

# ADVANCED PRACTICAL CHEMISTRY

Edited by

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# Advanced Practical Chemistry

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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

## Introduction

1

## Experiments in physical chemistry

1. Determining the Avogadro constant	2
2. Preparing a standard solution	5
3. An acid-base titration	7
4. A redox titration	10
5. A precipitation titration	12
6. A titration exercise	15
7. Estimating the ionization energy of a noble gas	17
8. Using a hand spectroscope to observe the emission spectra of some s-block elements	20
9. Determining an enthalpy change of reaction	22
10. Determining an enthalpy change of solution	24
11. Using Hess's law	25
12. Another application of Hess's law	27
13. Determining heats of combustion	31
14. A thermometric titration	35
15. Making models of two metallic structures	37
16. Recognising ionic, covalent and metallic structures	42
17. Determining the molar mass of a gas	43
18. Determining the molar mass of a volatile liquid	45
19. Determining the molar mass of a gas by diffusion	48
20. The effect of concentration changes on equilibria	51
21. Determining an equilibrium constant	53
22. Determining a solubility product	57
23. Illustrating the common ion effect	59
24. Distribution equilibrium	60
25. The pH of a weak acid at various concentrations	62
26. The pH of different acids at the same concentration	64
27. The action of a buffer solution	65
28. Preparation of buffers: testing their buffering capacity and the effect of dilution	67
29. Determining the pH range of some acid-base indicators	68
30. Determining the ionization constant for an indicator	70
31. Determining the dissociation constant of a weak acid using an indicator	73
32. Obtaining pH curves for acid-alkali titrations	75
33. The variation of boiling-point with composition for a liquid mixture	78
34. Measuring temperature changes on forming solutions	81
35. Determining the approximate strength of a hydrogen bond	83
36. The effect of hydrogen bonding on liquid flow	84
37. Testing liquids for polarity	85
38. Determining relative molecular mass by a freezing-point method	87
39. Determining enthalpy changes and volume changes of solution	89
40. Investigating the hydrolysis of benzenediazonium chloride	92
41. The kinetics of the reaction between iodine and propanone in aqueous solution	96
42. Determining the activation energy of a reaction	100
43. Determining the activation energy of a catalysed reaction	102
44. A bromine 'clock' reaction	103
45. Some simple redox reactions	107
46. Measuring the potential difference generated by some simple electro-chemical cells	108
47. Testing predictions about redox reactions	110
48. Variation of cell potential with concentration	113
49. Measuring the solubility products of some silver salts	115

## Experiments in inorganic chemistry

50.	Reaction between sodium peroxide and water	117
51.	Heating the nitrates and carbonates of the <i>s</i> -block elements	119
52.	The solubility of some salts of Group II elements	123
53.	The solubility of the halogens in inorganic solvents	125
54.	The action of dilute alkali on the halogens	127
55.	Halogen-halide reactions in aqueous solution	129
56.	Reactions of solid halides	131
57.	Reactions of halides in solution	134
58.	Reactions of the halates	136
59.	Balancing a redox reaction	138
60.	Observation and deduction exercise	140
61.	Observation and deduction exercise	142
62.	Investigating the properties of Period 3 chlorides	144
63.	Preparing anhydrous aluminium chloride	148
64.	Investigating the properties of Period 3 oxides	151
65.	The reactions of tin and lead and their aqueous ions	154
66.	The preparations and reactions of tin(IV) oxide and lead(IV) oxide	158
67.	Observation and deduction exercise	162
68.	Illustrating the oxidation states of vanadium	165
69.	Illustrating the oxidation states of manganese	168
70.	Relative stabilities of some complex ions	171
71.	Determining the formula of a complex ion	173
72.	Some redox chemistry of copper	176
73.	Investigating the use of cobalt(II) ions as a catalyst	178
74.	Catalysing the reaction between iodide ions and peroxodisulphate	179
75.	Observation and deduction exercise	182
76.	Reactions of aluminium	184
77.	Anodizing aluminium	187
78.	Reactions of oxoacids of nitrogen and their salts	189
79.	Investigating some reactions of the oxo-salts of sulphur	192
80.	Observation and deduction exercise	195

## Experiments in organic chemistry

81.	Chemical properties of alkanes	197
82.	Chemical properties of alkenes	201
83.	Hydrolysing organic halogen compounds	202
84.	Preparing a halogeno-alkane	204
85.	Chemical properties of ethanol	207
86.	Chemical properties of phenol	214
87.	Reactions of amines	217
88.	Observation and deduction exercise	221
89.	Reactions of aldehydes and ketones	224
90.	Identifying an unknown carbonyl compound	227
91.	Chemical properties of carboxylic acids	231
92.	Identifying salts of carboxylic acids	234
93.	Chemical properties of ethanoyl chloride	236
94.	Preparing an ester	239
95.	Observation and deduction exercise	242
96.	The glycine/copper(II) complex	245
97.	The biuret test for proteins	246
98.	Paper chromatography	247
99.	Reactions of carbohydrates	250

## 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 allocation 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.

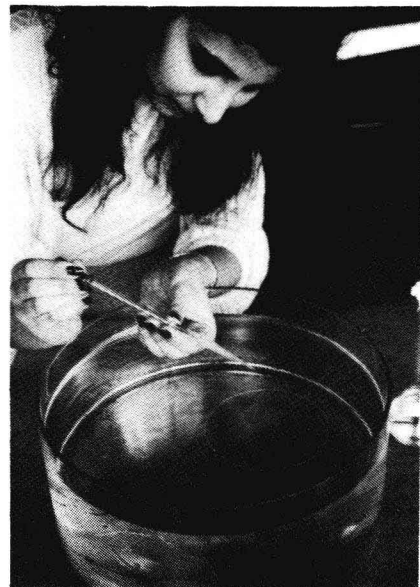
- TECHNICIANS' SHEET - a detailed requirements lists, with notes on apparatus and instructions for making up solutions, etc.
- TEACHERS' NOTES - brief comments, including likely sources of error and difficulty, possible alternatives, and the accuracy to be expected.
- \*SPECIMEN RESULTS - including step-by-step 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<sup>3</sup>  
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<sup>3</sup> of oleic acid per dm<sup>3</sup>)-----



### 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<sup>3</sup> measuring cylinder. Count the number of drops which must be delivered from the pipette to reach the 1 cm<sup>3</sup> mark. Enter your value in a copy of Results Table 1.

Results Table 1

Number of drops to deliver 1 cm <sup>3</sup> 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.

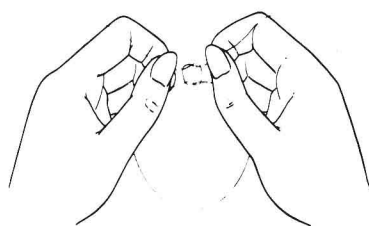


Fig. 1.

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)

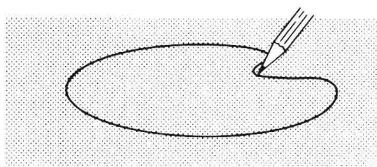


Fig.2.

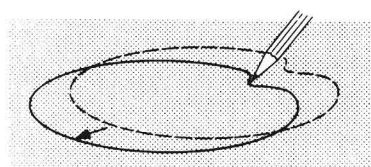


Fig.3.

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.

- 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

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

$$\text{Volume of 1 drop} = \text{_____ cm}^3$$

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

Remember that  $1000 \text{ cm}^3$  of this solution contains  $0.05 \text{ cm}^3$  of oleic acid.

$$\text{Volume of oleic acid in 1 drop} = \text{_____ cm}^3$$

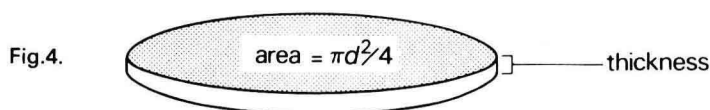
- Calculate the volume of oleic acid delivered to make the monomolecular layer; i.e. the volume of oleic acid in 1 drop x the number of drops required.

$$\text{Volume of oleic acid in monolayer} = \text{_____ cm}^3$$

- Calculate the surface area of the oleic acid layer.

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

- 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.



$$\text{Thickness} = \text{_____ cm}$$

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

$$\text{Volume of one molecule} = \text{_____ cm}^3$$

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

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

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

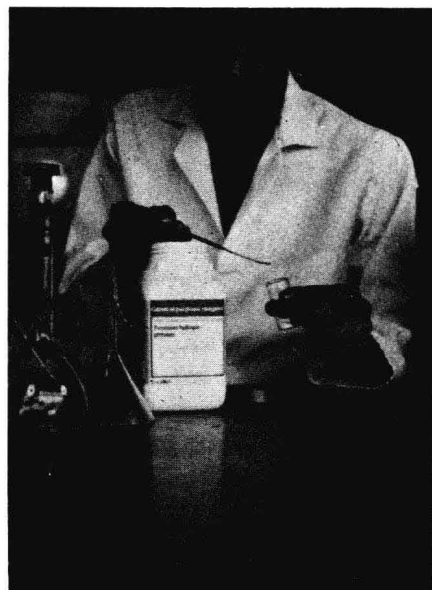
$$L = \text{_____ mol}^{-1}$$

### Questions

- 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  $L = 6.02 \times 10^{23} \text{ mol}^{-1}$ .
- Which of the values you used in your calculations is subject to the greatest error?
- 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  $0.10 \text{ mol dm}^{-3}$ . 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  $0.01 \text{ g}$   
beaker,  $250 \text{ cm}^3$   
wash bottle of distilled water  
stirring rod with rubber end  
volumetric flask,  $250 \text{ cm}^3$ , with label  
filter funnel  
dropping pipette

#### Procedure

1. Transfer between  $4.8$  and  $5.4 \text{ g}$  of potassium hydrogenphthalate into a weighing bottle and weigh it to the nearest  $0.01 \text{ g}$ .
2. Put about  $50 \text{ cm}^3$  of water into a  $250 \text{ cm}^3$  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  $0.01 \text{ g}$ .
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.

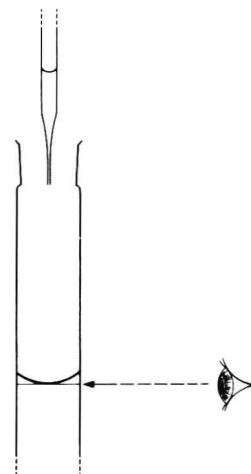


Fig.5.

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, $M$	$\text{g mol}^{-1}$
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, $V$	$\text{dm}^3$
Concentration of potassium hydrogenphthalate, $c = n/V$	$\text{mol dm}^{-3}$

### 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.

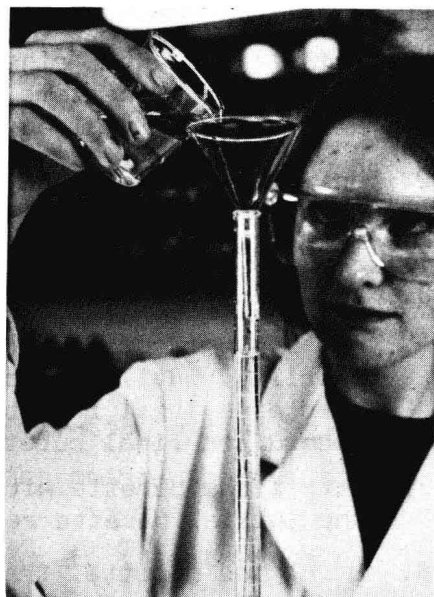


## EXPERIMENT 3

### An acid-base titration

#### Aim

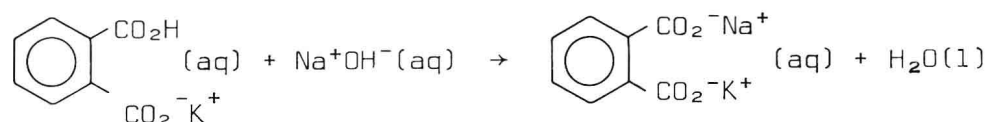
The purpose of this experiment is to determine the concentration of a solution of sodium hydroxide by titration against a standard solution of potassium hydrogenphthalate.



#### Introduction

In Experiment 2 you made a standard solution of potassium hydrogenphthalate, a primary standard. The substance has the formula  $\text{C}_8\text{H}_5\text{O}_4\text{K}$ , 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:



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<sup>3</sup>, and stand  
2 beakers, 100 cm<sup>3</sup>  
sodium hydroxide solution, approx. 0.1 M NaOH  
pipette, 25 cm<sup>3</sup>  
pipette filler  
standard potassium hydrogenphthalate solution (prepared in Experiment 2)  
4 conical flasks, 250 cm<sup>3</sup>  
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  $25.0\text{ cm}^3$  of the solution to a clean  $250\text{ cm}^3$  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  $0.05\text{ cm}^3$  (one drop).
6. Using the pipette, transfer  $25.0\text{ cm}^3$  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  $0.05\text{ cm}^3$  (approximately one drop). Consecutive titrations should agree to within  $0.10\text{ cm}^3$  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  $0.05\text{ cm}^3$ . 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 <sup>-3</sup>		cm <sup>3</sup>
Burette solution				mol dm <sup>-3</sup>		
Indicator						
		Trial	1	2	3	(4)
Burette readings	Final					
	Initial					
Volume used (titre)/cm <sup>3</sup>						
Mean titre/cm <sup>3</sup>						

### Calculation

1. Calculate the concentration of the sodium hydroxide solution.

### Questions

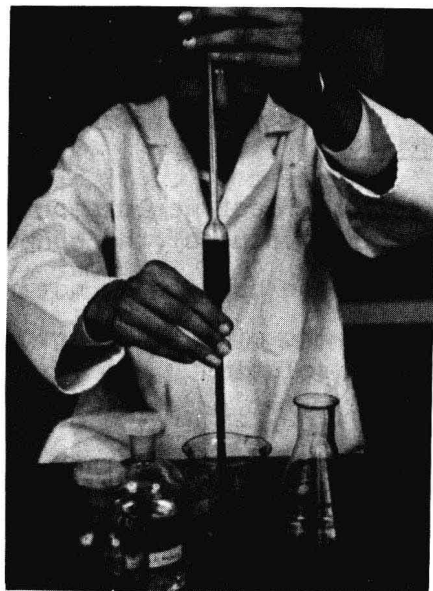
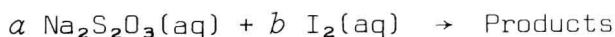
1. What effect would each of the errors described below have on the calculated value of the concentration of sodium hydroxide?
  - (a) The burette is not rinsed with the sodium hydroxide solution.
  - (b) The pipette is not rinsed with the potassium hydrogenphthalate solution.
  - (c) The tip of the 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.



#### 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<sup>3</sup>, and stand  
2 beakers, 100 cm<sup>3</sup>  
sodium thiosulphate solution, standardized  
pipette, 10 cm<sup>3</sup>  
pipette filler  
iodine solution, standardized  
4 conical flasks, 250 cm<sup>3</sup>  
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<sup>3</sup> of the solution to one of the conical flasks.
3. Titrate this solution until the colour of the iodine has almost gone (as indicated by a pale straw colour).