ADVANCED PRACTICAL CHEMISTRY

Edited by

Alec Thompson

Lambros Atteshlis

Advanced Practical Chemistry

Edited by

Alec Thompson MA
Advisory Teacher, ILEA

Lambros Atteshlis BSc
Formerly Advisory Teacher
and member of the Science
Support Team, ILEA

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I. Thompson, A. II. Atteshlis, L.
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PREFACE

The experiments in this book are those developed as part of the Independent Learning Project for Advanced Chemistry (ILPAC) produced by an Inner London Education Authority team. As with all the ILPAC materials, these were tested in schools and colleges inside and outside London and cover the requirements of all the major Examination Boards.

An important feature of the complete ILPAC scheme is that it enables students to work more effectively on their own and at their own pace. This is reflected in the way in which the procedures for the experiments are written: the style is personal and direct, and the instructions are far more detailed and precise than is usually the case in practical books.

However, the phrase 'independent learning' must not be interpreted too literally. It is not intended that students should be left unsupervised while they do practical work: rather, it is envisaged that a single teacher will be better able to supervise a number of different experiments going on at the same time without having to spell out detailed instructions to everyone.

An important feature of this book is the provision of specimen results, with detailed calculations where appropriate, and answers to the 'consolidation' questions which follow each experiment. These are available for use by teachers in the Advanced Practical Chemistry Resource Pack.

LONDON 1985

ACKNOWLEDGEMENTS

Our thanks are due to the University of London Entrance and Schools Examination Council for permission to use experiments which appeared in practical examinations:

Experiment 38 (1980), Experiment 60 (1979), Experiment 61 (1974), Experiment 67 (1973 & 1980), Experiment 75 (1980), Experiment 80 (1977), Experiment 88 (1978 & 1979), Experiment 95 (1978 & 1982)

Experiment 44 is based, with the kind permission of Longman Group Ltd, on an experiment which appeared in 'An Experimental Introduction to Reaction Kinetics' by M.A. Atherton & J.K. Lawrence (0582 32145 X)

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INTERNATIONAL HAZARD SYMBOLS


Harmful


Flammable


Corrosive


Toxic


Explosive


Oxidising


Radioactive

CONTENTS

Experiments in inorganic chemistry

Experiments in organic chemistry

INTRODUCTION

The original ILPAC Units, from which these experiments are taken, are organised into four Blocks (Starter, Physical, Inorganic and Organic) but, since the Starter Block Units are concerned mainly with introductory physical chemistry, the experiments are here arranged in three sections, as follows:

EXPERIMENTS 1-49	Physical Chemistry	(115 pages)
EXPERIMENTS 50-80	Inorganic Chemistry	(80 pages)
EXPERIMENTS 81-99	Organic chemistry	(58 pages)

The numbers of experiments in each section is not an indication of relative importance or time alllocation as experiments vary considerably in their length and complexity.

Many of the experiments may be used either for Practical Assessments or as preparation for Practical Examinations. In particular, Experiments 10, 28, 35 and 43 are suitable for assessment of planning skills, since we do not give the usual requirements lists or detailed instructions, while Experiments 60, 61, 67, 75, 80, 88 and 95 are especially useful in preparing for practical examinations which include the investigation of unknown substances.

The order in which the experiments are listed is almost identical to that in the ILPAC Units, and could provide a logical teaching sequence within each section. However, it is envisaged that the material in Experiments 1-16 might be covered first (they come from the Starter Block Units). Thereafter, experiments in the three sections can be "dovetailed".

Advanced Practical Chemistry Resource Pack

This pack of loose-leaf sheets is available as support material. It contains the following items for each experiment.

TECHNICIANS' SHEET	-	a detailed requirements lists, with notes on apparatus and instructions for making up solutions, etc.
TEACHERS' NOTES	-	brief comments, including likely sources of error and difficulty, possible alternatives, and the accuracy to be expected.
*SPECIMEN RESULTS	-	including step-by-step calculations, where appropriate.
*ANSWERS	-	suggested answers to the 'consolidation' questions.
*BLANK TABLES	-	for students to fill in (these are provided only for the observation and deduction experiments and for a few others where the Results Tables are large and/or complex).

*For these pages only, multiple copies may be produced for use within the establishment for which the pack was purchased.

EXPERIMENT 1

Determining the Avogadro constant

Aim

The purpose of this experiment is to estimate the value of the Avogadro constant and to compare this estimate with the accepted value.

Introduction

When a solution of oleic acid, $C_{17}H_{33}CO_2H$, in pentane is dropped on to water, the pentane evaporates leaving behind a layer of oleic acid one molecule thick. For this reason, this experiment has been called 'The Monomolecular Layer Experiment'.

You use a loop of hair or thread to contain the oleic acid and to give a measure of the surface area. By making certain assumptions about the shape of the molecule and its alignment on the surface, you can get a reasonably accurate value for the Avogadro constant.

The experiment has two parts. In the first, you calibrate the pipette. This gives the volume of one drop of solution. In the second part you determine how many drops of solution are required to just fill the loop with a layer of oleic acid molecules. Then we lead you, step by step, through the calculation.

Requirements

measuring cylinder, 10 cm^3
teat pipette and adaptor (for small drops)
trough
human hair or cotton thread, 40 - 50 cm
scissors
petroleum jelly or Vaseline
oleic acid solution in pentane (0.05 cm^3 of oleic acid per dm^3)-------

Hazard Warning

Pentane is highly flammable.

Therefore you MUST:

KEEP THE STOPPER ON THE BOTTLE WHEN NOT IN USE:

KEEP THE LIQUID AWAY FROM FLAMES.

Procedure

1. Fill the teat pipette with oleic acid solution and deliver it drop by drop into the 10 cm^3 measuring cylinder. Count the number of drops which must be delivered from the pipette to reach the 1 cm^3 mark. Enter your value in a copy of Results Table 1.

Results Table 1

Number of drops to deliver 1 cm^3 of solution	Number of drops delivered to make monomolecular layer	Diameter of monomolecular layer/cm

2. Tie the hair or cotton thread in a loop. Use a reef-knot (Fig. 1), rather than an overhand knot, so that the loop will make a flat circle. Cut the ends as close to the knot as possible. Hair is preferred because it does not need greasing but if you are using thread, thoroughly but lightly grease it with petroleum jelly. It is most important that no part of the thread escapes greasing. Run the knotted thread through your fingers several times before wiping off the excess.

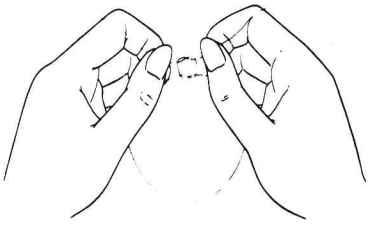


Fig. 1.

3. Fill the trough with water and float the loop on it, making sure that the entire circumference is in contact with the surface. Look very carefully for 'bridges' or submerged loops and move them into the surface with a clean glass rod or a pencil point.

4. Using the same pipette, add the oleic acid solution dropwise to the middle of the loop until it is filled. At first you will probably see the loop expand to a circle and then retract again.
Before the loop is filled, it 'gives' when you push it gently from the outside with a pencil. (Fig. 2)
When the loop is filled, it will slide across the surface, only denting very slightly when pushed gently with the pencil. (Fig. 3)

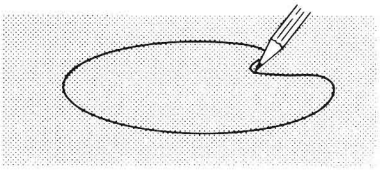


Fig.2.

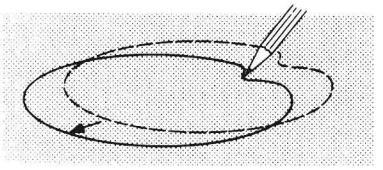


Fig.3.

Count the number of drops required to fill the loop and record this in a copy of Results Table 1.

5. Measure the diameter of the loop and complete Results Table 1.

6. If you have time, repeat the whole procedure. However, you must use a fresh hair or thread, and wash out the trough thoroughly to obtain a clean surface.

Calculation

1. Calculate the volume of 1 drop delivered from the teat pipette using the value in column one of Results Table 1.

   Volume of 1 drop = _____ cm^3

2. Calculate the volume of oleic acid in 1 drop of solution delivered from the teat pipette.

   Remember that 1000 cm^3 of this solution contains 0.05 cm^3 of oleic acid.

   Volume of oleic acid in 1 drop = _____ cm^3

3. Calculate the volume of oleic acid delivered to make the monomolecular layer; i.e. the volume of oleic acid in 1 drop x the number of drops required.

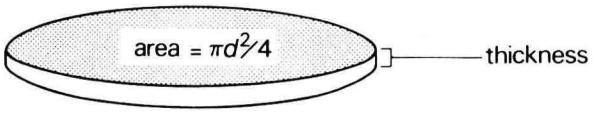
   Volume of oleic acid in monolayer = _____ cm^3

4. Calculate the surface area of the oleic acid layer.

   Area = $\pi d^2/4$ = _____ cm^2

5. You know the volume of oleic acid (from 3) and the surface area it covers (from 4). It is a simple matter to calculate the thickness of the layer because volume = area x thickness.

Fig.4.



   Thickness = _____ cm

6. Calculate the volume of one molecule of oleic acid by assuming it is a cube, with sides equal to the thickness of the layer.

   Volume of one molecule = _____ cm^3

7. Calculate the molar volume of oleic acid given that its density is 0.890 g cm^{-3} and its molar mass is 282 g mol^{-1}.

   Molar volume of oleic acid = _____ $cm^3\ mol^{-1}$

8. Divide the molar volume by the volume of one molecule to determine the Avogadro constant.

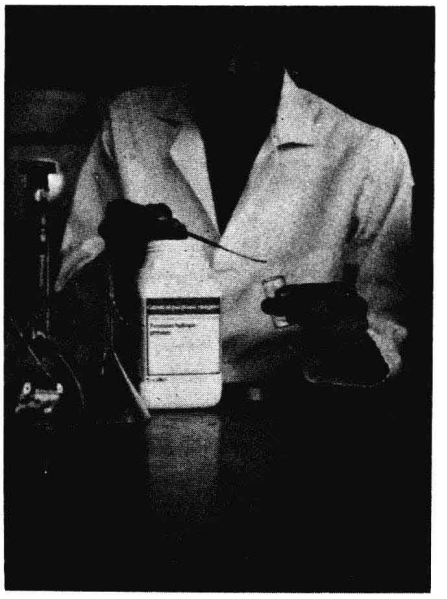
   L = _____ mol^{-1}

Questions

1. Suggest some sources of error in this experiment which account for the discrepancy between the value of L you obtained and the accepted value of $L = 6.02 \times 10^{23}\ mol^{-1}$.
2. Which of the values you used in your calculations is subject to the greatest error?
3. Pentane is not the only liquid that can be used in this experiment. Suggest four properties which a suitable substitute must have.

EXPERIMENT 2

Preparing a standard solution



Aim

The purpose of this experiment is to prepare a standard solution of potassium hydrogenphthalate.

Introduction

Potassium hydrogenphthalate, $C_8H_5O_4K$, is a primary standard because it meets certain requirements.

1. It must be available in a highly pure state.
2. It must be stable in air.
3. It must be easily soluble in water.
4. It should have a high molar mass.
5. In solution, when used in volumetric analysis, it must undergo complete and rapid reaction.

You weigh accurately a sample of potassium hydrogenphthalate and use it to make a solution of concentration close to 0.10 mol dm^{-3}. In Experiment 3 you use this solution to determine the concentration of a solution of sodium hydroxide.

Requirements

safety spectacles
weighing bottle
spatula
potassium hydrogenphthalate, $C_8H_5O_4K$
access to a balance capable of weighing to within 0.01 g
beaker, 250 cm^3
wash bottle of distilled water
stirring rod with rubber end
volumetric flask, 250 cm^3, with label
filter funnel
dropping pipette

Procedure

1. Transfer between 4.8 and 5.4 g of potassium hydrogenphthalate into a weighing bottle and weigh it to the nearest 0.01 g.
2. Put about 50 cm^3 of water into a 250 cm^3 beaker. Carefully transfer the bulk of the potassium hydrogenphthalate from the weighing bottle into the beaker.
3. Reweigh the bottle with any remaining potassium hydrogenphthalate to the nearest 0.01 g.
4. Stir to dissolve the solid, adding more water if necessary.

5. Transfer the solution to the volumetric flask through the filter funnel. Rinse the beaker well, making sure all liquid goes into the volumetric flask. (Some workers transfer the solid directly into the flask through a filter funnel, but you should only do this if you are sure the solid will dissolve easily and if your funnel has a wide enough stem to prevent blockage.)

6. Add distilled water until the level is within about 1 cm of the mark on the neck of the flask. Insert the stopper and shake to mix the contents.

7. Using the dropping pipette, add enough water to bring the bottom of the meniscus to the mark, as in Fig. 5. Insert the stopper and shake thoroughly ten times to ensure complete mixing. Simply inverting the flask once or twice does not mix the contents properly and is a very common fault.

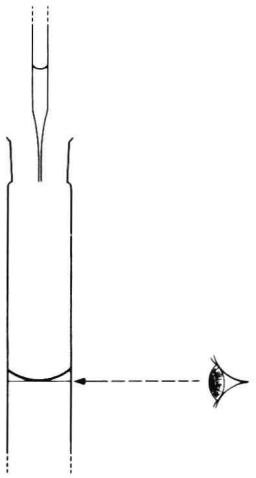


Fig.5.

8. Label the flask with the contents, your name and the date. Leave a space for the concentration to be filled in after you have calculated it. Set aside the flask for Experiment 3.

Results and Calculations

Using your data, you can fill in a copy of Results Table 2.

Results Table 2

Molar mass of potassium hydrogenphthalate, M	g mol^{-1}
Mass of bottle and contents before transfer, m_1	g
Mass of bottle and contents after transfer, m_2	g
Mass of potassium hydrogenphthalate, $m = (m_1 - m_2)$	g
Amount of potassium hydrogenphthalate, $n = m/M$	mol
Volume of solution, V	dm^3
Concentration of potassium hydrogenphthalate, $c = n/V$	mol dm^{-3}

Questions

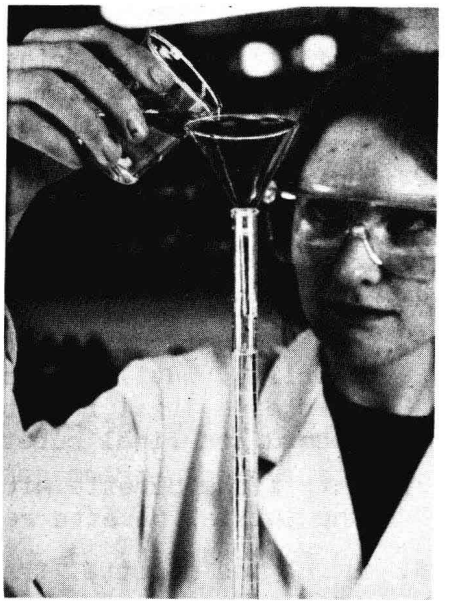
1. What effect would each of the errors described below have on the concentration of potassium hydrogenphthalate?

   (a) Some of the solid potassium hydrogenphthalate was spilled in making the transfer.

   (b) Not enough water was added to bring the volume up to the mark.

EXPERIMENT 3

An acid-base titration



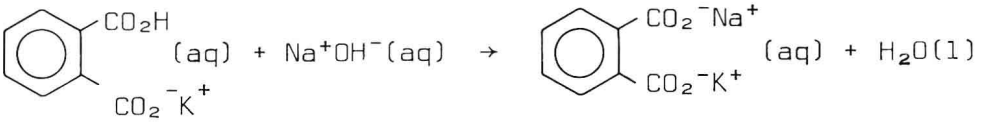
Aim

The purpose of this experiment is to determine the concentration of a solution of sodium hydroxide by titration against a standard solution of potassium hydrogenphthalate.

Introduction

In Experiment 2 you made a standard solution of potassium hydrogenphthalate, a primary standard. The substance has the formula $C_8H_5O_4K$, but because it behaves as a monoprotic (monobasic) acid in producing one mole of hydrogen ions per mole of compound, we can simplify the formula to HA. This simple formula is often used to represent an acid with a complicated structure.

Sodium hydroxide reacts with potassium hydrogenphthalate according to the equation:



or $HA(aq) + Na^+OH^-(aq) \rightarrow Na^+A^-(aq) + H_2O(l)$

To show you when the reaction is complete - the stoichiometric point or equivalence point - you use an indicator called phenolphthalein, which is colourless in acid and pink in alkaline solution. The point at which the addition of one drop (or even less) of alkali changes the solution from colourless to just faintly pink is called the end-point and, in this case, shows that the reaction is just complete.

Requirements

safety spectacles
filter funnel, small
burette, 50 cm^3, and stand
2 beakers, 100 cm^3
sodium hydroxide solution, approx. 0.1 M NaOH
pipette, 25 cm^3
pipette filler
standard potassium hydrogenphthalate solution (prepared in Experiment 2)
4 conical flasks, 250 cm^3
phenolphthalein indicator solution
white tile
wash-bottle of distilled water

Procedure

1. Using the funnel, rinse the burette with the sodium hydroxide solution and fill it with the same solution. Do not forget to rinse and fill the tip. Record the initial burette reading in the 'Trial' column of Results Table 3.
2. Using a pipette filler, rinse the pipette with some of the potassium hydrogenphthalate solution and carefully transfer 25.0 cm^3 of the solution to a clean 250 cm^3 conical flask.
3. Add 2-3 drops of the phenolphthalein indicator solution.
4. Run sodium hydroxide solution from the burette into the flask, with swirling, until the solution just turns pink. This first flask may be used as a trial run, because you will probably overshoot the end-point. Record the final burette reading.
5. Refill the burette with the sodium hydroxide solution, and again record the initial burette reading to the nearest 0.05 cm^3 (one drop).
6. Using the pipette, transfer 25.0 cm^3 of the potassium hydrogenphthalate solution to another clean conical flask. Add 2-3 drops of the phenolphthalein indicator solution.
7. Carefully titrate this solution to the end-point, adding the alkali drop by drop when you think the colour is about to change.
8. Repeat steps 5, 6 and 7 at least twice more.
9. Empty the burette and wash it carefully immediately after the titration, especially if it has a ground glass tap.

Accuracy

You should record burette readings to the nearest 0.05 cm^3 (approximately one drop). Consecutive titrations should agree to within 0.10 cm^3 and, strictly, you should repeat the titration until this is achieved. However, you may have neither the time nor the materials to do this. With practice, your technique will improve so that it is not necessary to do more than four titrations. Calculate the mean of the two (or preferably three) closest consecutive readings and quote this also to the nearest 0.05 cm^3. Note that this does not introduce a fourth significant figure; it merely makes the third figure more reliable.

Results Table 3

Pipette solution				mol dm^{-3}		cm^3
Burette solution				mol dm^{-3}		
Indicator						
		Trial	1	2	3	(4)
Burette readings	Final					
	Initial					
Volume used (titre)/cm^3						
Mean titre/cm^3						

Calculation

1. Calculate the concentration of the sodium hydroxide solution.

Questions

1. What effect would each of the errors described below have on the calculated value of the concentration of sodium hydroxide?

   (a) The burette is not rinsed with the sodium hydroxide solution.

   (b) The pipette is not rinsed with the potassium hydrogenphthalate solution.

   (c) The tip of the burette is not filled before titration begins.

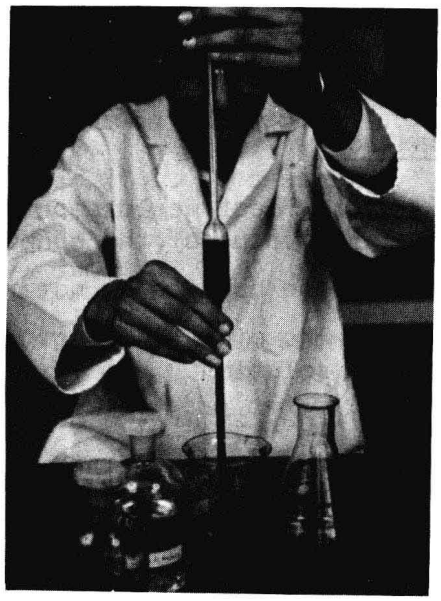
   (d) The conical flask contains some distilled water before the addition of potassium hydrogenphthalate.

2. In using phenolphthalein as an indicator, we prefer to titrate from a colourless to pink solution rather than from pink to colourless. Suggest a reason for this.

3. Why is it advisable to remove sodium hydroxide from the burette as soon as possible after the titration?

EXPERIMENT 4

A redox titration



Aim

The purpose of this experiment is to balance the equation for the reaction between sodium thiosulphate and iodine.

$$a\ Na_2S_2O_3(aq) + b\ I_2(aq) \rightarrow \text{Products}$$

Introduction

You are to determine the ratio of a to b and so determine the stoichiometry of the reaction. You do this by taking a known amount of iodine and titrating it with standard sodium thiosulphate.

The indicator you use in this titration is starch solution, which is deep blue in the presence of iodine; it is added near the end of the titration when the solution is straw-coloured. If you add starch too soon, you may get a blue-black precipitate which does not dissolve again easily even though there is an excess of thiosulphate. The end-point in this titration is the point at which the addition of one drop of sodium thiosulphate causes the disappearance of the deep blue colour.

Requirements

safety spectacles
filter funnel
burette, 50 cm^3, and stand
2 beakers, 100 cm^3
sodium thiosulphate solution, standardized
pipette, 10 cm^3
pipette filler
iodine solution, standardized
4 conical flasks, 250 cm^3
starch indicator solution
white tile
wash-bottle of distilled water

Procedure

1. Using the funnel, rinse the burette and tip with the sodium thiosulphate solution. Fill it with the same solution. Don't forget to fill the tip. Record the initial burette reading in Results Table 4.
2. Rinse the pipette with some of the iodine solution and carefully transfer 10.0 cm^3 of the solution to one of the conical flasks.
3. Titrate this solution until the colour of the iodine has <u>almost</u> gone (as indicated by a pale straw colour).