Practical 1 - Determination of Quinine in Tonic Water

Introduction

Quinine has a fluorescence and a UV absorbance and so can be quantified using either of these. In the method described here the absorbances of a series of aqueous solutions of quinine of known concentration are measured at 250nm in the UV and used to construct a calibration graph of Absorbance (Y-axis) versus Concentration in mg per litre (X-axis). This calibration curve can then be used to determine the quinine concentration in the drink provided.

Method

Weight out accurately, to four decimal places, approximately 0.1000g of quinine. Transfer this to a 100cm^3 volumetric flask and add, by measuring cylinder, approximately 5cm^3 of dilute sulphuric acid (2M). Make up to the mark with water. Label the flask *Quinine approx 1000mg per litre*.

Pipette 10cm^3 of the 1000mg per litre quinine into a 100cm^3 volumetric flask and make up to the mark with water. Label the flask *Quinine approx 100mg per litre*.

Pipette 10cm^3 of the 100mg per litre quinine into a 100cm^3 volumetric flask and make up to the mark with water. Label the flask *Quinine approx 10mg per litre*.

Calculate the actual concentration using the weight of quinine you weighed out. If you weighed out say, 0.1134g then the accurate concentration is 11.34mg per litre. This is your working stock solution.

Prepare a series of quinine standards by pipetting, using a 5cm^3 or 10 cm^3 graduated pipette, 1.00, 3.00, 5.00 and 7.00cm^3 of the working stock solution into 10cm^3 volumetric flasks, making each up to the mark with water.

Using a 1cm quartz cell, with the spectrometer set to 250nm, measure the absorbances of each of the four standard solutions and the undiluted working stock solution against water as the blank.

For each of the standards calculate the exact concentration of quinine in mg per litre. For example if you weighed out 0.1134g of quinine initially the concentration of the working stock solution would be (0.1134 x 1000 x 10) / (10 x 10) mg per litre = 0.1134 x 100 = 11.34mg per litre. The other concentrations would be as in the table below.
N.B. You must calculate YOUR OWN concentrations.

Volume of Working Stock Solution in cm^3 Diluted to 10cm^3	1	3	5	7	10 (Undiluted working stock)
Quinine Concentration / mg per litre	1.13	3.40	5.67	7.94	11.34

Plot *Absorbance* (Y-axis) against *Quinine Concentration / mg per litre* (X-axis) and draw the best straight line through the points. **DO NOT SIMPLY JOIN UP THE POINTS.**

By suitably diluting the Tonic Water provided (suggested dilution is 10cm^3 to 100cm^3) find its absorbance at 250nm and hence the concentration of the diluted tonic water.

Calculate the concentration of quinine in the original tonic water in mg per litre.

Determination of Quinine in Tonic Water

Raw Data and Results

School Name……

Weight of Weighing Boat + Quinine…….(g)

Weight of Empty Weighing Boat……..…...(g)

Weight of Quinine……....(g)

Volume of Working Stock Solution in cm^3 Diluted to $10cm^3$	1	3	5	7	10 (Undiluted working stock)
Quinine Concentration in mg per litre to two decimal places					
Absorbance					

Absorbance of Diluted Tonic Water……..…….

Draw a graph of Absorbance (Y-axis) against Concentration in mg per litre (X-axis) making sure that all points are clearly marked.

Label the X and Y axes appropriately.

Give your graph a title.

Draw the best straight line **DO NOT SIMPLY CONNECT THE POINTS**.

Read off from the absorbance of the diluted tonic water the concentration of quinine in it in mg/litre to one decimal place.

Conc of Quinine in Diluted Tonic Water…….

State the volume of Tonic you pipetted into the 100ml volumetric flask

Volume of Tonic Pipetted out……..

Calculate the concentration of Quinine in the original tonic water to the nearest whole number in mg per litre using the expression:-

$$\text{Conc of Quinine in Original Tonic} = \frac{\text{Conc in Diluted Tonic}}{\text{Volume of Tonic Pipetted into } 100\text{cm}^3 \text{ Volumetric}} \times 100$$

Conc of Quinine in original tonic water……...

Hand in your graph with this sheet.

Practical 2 - Does the Sample of Vinegar Supplied Comply with the Regulations?

Introduction

Vinegar is essentially a solution of ethanoic acid in water. There is a legal requirement that the ethanoic acid concentration should be a minimum of 4.0% weight/volume (%w/v), that is 4.0g of ethanoic acid in 100cm^3 of solution.

In this investigation a dilute solution of vinegar is titrated against standardised NaOH (0.0200M) and the reaction followed by measuring the pH. From the plot of *pH* (Y-axis) versus *Volume of NaOH Added* (X-axis) the concentration of ethanoic acid in the sample can be calculated.

The relevant equation is

CH_3CO_2H + NaOH = CH_3CO_2Na + H_2O
Ethanoic Acid

Procedure

(a) Standardising the pH Meter

Follow the instructions for the calibration of the pH meter. (These will depend on the meter to be used.)

(b) The pH Titration

Pipette 10cm^3 of the vinegar into a 100cm^3 volumetric flask, make up to the mark with deionised water and mix the contents thoroughly. Label the Flask Solution 1.

Pipette 25cm^3 of Solution 1 into a 100cm^3 volumetric flask, make up to the mark with deionised water and mix the contents thoroughly. Label the Flask Solution 2.

Pipette 10cm^3 of Solution 2 into a 250cm^3 beaker.

Add, by measuring cylinder, approximately 150cm^3 of deionised water and swirl to mix. (Hint:- the exact volume added in this step is not important as it plays no part in the results calculations.)

Put your school name on *the Raw Data Sheet* which has a table with two columns; *Volume NaOH Added in cm^3* and *pH*

Rinse the pH electrode and place it in the beaker. Let the pH reading stabilise and then note it on *the Raw Data Sheet*. This is at zero volume NaOH added.

Fill a burette with 0.0200M NaOH. Run 1cm^3 of NaOH from the burette into the beaker, mix the contents, let the pH reading stabilise, then note it together with the volume of NaOH added.

Continue adding 1cm^3 volumes of NaOH to the beaker, each time mixing and noting the pH, until 20cm^3 have been added.

Plotting the Graph **(See Figure 1)**

Plot on graph paper *pH* (Y-axis) against *Volume of NaOH Added in cm^3* (X-axis).

Label all axes appropriately, give the graph a heading and drawing a smooth curve through the data points.

Draw Lines A, B and C and label each. Line A is the linear portion before the end point extrapolated, line B is the linear portion after the end point extrapolated and line C is the line halfway between lines A and B.

Find and label the end point which is the intersection of the smooth pH curve and line C.

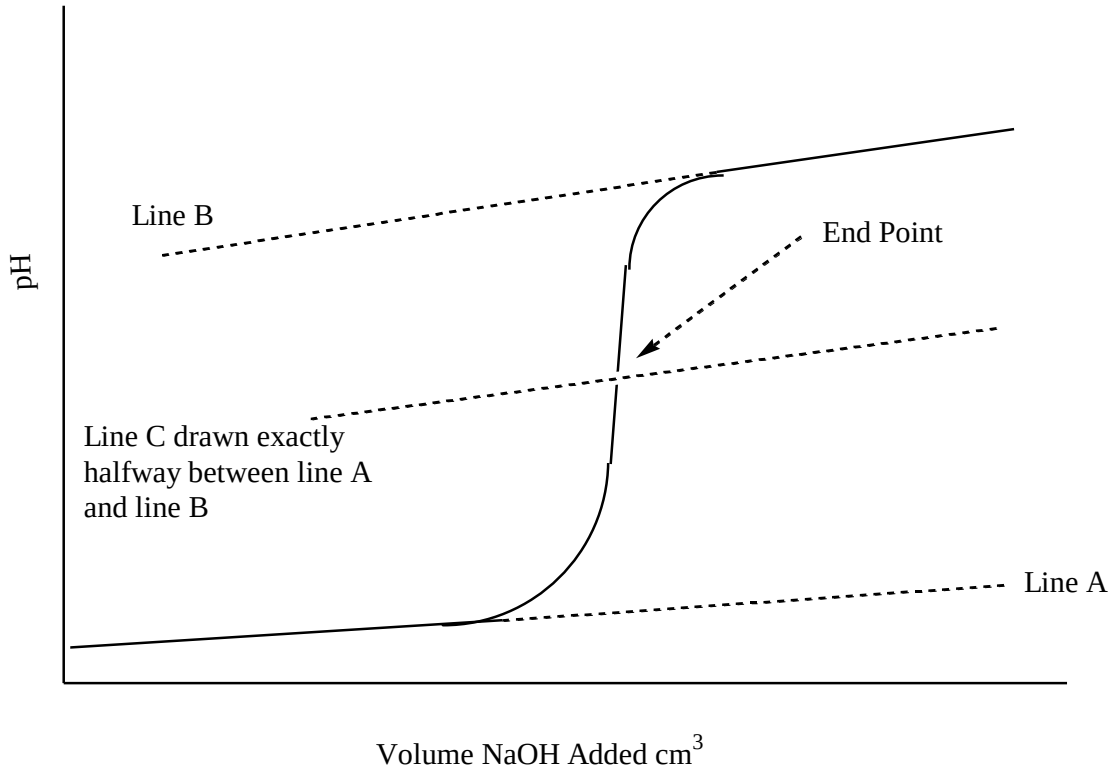

Figure 1 – Plot of pH (Y-axis) versus Volume of NaOH added (X-axis)

Does the Sample of Vinegar Supplied Comply with the Regulations?

Raw Data

School Name……

Volume NaOH Added in cm^3	pH
0	
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	

Does the Sample of Vinegar Supplied Comply with the Regulations?

Results

School Name ……

(1) (a) Submit:-

(i) your table of raw data i.e. *Volume of NaOH Added in cm^3* and *pH*;

(ii) a properly labelled and titled graph. Mark clearly on your graph Lines A, B and C. Mark and indicate the end point clearly.

(b) The volume of NaOH added at the end point in cm^3 (1 decimal place)

is……

(2) Find:-

(a) the molarity of the original vinegar (4 decimal places) which is given by:-

$$\text{Molarity of Orginal Vinegar} = \frac{\text{titre}(\text{cm}^3) \times 0.0200 \times 40}{10} = ...$$

(b) the molecular weight of ethanoic acid, CH_3CO_2H (C = 12, H = 1, O = 16)

molecular weight of ethanoic acid …….

(c) the concentration of ethanoic acid in the vinegar in grams of ethanoic acid per 100cm^3 solution.

concentration in grams per 100cm^3 (2 decimal places)…….

(3) State from your results whether the vinegar supplied complies or does not comply with legislation. Circle the appropriate answer.

The vinegar DOES DOES NOT comply with legislation.

Practical 3 - What Did the Victim Overdose On?

Introduction

It is an unfortunate truth that some people use commonly available over-the-counter pharmaceuticals to attempt to take their own life. It is essential that the Hospital can be advised as quickly as possible what the victim has taken. You will be supplied with a crushed tablet found by the victim's bed and it will be your job to identify what it is rapidly enough to help save the victim's life.

TLC

The technique we will use to determine which drug was used is called Thin Layer Chromatography or or TLC, a simple and versatile technique that can provide useful information about drugs and drug products. Like other chromatographic processes, the separation of analytes in a TLC analysis depends on the relative interaction of the analyte between a solid stationary phase and a liquid mobile phase. The two most common types of TLC modes are Adsorption, sometimes called Forward Phase TLC, and Partition, sometimes called Reverse Phase TLC. Forward phase TLC, the most popular type and the type used in this experiment, acts through an adsorption, or hydrogen bonding interaction between the analyte and a polar stationary phase such as silica gel.

Rf Values

In both forward and reverse phase TLC analyses, the analyte is observed as a spot on the TLC plate. The extent of interaction is quantified with an Rf or retardation factor value. Rf values are calculated as the ratio of the distance travelled by the analyte to that travelled by the solvent. We will try to match the Rf value of the unknown sample, the tablet, to that of a series of known standards to identify which tablet the victim took. Implicit in this is that we have a limited range of possible tablets to test the unknown against, and that all of these possibilities have different Rf values.

Procedure

Preparation of Mixed Solvents

Extracting Solvent:- 10cm^3 of 60-80 pet ether and 10cm^3 of methylated spirits.
Developing Solvent:- 10cm^3 ethyl acetate, 2cm^3 68-80 pet ether, 0.2cm^3 glacial acetic acid

Preparation of Standards

There are three standards; aspirin, paracetamol and ibuprofen. For each of the three standards weigh out about 50mg into a sample tube, add 3cm^3 of extraction solvent, and shake for three minutes. Label the sample tubes.

Preparation of Unknown Tablet

You receive the crushed tablet white powder ready for extraction in a sample tube. Add about 5 cm^3 of the extraction solvent and shake gently for three minutes. Allow the sample tube to stand for five minutes to allow the powder to settle. Pack a little glass wool into a Pasteur pipette and mount it on a clamp stand with a clean empty sample bottle directly below the pipette. Filter the crushed tablet solution through the cotton wool plug. This solution is too concentrated to be run directly. Dilute it by a factor of 3 by adding 5 drops of the tablet solution to 15 drops of extracting solvent.

Loading the Plates

Take a careful note of how the demonstrator spots the sample plates. (See Diagram of TLC Plate). Load four spots, one each of the three standards and one of the unknown tablet solution.

Helpful Hints:

- Do not spot too heavily. The spots will run and give poor results.
- Make sure your spotting capillaries and developing jar are free from contaminants.
- Use a new capillary for each standard and for the sample.
- Only label your TLC plates with pencil, as ink is organic and will run along with your spots. Do not touch the absorbent side of the plate. The oils from your fingers will also give spots and confuse your results.
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Visualising the Plate

You cannot see the spots in normal light. Use Ultra Violet (UV) light to visualise the spots on the plate. You must take care and DO NOT LOOK AT THE UV LIGHT DIRECTLY. Ask the instructor to check your plate before you run it.

Developing the Plate

Add 10 cm^3 developing solvent to the TLC development jar. Carefully lower the TLC plate into the jar. Screw on the lid. The solvent will start to run up the TLC plate**. You must not move the jar while the solvent is running!** When the solvent has run to within 1cm of the top of the plate, unscrew the lid from the TLC development jar and carefully remove the plate. Mark with a pencil the position of the solvent front. Let the plate dry.

Visualising the Developed Plate

View your plate under UV light. Outline all the spots with a pencil. Mark the centre of each spot with a dot. Measure the distance from the starting line to the solvent front. This is the *solvent front distance,* $Solv_{dist}$

For each spot :- measure the distance from the starting line to the dot in the middle of the spot. This is the *spot distance,* $Spot_{dist}$.
Calculate the R_f value as $Spot_{dist}$ / $Solv_{dist}$

<u>Conclusions</u>

By comparing the R_f values from the tablet and the standards determine which type of tablet the victim has taken

Diagram of TLC Plate