where a demonstrator will show you how to inject your samples into the HPLC instrument. Please be patient if you have to wait a few minutes while other samples are run. Once you have the chromatograms and data for your two samples, you can calculate the amount of caffeine in your coffee extract (see results sheet for instructions on how to do this).

(If you have to wait for the instruments to become free, I suggest that one team member should do this, while the others start the next exercise).