

Nuffield Chemistry
Collected Experiments

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Foreword

This volume is one of the first to be produced by the Nuffield Science Teaching Project, whose work began early in 1962. At that time many individual schoolteachers and a number of organizations in Britain (among whom the Scottish Education Department and the Association for Science Education, as it now is, were conspicuous) had drawn attention to the need for a renewal of the science curriculum and for a wider study of imaginative ways of teaching scientific subjects. The trustees of the Nuffield Foundation considered that there were great opportunities here. They therefore set up a science teaching project and allocated large resources to its work.

The first problems to be tackled were concerned with the teaching of O-Level physics, chemistry, and biology in secondary schools. The programme has since been extended to the teaching of science in sixth forms, in primary schools, and in secondary school classes which are not studying for O-Level examinations. In all these programmes the principal aim is to develop materials that will help teachers to present science in a lively, exciting, and intelligible way. Since the work has been done by teachers, this volume and its companions belong to the teaching profession as a whole.

The production of the materials would not have been possible without the wholehearted and unstinting collaboration of the team members (mostly teachers on secondment from schools); the consultative committees who helped to give the work direction and purpose; the teachers in the 170 schools who participated in the trials of these and other materials; the headmasters, local authorities, and boards of governors who agreed that their schools should accept extra burdens in order to further the work of the project; and the many other people and organizations that have contributed good advice, practical assistance, or generous gifts of material and money.

To the extent that this initiative in curriculum development is already the common property of the science teaching profession, it is important

that the current volumes should be thought of as contributions to a continuing process. The revision and renewal which will be necessary in the future, will be greatly helped by the interest and the comments of those who use the full Nuffield programme and of those who follow only some of its suggestions. By their interest in the project, the trustees of the Nuffield Foundation have sought to demonstrate that the continuing renewal of the curriculum – in all subjects – should be a major educational objective.

Brian Young,
Director of the Nuffield Foundation

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Introduction

The Nuffield approach is a way of teaching chemistry which, among other things, lays particular emphasis on the pupil finding out things for himself under careful direction, rather than being told facts and made blindly to learn them. The *Handbook for Teachers* describes in general terms this approach to the teaching of chemistry, and it is illustrated in detail by means of a Sample Scheme, in the books, the *Basic Course* and the *Course of Options*.

The Sample Scheme is a complete five-year teaching course for children of eleven to sixteen years, and consists of lesson notes and details of all the necessary experiments required for it. It is intended to be a sample only, and many other items of chemical subject-matter can of course be taught using the Nuffield approach.

Collected Experiments contains all the experiments used in the Sample Scheme together with many others. It is intended for the use of teachers, and its purpose is two-fold:

1. To provide alternative experiments in case those given for the Sample Scheme are not suited to the particular conditions under which the teacher is working.
2. To provide some experimental material for teachers who want to use the Nuffield approach, but to teach subject-matter other than that given in the Sample Scheme.

The experiments are arranged in this book by 'themes'; that is, experiments on related topics are grouped together. For example, experiments on isolating single substances are collected together, whether they are best used for first-year or fifth-year work, but an indication is given of the stage for which they are thought most suitable. It is hoped that this arrangement will make it easy for the reader to find details of experiments to meet his particular requirements.

The experiments are identified by means of two numbers, which are separated by a full stop. The number before the full stop is the number of the chapter in which the experiment appears, and the number after the full stop is the number of the experiment within the chapter. The first number is preceded by the letter E, to distinguish the numbering from that used in other publications of the Chemistry section of the Nuffield Science Teaching Project. Thus a reference to experiment E2.20 refers to *Collected Experiments*, Chapter 2, experiment 20.

Throughout the experiments the word *measure* in sentences such as 'Take a measure of zinc oxide' refers to the quantity obtained using the measuring device which forms part of the Nuffield spatula.

Many of the experiments have, as the first item in the list of apparatus, the phrase 'Page from *Laboratory Investigations*', followed by an experiment number. The publication *Laboratory Investigations* is obtainable as separate pages, and the experiment number referred to identifies the relevant page. These pages provide the pupils with instructions for the experiment, stimulate them into adopting an investigational attitude to their work, and enable them easily to build up a permanent record of their laboratory activities. Pages have been prepared only for pupils' experiments that appear in the Sample Scheme, but others can easily be prepared by the teacher if he so wishes.

Part I

What chemical changes are taking place, and what representation of them can be given at the molecular level?

Chapter 1

The effects of heating substances

Introduction

Heating substances is one of the basic operations of chemistry. From experiments in which a large number of substances are heated, and from noting carefully the changes which take place, the pupils are introduced to many other lines of investigation. Some of these are now suggested, and they are developed more fully in later chapters.

When many substances are heated, they catch fire and burn, because air has not been excluded. This result will provide a start to the investigation of 'Air and combustion' described in Chapter 3.

Some substances decompose on heating but others cannot be broken apart in this way. Those that do not decompose may be thought to be incapable of further subdivision, and thus the idea of elements as the ultimate product of thermal decomposition is made real. This idea is developed further in Chapter 6.

When liquids are heated they may boil, and the vapour obtained may condense on cooling and re-form the liquid. This introduces the idea of distillation. When solutions are heated, the solvent evaporates and, on cooling, the stronger solution which is obtained may deposit crystals which can be separated. From experiments such as these the whole field of 'Isolation and recognition of a single substance' (Chapter 2) is opened up.

'Energy' (Chapter 16), 'Rates of reaction' (Chapter 14), and 'Structure' (Chapter 13) may all be introduced and given some meaning using heating experiments, and thus this theme contains the germ of many ideas.

All of the experiments described in this chapter are suitable for use during the first stage of a chemistry course, and some of the ideas which they introduce are indicated after the titles in the list of experiments given below.

References

Two ways in which this theme can be presented to classes are described in the Sample Scheme, Part I, *Basic Course*, at the following points:

Stage I A, Topic 2: 'The effects of heating substances'

Stage I B, Topic 5: 'Finding out more about substances by heating them'

List of experiments

		<i>Ideas introduced</i>
E1.1	To find out what happens when substances are heated	Observation of change Rates of reaction
E1.2	To find out if a change in weight takes place when substances are heated (rough method)	Decomposition by evolution of vapour Combustion
E1.3	To find out if a change in weight takes place when substances are heated (more accurate method)	Decomposition by evolution of vapour Combustion
E1.4	To find out what is lost when copper(II) sulphate crystals are heated	Reversibility of some changes
E1.5	To find out what is lost when red lead is heated	Gases other than air Collection of gases by various methods
E1.6	Is copper changed in chemical nature when heated in air?	Combustion
E1.7	To find out if copper remains unchanged when heated if protected from the air	Combustion
E1.8	To find the temperature at which a liquid boils	Identification of pure substances Distillation

Experiment E1.1

To find out what happens when substances are heated

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A2.1)

Bunsen burner and asbestos square

Six hard-glass test-tubes, 75×12 mm

Tongs or test-tube holder

Iodine crystals

Zinc oxide

Powdered roll sulphur

Copper(II) sulphate crystals

Cobalt(II) chloride crystals

Magnesium ribbon, about 2 cm

Copper foil

Red lead or potassium permanganate

PROCEDURE

Let the class heat a variety of substances and meet a number of changes, one or two of which can be followed up in more detail shortly. The substances chosen are grouped into:

1. Those that return to their previous state on cooling. Possible substances are iodine crystals, zinc oxide, powdered roll sulphur (flowers of sulphur is not suitable for this experiment).
2. Those that split up but can be re-formed by putting together the products that resulted from heating them – copper(II) sulphate crystals, cobalt(II) chloride crystals.
3. Those that seem to have changed irreversibly – magnesium ribbon, copper foil, red lead (or potassium permanganate).

The pupils should not be told of this grouping in the first instance.

Warning: Iodine crystals attack the skin and the vapour is poisonous. Use one small crystal only for the experiment.

Each substance can be put out on a piece of paper and labelled, and the pupils told to take a measure of each in turn. Most of the substances are easily examined by heating in a 75×12 mm test-tube, held in a test-tube holder, and heated with a 3 cm Bunsen flame. The magnesium ribbon and copper foil should be held in tongs and heated directly by the flame. The Bunsen burners should be on asbestos squares. The changes observed should be noted down and discussed later.

Note: For experiments to find out what happens when various carbon compounds are heated, see experiments E10.1, E10.2, and E10.3.

Experiment E1.2

To find out if a change in weight takes place when substances are heated (rough method)

The simple type of experiment given above (experiment E1.1) may also be used to find out if a change in weight takes place when substances are heated. For this purpose, test-tubes with rims should be supplied, so that a wire support can be used to hang them up on a balance. The tube and contents should be weighed before and after heating, using a laboratory balance capable of detecting changes of 0.1 g. Sufficient evidence for many purposes can be obtained merely by deciding whether a gain or loss in weight has taken place.

It is a good plan to include some substances that do not change in weight on heating. Iodine, sulphur, and zinc oxide were suggested in experiment E1.1, but old samples of zinc oxide may have absorbed carbon dioxide from the air and if so, will lose weight on heating. Other possible substances which will not change in weight on heating are:

Nichrome wire from an electric heating element.

Thoroughly-dried silver sand.

Experiment E1.3

To find out if a change in weight takes place when substances are heated (more accurate method)

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A2.3)

Small crucible and lid

Tongs

Pipe-clay triangle

Bunsen burner, tripod, and asbestos square

Use of laboratory balance capable of detecting changes of 0.005 g

Hard-glass test-tube, 75 × 12 mm

Test-tube holder

Magnesium ribbon

Copper, made by reducing wire-form copper(II) oxide

Nichrome wire

Red lead or potassium permanganate

Thoroughly dried silver sand

PROCEDURE

The following are the procedures for each of the substances given in the list above.

A. Magnesium

Coil about 30 cm of magnesium ribbon into a small volume and place the coil in a crucible. Put the lid on and weigh the crucible and its contents. Next place the crucible and lid on a pipe-clay triangle on a tripod, standing on an asbestos square to protect the bench from reflected heat. Heat the crucible by means of a Bunsen burner, using a low flame at first and gradually increasing it until the magnesium ignites. Raise the crucible lid with a pair of tongs for a few moments at a time so that air may be admitted, but take care to see that as little smoke as possible escapes. When the reaction appears to be over, remove the crucible lid and heat the crucible strongly for two or three minutes. Allow the crucible to cool, replace the lid, and weigh the crucible and contents again. A gain in weight will be observed.

B. Copper

Take about 5 g of copper (preferably made by reducing wire-form copper(II) oxide) and heat it in a crucible without the crucible lid, until it is red hot. Continue the heating for five minutes. A gain in weight will be observed.

C. Red lead (*CAUTION: red lead is poisonous*) or *potassium permanganate*
Place some red lead in a 75×12 mm hard-glass test-tube so as to fill it about one-third full. Weigh the test-tube and contents, and then, holding the tube in a test-tube holder, heat it strongly by means of a Bunsen burner. After at least five minutes' strong heating, leave the tube and contents to cool on the asbestos square, and then reweigh them. A loss in weight will be obtained.

D. Sand

Put about 5 g of dry sand in a crucible and weigh the crucible and its contents. Heat the crucible strongly for two or three minutes, allow it to cool and weigh it again. No change in weight will be observed.

E. Nichrome wire

Put about 10 cm of coiled nichrome wire in a crucible and proceed as in (D). No change in weight will be observed.

Experiment E1.4

To find out what is lost when copper(II) sulphate crystals are heated

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A2.2)

Two hard-glass test-tubes, 100×16 mm

Cork or bung to fit test-tube, carrying a delivery tube

Bunsen burner and asbestos square

Thermometer, -10° to $+110^{\circ}\text{C}$

Stand and two clamps

Beaker, 100 cm^3

Hard-glass watch-glass

Copper(II) sulphate, small crystals

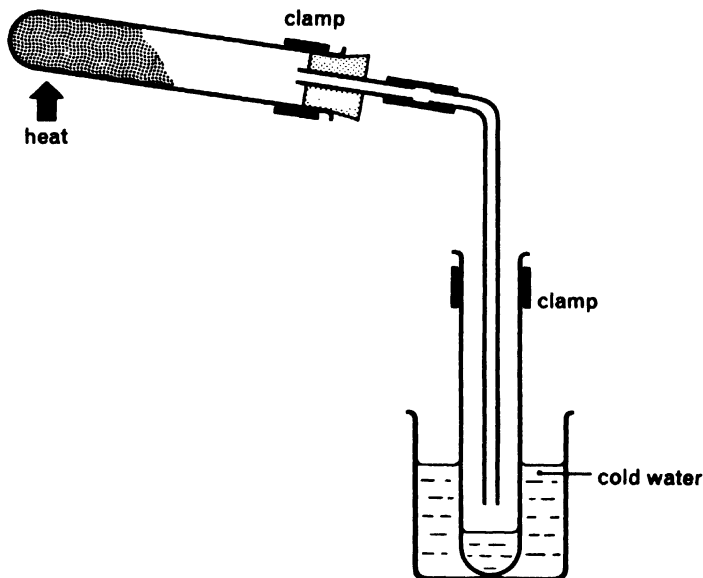


Figure 1.1

PROCEDURE

Set the apparatus up as shown in the diagram, filling the left-hand test-tube about half full with finely-powdered dry crystals of copper(II) sulphate. Heat this test-tube gently until the liquid driven off collects in the right-hand tube. From this amount of crystals about 2 cm depth of water will be obtained. Continue to heat gently until no more vapour is driven off. (The pupils should be warned to keep the end of the delivery tube above the level of the liquid collected, or some of the liquid may be drawn back into the heated tube.) Investigate the products in the following way.

1. Take the apparatus apart, and boil the liquid in the test-tube, with a thermometer clamped so that its bulb is in the air space above the liquid, to find out if it has the same boiling point as water. A small flame no more than 3 cm high should be used.
2. When the white residue left after heating the crystals is cold, transfer

some of it to a hard-glass watch-glass held in the palm of the hand and add some of the cold liquid obtained in (1).

Experiment E1.5

To find out what is lost when red lead is heated

Note: The same object can be achieved using potassium permanganate in this experiment in place of red lead; less heating is required and the permanganate does not attack the glass. A modification is required, however, and details of this are given in the last paragraph.

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A2.4)

Bunsen burner and asbestos square

Stand and clamp

Hard-glass test-tube, 100×16 mm

Wood splints

Red lead or potassium permanganate

Further apparatus needed will depend upon which method of collection is used:

a. Collection by syringe.

Syringe, 100 cm^3 glass or 50 cm^3 plastic

Stand and syringe holder

Short curved delivery tube fitted with bung and rubber connection tubing

b. Collection over water.

Delivery tube fitted with bung and rubber connection tubing

Small trough

Two test-tubes 150×25 mm and corks

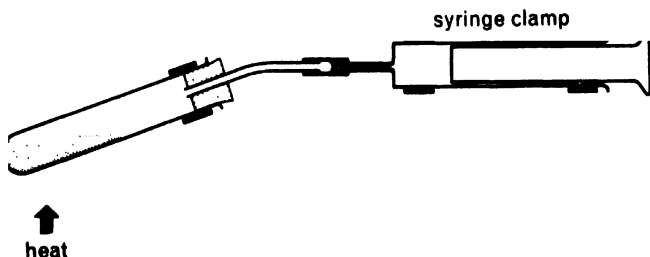


Figure 1.2a

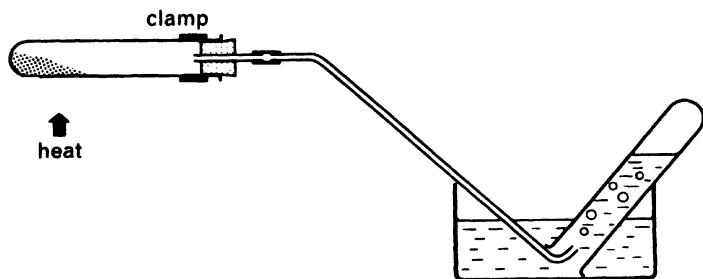


Figure 1.2b

PROCEDURE

A. Collection by syringe

About 2 cm depth of red lead (i.e. about 4 to 5 g) is placed in the 100×16 mm hard-glass test-tube which is clamped at an angle of about 30° to the horizontal and fitted with a short curved delivery tube. The syringe is joined to this by a short piece of rubber tubing and is clamped lightly in a horizontal position. The piston is pushed right in at the beginning of the experiment and is gradually pushed back as gas is evolved from the heated red lead. The test-tube should be heated with a non-luminous Bunsen flame which must be moved about to prevent over-heating in any part of the test-tube. The volume of gas evolved can easily be measured.

B. Collection over water

Quarter fill the 100×16 mm hard-glass test-tube with red lead (i.e. about 8 g), insert the delivery tube and clamp the test-tube with the end of the delivery tube about 1 cm below the surface of some water contained in a small trough.

Heat the red lead by means of the Bunsen burner, starting at the point shown, and watch the end of the delivery tube to find out if a gas is given off. A non-luminous flame should be used which must not be allowed to overheat the tube in any one place.

When about half a test-tube of gas (which will be largely displaced air) has been collected, collect a further two test-tubes full of the gas by displacement of water.

When sufficient gas has been collected the end of the delivery tube must be taken out of the water before heating is stopped, otherwise water will be drawn into the test-tube and cause it to crack.

Examination of the gas obtained

When the gas has been collected ask the pupils to find out if it is like air in supporting the combustion of a burning splint. This may be done by gently expelling some of the gas from the syringe or by using one of the test-tubes full of gas. Then suggest using a glowing splint in the same way so that they may see the splint re-kindle in the remaining sample of the gas.

Use of potassium permanganate

Red lead has the disadvantage that it spoils the test-tubes. Potassium permanganate may be used as an alternative. If potassium permanganate is substituted and collection is by method (A), a 150×25 mm hard-glass test-tube should be used and loosely packed asbestos wool placed in the upper part of the test-tube to prevent solid products of decomposition entering the syringe.

Note: A quantitative version of this experiment is given as experiment E3.5.

Experiment E1.6

Is copper changed in chemical nature when heated in air?

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 150×25 mm

Dilute sulphuric acid

Tongs

Bunsen burner and asbestos square

Clean copper foil or wire (two 2 cm squares of foil would be suitable)

PROCEDURE

Take two pieces of copper foil or wire and hold one of them by means of tongs in a Bunsen burner flame for a few moments. Place the heated copper foil in one 150×25 mm test-tube and the unheated piece in another. Find out if the heated copper has been changed in chemical nature by covering both pieces of copper with dilute sulphuric acid, and then warming the tubes gently. The colour of the solution above the heated copper foil will become blue, but that above the other piece will remain colourless.

As an additional test, the unheated copper could be weighed before immersion in the acid, and then washed and dried afterwards and re-weighed; it will be found that no change takes place.

Experiment E1.7

To find out if copper remains unchanged when heated if protected from the air

A. This experiment should be done by the teacher.

APPARATUS

Soft-glass test-tube, 100×16 mm, and cork to fit

Hard-glass test-tube, 100×16 mm

Bung and delivery tube to fit hard-glass test-tube

Vacuum pump

Pressure tubing to connect delivery tube and pump

Test-tube holder, or stand and clamp

Bunsen burner and asbestos square

Copper foil (2 cm squares of foil are suitable)

PROCEDURE

Two possible ways of heating copper protected from the air are as follows. To avoid accidents they are best performed by the teacher.

1. Place the foil in a 100×16 mm soft-glass test-tube with a few drops of water and place a cork *loosely* in the mouth of the test-tube. Hold the test-tube by means of a test-tube holder, or in a clamp, and heat it gently until all the water has become steam and the air has been displaced. Next, push the cork in firmly, while continuing to heat the tube to redness. The glass may collapse onto the copper, but, whether protected by steam or glass, the copper is not exposed to the air and does not turn black.

2. Place the foil in a 100×16 mm hard-glass test-tube fitted with a bung and delivery tube attached to a vacuum pump. Pump out the air from the tube and heat it to redness. On cooling it will be seen that the copper has not acquired a black coating.

B. Pupils' experiment.

APPARATUS

Each pupil or pair of pupils will require:

Bunsen burner and asbestos square

Tongs

Copper foil

A light hammer should also be available

PROCEDURE

Make an 'envelope' out of a piece of copper foil by folding it over itself and flattening the edges with a hammer. Heat the envelope by holding it in a Bunsen burner flame with a pair of tongs. Allow it to cool and then open it out. The inside remains unblackened.

Experiment E1.8

To find the temperature at which a liquid boils

This experiment should be done by the teacher.

APPARATUS

Beaker, 250 cm³

Bunsen burner and asbestos square

Tripod and gauze

Hard-glass test-tube, 150 × 25 mm

Pumice stone or pieces of broken porcelain

Thermometer, -10° to +110°C

Stand and two clamps

Ethanol or acetone

PROCEDURE

Place a 250 cm³ beaker half-full of water on a tripod and gauze, over a Bunsen burner, and by means of a stand and two clamps support a 150 × 25 mm test-tube containing not more than 10 cm³ of ethanol in the beaker, and a thermometer in the test-tube so that its bulb is held just above the level of the ethanol. Put one or two small pieces of pumice stone or broken porcelain in the ethanol. Heat the water in the beaker to about 80°C and when the ethanol begins to boil note the steady reading on the thermometer. *Ethanol should not be heated directly by the Bunsen burner because of the risk of fire.* As an alternative, acetone (which is also inflammable) may be used in place of ethanol.

Boiling points are: ethanol 78°C

acetone 56°C

Chapter 2

Isolation and recognition of a single substance

Introduction

Before a chemical investigation into a substance can begin, the substance must first be isolated from any others with which it may be mixed. This could involve separating this substance either from its naturally-occurring source, or from the other products of the reaction by which it was made.

The experiments in this chapter show the pupil how these separations can be carried out. They are designed (a) to illustrate useful laboratory techniques, such as filtration and distillation, and (b) to introduce various physical and chemical properties that can be used to separate mixtures of substances, and to characterize them when separated.

Before they do such experiments, it is doubtful if the pupils will have any real idea of what is meant by a *pure* substance, and the word 'pure' is probably best avoided until they have performed a few separations. They will, however, readily appreciate that they must deal with substances one at a time, and the common object of these experiments is therefore best described as obtaining a *single* substance from a mixture.

The experiments are divided into sections depending on the state of the materials being separated, solid, liquid, or gas. Additional sections describe experiments on chromatographic techniques, and the separation of elements from naturally occurring compounds.

References

Two ways in which this theme can be introduced at an early stage are described in the Sample Scheme, Part I, *Basic Course*, at the following points:

Stage I A, Topic 1: 'Getting pure substances from the world around us'

Stage I B, Topic 1: 'Separating pure substances from common materials'

The theme is developed throughout the Sample Scheme, but especially in the *Course of Options*, Option 11, 'Analysis with a purpose'.

List of experiments

Solids		<i>Ideas introduced</i>
E2.1	Simple separations of mixtures of solids a. salt and pepper b. iron and sulphur c. salt and sand	Electrostatic separation Magnetic separation Separation by solution and filtration
E2.2	How can we extract a substance from rock salt?	Use of solution method
E2.3	How can crude alum be purified?	Use of solution method
E2.4	How can crude naphthalene be purified?	Solution method with non-aqueous solvent
E2.5	How to recognize naphthalene	Determination of melting point
E2.6	To find out if tap water and sea water contain dissolved substances a. dissolved solids b. dissolved gases	
E2.7	How to obtain solids from sea water	Fractional crystallization
E2.8	MORE ADVANCED LEVEL How to separate two soluble solids by crystallization	Extension of solution method
E2.9	Compounds that crystallize together	Limitation of solution method
Liquids		
E2.10	How to recover pure water (simple method)	Distillation

E2.11	How to recover pure water (more efficient method)	
E2.12	How to recognize pure liquids	Use of physical constants
E2.13	Use of distillation to separate the components of crude oil	
E2.14	MORE ADVANCED LEVEL Illustration of fractional distillation	Extension of distillation
E2.15	Illustration of steam distillation	Extension of distillation

Gases

E2.16	Separation of gases by liquefaction	
E2.17	How to recognize pure gases	Density determination

Chromatographic techniques

E2.18	Separation of grass extract by paper chromatography	
E2.19	Separation of grass extract using blackboard chalk	
E2.20	Separation of dyestuffs by thin- layer chromatography	
E2.21	MORE ADVANCED LEVEL Separation of volatile liquids by gas chromatography	
E2.22	Breakdown of proteins and chromatographic identification of the amino-acids formed	

Separation of elements from compounds

E2.23	Separation of lead from an ore	Reduction
E2.24	Separation of iodine from seaweed	Oxidation

E2.25 Separation of copper from an ore Electrolysis

E2.26 Recognition of some elements using flame tests Flame tests

Isolation of solids

Experiment E2.1

Simple separations of mixtures of solids

A. *Salt and pepper*

APPARATUS

Each pupil, or pair of pupils, will need:

Comb

Supply of a mixture of salt and pepper

PROCEDURE

The pupils should be provided with a mixture of salt and pepper, in which the salt is as coarse and the pepper as fine as possible, and everything must be very dry. They could be asked to suggest ways of separating this mixture, and then told to comb their hair and hold the comb over the mixture. They should find out if the separation is complete.

B. *Iron and sulphur*

APPARATUS

Each pupil, or pair of pupils, will need:

Magnet

Supply of a mixture of iron filings and powdered sulphur

PROCEDURE

Hold a magnet over the mixture of iron filings and powdered sulphur and see that one of the components is picked up, but the other is not. Try to find out if the separation is complete.

Note: It is sometimes difficult to remove iron filings from a magnet after use and it is a good plan to get the pupils to wrap each magnet in paper before use.

C. *Salt and sand*

APPARATUS

Each pupil, or pair of pupils, will need:

Test-tube, 100 × 16 mm

Glass funnel

Filter paper

Funnel stand
Evaporating basin
Supply of a mixture of salt and sand

PROCEDURE

Shake about a 2 cm depth of the mixture in half a test-tube full of cold water for about two minutes. This will dissolve the salt. Pour the contents of the tube into a filter paper in a glass funnel held by means of a funnel stand over an evaporating basin. Rinse the test-tube with some more water and pour the extra water through the funnel. By doing this, the sand will be retained on the filter paper, and the salt can be recovered by warming the evaporating basin, as described in experiment E2.2.

Filtration will be seen to be a slow process, but it can be speeded up in two ways:

1. By use of a Buchner filter and flask.
2. By means of a centrifuge.

This simple experiment can be used as a basis for a comparison of these methods of filtration.

Experiment E2.2

How can we extract a substance from rock salt?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.1)

Pestle and mortar

Glass stirring rod

Filter funnel and quick filter paper (or Buchner funnel, Buchner flask, filter paper, and filter pump)

Evaporating basin

Beaker, small enough for evaporating basin to rest on top

Tripod and gauze

Hand lens

Bunsen burner and asbestos square

Sample of rock salt

PROCEDURE

Take as much rock salt as will cover a penny, crush the lumps, and dissolve them in hot water in a small beaker, stirring with a glass rod. A quantity of water a little more than three times the bulk of the rock salt will be needed.

After filtering (either with a quick-filter paper or with a Buchner funnel

and filter pump) heat the salt solution in an evaporating basin until a crust forms on the liquid. Heat with a Bunsen burner and put the basin on a gauze.

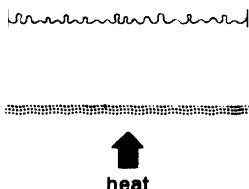


Figure 2.1

When most of the liquid has gone, transfer the evaporating basin to a steam-bath for further heating, otherwise the solid is likely to jump out. A small beaker half full of water, as shown in the diagram, makes a suitable steam-bath.

After the evaporation the crystals of salt produced can be examined under a hand lens and their shape and regularity noted.

Experiment E2.3

How can crude alum be purified?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B1.1)

Beaker, 100 cm³

Crystallizing dish

Funnel and quick filter paper (e.g. Green's No. 802 or Whatman No. 41)

Tripod and gauze

Bunsen burner and asbestos square

Glass rod

Stand and clamp or a conical flask for supporting funnel

Measuring cylinder, 25 cm³

Hand lens or access to low-power microscope

Sample of dirty alum, about 5 g; if none is available, a suitable mixture may be made by adding dried soil or laboratory dust to alum crystals

PROCEDURE

Place enough of the dirty alum in a 100 cm³ beaker to cover the bottom and cover it with about 10 cm³ of water. Put the beaker on a tripod and gauze and heat it with a Bunsen burner. Stir the contents of the beaker with a glass rod. If some of the alum remains undissolved when the water is boiling, add more water until all the alum has dissolved and only the dirt remains as a solid.

Pour the hot solution through a quick filter paper in a filter funnel and collect it in a crystallizing dish or a conical flask.

On cooling, crystals of pure alum will form, as its solubility in cold water is much less than in hot water. If crystals fail to appear, boil off some of the water, and allow to cool in the crystallizing dish once more. When quite cool, pour the liquid away and dry the crystals by placing them on filter papers. To get crystals large enough to form 'seeds' for further growth, the solution must be only slightly supersaturated at room temperature and left to crystallize overnight.

Experiment E2.4

How can crude naphthalene be purified?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B1.2)

Two test-tubes, 100 × 16 mm

Beaker, 100 cm³

Glass rod

Tripod and gauze

Bunsen burner and asbestos square

Buchner or Hirsch funnel, filter paper, filter flask, and filter pump

Teat pipette

Spatula

Filter papers for drying crystals

Thermometer, -10° to +110°C

Crude naphthalene (this is available from laboratory suppliers. Dirty naphthalene can be made by adding a little soil or laboratory dust to a quantity of the clean compound)

1,1,1-trichloroethane or ethanol (industrial methylated spirits)

Activated charcoal powder

Pupils will require access to a centrifuge.

PROCEDURE**Making the solution:***1. Using 1,1,1-trichloroethane as solvent*

Put about 2 cm depth of crude naphthalene in a 100×16 mm test-tube and add 2 cm^3 of trichloroethane. Warm the test-tube over a small Bunsen flame, stirring the contents with a glass rod to aid solution.

2. Using ethanol (industrial methylated spirits) as solvent

Caution: ethanol is inflammable

Take a 100 cm^3 beaker, fill it to two-thirds with water, and heat it to about 75°C on a tripod and gauze. Then place the beaker at bench level on an asbestos square and extinguish all Bunsen flames. Put about 2 cm depth of crude naphthalene in a 100×16 mm test-tube and half fill the tube with ethanol. Place the test-tube in the beaker of hot water, stirring with a glass rod to aid solution.

Obtaining pure naphthalene from the solution:

As soon as the naphthalene has all dissolved, centrifuge the contents of the test-tube. Pour off the supernatant liquid as quickly as possible into another test-tube to prevent the naphthalene from beginning to crystallize in the centrifuged test-tube. Now cool the solution under a tap whereupon naphthalene crystals will appear. Separate the crystals of naphthalene by filtering them through a Buchner funnel and washing them with a few drops of cold trichloroethane or ethanol. Finally dry the crystals by placing them on a few pieces of filter paper.

If a discoloured sample of naphthalene is obtained, as is most probable with the industrial crude naphthalene sample, repeat the entire procedure, using a fresh sample of crude naphthalene, but this time adding a spatula measure of activated charcoal to the solution just before warming the solvent and crude naphthalene. A white sample of naphthalene will be obtained this time.

After an impure sample of a solid has been treated by one of the methods given above, an experiment is needed which will tell us whether the product obtained is really pure. For many solid substances, this can best be done by a determination of the melting point of the product, which is then compared with the melting point of a sample known to be pure. This is described in the next experiment.

Experiment E2.5

How to recognize naphthalene by a determination of its melting point

APPARATUS

Each pupil or pair of pupils will need:

Pure naphthalene

Purified naphthalene from experiment E2.4

Two ignition tubes

Rubber band

Thermometer, -10° to $+110^{\circ}\text{C}$

Beaker, 100 cm^3

Tripod and gauze

Bunsen burner and asbestos square

PROCEDURE

Compare the melting point of the sample of naphthalene that has been purified with that of the pure material. Place a sample of pure naphthalene in one ignition tube and one of purified naphthalene in another. Attach the two ignition tubes to a thermometer by means of a rubber band and place them in a beaker of water on a tripod and gauze over a lighted Bunsen burner. The thermometer may be used to stir the water but care must be taken to see that no water gets in the ignition tubes. Slowly raise the temperature of the water and watch the ignition tubes until each sample of naphthalene has melted.

The presence of impurities lowers the melting point of naphthalene. Pure naphthalene melts at 80°C .

Experiment E2.6(a)

To find out if tap water and sea water contain dissolved solids

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B1.3)

Watch-glass, hard glass, approximately 75 mm diameter

Beaker, 100 cm^3

Bunsen burner, tripod, gauze, and asbestos square

A cloth should be available

Alternatively:

Microscope slide for each pupil

Infra-red lamp for class use

PROCEDURE

Put on the watch-glass enough tap water to cover about half of its area. Place the watch-glass on top of a 100 cm³ beaker half-filled with water, which is supported on a gauze by a tripod above a lighted Bunsen burner.

Boil the water in the beaker fairly briskly so that the steam produced causes the water in the watch-glass to evaporate. As soon as the water in the watch-glass has all gone (about ten minutes will be required), remove the burner and take the watch-glass off the beaker, drying the drops of water adhering to the underside with a cloth. A residue will be seen on the watch-glass.

Do not heat the watch-glass directly with a burner or it may crack.

A parallel experiment may be performed using distilled, or deionized, water on the watch-glass instead of tap water, and a third experiment using sea water could also be run concurrently.

If the evaporation is done under an infra-red lamp, drops of water may be placed on a microscope slide. (They will not run off the slide if it is first made slightly greasy by wiping with the finger.)

Experiment E2.6(b)

To find out if tap water and sea water contain dissolved gases

APPARATUS

Each pupil or pair of pupils will require:

Round-bottomed flask, 250 cm³

Bung and delivery tube to fit flask (see diagram on p. 24)

Tripod and gauze

Bunsen burner and asbestos square

Stand and clamp

Beaker, 250 cm³, or small trough

Test-tube, 125 × 16 mm

Support for beaker or trough (stand and clamp, box, pile of books, etc.)

PROCEDURE

Fill the round-bottomed flask right up to the top with tap water, and insert the bung carrying the delivery tube so that the tube itself becomes completely filled with water. If this cannot easily be done, place the bung in the flask having the whole apparatus immersed in a sink of water. Set up the apparatus as shown in the diagram and heat the flask by means of the Bunsen burner. Bubbles of gas will be released from the water and travel into the test-tube. Continue until the contents of the

flask are boiling. About half a test-tube full of gas will be collected, all of which has been displaced from solution by heating.

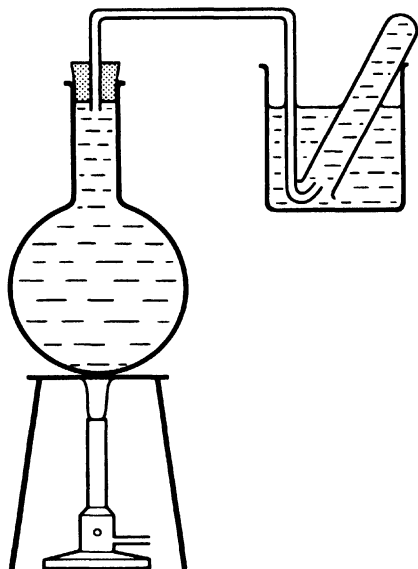


Figure 2.2

Another version of this experiment, in which the composition of the dissolved air can be determined, is given as experiment E4.9.

Experiment E2.7

How to obtain solids from sea water

This experiment can conveniently be done by the teacher and pupils working together, the teacher doing the evaporation of the sea water, and the pupils performing the tests on the solids obtained.

APPARATUS

Some large beakers, say 2 litre, 1 litre, 600 cm³, 250 cm³, 100 cm³

Bunsen burner, tripod, gauze, and asbestos square

Supply of sea water

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A10.1)

Small Buchner funnel, filter flask, and access to filter pump

Six test-tubes, 100 × 16 mm in rack

5 cm length of nichrome wire, supported in glass rod or cork, for flame tests

Watch-glass

Blue glass, approximately 2×5 cm

One or more simple microscopes or a microprojector should be available

Lime water

Silver nitrate solution

Barium chloride solution

Magneson reagent

Concentrated hydrochloric acid

2M hydrochloric acid

2M nitric acid

PROCEDURE

Start with 1 litre of sea water, and filter it if necessary. Evaporate it in stages, examining the solids that come out of solution at each stage. The solids can be removed by filtration, preferably using a Buchner funnel, and the following observations can be made. (*N.B.* The final stages of the evaporation are best done on a steam-bath.)

Stage 1

Volume reduced to 250 cm³. The amount of solid matter is small, but the presence of the carbonate ion is easily shown by the action of 2M hydrochloric acid, which gives carbon dioxide (test with lime water). The calcium ion can be detected by means of a flame test. This indicates that calcium carbonate is present.

Stage 2

Volume reduced to 125 cm³. Somewhat greater deposition of solid matter. Calcium and carbonate ions may again be detected, but in addition the formation of a precipitate on adding barium chloride to the solution of the solid matter in 2M hydrochloric acid demonstrates the presence of a sulphate. This indicates the presence of calcium sulphate. On viewing the solid in the microscope, spiky needle-like crystals will be seen, coloured when viewed between crossed polaroids.

Stage 3

Volume reduced to 75 cm³. A similar amount of solid matter obtained as at stage 2. The solid gives a strong yellow flame, indicating the presence of sodium ion, a faint precipitate for the sulphate test, but a strong indication of chloride on addition of 2M nitric acid and silver nitrate solution. Under the microscope the crystals nearly all appear to be rectangular and isotropic. This indicates that sodium chloride is present.

Stage 4

Volume reduced to 50 cm³. Considerable deposition of solid matter, which is found to be sodium chloride again.

Stage 5

Volume reduced to 25 cm³. Considerable further deposition of solid matter, which is found to be sodium chloride again.

Stage 6

Volume reduced to 15 cm³. Somewhat smaller deposition of solid, which gives a clear indication of sulphate ions, and a positive result to the test for magnesium ions using the Magneson reagent. Compare the result with that of a magnesium salt from a labelled bottle off the shelf. Under the microscope the crystals appear to be mainly prismatic, and are coloured when viewed between crossed polaroids. This shows that magnesium sulphate is present.

Stage 7

Volume reduced to 10 cm³. More magnesium sulphate is deposited.

Stage 8

Volume reduced to 5 cm³. The solid deposited at this stage gives a lilac flame colour on viewing through blue glass, indicating the presence of potassium ions (compare this with the flame colour of a known potassium salt), and a heavy precipitate with 2M nitric acid and silver nitrate solution. It consists of *potassium chloride* and *magnesium chloride*. The filtrate from this stage should be kept for experiment E5.3, part 2.

Note: This description refers to sea water obtained from the South Coast. The experiment is adapted from one described in Fowles, G., *Lecture Experiments in Chemistry* (Bell).

For an experiment to examine the effect of electrolysis sea water, see experiment E5.3, part 2.

Isolation of solids – more advanced level**Experiment E2.8**

How to separate two soluble solids by crystallization (an extension of the solution method of separation)

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Tripod and gauze

Beaker, 100 cm³

Glass stirring rod

Finely powdered mixture of copper(II) sulphate and potassium dichromate

PROCEDURE

Add two or three measures of the mixture of copper(II) sulphate and potassium dichromate to some hot water in a 100 cm³ beaker on a tripod and gauze over a lighted Bunsen burner. Stir the mixture well with the glass rod until it is all dissolved, adding more water if necessary.

Allow the solution to cool and examine the crystals which are formed. If none appears, heat the solution to boiling, evaporate some of the water, and allow to cool again. Examine the crystals obtained. They will all be orange (potassium dichromate) or, if the solution was very strong, of two sorts, orange and blue (copper(II) sulphate). It can be seen that the substances form separate crystals, and these can be separated with tweezers.

Experiment E2.9

Compounds that crystallize together (limitations of the solution method of separation)

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Three test-tubes, 100 × 16 mm, in rack

Three microscope slides

Glass stirring rod

Hand lens or microscope

Iron(II) sulphate

Ammonium sulphate

Dilute sulphuric acid

PROCEDURE

Fill two test-tubes with water each to a depth of about 3 cm. In one of the test-tubes place two or three drops of dilute sulphuric acid and as much iron(II) sulphate as will dissolve in the water when it is heated nearly to boiling. In the other test-tube place as much ammonium sulphate as will dissolve in the water when it is heated nearly to boiling.

While the solutions are still hot, pour about one-third of each into the third test-tube and mix well. Using the glass rod place one or two drops from each solution on separate microscope slides and leave to cool. Examine the crystals which form using a hand lens or a low-power microscope. The crystals obtained from the mixture will be seen to be

all of one sort, and different from those obtained from the other two solutions. They are of the double salt, ammonium iron(II) sulphate. When double salts form it can be seen that the constituents cannot be separated by a crystallization method.

Note: Further experiments on crystallization are given in Chapter 13, 'Structure'; experiments E13.3 to E13.13.

Isolation of liquids

Experiment E2.10

How to recover pure water (simple method)

In this experiment, pure water is obtained from ink, but any other solution could be used. To be convincing, there should be some colouring matter mixed with the water being heated; the water collected will be seen to be colourless.

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.2a)

Filter flask, 100 cm³, or conical flask, 100 cm³

Cork or bung to fit flask

Lengths of glass tubing bent as shown in figure 2.3

Rubber connection tubing

Stand and two clamps

Tripod and gauze

Bunsen burner and asbestos square

Test-tube, 100 × 16 mm

Measuring cylinder, 25 cm³

Supply of ink (Stephen's black, or black Quink are suitable. Some inks froth excessively when boiled)

PROCEDURE

Place about 10 cm³ of ink in the conical flask, insert the cork or bung, and connect the delivery tube as shown in the appropriate diagram. Clamp the test-tube at the end of the delivery tube. Now heat the flask *very carefully* with a small (3 cm) Bunsen flame. Take care to prevent the ink from frothing over or splashing into the delivery tube. After the ink has been boiling for a few moments, a few drops of colourless liquid will collect in the test-tube.

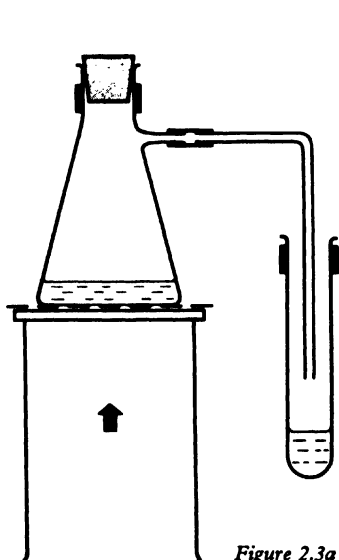


Figure 2.3a

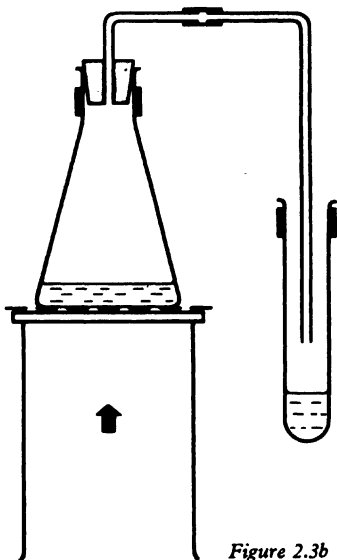


Figure 2.3b

Experiment E2.11

How to recover pure water (more efficient method)

In this experiment, as with experiment E2.10, pure water is obtained from ink, but any other solution could be used. To be convincing, there should be some colouring matter mixed with the water being heated; the water collected will be seen to be colourless. For elementary classes, this experiment should be done by the teacher.

APPARATUS

Distillation flask (at least 100 cm³ capacity)

Liebig condenser and connection tubing to tap and sink

Thermometer, -10° to $+110^{\circ}\text{C}$

Two stands and clamps

Bunsen burner, tripod, gauze, and asbestos square

Corks or bungs to assemble apparatus

Beaker, 100 cm³

Pumice stone or pieces of broken porcelain

Filter paper or blotting paper

Ink

PROCEDURE

The distillation apparatus should be set up as shown in the diagram on p. 30. Important points to note are:

1. The thermometer bulb should be in line with the side-arm of the distillation flask to record the temperature of the vapour actually being collected.
2. The cooling water should enter through the lower tube and leave by the upper one, so as to keep the condenser jacket full of cooling water.
3. A few pieces of pumice stone or broken porcelain should be placed in the distillation flask to ensure even boiling.
4. The clamp holding the distillation flask should be above the side-arm and not below it, otherwise heat will be conducted away by the clamp and distillation will not be so rapid.

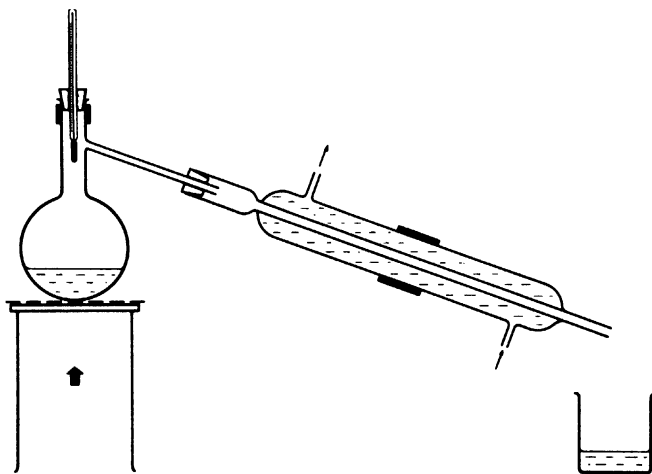


Figure 2.4

Collect sufficient water for all the pupils to see but do not boil the contents dry or the flask may crack.

Finally, mix the water with the contents of the flask to show that 'ink' is formed again.

Experiment E2.12

How to recognize pure liquids

After a liquid has been isolated from a mixture its purity can best be established by comparing its boiling point with that of a sample known to be pure.

Boiling points are easily determined by observation of the thermometer

during distillation, but if distillation is not being done, the technique described in experiment E1.8 can be used.

To investigate the method, pupils can determine the boiling points of several different liquids, finding that each has its own value.

An alternative physical constant which can be investigated is specific gravity. If hydrometers are available, pupils can compare the specific gravities of water, some aqueous solutions, and other liquids which may be available.

Experiment E2.13

Use of distillation to separate the components of crude oil

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.5)

Stand and clamp

Bunsen burner and asbestos square

Hard-glass test-tube with side-arm, 125×16 mm

A bent delivery tube and rubber connection tubing

Four test-tubes, 75×12 mm

Thermometer, 0° – 360°C , and cork

Teat pipette

Beaker, 100 cm^3

Watch-glass, hard glass

Asbestos wool

About 2 cm^3 of crude oil

PROCEDURE

Place a loose asbestos plug in the bottom of the test-tube with side-arm and add about 2 cm^3 of crude oil, using the teat pipette. Set up the apparatus as shown in figure 2.5 and heat the test-tube carefully until the first drops of liquid distil over. The four test-tubes may be used to collect four fractions of roughly the following boiling ranges. The crude oil must be distilled *slowly* and the first fraction should be collected in a test-tube cooled in a beaker of water.

1. Room temperature to 70°C

2. 70° – 120°C

3. 120° – 170°C

4. 170° – 220°C

A black residue will remain in the test-tube. Test the four fractions for viscosity (how easily do they pour?), colour, and inflammability. For the inflammability test, pour a sample of each fraction in turn onto a watch-glass (hard glass only) or crucible, and light the fraction with a match or

wood splint. If an asbestos plug is added the heavier fractions burn easily and the sooty flames show up well.

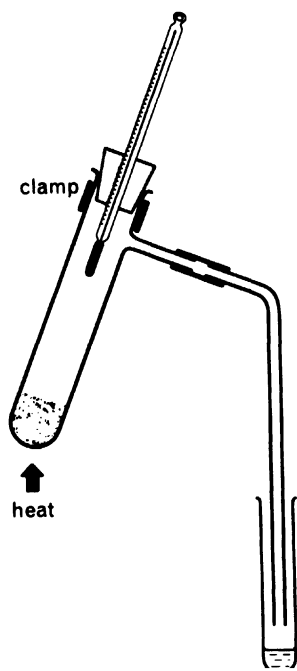


Figure 2.5

Do not burn the remaining sample of each of the four fractions, but mix them together again. A substance very like the original crude oil will be obtained.

Isolation of liquids – more advanced level

Experiment E2.14

Illustration of fractional distillation

For elementary classes, this experiment should be done by the teacher.

APPARATUS

Round-bottomed flask, about 500 cm³

Fractionating column and packing

Liebig condenser with rubber tubing to connect to water supply and sink

Bunsen burner, asbestos square, tripod, and gauze

Thermometer, -10° to $+110^{\circ}\text{C}$, in cork or bung to fit fractionating column

Several beakers, 100 cm^3 , to collect distillate

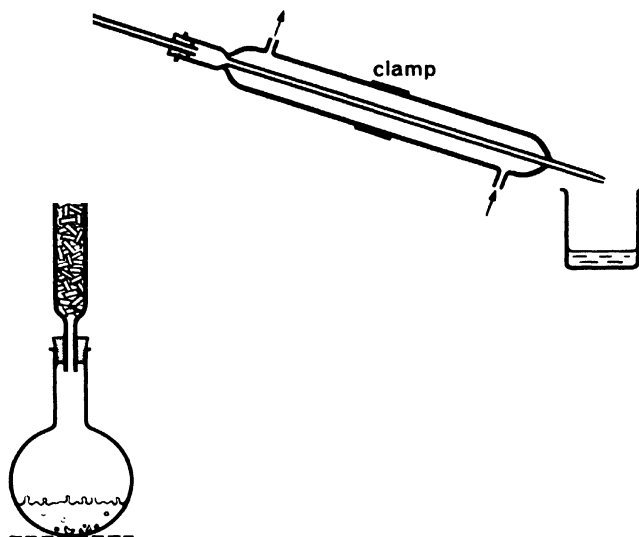
Three stands and clamps

Corks or bungs to assemble apparatus

Mixture of alcohol (industrial methylated spirits) and water, one part of alcohol to three parts of water

Pumice stone or pieces of broken porcelain

clamp



PROCEDURE

Demonstrate fractional distillation by setting up the apparatus as shown in the diagram. The fractionating column may be packed with small pieces of glass rod or glass tubing. Important points to note are (1) the round-bottomed flask should not be more than a third full at the beginning of the distillation; (2) the thermometer bulb should be in line with

the side-arm at the top of the fractionating column, to record the temperature of the vapour actually being collected; and (3) the cooling water should enter the condenser through the lower tube and leave by the upper one, so that the condenser jacket is always full.

Before starting the experiment, show that the mixture of water and alcohol will not burn.

Place a few pieces of broken porous porcelain or pumice stone in the round-bottomed flask to promote even boiling. Boil the liquid in the flask so that the distillate collects at a rate of two to three drops a second. Take care to avoid boiling too rapidly or the column may 'flood' (become filled with liquid) and a quantity of liquid be forced over into the condenser. Watch the thermometer and collect different fractions in separate receivers. Note the boiling ranges of these distillate fractions and show that some, at least, of these fractions will burn. The proportions of water and alcohol present in mixtures of various boiling points are as follows:

<i>Boiling point</i>	<i>Percentage of ethanol</i>
79°C	86 per cent
80°C	83 per cent
82°C	79 per cent
84°C	76 per cent
86°C	72 per cent
90°C	62 per cent
94°C	44 per cent
98°C	19 per cent

Experiment E2.15

Illustration of steam distillation

An experiment to be carried out by the teacher, or with advanced classes, by the pupils.

APPARATUS

The teacher will need:

Steam generator

Round-bottomed flask

Liebig condenser and connection tubing to tap and sink

Adaptor

Several small beakers to collect distillate

Corks and glass tubing to assemble apparatus (see diagram)

Two tripods and gauzes

Two retort stands and clamps

Two Bunsen burners Orange peel

PROCEDURE

The point of this operation can well be illustrated by letting the pupils find out what happens if small pieces of orange peel are heated in a test-tube. An unpleasant smell and a black residue of carbon are obtained.

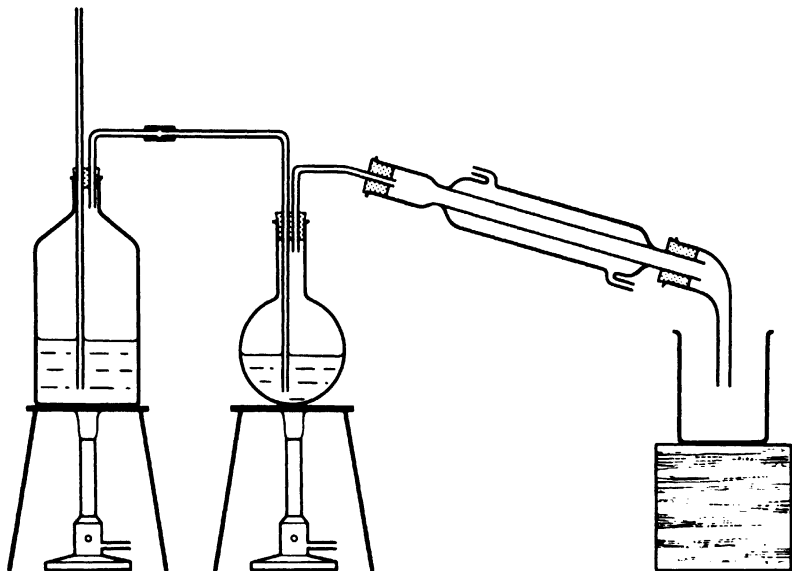


Figure 2.7

If however the apparatus for steam distillation is set up as shown in the diagram and pieces of chopped orange peel are placed in the round-bottomed flask, covered with water, and both containers of water heated until the water boils, the condensate obtained will carry a strong smell of the oil from the peel.

This happens because when the volatile component of the orange and water are heated together, *both* substances boil when the sum of their vapour pressures is equal to the atmospheric pressure, even if the temperature is below the boiling points of *either* substance. When the orange peel is heated alone, however, before the boiling point of the volatile component is reached, the temperature is sufficient to cause thermal decomposition of the substances present in the peel.

The purpose of the steam generator is to provide a source of internal heating in the round-bottomed flask, and to stir the contents so as to promote even boiling. Without it the contents of the round-bottomed flask are likely to be expelled into the condenser.

Isolation of gases

The handling of gases. There are a number of ways in which gases can be handled, and these are described in detail in various other experiments. Collection over water is probably too well-known to require any description here. The use of plastic bags has much to commend it; in this way dry samples of gases can be collected and carried about in a container of negligible weight.

The use of syringes is a major advance on older methods and several experiments illustrate their use. These include experiments E1.5, E3.3, E12.11, E12.18, E12.19, and E12.20.

A helpful commentary on the use of aspirators is to be found in Fowles, G., *Lecture Experiments in Chemistry* (Bell).

Experiment E2.16

Separation of gases by liquefaction

An experiment to be carried out by the teacher, or, with advanced classes, by the pupils.

APPARATUS

The teacher will need:

Bunsen burner and asbestos square

Stand and clamp

Four soft-glass test-tubes, 150×25 mm

Hard-glass test-tube, 150×25 mm

Bung to fit test-tube, carrying right-angle delivery tube

U-tube, and bungs to fit

Beaker, large enough to hold U-tube

Delivery tube from U-tube to trough (see diagram)

Small trough

Lead(II) nitrate, finely powdered

Ice and salt to make a freezing mixture

PROCEDURE

Place a 3 cm depth of finely-powdered lead(II) nitrate in a hard-glass test-tube, and connect it to a U-tube and trough by means of delivery tubes as shown in figure 2.8. Surround the U-tube with crushed ice and salt, in alternate layers, using about four parts of ice to one of salt.

Fill the trough and the soft-glass test-tubes with water. Heat the test-tube containing the lead(II) nitrate, gently at first, when it will decompose giving both oxygen and nitrogen dioxide as gaseous products. The brown gas (nitrogen dioxide) will condense in the U-tube and the oxygen will pass through and be collected in the test-tubes over water. A separation of the two gases will thus have been made.

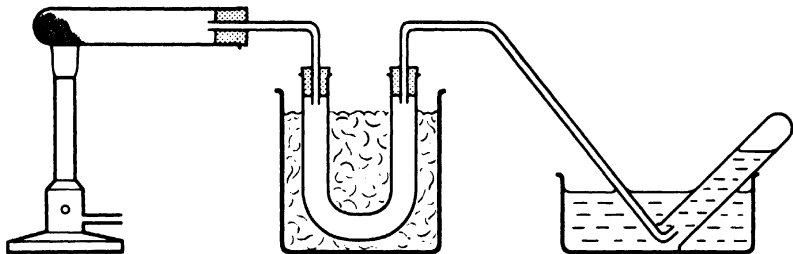


Figure 2.8

Experiment E2.17

How to recognize pure gases

A physical method by which gases can be recognized is by the measurement of their densities. The following experiment illustrates a method by which reasonably accurate values can be obtained in a simple manner.

APPARATUS

Each pupil or pair of pupils will need:

Round-bottomed flask, 500 cm³, as light as possible

Bung to fit flask, carrying two delivery tubes (see figure 2.9)

Two short lengths of rubber tubing

Two Mohr clips

Balance

Sources of oxygen, nitrogen, carbon dioxide, etc.

Measuring cylinder

PROCEDURE

The main errors in this experiment arise owing to the fact that the weights of gases involved are very small compared to the weight of the flask.

First, weigh the round-bottomed flask, bung, and delivery tubes full of air.

Next, pass a stream of gas, for example nitrogen from a cylinder, into the flask through the delivery tube reaching to the bottom of the flask

(see diagram) having both clips open. Continue passing the gas until you think that it has displaced all the air. Close both clips and weigh the flask full of nitrogen.

Finally, fill the flask with water and empty into a measuring cylinder. Measure the volume of water tipped out; this is the volume of the round-bottomed flask.

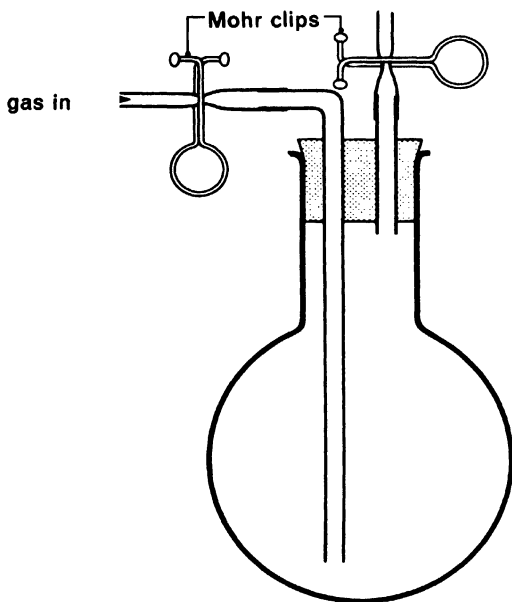


Figure 2.9

Look up the density of air, and make a correction for atmospheric temperature and pressure if you can. Multiply this by the volume of the flask; the result will be the weight of air in the flask.

Subtract the weight of air from the weight of the flask full of air; this gives the weight of the flask alone.

Subtract the weight of the flask alone from the weight of the flask full of nitrogen; this gives the weight of the nitrogen.

Divide the weight of the nitrogen by the volume of the flask; this gives the density of nitrogen.

Different pupils can carry out the experiment using different gases, and the results can then be collected.

It is found that use of the bung and delivery tube system illustrated in the diagram gives better results than just passing the gas into the flask through a single tube for some time, withdrawing the tube and inserting a solid bung.

Chromatographic techniques

Experiment E2.18

Separation of grass extract by paper chromatography

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.3)

Pestle and mortar

Test-tube, 100×16 mm

Beaker, 100 cm^3 , or crystallizing dish as support for filter paper

Filter paper (preferably Whatman's No. 1)

Teat pipette, drawn out to a fine point

Scissors (unless the grass is already cut up)

Supply of grass (or spinach)

Acetone or ethanol (*Caution.* Acetone and ethanol are highly inflammable.)

PROCEDURE

Cut up a handful of grass (or spinach) into small pieces with scissors. Grind the pieces with enough acetone or ethanol to produce 2 to 3 cm^3 of free liquid in a mortar, and then decant the green liquid into a test-tube. *The extract should be as concentrated as possible.*

Place a filter paper on top of a beaker or crystallizing dish or other support, and using a teat pipette place a drop of the extract in the centre of the paper. When it has finished spreading, add another drop and repeat until three or four drops have been added.

Allow a minute or so for the last drop to finish spreading, and then add, one at a time from a teat pipette, more drops of acetone or ethanol. Well-separated green and yellow bands will develop as the coloured substances travel towards the edge of the paper. The use of a different solvent (e.g. toluene) for elution may be tried. In this case the original solution must evaporate completely before the new solvent is added.

A better separation of the mixture can be obtained in the following manner, but it will take rather longer to obtain the result:

Using a pair of scissors, make two parallel cuts from the edge of a circle of filter paper to near the centre, about 1 cm apart from each other. Bend the strip of paper so formed so that it is at right angles to the rest of the paper, and place it on top of a crystallizing dish containing the solvent, so that the strip bent at right angles dips into the solvent. Place the drops of extract on the centre of the paper as described for the first method and leave for the chromatogram to develop.

The outer orange band is xanthophyll, and the green band is chlorophyll. There are two types of chlorophyll, chlorophyll *a* and *b*, but these are not separated by this technique. Carotene is also present. An inner ring of carotene can be seen if toluene is used as the eluent.

The bands can be separated by cutting them out from the filter paper, and the teacher can obtain the coloured materials by collecting the rings of paper from all the pupils and extracting the chlorophyll and xanthophyll with a little warm acetone.

For the use of paper chromatography in identifying the products of the hydrolysis of proteins, see experiment E2.22, and for a similar experiment on the products of hydrolysis of starch, see experiment E10.9.

Experiment E2.19

Separation of grass extract using blackboard chalk

APPARATUS

Each pupil or pair of pupils will need:

Grass extract (for preparation see previous experiment)

Small dish

Stick of blackboard chalk (plain white: not the 'dustless' or yellow-covered varieties)

PROCEDURE

Place a little grass extract in a small dish, and stand a stick of blackboard chalk on end in the extract. The liquid will be drawn up the chalk, and coloured bands of the various components will be formed on the chalk

Experiment E2.20**Separation of dyestuffs by thin layer chromatography****APPARATUS**

Each pupil or pair of pupils will need:

Two or three microscope slides

Beaker, 100 cm³

Glass sheet to cover beaker

Glass stirring rod

Short length of iron wire

Access to a steam oven

Camag Kieselgel

Chromatography solvent (butanol, 60 volumes; ethanol, 20 volumes; 2M ammonium hydroxide, 20 volumes)

Mixture of inks (brown ink is a good mixture), or of an aqueous solution of dyestuffs, e.g. eosin, magenta, and malachite green

PROCEDURE

Place about 10 cm³ of water in the 100 cm³ beaker and gradually add sufficient Kieselgel to make a thin paste, stirring well with a glass rod.

Tip the paste onto two or three microscope slides, spreading it out with the glass rod and tipping the slides backwards and forwards until it is evenly spread. The paste should be thin enough for it to spread quickly and evenly. Leave the slides to stand on a flat surface for a few minutes when the layer will be set.

Activate the slides by placing them in a steam oven, or an electric oven maintained at about 110–120°, for half an hour. During this time, rinse out the beaker, dry it, add about 0.5 cm depth of the chromatography solvent, and cover it with a sheet of glass to allow the atmosphere inside to become saturated with the solvent vapour.

Remove the slides from the oven. Make a very small loop in a piece of iron wire, dip this into the mixture to be separated, and transfer a *small* drop to a point about 1 cm from one end of the slide. Place the slide so as to stand nearly vertically in the beaker, with the spot of mixture at the lower end. Other slides may also be put in; take care that they do not touch.

In about 20 minutes the solvent will have climbed to the top of the slides, separating the coloured mixtures as it does so.

Polyfilla can be used as an alternative to Kieselgel.

Chromatographic techniques – more advanced level

Experiment E2.21

Separation of volatile liquids by gas chromatography

In this experiment a mixture of hydrocarbons is separated, using Tide as the column filling, and town gas as the carrier gas.

APPARATUS

Each pupil or pair of pupils will need:

U-tube 80–100 cm long, 6–7 mm diameter

Jet about 2 mm diameter, brass or pyrex glass

Rubber tubes to join jet to U-tube, and gas supply to U-tube

Cylinder tall enough to hold U-tube

Dropping tube or teat pipette with fine jet

Splints or a taper

Stand and clamp

Tide detergent (Note. The Tide should be dried in a steam oven for a few hours to obtain a free-running sample, and then sieved to remove the finest particles)

Pupils will also require access to the following:

Pilot light for lighting gas jet

Clock with a seconds hand

Thermometer, -10° to $+110^{\circ}\text{C}$

Hot water

n-Pentane, n-Hexane, n-Heptane, and n-Octane

PROCEDURE

Fill the U-tube with the prepared Tide without any attempt at packing, and immerse it in a cylinder of water kept at about $50-55^{\circ}\text{C}$. Connect the apparatus to the town-gas supply and adjust the gas pressure so as to obtain a small blue flame about 0.5 cm high. If the gas pressure is too high a luminous flame will be obtained, and the supply must be turned down or the length of the column of Tide increased.

Switch off the gas supply, remove the rubber tube from the U-tube, and drop into the U-tube the smallest drop that can be induced to fall from a fine dropping tube of n-pentane. Replace the rubber tube, switch on the gas supply, light the gas at the jet, and measure the time interval until the gas flame becomes luminous. This luminosity persists for a time and then dies away.



Figure 2.10

Repeat the operation for each of the other hydrocarbons in turn. Typical results are as follows:

<i>Hydrocarbon</i>	<i>Luminosity appears after</i>	<i>Luminosity goes after</i>
n-Pentane	17 sec	32 sec
n-Hexane	„ 28 „	„ 45 „
n-Heptane	„ 63 „	„ 90 „
n-Octane	„ 180 „	„ 240 „

Next add a drop of a mixture of all four hydrocarbons to find out the efficiency of the column at separating them. Other suitable mixtures can be tried; a fraction from the distillation of crude petroleum (see experiment E2.13) or a sample of petrol (motor spirit). Other organic liquids suitable for investigation include diethyl ether, carbon disulphide, chloroform, carbon tetrachloride, benzene, toluene, ethyl benzene, etc.

Experiment E2.22**Breakdown of proteins and chromatographic identification of the amino-acids formed****APPARATUS**

Each pupil or pair of pupils will need:

Source of protein (egg albumin, milk powder, hair, nail clippings, gelatin, cheese)

Pepsin powder

3 per cent hydrochloric acid solution

Small beaker or boiling tube, 150 × 25 mm

Water bath (250 cm³ beaker) at 36–37°C

Concentrated hydrochloric acid

Small distilling flask

'Cold finger' condenser

Water pump

Rubber bung and capillary

Porous pot

Paper for chromatography

Propan-1-ol + 20 per cent water

1 per cent ninhydrin in alcohol

Gas jar 30 cm high and lid

Thermometer –10° to +110°C

Stirring rod

Apparatus for spotting (platinum wire loops or capillary tubes)

Dip tray or mouth spray

Amino-acid solutions (see note 1 below)

Note 1. It is necessary to use different amino-acids as controls with different proteins, but the following would be fairly typical:
milk: leucine, glutamic acid, lysine, and serine.

Note 2. As proteins may be made up of over twenty amino-acids, it will not be possible to get a complete chromatographic separation by this method.

PROCEDURE

Using enzymes for hydrolysis. For this hydrolysis use powdered milk, egg albumin or gelatin as a source of protein, and pancreatin powder as the enzyme. The enzyme must be used fresh, but it will keep in a refrigerator. Digest about 0.1 g of protein material with a pinch of enzyme in a few drops of 3 per cent hydrochloric acid solution at 36–37°C for about one hour. The amino-acids can then be separated using the chromatographic method described below.

Using strong hydrochloric acid for hydrolysis. For this hydrolysis add about 0.1 g of protein-containing material (gelatin, hair, powdered milk, etc.) to 5 cm³ of hydrochloric acid made by diluting 30 cm³ of concentrated acid with 2 cm³ of water. Place the mixture in a small distilling flask, add a small piece of porous pot to promote even boiling, and fit the flask with a 'cold finger' as a reflux condenser.

Boil the contents of the flask under reflux for an hour, and at the end of this time, remove the 'cold finger' and place a rubber stopper in its place. Connect the side-arm of the flask to a water pump and evaporate the contents of the flask under reduced pressure until they have become a syrup. Stir in 1 cm³ of water, and evaporate again under reduced pressure. Dissolve this syrup in 0.5 cm³ of water, and use the solution obtained (which should be as concentrated as possible) for separation by chromatography.

Chromatographic identification of amino-acids

Place a spot of the solution 1 cm from the bottom of a piece of chromatographic paper 25 cm deep by 10 cm wide. Place spots of approximately 1 per cent solutions of known amino-acids in line with the spot of unknown and about 2 cm from each other, and label them in pencil, not ink. Roll the paper into a cylinder and fasten it at the top with a paper clip. Allow the spots to dry and stand the cylinder spots downwards, in a gas jar containing propan-1-ol, with about 20 per cent of water added. Close it with a ground glass lid and leave it for 24 hours or until the liquid has risen three-quarters of the way up the paper. Remove the paper, dry it in air, spray it with 1 per cent ninhydrin in ethanol and develop it by heating for a few minutes over an electric fire or in a drying cabinet.

The positions taken up by the various amino-acids will be indicated by violet spots. Compare the positions of the spots obtained from the protein hydrolysate with those of the known amino-acids, and thus identify the constituents of the hydrolysate.

Note: The breakdown of starch and the chromatographic identification of the products are described in experiment E10.9.

Separation of elements from compounds

Experiment E2.23

Separation of lead from an ore

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.6)

Bunsen burner and asbestos square

Tongs

Asbestos paper strip, about 7×2 cm

Sample of crushed lead ore, e.g. lead carbonate ('cerussite')

Powdered wood charcoal

PROCEDURE

A bead of lead can be obtained from cerussite by heating a mixture of a little crushed ore with charcoal on a piece of asbestos paper. Mix the ore and the charcoal thoroughly and heat with a roaring Bunsen burner.

Experiment E2.24

Separation of iodine from seaweed

A This experiment should be done by the teacher.

APPARATUS

Bunsen burner and asbestos square

Tripod and tin lid

Beaker, 250 cm³

Separating funnel, 100 cm³

Evaporating basin

Access to fume cupboard

20-volume hydrogen peroxide

Carbon tetrachloride

Distilled water

1M sulphuric acid

Supply of ribbon seaweed (*Laminaria*—obtainable from biological supply agencies)

PROCEDURE

Iodine may be obtained from ribbon seaweed in the following manner. Collect and dry about a dozen 50 cm lengths of the seaweed and heat them strongly on a tin lid supported by a tripod in a fume cupboard until they are reduced to ash; about a dessertspoonful will remain. Boil the ash with about 20 cm³ of distilled water in a small beaker, and filter. If hydrogen peroxide solution is added to the acidified cool filtrate, a deep brown colour of iodine is formed as hydrogen peroxide

liberates iodine from the iodide ions present. Transfer the mixture to a separating funnel and extract it using carbon tetrachloride. Note the colour of the carbon tetrachloride solution. If desired, the solvent may be allowed to evaporate at room temperature by placing it in an evaporating basin in a fume cupboard. Crystals of iodine will be formed.

B A simple method of iodine extraction.

APPARATUS

Each pupil or pair of pupils will need:

Beaker, 100 cm³

Test-tube, 150 × 25 mm

Bunsen burner, tripod, gauze, and asbestos square

Filter funnel and filter paper

About 1 g of dried seaweed (*Laminaria*)

20-volume hydrogen peroxide

1M sulphuric acid

Carbon tetrachloride

Distilled water

PROCEDURE

Gently boil about 1 g of dried seaweed with about 10 cm³ of distilled water in the beaker for a few minutes. Filter the solution and collect the filtrate in the 150 × 25 mm test-tube. To the filtrate in the test-tube add about 2 cm³ of molar sulphuric acid and about 10 cm³ of 20-volume hydrogen peroxide. Now add about 3 cm³ of carbon tetrachloride and shake. The free iodine dissolves in the carbon tetrachloride layer causing it to go purple. There is not sufficient iodine in the layer to form crystals if the carbon tetrachloride is evaporated, but the class results could be pooled in order to attempt the crystallization, which should be done in a fume cupboard.

Experiment E2.25

Separation of copper from an ore

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A1.6)

6-volt battery or alternative d.c. supply

Two carbon electrodes with connecting wires and crocodile clips

Beaker, 100 cm³

Spatula

Bunsen burner and asbestos square

Tongs

Access to filtration apparatus

Pestle and mortar

Asbestos paper strip, about 7×2 cm

A clean nail

1M sulphuric acid

Crushed copper pyrites

Before the experiment begins, the teacher should ensure that his sample of copper pyrites really contains some copper, as samples of copper pyrites are sometimes confused with iron pyrites. To do this, heat about a gram of the powdered mineral with 0.5 cm^3 of aqua regia (concentrated hydrochloric acid 3 volumes, concentrated nitric acid 1 volume) in a test-tube in a fume cupboard. Pour the mixture into about 10 cm^3 of water and add an excess of ammonia solution. A blue colour indicates the presence of copper.

PROCEDURE

Heat strongly a spatula measure of the crushed ore on a small piece of asbestos paper in the air for a few minutes. Tip the product into about 15 cm^3 of molar sulphuric acid in a beaker, heat the mixture for five minutes, and then filter it. A pale blue solution should result. The solution can be shown to contain copper by dipping a clean nail into it so that half the nail is in the solution—a coppery deposit should form.

It is also possible to get copper out of the blue solution using electricity. Immerse two carbon electrodes in the solution and connect them to a 6V d.c. supply. A deposit of copper should be visible in a few minutes.

Experiment E2.26

Recognition of some elements using flame tests

A. *A simple method*

APPARATUS

Each pupil or pair of pupils will need:

Watch-glass

Nichrome wire, with a cork fixed to one end

Bunsen burner and asbestos square

Compounds of sodium, potassium, calcium, strontium, barium, lead, and copper

Concentrated hydrochloric acid

PROCEDURE

To carry out flame tests, mix a little of the salt of the metal with concentrated hydrochloric acid in a watch-glass, moisten one end of a piece of nichrome wire about 5 cm long with this solution, and then hold the wire in a non-luminous Bunsen flame. The wire may be

cleaned between each test by dipping it into concentrated hydrochloric acid on a watch-glass and heating it to redness.

B. *Method using a spectroscope*

APPARATUS

In addition to the apparatus for part (a) above, each pupil or pair of pupils will need:

Small wooden or cardboard box

Razor blade, double-edged

Sellotape

Transparent replica diffraction grating, 15×15 mm approximately

Scissors

White paper

Instructions for making a simple spectroscope

First obtain a small wooden box, such as a flat cigarbox. Drill two small holes about 1 cm in diameter opposite each other, one in each of the two short sides of the box near the corners. Make one into a narrow slit (running from top to bottom of the box) by sticking two pieces of razor blade on the outside with Sellotape. Over the other hole, but on the inside of the box, fix a small piece of a transparent 'replica' diffraction grating. Finally, stick a strip of white paper inside the box along the short side in which the slit was made.

When the box is held so that a bright light enters the slit, and the grating is close to the eye, a spectrum is seen inside the box on the white paper strip.

PROCEDURE

Repeat the procedure described in part (A) above, but look at the flame through a spectroscope. Have the Bunsen burner flame as close as possible to the slit of the spectroscope without risking damage to it. Each time the moistened wire is put into the flame, the spectrum will appear for a few moments.

Describe the spectrum seen for each element in terms of the lines that can be seen – how many lines, what colour, and how they are grouped. It will be evident that each element has a characteristic spectrum by which it can be identified.

Chapter 3

Air and combustion

Introduction

An enquiry into the composition of the air is a useful subject for a simple chemical investigation. In this chapter a number of experiments which can be used for this purpose are described, all of which are suitable for use at an early stage of the pupils' progress.

The chapter also includes experiments which can be used to investigate some special aspects of combustion. Examples include the burning of a candle; the burning of town gas, leading to the formation of water and its investigation (see Chapter 4); the similarities between burning and breathing; and an investigation of rusting.

Comparison of the burning ability of different metals leads to the idea of a reactivity series, which is developed further in Chapter 7. Testing the products of combustion of various elements with indicators introduces the ideas of types of elements (Chapter 6), and of acidity-alkalinity (Chapter 8).

References

Two contrasting ways by which an investigation of air and combustion can be carried out are described in the Sample Scheme, Stage I, *Basic Course*, at the following points:

Stage I A, Topic 3: 'Finding out more about the air', and

Topic 4: 'The problem of burning'

Stage I B, Topic 4: 'The major gases of the air', and

Topic 9: 'Investigation of some common processes involving the air'

List of experiments

E3.1 What happens when metals are burnt?

E3.2 The combustion of phosphorus in a closed container

E3.3 To find the proportion of air responsible for burning, using copper:

- a. syringe method
 - b. method for use if syringes are not available
- E3.4 To find the proportion of air responsible for burning, using phosphorus
- E3.5 To find out if the gas released by red lead (or potassium permanganate) is the same as the active part of the air
- E3.6 How active air was first obtained by Priestley
- E3.7 How mercury(II) oxide was obtained by Lavoisier
- E3.8 What happens when substances are burned in oxygen?
- E3.9 An investigation of the burning of a candle
- E3.10 Does a change in weight take place when a candle burns?
- E3.11 An investigation of the burning of town gas:
 - a. simple experiment
 - b. more efficient experiment
- E3.12 A comparison of burning and breathing
- E3.13 An investigation of rusting

Experiment E3.1

What happens when metals are burnt?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A5.1b)

Crucible lid or piece of broken porcelain

Asbestos paper strip, approximately 7×2 cm

Tongs

Steel knitting needle or rod

Bunsen burner and asbestos square

Four test-tubes, 100×16 mm

Small quantities of lead foil, zinc dust, calcium turnings, and sodium

Universal Indicator solution

PROCEDURE

1. Heat a piece of sodium the size of a rice grain on a crucible lid or piece of broken porcelain held with tongs. Note the ease with which it burns and the colour of the flame.

Note: The pupils must be warned about the precautions needed when handling sodium and teachers should cut and give out the pieces of sodium themselves. Store small pieces of the metal in liquid paraffin and remove them with a pair of tongs. The pieces of sodium must then be cleaned by dropping them into a beaker containing petroleum ether. Remove the pieces from the beaker and allow them to dry on filter paper. They may be cut with a pen-knife but must not be touched nor allowed to come into contact with any water. Place and leave any small pieces not used in a beaker containing methylated spirits until effervescence stops; the liquid may then be safely thrown away.

2. Heat one or two small pieces of lead foil on a crucible lid or piece of broken porcelain, held with tongs, for several minutes. Stir the molten globule with a steel knitting needle to expose the metal to the air. A yellow coloration will appear. Allow the material to cool, and examine the residual ash.

3. Place a spatula measure of zinc dust near the edge of a strip of asbestos paper held with tongs and heat it. After the zinc catches fire and burns, take the paper out of the flame and gently prise up the yellow crust that forms, with a steel knitting needle. The metal, now exposed to the air again, burns, forming the white fluff of 'Philosopher's Wool'.

4. Hold a piece of calcium (usually supplied in the form of turnings) with tongs and heat it strongly in a roaring Bunsen flame. It is not easy to ignite the calcium because the tongs conduct the heat away and make it difficult to raise the temperature of the calcium to its ignition point. However, by using asbestos paper to sandwich the calcium turning this difficulty can be avoided. Note the colour of the flame and the nature of the product.

Examine the action of water on the 'ashes' formed from all these metals, and test the resulting liquid with Universal Indicator solution.

Note: An amplified version of this experiment is given as experiment E.7.1

Experiment E3.2

The combustion of phosphorus in a closed container

If yellow phosphorus is ignited in a closed, weighed container no increase in weight is observed. On opening the container, however, there is an inrush of air and the weight increases.

Details of this experiment are given in Fowles, *Lecture Experiments in Chemistry*, 6th edition, p. 23. (Bell).

Experiment E3.3

To find the proportion of the air responsible for burning, using copper

A. Syringe method

This experiment may be done by the teacher, or by the pupils if enough syringes are available.

APPARATUS

a.

Two stands and syringe clamps

Two syringes, 100 cm³ glass

15 cm length of 7 mm o.d. transparent silica tubing

Bunsen burner and asbestos square

Thick-walled rubber tubing

Three-way stopcock in capillary tubing (optional)

Two pieces of hard-glass rod, 5 mm diameter

Copper made by freshly reducing wire-form copper(II) oxide

or b.

Two stands and clamp

Syringe, 100 cm³ and syringe clamp

Hard-glass test-tube, 150 × 25 mm, fitted with bung and straight delivery tube

Rubber connecting tubing

Bunsen burner and asbestos square

Spatula

Vaseline

Copper made by freshly reducing wire-form copper(II) oxide

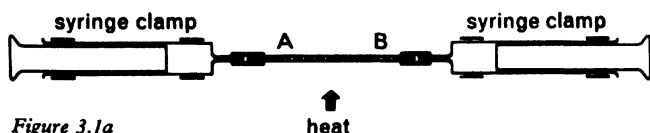


Figure 3.1a



Figure 3.1b

PROCEDURE

Place some copper (preferably reduced wire-form copper(II) oxide) in a horizontal silica tube, between two syringes. There must be no gaps between the syringes and the tube. Fill in the ends of the combustion tube with pieces of hard-glass rod which are slightly smaller in diameter than the inside diameter of the tube (see diagram *a*), and fill one syringe with 100 cm³ of air. *At this point the apparatus must be tested for air-tightness.*

Heat the copper at A fairly vigorously, and when hot move the syringe plungers slowly backwards and forwards whilst the heating is continued. After about two minutes have elapsed stop the heating and cool the combustion tube with a damp cloth. Note the volume reading on the syringe. Repeat the heating of the copper at A for a further minute, cool as before, and continue this procedure until the volume reading is constant. Now use the Bunsen burner to heat the copper at B (see diagram). Ideally the copper at B will remain bright and untarnished, and the volume reading constant, indicating that the active part of the air has been removed completely.

The volume occupied by the active part of the air is the difference between the first and the final volume readings. From these two volumes the percentage of the active part of the air can be calculated.

This experiment may be enlarged in scope by weighing the tube with copper before and after the reaction. This will allow the density of the gas absorbed to be worked out, in which case the atmospheric temperature and pressure should be recorded.

The experiment may be performed with only one syringe if the apparatus shown in diagram *b* is used. In this case the syringe should be lightly greased with Vaseline and contain about 30 cm³ of air initially. Two or three spatula measures of reduced wire-form copper(II) oxide are needed in the test-tube.

B. Method for use if syringes are not available

APPARATUS

Each pupil or pair of pupils will need:

Two hard-glass test-tubes with rubber bungs carrying delivery tubes about 50 cm long, and of 7-8 mm bore

Two small troughs

Two Bunsen burners

Two stands and clamps

Fine copper wire, gauze, or foil

PROCEDURE

One piece of apparatus 'A' (see figure 3.2) is set up containing copper in the form of fine wire gauze or foil, and another completely identical piece 'B' but containing no copper is set up beside it in such a position that the Bunsen burners can be switched from one apparatus to the other rapidly.

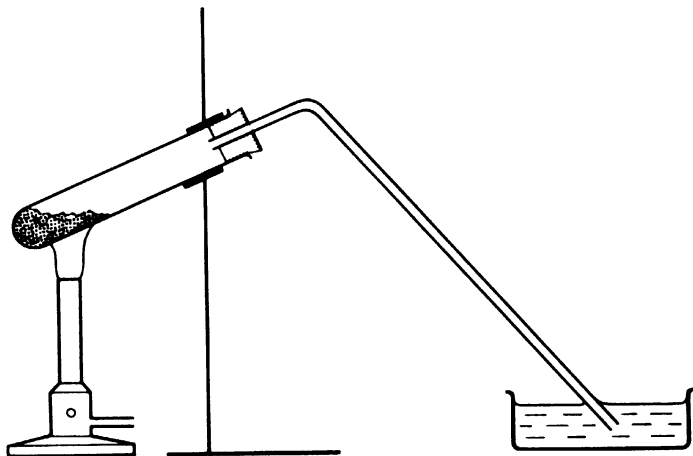


Figure 3.2

Both pieces of apparatus must be clamped firmly to remain at exactly the same height above the bench during the heating. Set the air ring of the Bunsen burners to give the hottest (roaring) flames and if the flame sizes are not identical try to make them so by adjusting the gas supply or exchanging burners.

Try to keep all the heating and cooling conditions of 'A' and 'B' the same.

Begin heating both test-tubes at the same moment and observe the effects. Heat until no more bubbles are delivered through the water from either piece of apparatus, and then quickly change the burners over and continue heating.

After a time it will be found that water begins to be drawn up by the delivery tube of the 'A' apparatus. When no more is drawn up, quickly change the burners over again and continue heating until steady states have again been obtained, and then at the same moment remove both burners.

Noting the colour of the copper, allow both pieces of apparatus to cool slowly and watch the effects.

Water will be drawn into the delivery tube of apparatus 'B' as the air inside cools; any *extra* amount of water drawn into delivery tube 'A' will indicate the quantity of air used up by the burning copper.

Experiment E3.4

To find the proportion of the air responsible for burning, using phosphorus

A simple method is described in Fowles, *Lecture Experiments in Chemistry*. 6th edition, p. 63 (Bell).

Experiment E3.5

To find out if the gas released by red lead (or potassium permanganate) is the same as the active part of the air

In this experiment the active part of the air is compared with the gas obtained by heating red lead (or potassium permanganate) and is found to be the same. Density measurements show it to be oxygen.

The density of the active part of the air can be found using the method of experiment E3.3. If the copper is weighed before and after it is heated, the density of the active part of the air is given by:

$$\frac{(\text{weight of copper oxide} + \text{tube}) - (\text{weight of copper} + \text{tube})}{\text{decrease in volume of air}}$$

the units being g cm^{-3} .

The density of the gas obtained by heating red lead (or potassium permanganate) can be found using the method of experiment E1.5, a quantitative version of which is now described. Results are most easily obtained using the syringe method, but if syringes are not available the gas must be collected over water in a gas jar.

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A3.2)

Hard-glass test-tube, 100×16 mm or 125×16 mm (carefully dried)

Use of a direct reading balance

Bunsen burner and asbestos square

Stand and clamp

Red lead or potassium permanganate which has been dried in a desiccator or steam oven

and *a*.

if syringe method is used:

Stand and syringe clamp

Bung with 8 cm glass tube 7 mm o.d. bent through about 120° (see diagram 'a')

Glass syringe, 100 cm³ glass, or 50 cm³ plastic

Thick-walled rubber tubing

or *b*.

if no syringes are available:

Bung and delivery tube for test-tube

Trough, or other suitable vessel

Gas jar and lid

Measuring cylinder, 100 cm³

Beehive shelf

Vaseline

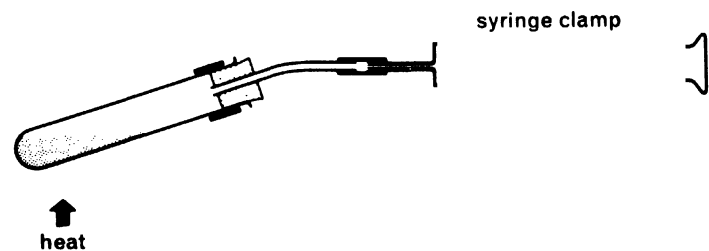


Figure 3.3a

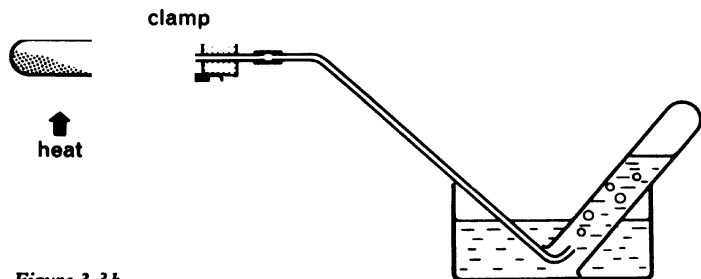


Figure 3.3b

PROCEDURE

a. Set up the test-tube and syringe in the way shown in the diagram. Place about 4 g of well-dried red lead (or 0.5 g of potassium permanganate), enough to produce about 80 cm³ of oxygen, in the dry test-

tube and weigh it to the nearest milligram. Heat the test-tube gently as too strong a heating of red lead in glass will crack the test-tube. When no more gas is evolved, allow the apparatus to cool before measuring the volume of the gas evolved.

b. If no syringe is available place about 8 g of well-dried red lead (or 2 g of potassium permanganate) in a dry test-tube and weigh it to the nearest milligram. Place a delivery tube in the test-tube so that the other end is immersed in water in a trough and support a gas jar full of water over it to collect the gas evolved. Heat the test-tube until no more gas is evolved. Close the submerged open end of the gas jar with a vaselined lid and stand the jar upright on the bench.

Stop the heating and remove the delivery tube carefully from the test-tube before any water is drawn up.

To measure the volume of gas delivered to the gas jar fill a measuring cylinder to its top mark with water, and pour water from it into the gas jar. The volume of water poured out of the measuring cylinder is the volume of gas obtained by heating the red lead, measured at room temperature and pressure. All this is more easily done, of course, if a syringe is used.

To measure the weight of this volume of gas, wait until the test-tube is cool, then unclamp it and examine it carefully for any fragments of rubber from the bung which may be sticking to it. If any are found, they must be removed. The test-tube should now be reweighed accurately, and the loss in weight calculated. This is the weight of gas evolved.

Treatment of class results

If a graph is constructed having axes labelled 'Volume of gas in cm^3 ' and 'Loss in weight in g' then a straight line can be drawn connecting the origin (0, 0) with the point representing the experimental result, and this can be produced so as to show the weight of (say) 1 litre of the gas.

The teacher can plot such a graph on the blackboard, and insert points for each pupil's result.

If the pupils are not familiar with graphs some time must be spent on explaining their meaning. The pupils will see from the graph that their results all vary, but they vary (except for odd exceptions) round an average value. This point is very significant. Some time should be

spent in discussing the reasons for the variations and the fact that there is really no 'right' answer, but only one which we can say is correct within certain limits. The pupils will thus realize:

1. The results they get vary and are bound to vary.
2. You can see from the variation approximately how certain you can be about your results.

The value of the density of the gas obtained from the graph can be compared with accepted values of densities for several gases including oxygen.

Experiment E3.6

How active air was first obtained by Priestley

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A3.3)

Four test-tubes, 100×16 mm

Bung to fit test-tube carrying delivery tube

Small trough

Stand and clamp

Bunsen burner and asbestos square

Splints

Spatula

Mercury(II) oxide

PROCEDURE

In this experiment pupils each make a small quantity of oxygen by Priestley's method, but using school apparatus.

Place a spatula measure of mercury(II) oxide in a 100×16 mm test-tube which is fitted with a bung carrying a delivery tube. Clamp the test-tube so that the lower end of the delivery tube is held beneath the surface of the water in a small trough. Fill two or three other test-tubes with water taken from the trough and invert them in it.

Heat the mercury(II) oxide gently using a low non-luminous Bunsen burner flame. Take care to heat only the lowest part of the test-tube. The gas evolved should be collected in the inverted test-tubes in the trough; the first test-tubeful may be tested but it will contain, mainly, displaced air.

A ring of droplets of condensed mercury will be seen above the heated portion of the test-tube. When no more gas is evolved, the delivery tube should be removed from the trough *before* heating is stopped, otherwise water will be forced into the hot test-tube.

The drops of mercury may be examined and then returned to the laboratory stock of impure mercury; they should not be heated as mercury vapour is very poisonous. The oxygen can be tested with a glowing splint.

Experiment E3.7

How mercury(II) oxide was obtained by Lavoisier

This experiment **must** be done by the teacher.

APPARATUS

Conical flask, 250 cm³

Filter funnel

Tripod and sand tray

Bunsen burner and asbestos square

Beaker, 250 cm³ or filter funnel and paper

Stand and clamp

Test-tube, 125 × 16 mm

Spatula

Splints

Mercury

An efficient fume cupboard is also required

PROCEDURE

Mercury vapour is very poisonous indeed, and *the experiment must be carried out in a well-ventilated fume cupboard.*

Place just enough mercury in a 250 cm³ conical flask to cover the bottom, and stand the flask in a sand tray placed on a tripod over a Bunsen burner. Place a funnel in the mouth of the flask, with its stem inside the flask; this will act as a condenser for mercury vapour.

A small flame should be lit under the sand tray and kept burning for at least a whole day; two or three days or even a week is better.

At the end of this time the flask should be allowed to cool and the oxide floating on the mercury removed. This may be done by transferring the contents of the flask to a beaker, and scraping the crust to one side; alternatively the contents can be poured into a filter paper folded in the normal way in a filter funnel but with a small pin-hole at the lowest point. The mercury will run through but the oxide coating will remain on the paper.

The oxide, which is not red at this stage but is grey owing to mixture with mercury, can then be transferred to a test-tube and heated very gently, when any mercury present will be distilled to a higher place in the test-tube, leaving the mercury(II) oxide. (*Avoid breathing any mercury vapour.*) Using care, the mercury can then be separated and the mercury(II) oxide tapped out of the tube.

Its identity can be shown by the action of heat followed by the glowing splint test.

Experiment E3.8

What happens when substances are burned in oxygen?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A4.3)

Four hard-glass test-tubes, 150×25 mm, and corks in test-tube rack

Small trough

Combustion spoon

Bunsen burner and asbestos square

Teat pipette

Stick or small piece of wood charcoal

Powdered sulphur

Steel wool

Magnesium ribbon (about 3 cm)

Universal Indicator solution

The teacher will need:

Cylinder of oxygen

Length of rubber tubing to lead oxygen from cylinder to pupil's trough

Lime water

Note: If the oxygen is being supplied from a cylinder, the pupils can fill test-tubes with water, and invert them over water in a small trough. They can then bring their troughs and test-tubes to the oxygen cylinder to have the test-tubes filled with oxygen. On return to their places they can remove the test-tubes from the water quickly closing them with corks, and place them in a rack ready for use.

Alternatively prepare a supply of 150×25 mm test-tubes filled with oxygen and closed with a cork, before the lesson.

If an oxygen cylinder is not available, or the teacher prefers to let each pupil prepare his own supply of oxygen, a convenient method is by the addition of 20-volume hydrogen peroxide solution to granules of

manganese dioxide, using the apparatus shown in the diagram. The manganese dioxide catalyses the decomposition of the hydrogen peroxide to water and oxygen, and this affords a safe and steady supply of the gas.

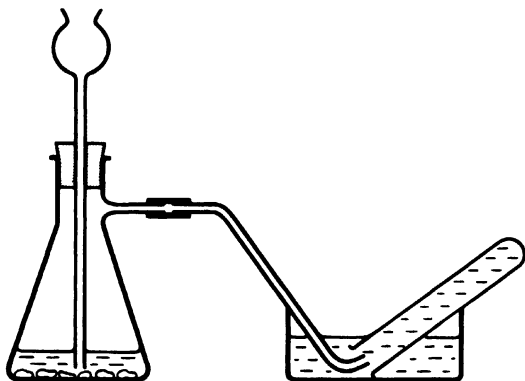


Figure 3.4

PROCEDURE

Investigate the burning of the elements mentioned below. Do not remove the test-tube cork until the burning sample is ready to be inserted, and make sure that the pieces of the elements are small enough to be placed easily and quickly into the test-tubes.

After each experiment, place a few drops of Universal Indicator and a little water in the test-tube, insert the cork, and shake the tube to mix the contents.

a. Carbon. Hold one end of a stick of wood charcoal in tongs, or place a small piece of wood charcoal in a combustion spoon. Heat the charcoal to redness in a Bunsen burner flame and then insert it into a test-tube of oxygen. It will be seen to glow much more strongly.

This experiment can be performed twice, the first time so that Universal Indicator can be added to the product, and the second time for the addition of lime water, so as to introduce this test for carbon dioxide.

b. Sulphur. Pick up some powdered sulphur by thrusting a slightly moistened pipe-cleaner into a pile of the powder; alternatively place some sulphur in a combustion spoon. Ignite the sulphur and plunge it into a test-tube of oxygen. A bright blue flame will be seen.

c. *Iron.* Wrap a little iron wool round a combustion spoon and make it red hot in a Bunsen burner flame. Push it into a test-tube of oxygen. Brilliant sparkles will be seen.

d. *Magnesium.* Wrap a short piece of magnesium ribbon round a combustion spoon. Ignite it and place it quickly in a test-tube of oxygen supported in the rack and *not* held in the hands. An exceedingly bright flame will be seen. Do not look at it directly as your eyes may be harmed.

The teacher may note that the results of the use of Universal Indicator will introduce the concept of acidity-alkalinity; if preferred litmus solution would also serve.

The idea of two sorts of elements, those that have acidic oxides and those that have basic oxides, opens up the possibility of classifying elements by their chemical properties.

Experiment E3.9

An investigation of the burning of a candle

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A4.1a)

A short candle, about 5 cm long

A large beaker, approximately 400 cm³ with no spout

Cobalt chloride paper

Universal Indicator paper

Watch-glass

Splint

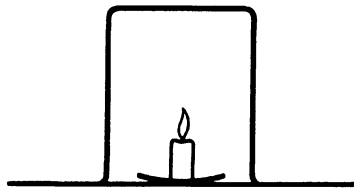


Figure 3.5

PROCEDURE

Burn a candle on a watch-glass in a confined volume of air under a large beaker, placed on the bench. The candle soon begins to go out. Before it does so, admit more air by raising the beaker slightly at one side. Note the formation of mist on the beaker. Test it with cobalt chloride paper. Test the remaining gas with a glowing splint. The splint will go out. (Note that despite the fact that the splint and the candle flame are extinguished there is still a significant amount of oxygen left in the

beaker. You can check this by analysis with pyrogallol or copper, using a syringe full of gas and proceeding as described in experiment E3.3, part A.)

Cobalt chloride paper is used to test the mistiness to see if it is water. If it is tested with litmus or Universal Indicator paper, it is found to be acidic owing to the formation of carbon dioxide.

Experiment E3.10

Does a change in weight take place when a candle burns?

This experiment should be done by the teacher.

APPARATUS

Thistle funnel with bent stem

U-tube, 10 cm limbs fitted with bungs and delivery tube

Filter pump

Stand and clamp

Watch-glass

Candle

Glass wool and tongs or forceps

Soda lime, granular form

Access to balance

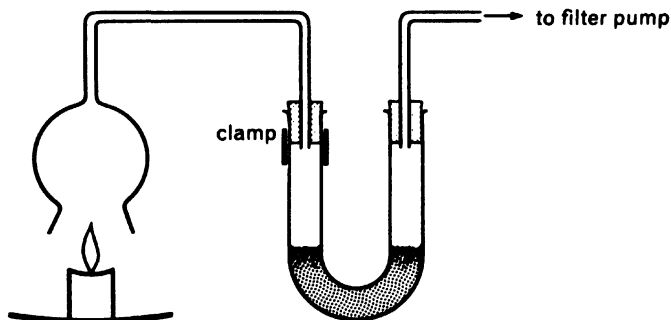


Figure 3.6

PROCEDURE

Loosely pack the U-tube with soda lime granules to a depth of about 5 cm in each limb, holding the granules in place by small tufts of glass wool. Fit the thistle funnel and delivery tube and weigh the entire apparatus complete with the candle and watch-glass.

Support the apparatus by a clamp and connect it to the filter pump. Light the candle and turn on the filter pump. Allow the candle to burn

steadily for two or three minutes before it is extinguished and at the same time disconnect the pump. Do not turn the filter pump off. When the apparatus has cooled, reweigh it together with the candle and watch-glass.

Finally, repeat the experiment without lighting the candle but maintaining the same rate of flow by reconnecting the filter pump for the same length of time. This increase in weight will indicate the amount of moisture and carbon dioxide absorbed from the atmosphere during the combustion of the candle.

The total weight will probably increase by at least 0.3 g in three minutes.

Experiment E3.11

An investigation of the burning of town gas

A. *By a simple experiment*

This experiment **must** be done by the teacher.

APPARATUS

Glass jet and rubber tube to connect to gas supply

Round bottomed flask, 250 cm³

Cobalt chloride paper

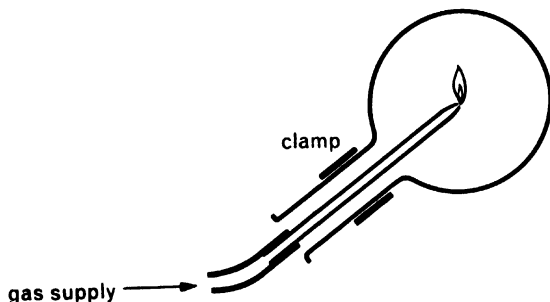


Figure 3.7

PROCEDURE

Light a jet of town gas at the end of a glass tube and turn down the gas till the flame is about 1 cm long. Insert the tube into a flask (250 cm³ is convenient) preventing the entry of air with the hand. The flame goes out. Note the formation of mist on the flask.

Test the mist with cobalt chloride paper.

Caution: If after one demonstration it is thought necessary to repeat the experiment, remember that the flask may now contain an explosive mixture of town gas and air.

B. By a more efficient experiment

This experiment **must** be performed by the teacher.

APPARATUS

U-tube or calcium chloride tube

Thistle funnel with stem bent as in diagram, fitted with bung for the test-tube

Test-tube with side arm, 125×16 mm

Beaker, 250 cm^3

Two stands and clamps

Filter pump and connecting tubing

Glass jet and connecting tubing

Test-tube, 100×16 mm

Thermometer, -10° to $+110^\circ\text{C}$

Bunsen burner and asbestos square

Silica gel or anhydrous calcium chloride

Anhydrous copper sulphate or cobalt chloride paper

Access to gas tap

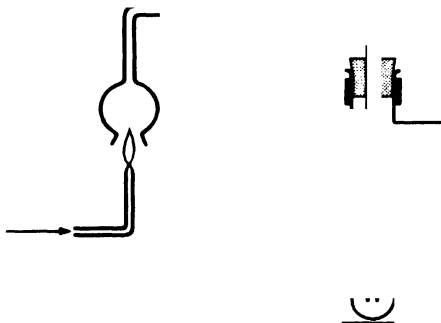


Figure 3.8

PROCEDURE

First dry the town gas by passing it through a tube containing silica gel or anhydrous calcium chloride, and then burn it at a jet. Have the flame about 3 cm high and allow it to burn beneath a thistle funnel as illustrated in the diagram. Keep a steady flow of the burnt gas passing through the apparatus using a filter pump. A liquid soon condenses in

the side-arm test-tube which is cooled in a beaker of water. After about ten minutes 2 or 3 cm³ of liquid will have collected, which should be sufficient to check its boiling point and action on cobalt chloride paper or on anhydrous copper sulphate. The cooling will be more effective if ice is added to the beaker of water.

Experiment E3.12

A comparison of burning and breathing

Part 1. Burning some carbon compounds

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B9.1)

Teat pipette

Four test-tubes, 100 × 16 mm

Four test-tubes, 150 × 25 mm, fitted with corks

Combustion spoon

Bunsen burner and asbestos square

Wood

Paper

Polythene

Naphthalene

Sugar

Starch

Ethanol (industrial methylated spirits)

Wax taper

Lime water

Cobalt chloride paper

PROCEDURE

Take amounts of wood, paper, Polythene, naphthalene, sugar, starch, ethanol, and a portion of wax taper, and in turn ignite them on a combustion spoon, placing the spoon in a dry 150 × 25 mm test-tube containing air. Sugar and starch require strong heating before they will ignite.

As soon as the flame is extinguished, withdraw the spoon and cork the test-tube.

Test the gas in the test-tube for carbon dioxide by extracting a portion with a teat pipette and bubbling it through about 2 cm³ of lime water. The lime water will go milky. Confirm that the mist which condenses on the side of the test-tube is water by testing it with cobalt chloride paper.

As an example of burning another carbon compound, the teacher can perform experiment E3.11(a).

Part 2. An investigation of breathing

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B9.2)

Two test-tubes, 100×16 mm

Side-arm test-tube with cork or bung and glass delivery tube (see diagram)

Filter pump and connecting tubing

Thermometer, -10° to $+110^\circ\text{C}$

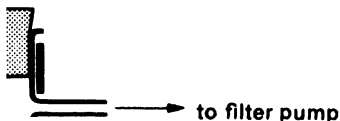
Beaker, 100 cm^3

Stand and clamp

Ice

Lime water

Anhydrous copper sulphate or cobalt chloride paper



lime water



Figure 3.9

PROCEDURE

1. Breathe out slowly and steadily through a piece of glass tubing or a drinking straw into some lime water in a 100×16 mm test-tube. Note the time required for the solution to turn milky (this may be about ten seconds).

Next, draw air through some lime water using a filter pump, as in the diagram. The volume of lime water should be the same as that used in the breathing experiment, and the air should be drawn through at the same rate. Note the length of time which it takes for the milkiness to appear (this may be about ten minutes). Leave the filter pump on for the next experiment.

2. Carry out a similar pair of experiments with empty and dry test-tubes, this time placed in ice-cold water. Maintaining the same gas flow rates, breathe into one tube and draw air through the other and record the times taken for the condensation of detectable amounts of liquid in each case. Identify the liquid as water by the addition of anhydrous copper sulphate or by using cobalt chloride paper.

3. To find out about the difference in temperature between inspired and expired air, read a thermometer while in ordinary air, and then again while breathing on to the bulb gently, the bulb being held about 5 cm from the mouth. Body temperature may also be recorded from the closed palm of the hand.

A comparison between the processes of burning and breathing can now be made.

Experiment E3.13

An investigation of rusting

Part 1. To find out if air and water are necessary for rusting, and if salt water is more effective than ordinary water

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B9.3)

Four test-tubes, 100×16 mm, fitted with corks

Test-tube rack

Eight iron nails, about 3 cm long

Spatula

Beaker, 100 cm³

Tripod and gauze

Bunsen burner and asbestos square

Vaseline, or olive oil

Calcium chloride, anhydrous, small lumps

Cotton wool

Salt water (see Procedure)

Distilled water

PROCEDURE

Take four test-tubes. In the first put two ordinary wire nails followed by distilled water, enough to half-cover the nails. These nails are in contact with air and water, and will be the control experiment. Then place a cork loosely in the mouth of the test-tube to keep out dust, and prevent the water from evaporating unduly.

In the second tube place a few pieces of anhydrous calcium chloride, followed by a small plug of cotton wool and then two nails, finally corking the test-tube firmly. These nails will be in contact with air but no water.

In the third tube place enough boiled water to cover the nails completely. The water should have been boiled in a beaker for several minutes to expel dissolved air. Then put the nails in, and place a little Vaseline, or a few drops of olive oil, onto the water by means of a spatula. The Vaseline will melt and form an air-proof layer on the hot water, solidifying as the water cools. Place a cork loosely in this test-tube which now contains nails in contact with water only. The fourth test-tube is like the first, but contains salt water instead of distilled water.

Stand the four test-tubes in a rack and leave for several days, at the end of which time only the 'control' nails and those in contact with salt water will be rusty.

Part 2. To find out if oxygen or nitrogen in the air is responsible for rusting

*First method***APPARATUS**

Each pupil or pair of pupils will need:

Test-tube, 150 × 25 mm

Beaker, 100 cm³

Spatula

Iron filings or wool

Wood splint

PROCEDURE

Moisten the inside of the test-tube with water, sprinkle in about a spatula measure of iron filings, and rotate the test-tube horizontally so that the filings spread and adhere to the walls. Alternatively a small plug of moistened iron wool may be inserted in the upper part of the test-tube.

Invert the test-tube in a beaker about one-third full of water, using the beaker lip to support the inclined test-tube. The water levels inside and outside the tube should be the same and this level noted. Leave the apparatus to stand for a few days, adding water to make up for any lost by evaporation. The iron will rust and the water level will rise inside the tube, finally becoming steady. When the final water level inside the test-tube is noted, once more making the levels inside and outside the tube the same, it will be seen that only one-fifth of the air volume has been used up, suggesting that oxygen rather than nitrogen has been involved. The residual gas does not support combustion of a lighted splint, confirming that the gas used up during the rusting of the iron must have been oxygen.

Second method

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 100×16 mm, fitted with rubber bungs

Test-tube rack

Four nails

Bunsen burner and asbestos square

Tripod and gauze

Beaker, 100 cm^3

Distilled water

The teacher will require:

Cylinders of oxygen and nitrogen (delivery tubes attached to both)

PROCEDURE

Boil some distilled water in a beaker for a few minutes to expel dissolved air. Place two nails in a test-tube, and half cover them with boiled distilled water. Lead oxygen from a cylinder at a steady rate into the test-tube through a delivery tube dipping into the water, until it is thought that all the air is displaced. Remove the delivery tube, and quickly close the test-tube by means of a well-fitting rubber bung.

The entire operation should then be repeated but this time using nitrogen in place of oxygen.

Leave the two test-tubes for a week, at the end of which time the nails in contact with oxygen will be found to be rusty, whereas those in contact with nitrogen will not.

Part 3. What is rust?**APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B9.4)

Beaker, 100 cm³

Watch-glass, hard glass

Tripod and gauze

Bunsen burner and asbestos square

Test-tube holder

Test-tube, 100 × 16 mm

Spatula

Cobalt chloride paper, or anhydrous copper sulphate

Rust

PROCEDURE

Place a spatula measure of the dry rust in a dry 100 × 16 mm test-tube and heat it. Drops of water will be seen to condense on the cooler upper parts of the test-tube. This can be tested, using cobalt chloride paper or a little anhydrous copper sulphate. Examine the residue and note its appearance. Compare it with a sample of iron(III) oxide.

Chapter 4

Water

Introduction

Another simple chemical investigation can be carried out using water as the subject for study, and this chapter contains experiments which are suitable for this purpose. There are also a number of experiments which illustrate some of the special properties of water.

One method by which a simple investigation can be made is to begin by finding out what happens when various metals are placed in water (experiment E4.2). This will lead to the discovery of hydrogen, a gas which must have come from the water. An examination of its properties will show its inflammable nature, and, if some is collected in a dry container and ignited, a mist will be seen. This will prompt the question 'Is water really burnt hydrogen?' and this can be answered by experiment E4.4, in which hydrogen is burnt and the product is collected and identified as water.

References

Another investigation of water is described in the Sample Scheme, Part I, *Basic Course*, at the following point:
Stage I A, Topic 7: 'Water as a product of burning'

The properties of water are investigated more fully in the *Course of Options*, Option 1, 'Water'.

List of experiments

- E4.1 What happens when magnesium and water are mixed?
- E4.2 What happens when metals are placed in water?
- E4.3 How to make hydrogen in the laboratory
- E4.4 Is water formed when hydrogen burns?
- E4.5 What substances does water dissolve?

- E4.6 Measurement of the solubility of a solid in water
- E4.7 Supersaturation
- E4.8 The effect of adding salt to ice.
- E4.9 Does water contain dissolved gases?
- E4.10 What is the effect of detergents on water?
- E4.11 Comparison of light- and heavy-duty detergents
- E4.12 What ions cause hardness in water?
- E4.13 How hardness is formed in water
- E4.14 How hardness can be removed
- E4.15 Experiments with a charged conductor

Experiment E4.1

What happens when magnesium and water are mixed?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A7.2)

Test-tube, hard glass, 125×16 mm fitted with bung and short delivery tube

Bunsen burner and asbestos square

Stand and clamp

Teat pipette

Sand paper

Taper

Magnesium ribbon, about 15 cm long

Asbestos wool

PROCEDURE

Place sufficient loosely packed asbestos wool in a 125×16 mm hard-glass test-tube to fill it to a depth of about 3 cm. Add about 2 cm^3 of water, which will be as much as the asbestos will absorb completely, and clamp the tube horizontally.

Clean about 15 cm of magnesium ribbon by means of sand paper, form the cleaned ribbon into a compact cylinder and place it about 2 to 3 cm

from the end of the wet asbestos. Fit the bung carrying the short delivery tube.

Gently heat the part of the test-tube containing the magnesium by means of a non-luminous Bunsen flame until the magnesium first appears to melt. Just touch that part of the tube containing the wet asbestos with the flame so that some steam is generated and passes over the hot magnesium. A reaction occurs; and the gas which is produced can be burnt at the end of the delivery tube if a lighted taper is applied. Continue heating the magnesium and generating the steam until the reaction is complete.

When the tube has cooled the contents may be tipped out and examined. Notice the black markings on the walls of the test-tube. This is due to a reaction of magnesium with the glass to form magnesium silicide.

See also experiment E4.2, part 3.

Experiment E4.2

What happens when metals are placed in water?

APPARATUS

Each pupil or pair of pupils will need:

Beaker, large enough to hold an inverted funnel

Funnel, and quick filter paper

Test-tube, 150×16 mm

Small quantities of calcium (a turning), magnesium (about 10 cm of ribbon) and copper (a few turnings)

Universal Indicator solution

The teacher will need:

Plastic safety screen

Two plastic troughs

Penknife

Filter papers

Small quantities of sodium and potassium

PROCEDURE

1. Experiments on sodium and potassium should be done by the teacher.

Stand two plastic troughs side by side on the demonstration bench, each half-filled with water. Remove a piece of sodium from the bottle, free it from adhering paraffin oil by means of filter papers, and cut a small piece free from surface corrosion. A cube of side 2–3 mm is suitable. Place the sodium on the water in one trough.

Repeat the experiment with potassium, using the other trough.

Notice that sodium melts and skates about the surface of the water; potassium melts, skates about the surface of the water, and may even catch fire.

Pupils should not come too near this experiment in case pieces of molten metal jump out of the troughs.

Finally, put a few drops of Universal Indicator into each trough of water and notice the result.

The remainder of the experiment can be done by the pupils.

2. Place a small piece of calcium in a beaker half-filled with water. It will sink, and a stream of bubbles will be seen coming to the surface. Notice that the water turns 'milky'. When the action has ceased filter a little of the suspension into a test-tube and blow through it. It is lime water and will turn milky. Add some Universal Indicator solution to the water in the beaker.

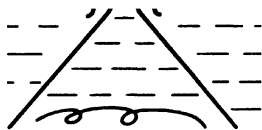


Figure 4.1

3. Arrange the beaker, funnel, and test-tube as shown in the diagram, placing under the funnel either about 10 cm of magnesium ribbon or a few copper turnings. Leave the apparatus for a week. Bubbles of gas will have collected in the test-tube above the magnesium, but not above the copper.

Note: An amplified version of this experiment is given in experiments E7.2 and E7.3.

Experiment E4.3

How to make hydrogen in the laboratory

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A7.4)

Filter flask, fitted with delivery tube, bung, and thistle funnel

Taper for igniting the hydrogen

Small trough

Four test-tubes, 150×25 mm, and corks

1M sulphuric acid

Zinc, granulated

Copper sulphate solution

Detergent solution

Caution: In no circumstances light the hydrogen as it comes out of the delivery tube.

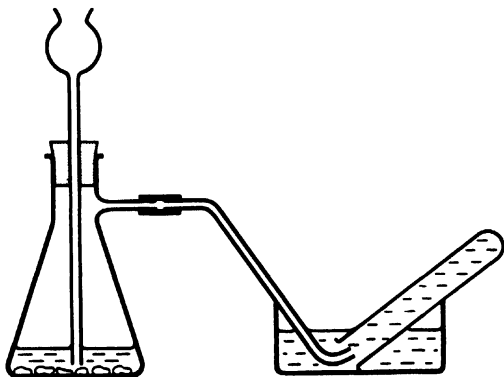


Figure 4.2

PROCEDURE

Place a few pieces of granulated zinc in the filter flask, add a few drops of copper sulphate solution, and assemble the apparatus. (The effect of the copper sulphate is to speed up the reaction.) Pour enough molar sulphuric acid down the thistle funnel onto the zinc to cover the bottom of the funnel stem, and collect the hydrogen which is generated in test-tubes over water in a small trough. The first two or three test-tubes full should be discarded as they will contain mainly displaced air.

Apply a lighted taper to a test-tube full of the gas. It will be found to burn, and thus its presence can be revealed. Find out its density, relative to the air, by 'pouring' experiments, using a lighted taper to find out where the gas has gone.

By pouring upwards into a dry test-tube and igniting, the hydrogen mixed with air will explode with a characteristic 'pop', and a mist will be seen on the inside of the test-tube.

Blow some soap bubbles by holding the delivery tube of the apparatus producing hydrogen in some detergent solution. The bubbles will rise rapidly into the air, again showing the low density of the gas.

Experiment E4.4

Is water formed when hydrogen burns?

This experiment **must** be performed by the teacher.

APPARATUS

Plastic safety screen

U-tube or calcium chloride tube

Thistle funnel with stem bent as in diagram, fitted with bung for the test-tube

Test-tube with side arm, 125×16 mm

Beaker, 250 cm³

Two stands and clamps

Filter pump and connection tubing

Metal or ceramic jet and connection tubing

Test-tube, 100×16 mm

Thermometer, -10° to $+110^{\circ}\text{C}$

Bunsen burner and asbestos square

Hydrogen cylinder

Silica gel or anhydrous calcium chloride

Anhydrous copper sulphate or cobalt chloride paper

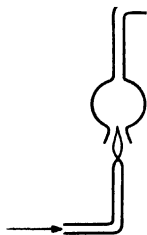


Figure 4.3

PROCEDURE

The hydrogen is first dried by passing it through a tube containing silica gel or anhydrous calcium chloride. When all air has been displaced completely from the apparatus the hydrogen is burnt at a metal or ceramic jet. The hydrogen flame should be about 3 cm high and allowed to burn beneath the thistle funnel as indicated in the diagram. The gas formed by the combustion is drawn through the apparatus by a filter pump, and a liquid soon condenses in the cooled test-tube. After about ten minutes 2 or 3 cm³ of liquid will have collected. The nature of the liquid should be investigated by finding its boiling point and its reactions towards anhydrous copper sulphate or cobalt chloride paper.

Experiment E4.5

What substances does water dissolve?

APPARATUS

Each pupil or pair of pupils will need:

Test-tubes, 100 × 16 mm, in rack

Beaker, 100 cm³ } or a microscope

Watch-glass } slide and holder

Teat pipette

Bunsen burner, tripod, gauze, and asbestos square

Access to a centrifuge

Potassium nitrate, iodine, naphthalene, glucose, paraffin wax, sulphur, potassium chlorate, and sodium chloride

PROCEDURE

Shake small amounts of different materials separately with distilled water in a test-tube. Notice whether the solution gets warm or cool.

Evaporate two or three drops of each solution on a watch-glass over a beaker of boiling water (or on a microscope slide held well above a small non-luminous flame).

Examine the substances in each case and decide which substances are:

1. very soluble,
2. only slightly soluble,
3. insoluble in water.

Compare the solubility of these substances in benzene, carbon tetrachloride, and kerosine with that in water.

Experiment E4.6**Measurement of the solubility of a solid in water****APPARATUS**

Each pupil or pair of pupils will need:

Test-tube, 150×25 mm

Thermometer, -10° to $+110^{\circ}\text{C}$

Measuring cylinder, 25 cm^3

Access to a balance

A selection of the following salts so as to obtain a variety of curves over as wide a temperature range as possible:

Sodium chloride

Sodium sulphate

Potassium chloride

Potassium chlorate

Calcium acetate

Ceric sulphate

PROCEDURE

This method requires only one weighing of the solute. The solubility at a given temperature is found by measuring amounts of solvent necessary to form a saturated solution at that temperature.

Place a weighed amount of solute in a 150×25 mm test-tube. Add sufficient distilled water from a 25 cm^3 measuring cylinder just to dissolve the solute on warming the solution a few degrees above the highest temperature at which solubilities are to be found. Allow the solution to cool, stirring continuously with the thermometer. Note the temperature at which crystals first appear. It is important to stir well to avoid the possibility of supersaturation and it may be necessary to add a small crystal of solute.

Knowing the mass of solute and the volume of water added, calculate the solubility, that is, the number of grams of solute required to saturate 100 g of water at the temperature in question.

Add a little more water to the mixture and note the new (lower) temperature at which the crystals now appear. Repeat this several times and plot a graph of solubility against temperature.

Experiment E4.7**Supersaturation****APPARATUS**

Each pupil or pair of pupils will need:

Two test-tubes, 100×16 mm

Measuring cylinder, 25 cm³

Thermometer, -10° to $+110^{\circ}\text{C}$

Bunsen burner

Two beakers, 600 cm³

Cotton wool plug to fit test-tube

Sodium thiosulphate

PROCEDURE

Fill a 100×16 mm test-tube to two-thirds with a known weight of sodium thiosulphate crystals. Place the tube in a beaker half-full of water and heat the beaker until all the crystals have dissolved in their own water of crystallization. Plug the top of the tube with cotton wool to keep out dust and stand it in an empty 600 cm³ beaker to protect it from draughts. Allow the tube to cool. Meanwhile fill another tube with water to the same depth as appears in the cooling tube and measure this water in a measuring cylinder. Refer to the solubility data. The solution appears to have more dissolved solute than is possible for a saturated solution and we therefore call it 'supersaturated'.

Let half the class drop a small sodium thiosulphate crystal into the supersaturated solution (thus supplying an excess of undissolved solute) and get the others to stir the supersaturated solution with a thermometer.

Experiment E4.8

The effect of adding salt to ice

APPARATUS

Each pupil or pair of pupils will need:

Filter funnel supported over a beaker

Thermometer, -10° to $+110^{\circ}\text{C}$

Crushed ice

Salt

PROCEDURE

Fill the funnel with crushed ice and insert the thermometer in the ice. When it has reached a steady temperature, cover the ice with coarse salt. Watch any changes in temperature. What else is observed to happen?

Two observations will be made:

1. Ice lowers the freezing point of water.
2. The addition of salt to ice makes it melt.

Experiment E4.9**Does water contain dissolved gases?**

The air dissolved in tap water may be boiled out of the water and its constitution investigated by the following demonstration.

APPARATUS

Round-bottomed flask, 1 litre

Screw clip

Two-holed rubber bung to fit flask carrying one 5 cm length of glass tubing and one glass delivery tube to lead to a trough

Rubber tubing to connect water tap to 5 cm length of glass tubing

Large pneumatic trough

Stand and clamp

Bunsen burner and asbestos square

Tripod and gauze

Gas measuring tube, 50 cm³

Sodium hydroxide pellets

Pyrogallol

PROCEDURE

A 1-litre flask is connected to the bench tap by means of a rubber tube carrying a screw clip close to the junction between the tube and a glass tube which terminates within the flask about an inch below the bung. The bung also carries an all-glass delivery tube which terminates within the bung – i.e. does not enter the flask, and has its other end below water in a pneumatic trough.

The flask is filled with water by opening the tap and in this way the whole apparatus, including the delivery tube, is swept clear of air and filled with water. The screw clip is now closed but the rubber tube left connected to the tap.

The water in the flask is boiled vigorously for about half an hour and the air coming off collected in a gas measuring tube until no more gas comes over. After the experiment the volume of water required to fill the flask may be measured and the solubility of air in water thus found.

The percentage of oxygen in dissolved air can then easily be found as follows:

Close the end of the gas measuring tube with your thumb and remove the tube from the water. Hold the tube vertically, and slip in a sodium hydroxide pellet by momentarily removing your thumb. Invert the tube once or twice and open it under water again. Measure the volume of air now free from carbon dioxide. Remove the tube again and slip in a measure of pyrogallol. Again invert the tube once or twice. Reopen it under water. The contraction gives the volume of oxygen absorbed.

The percentage of oxygen in dissolved air is greater than that in the atmosphere.

Experiment E4.10

What is the effect of detergents on water?

APPARATUS

Each pupil or pair of pupils will need:

Beaker, 250 cm³

Lycopodium powder, or powdered carbon

Distilled water

Liquid detergent (e.g. Teepol) in a dropping bottle

Soap solution (e.g. solution of sodium oleate in aqueous ethanol) in a dropping bottle

PROCEDURE

Fill the beaker with distilled water, sprinkle lycopodium powder (or powdered carbon) on the surface of the water and allow one drop of detergent to fall in the centre of the circle of powder. The lycopodium powder will still float. Empty and clean the beaker, refill it with fresh clean water, and lower a needle down carefully onto the surface of the water so that it floats. Allow one drop of detergent to fall on the surface of the water close to the needle. The needle will sink, as the presence of the detergent reduces the surface tension of the water.

The above experiments may be repeated using hard water and they will show that detergents are equally effective in hard water. Replacing the detergent by soap solution will show that soap solution is not as effective in hard water.

Experiment E4.11

Comparison of light- and heavy-duty detergents

APPARATUS

Each pupil or pair of pupils will need:

Two beakers, 1 litre

Glass stirring rod

Bunsen burner, tripod, gauze, and asbestos square

Thermometer, -10° to $+110^{\circ}\text{C}$

Two 2 inch squares of soiled cloth (see Procedure)

0.25 per cent solutions of Tide and Dreft (250 cm³ of each)

PROCEDURE

Detergents are of two kinds – light duty and heavy duty. The former are of low alkalinity and are not suited for heavily soiled cloth as in the weekly wash; they are however quite effective on lightly soiled fine fabrics. Their lower alkalinity gives them an added advantage with fabrics having pH-sensitive dyes. Liquid detergents are of this type. Many woollen dyes such as lissamine green and lissamine blue are quickly discoloured at pH 10 but are unaffected at pH 8. (I.C.I. Dyestuffs Division will provide samples of woollen fabrics dyed with these dyes if experiments using detergents on such materials are contemplated.)

Heavy-duty detergents contain a high level of alkaline phosphate so that they can remove greasy dirt present in the weekly cotton wash. Many of the detergents supplied in powder form are of this kind.

The behaviour of different detergents can be compared by means of the following experiment. Suitable detergents for study are:

Light duty – Dreft

Heavy duty – Tide or Daz

Each pupil will require two 2 inch squares of standard soiled cloth, prepared beforehand by coating a large square of clean cloth evenly with a mixture of lampblack and olive oil, applying the coating with a roller.

A comparison of the effects produced by the two types of detergent may be made as follows:

1. Place a 2 inch square of standard soiled cloth in a 1 litre beaker with 250 cm³ of a 0.25 per cent solution of Tide at 50°–60°C.
2. Repeat using a 0.25 per cent solution of Dreft also at 50°–60°C.
3. Agitate the solution, with propeller agitation if possible, but not too vigorously.
4. After four minutes remove the cloths from the two different solutions of washing products and examine the differences in washing.

If a contrast between the two pieces of cloth is not seen the first time, vary the degree of agitation and the washing time until the right conditions are found to show the differences between a light-duty and a heavy-duty detergent. (The times given may not be right for everyone's degree of agitation, nor for everyone's idea of a standard soiled cloth.) A repeated series of washes using the same detergent in each case usually shows the differences sought after.

The experiment may be extended by the use of woollen fabrics dyed with lissamine green or lissamine blue to show the importance of using the right kind of detergent for any particular washing task.

For the preparation of a common soapless detergent see the Unilever laboratory experiment *The preparation of sodium alkylbenzene sulphonate* (obtainable from Unilever Ltd., Information Division, Unilever House, Blackfriars E.C.4).

Experiment E4.12

What ions cause hardness in water?

APPARATUS

Each pupil or pair of pupils will need:

Test-tubes, 100×16 mm, in rack

Soap solution. (This may be made from soap flakes, about 10 g/litre in distilled water, mixed with an equal volume of industrial methylated spirits.)

The following substances dissolved separately in about 2 cm^3 of distilled water in different test-tubes using about 20 mg of solute in each case: Sodium chloride, calcium chloride, magnesium chloride, potassium nitrate, sodium sulphate, iron(II) sulphate, and magnesium sulphate.

PROCEDURE

Add each solution separately to the same volume of soap solution in another test-tube and shake well. Observe the resulting lather.

The positive ions may be divided into those which prevent the soap from forming a fairly stable lather and those which do not. The former are said to make the water hard.

It can be seen from this experiment that negative ions are not responsible for hardness.

Experiment E4.13

How hardness is formed in water

The first part of the experiment may be conveniently done by the teacher.

APPARATUS

Beaker, 600 cm^3

Carbon dioxide generator

Bunsen burner, tripod, gauze, and asbestos square

Water containing permanent hardness:

Dilute a quantity of saturated calcium sulphate solution with an equal volume of distilled water.

Water containing temporary hardness:

Dilute about 150 cm^3 of lime water with an equal volume of distilled water. Pass in carbon dioxide, taking care that the gas carries over no acid spray, whereupon calcium carbonate is soon precipitated. Continue the passage of gas until all the precipitate dissolves, giving a solution of calcium hydrogen carbonate.

In addition, each pupil or pair of pupils will need:

Test-tube, $150 \times 25\text{ mm}$, and cork to fit.

Measuring cylinder, 25 cm^3

Graduated teat pipette or burette

Access to microscope and microscope slides

Soap solution (see experiment E4.12)

PROCEDURE

Divide the water containing temporary hardness into two equal portions and boil one portion for about fifteen minutes, pouring off the supernatant liquid into another beaker. Add dilute acid to the solid residue in the beaker or transfer the precipitate to a test-tube before adding the acid. Effervescence occurs and the gas evolved is confirmed as carbon dioxide. Thus the precipitate must be a carbonate.

Distribute portions of the boiled and unboiled solutions to the class and let them test with soap solution until a permanent lather is obtained.

The method of using the soap solution can be either:

1. Addition of 1 cm^3 portions from the graduated teat-pipette to a known volume of hard water, say 10 cm^3 portions, measured by a measuring cylinder into a $150 \times 25\text{ mm}$ test-tube. This may be corked and shaken to produce a lather.
2. Delivery of soap solution from a burette into a measured volume of hard water in a conical flask.

What difference did boiling make to the water? By this means introduce temporary hardness as being caused by calcium hydrogen carbonate, which is removable by boiling since it is decomposed and calcium carbonate precipitated.

The relation between calcium hydrogen carbonate solution and calcite, stalactites and stalagmites, may be shown.

Leave the calcium hydrogen carbonate solution in a beaker for a few days. A 'dust' appears on the top; remove this on a microscope slide and examine it through the microscope. The 'dust' consists of little calcite crystals.

Then let the class test some calcium sulphate solution to see if boiling makes any difference to its hardness. Thus permanent hardness is introduced.

Experiment E4.14

How hardness can be removed

APPARATUS

Each pupil or pair of pupils will need:

Test-tube, 150×25 mm, and cork to fit

Spatula

Hard water (see experiment E4.13)

Soap solution (see experiment E4.12)

Sodium carbonate crystals

Column containing a sodium zeolite ion exchange resin

PROCEDURE

Removal of calcium ions can be effected in two ways:

1. Precipitating the calcium as an insoluble compound.
2. Exchanging the calcium ions for those of another metal such as sodium which do not cause hardness.

1. Add a measure of sodium carbonate crystals to about 20 cm^3 of hard water in a 150×25 mm test-tube. Shake well, and add a few cm^3 of soap solution. Does the water lather any more easily than untreated water? Is any scum formed? Was anything observed in the solution as soon as the sodium carbonate dissolved?

2. Run a little hard water through a column packed with a sodium zeolite ion exchange resin, and test the water coming out with soap solution. When the zeolite has exchanged all its sodium ions for the calcium ions in hard water it may be regenerated by passing strong sodium chloride solution through it.

Experiment E4.15**Experiments with a charged conductor****APPARATUS**

Each pupil or pair of pupils will need:

Burette

Stand and clamp

Beaker, large size

Ebonite rod, nylon comb, or other material which can be electrostatically charged

Benzene

Carbon tetrachloride

Acetone

PROCEDURE

Arrange for a thin, steady stream of water to run from a bench tap or a burette into a beaker and bring up to it a charged source, e.g., a Biro type pen previously rubbed on a jacket sleeve, a nylon comb run briskly through the hair, or any of the usual electrostatic demonstration materials. What happens to the water stream? What is a possible reason for this? Would benzene or carbon tetrachloride behave in a similar manner? Try them and also try acetone. What conclusions can you draw from the results?

It will be found that the stream of acetone is slightly affected, but that benzene and carbon tetrachloride are not. In interpreting the behaviour, two factors must be taken into account:

1. The polarity of the molecules.
2. The polarizability of the molecules.

The liquid stream would be deflected in a strong electrical potential gradient if the molecules were polarizable – even if they were not themselves polar. The fact that the water stream is strongly deflected suggests, but does not prove, that the molecules are polar.

Chapter 5

The effect of electricity on substances

Introduction

The effect of electricity on substances is as fundamental and important as that of heating them; the battery is as important a weapon in the chemist's armoury as is the Bunsen burner.

The investigation can be started by examining the effect of electricity on a wide range of single substances, both at room temperature, and when heated until they are molten. This can then be extended to an investigation of the effect of electricity on solutions; but it would be confusing to begin with solutions, as these present a more complicated situation.

In the course of these experiments the pupils will see that chemical changes sometimes occur at the electrodes, and this will prompt an investigation of the products obtained. Experiments E5.1–E5.3 cover this ground, and all are quite suitable for use at an early stage of the pupils' progress.

A good deal of extra information can be obtained by a quantitative study, and if an investigation of the effect of heating substances has been done in this way, the idea of attempting to weigh the products of electrolysis should come to the pupils' minds. Such an enquiry could be done at an early stage, but it is best left until after the concepts of atoms and of reacting weights have been discussed. At this later stage the evidence for the ionic theory which these experiments provide can be discussed. Experiment E5.9 can be used to illustrate the movement of ions.

The chapter also includes some experiments in which the dependence of the resistance of electrolyte solutions on various factors can be found. Illustrations of industrial processes in which electrolysis is used are given in Chapter 11, 'Raw materials and Industry'.

References

The development of this theme is discussed in the *Handbook for Teachers*, Part 3, *Electrochemistry*.

The theme is introduced in the Sample Scheme, Part I, *Basic Course*, using two alternative methods of approach, as follows:

Stage I A, Topic 8: 'The effect of electricity on substances'

Stage I B, Topic 6: 'Using electricity to decompose substances'

It is then developed in:

Stage II, Topic 16: 'Explaining the behaviour of electrolytes'

List of experiments

Qualitative experiments

- E5.1 Which substances conduct electricity?
- E5.2 Do solutions conduct electricity?
- E5.3 What products are formed at the electrodes when solutions conduct electricity?
- E5.4 Using electricity for plating
- E5.5 The qualitative electrolysis of lead(II) bromide

Quantitative experiments

- E5.6 The quantitative electrolysis of lead(II) bromide
- E5.7 How to compare the weights of different metals formed by the same amount of electricity
- E5.8 How to compare the weights of the same metal formed when carrying different quantities of electricity

Other electrolysis experiments

- E5.9 Demonstrations of the movement of ions during electrolysis
- E5.10 Are different cations discharged preferentially?
- E5.11 How does the resistance of an electrolyte solution vary with distance between the electrodes?
- E5.12 How does the resistance of an electrolyte solution vary with concentration of the electrolyte?

E5.13 How does the resistance of an electrolyte solution vary with the nature of the electrolyte (strong or weak)?

E5.14 How does the resistance of an electrolyte solution vary with the size of the electrode?

Qualitative experiments

Experiment E5.1

Which substances conduct electricity?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A8.1)

D.C. supply, 6 volts approximately

6-volt bulb in holder

Carbon or steel electrodes in holder

Three lengths of connecting wire fitted with crocodile clips

Bunsen burner and asbestos square

Tripod and pipe-clay triangle

Access to:

1. Small pieces of lead, copper, and other metals
2. Crucibles or hard-glass test-tubes, 150×25 mm, containing samples of naphthalene, sulphur, polythene, wax, sugar, lead(II) iodide, potassium iodide

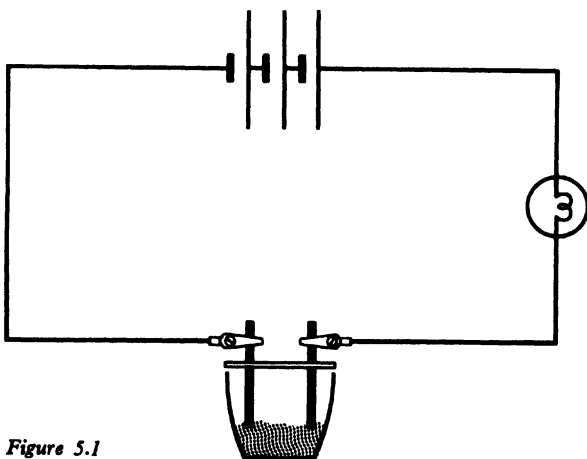


Figure 5.1

PROCEDURE

Set up the electrical circuit as shown in the diagram. The source of electricity can be dry cells giving 6 volts, or if a laboratory low-voltage supply is available, this should be arranged so as to give 6 volts. The 6-volt bulb mounted in an ordinary bulb holder is included to indicate when the current is flowing. Electrodes may be carbon or steel, mounted in a wooden support, cork, or bung, so as to keep them at a constant distance apart.

Select one of the sample materials above, and by holding the electrodes in contact with it, find out whether it conducts electricity or not. The electrodes must be cleaned after each experiment.

The metals will be found to conduct, but not the salts and other solids in part 2 of the list above.

Gently heat samples of the solid non-conductors until they just melt, to find out whether the molten material conducts electricity. Care must be taken, for some substances (naphthalene, sulphur, wax, Polythene) will burn if heated too much. Electrodes must be scraped clean before testing the next substance. It will be found that potassium iodide and lead(II) iodide conduct when molten.

If time and materials allow, the entire range of materials can be investigated. If not, the teacher can suggest that a pair of pupils investigates a particular group of four or five substances (selected to give experience of conductors and non-conductors in the solid and liquid state), while other pupils examine alternative groups. Results from all the class can then be pooled.

Experiment E5.2

Do solutions conduct electricity?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A8.2)

D.C. supply, 6 volts approximately

6-volt bulb in holder

Carbon electrodes and holder

Two lengths of connecting wire, fitted with crocodile clips

Beaker, 100 cm³

Paper tissues or a cloth

Distilled water

Access to 100 cm³ beakers containing approximately M or 0.5M solutions of:

Potassium iodide
Sulphuric acid
Sodium hydroxide
Sodium chloride
Copper(II) sulphate
Zinc sulphate
Cane sugar

PROCEDURE

Arrange a circuit to contain the electrodes, bulb and holder, as shown in figure 5.1, using the low voltage supply available. The bulb is included to indicate when current is flowing.

An experiment to find out if distilled water appears to conduct electricity should be done first since this has been used to prepare the other solutions.

Dip the electrodes into a solution selected from those above to see if it conducts electricity, and note the result. After cleaning the electrodes and wiping them dry, repeat the procedure with further solutions.

It will be found that, except for the sugar solution, they all conduct electricity.

Experiment E5.3

What products are formed at the electrodes when solutions conduct electricity?

Part 1. An examination of some simple solutions

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B6.3)

D.C. supply, 6 volts approximately

6-volt bulb in holder

Electrolysis cell shown in diagram and described below, with carbon electrodes, and two 75 × 10 mm rimless test-tubes for collecting gases

Two lengths of connecting wire fitted with crocodile clips

Stand and clamp

Paper tissues or a cloth

Approximately 1M solutions of:

Copper(II) chloride

Zinc bromide

Potassium bromide

Potassium iodide

or enough of the solid compounds to allow the pupils to make up their own solutions

Distilled water

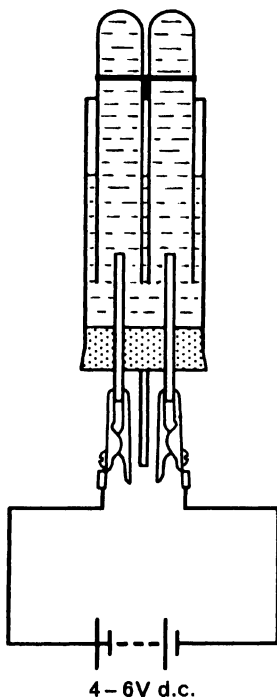


Figure 5.2

ELECTROLYSIS CELL

Description

This comprises an open glass cylinder approximately 75 mm tall and 25 mm diameter, a rubber bung carrying two carbon electrodes and metal connecting leads, the bung making a liquid-tight fit in one end of the cylinder. A strip of insulating material let into the bung prevents short-circuiting when crocodile clips are fastened to the connecting leads.

Two rimless 75×10 mm test-tubes are supported by a wood splint and elastic band over the electrodes to collect any gases evolved.

The assembled cell is conveniently mounted by a Terry clip on a wood block approximately $5 \times 5 \times 2$ cm.

Method for inserting electrodes in the rubber bung

A rubber bung is selected so as to give a good liquid-tight seal for the glass cylinder being used. Two holes to accommodate the electrodes are drilled through the bung with a $\frac{1}{32}$ inch drill.

A piece of stout copper wire is soldered to the length of braided metal screening lead to act as the 'needle' whereby the lead can be threaded through the bung leaving about 1 cm projecting. Into the core of the exposed lead insert securely a length of pencil lead as the carbon electrode. Pull the lead further into the body of the bung so that only 1.5 cm of the electrode projects. The excess lead is cut off, leaving about 2 cm length for attaching crocodile clips.

The procedure is then repeated to insert the other electrode. Finally a cut about 1 cm deep should be made across the bung, midway between the two projecting lead ends and an insulating strip inserted to prevent short circuiting when the crocodile clips are attached. The insulating strip can be secured in place by a turn of wire around the circumference of the bung to hold the cut closed.

In a similar way the electrolysis cell may be fitted with metal electrodes, though it is desirable to solder the electrode metal to the braided lead in this case. A suitable metal for such electrodes was found to be the alloy leads supporting the coiled filament in electric lamp bulbs.

PROCEDURE

Pass electricity through the solutions in the apparatus described above. To emphasize the fact that both a solid and a liquid are involved the pupils may make up their own solutions. Once it has been seen that the solution conducts electricity, the bulb should be removed from the circuit in order to allow a larger current to flow. When a gas is given off, collect it in a 75×10 mm test-tube and test its properties. The following results will be found using carbon electrodes.

1. Copper(II) chloride solution

Copper is deposited on the carbon cathode, and chlorine is evolved at the anode. The chlorine may be collected and its colour and smell noted, but take care!

2. Zinc bromide solution

Zinc is deposited on the carbon cathode and bromine is evolved at the anode. Most of the bromine dissolves in the water, colouring it brown.

3. Potassium bromide solution

Hydrogen is evolved at the cathode, and can be collected and tested by burning. The bromine evolved at the anode dissolves in the water, turning it brown.

4. Potassium iodide solution

Hydrogen is evolved at the cathode as above. The iodine evolved at the anode does not vaporize, but stains the solution dark brown.

Part 2. An experiment using sea water

A. Direct from the sea

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A10.2)

Electrolysis cell as figure 5.2, with two rimless test-tubes, 75×10 mm

Two lengths of connecting wire, fitted with crocodile clips

D.C. supply, 6 volts approximately

Splints

Stand and clamp

Universal Indicator paper

Starch-iodide paper for testing gases

Supply of sea water

PROCEDURE

Half fill the cell with sea water and connect the d.c. supply. No bulb is needed in the circuit for this experiment. Chlorine will appear at the anode and hydrogen at the cathode. Do the starch-iodide test for chlorine by holding a piece of moist starch-iodide paper in the gas. Iodine is liberated and forms a deep blue colour with the starch: the paper therefore turns from white to deep blue. Test the gas liberated at the cathode using Universal Indicator paper and a lighted splint. It will be found to burn.

B. After concentration by the method outlined in experiment E2.7

This experiment should be done by the teacher.

APPARATUS

Two carbon or steel electrodes

Two lengths of connecting wire, fitted with crocodile clips

D.C. supply, 6 volts approximately

Crucible

Two test-tubes, 100×16 mm

Test-tube holder

Bunsen burner and asbestos square

Teat pipette

Concentrated sea water from experiment E2.7

Manganese(IV) oxide (manganese dioxide)

Concentrated sulphuric acid

Potassium iodide solution, approximately 0.5M

PROCEDURE

The teacher can electrolyse the small amount of residual solution kept from experiment E2.7. This is insufficient to place in the pupils' apparatus, but a pair of electrodes can be dipped into the small amount of solution available. A yellow-brown colour will be seen to develop around the anode (bromine).

The teacher should next evaporate this last solution to dryness in a crucible, mix the residue in a 100×16 mm test-tube with a measure of manganese(IV) oxide (manganese dioxide), and add concentrated sulphuric acid and warm.

A reddish-brown vapour will form which will condense to red drops of bromine higher up the tube. A little of the bromine vapour can be transferred by means of a teat pipette to another test-tube and bubbled through 1 or 2 cm³ of water contained in a second test-tube; the addition of potassium iodide solution will yield a brown coloration of iodine.

Experiment E5.4**Using electricity for plating****APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B6.4)

D.C. supply, 6 volts approximately

6.2 volt, 0.3 amp bulb in bulb holder

Two copper foil cathodes, 5×3 cm² approximately

Nickel and zinc foil anodes, 5×3 cm² approximately

Three lengths of connecting wire fitted with crocodile clips

Support for electrode foils

Beaker, 100 cm³

Steel wool

Paper tissues

Plating solution, about 100 cm³ each

1M sodium hydroxide solution

Distilled water

PROCEDURE

For all plating experiments the cathode on which the metal is to be deposited must be thoroughly clean and free from grease, and the correct plating solution and current density must be used. The circuit has been designed to give the correct current density provided that electrodes of the recommended size are used. The bulb indicates that current is flowing and limits this so that no rheostat is necessary. Immerse 10 to 15 cm² of the electrode. Clean the objects to be plated by rubbing them with steel wool and paper tissues moistened with molar sodium hydroxide solution, and then wash them well with distilled water.

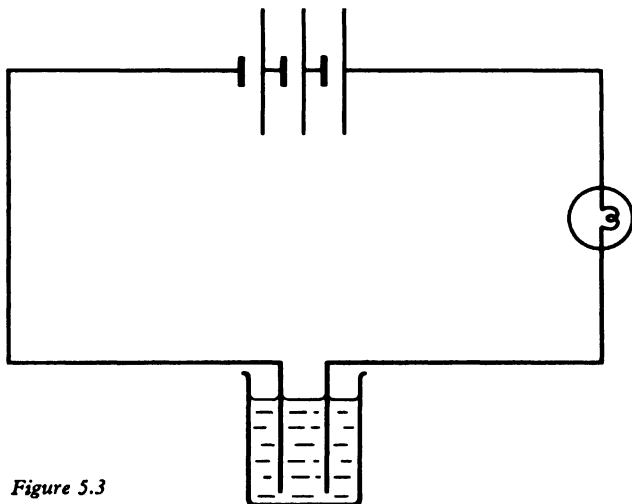


Figure 5.3

Other details are as follows:

1. Nickel plating

A simple solution of nickel ammonium sulphate, about 5 g in 100 cm³ of water, will give reasonable results. A current density of 0.01 to 0.03 amps per cm² gives good results after five minutes. For even plating on both cathode surfaces the cathode should be turned round after half the time has elapsed.

2. Zinc plating

A solution suitable for zinc plating may be made as follows:

Add an equal volume of water to about 50 cm³ of a saturated solution of zinc sulphate. To this solution add a few drops of 1M sulphuric acid and two spatula measures of boric acid. A current density of 0.01 to

0.03 amps per cm^2 will give good results in five to ten minutes. Turn the cathode round after half the time has elapsed to obtain even plating.

Experiment E5.5

The qualitative electrolysis of lead(II) bromide

Caution: Lead salts and the vapours produced during the experiment are toxic; molten lead bromide can cause severe burns. Pupils must be forewarned and supervised.

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 16.1)

Hard-glass test-tube, 150×25 mm

Two carbon electrodes and holder

Source of 6 volts d.c.

6-volt bulb in holder

Three lengths of connecting wire, fitted with crocodile clips.

Stand and clamp

Bunsen burner and asbestos square

Lead(II) bromide: this must be good quality material or much bromine may be given off merely by melting it. In particular, nitrate should not be present

PROCEDURE

Place about 3 cm depth of lead(II) bromide in a 150×25 mm test-tube and heat with a Bunsen burner until the lead bromide melts. Electrolyse the molten salt using carbon electrodes, having a 6-volt bulb and holder in circuit with the 6-volt d.c. supply. For a diagram of this apparatus, see p. 100. Once the fact that the fused lead bromide conducts the current has been established, the bulb and holder should be removed from the circuit to allow a larger current to flow. This larger current is necessary to obtain a reasonable amount of decomposition in a limited time. Lead bromide is expensive, but only small quantities are required and the substance can be re-used many times. When heating the lead bromide the flame should be just high enough to maintain the lead bromide molten, since the higher the temperature, the greater the formation of poisonous vapours.

During electrolysis observe the bubbles of gas discharging on the anode. After about five minutes stop the electrolysis and allow the apparatus to cool. When it is cold enough to handle examine the electrolyte. A small bead of lead will be found in the region of the cathode. It is usually best seen from under the test-tube.

Lead(II) iodide may be used as electrolyte, when the iodine formed is more clearly visible than bromine, but it is more expensive.

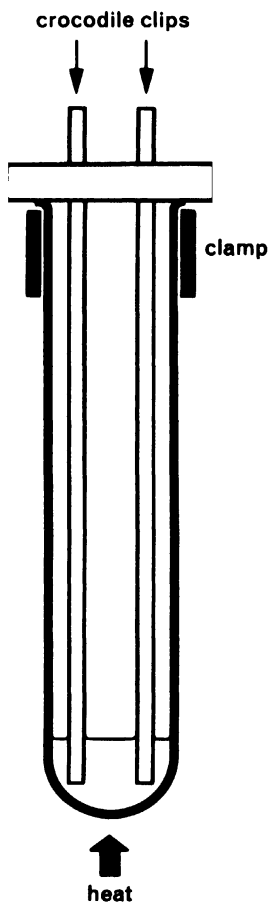


Figure 5.4

Quantitative experiments

Experiment E5.6

The quantitative electrolysis of lead(II) bromide

This experiment should be done by the teacher.

APPARATUS

Two carbon electrodes and holder

Two crystallizing dishes

Source of 10 volts d.c. from accumulators or other steady source

Switch

Several lengths of connecting wire, including two fitted with crocodile clips

Ammeter (5 amp maximum)

Rheostat

Stopclock

Balance

Spatula

Tripod and gauze

Stand and clamp

Bunsen burner and asbestos square

Tongs

Lead(II) bromide

PROCEDURE

This experiment should be demonstrated by the teacher. In this case a higher voltage (10 volts) than that used in experiment E5.5 is desirable and a rheostat, ammeter, and switch are included in the circuit.

The electrodes should be on opposite sides of the crystallizing dish and the depth of the electrolyte should be about 0.5 cm. When molten, place the electrodes in position and give time for remelting the solid electrolyte round the electrodes, which will form owing to local cooling. Switch on and adjust the current immediately to a value between 2 and 4 amps. Maintain the current at this constant value with the rheostat as it may fluctuate during electrolysis.

After ten or fifteen minutes of electrolysis, switch off, withdraw the electrodes and decant the melt carefully into another dry crystallizing dish, taking care to retain the bead of lead in the original dish. Hold this dish angled in the decanting position until the bead of lead is seen to solidify and stand it down while it cools to room temperature. The bead may now be cleanly prised away from the glass with the spatula and adhering lead bromide may easily be broken off giving a clean lead bead, which is weighed.

Calculate how many coulombs and hence faradays of electricity are needed to release 1 gram-atom (207 grams) of lead. (Amperes $\times$ seconds = coulombs.)

As in experiment E5.5 lead(II) iodide may be used as electrolyte in place of lead(II) bromide.

Experiment E5.7

How to compare the weights of different metals formed by the same amount of electricity

APPARATUS

Each pair of pupils will need:

Page from *Laboratory Investigations* (experiment 16.5a)

Source of 6 volts d.c.

Rheostat

Ammeter (0.5 amp maximum)

Two beakers, 100 cm³

Five lengths of connecting wire

Access to balance

Two copper foil electrodes and support } electrodes about

Two silver foil electrodes and support } 5 cm $\times$ 3 cm

Approximately 0.05M copper(II) sulphate solution

Approximately 0.05M silver nitrate solution

Distilled water

Ethanol

Acetone

PROCEDURE

This experiment is best done by pairs of pupils working together.

Connect up an electrical circuit as shown in figure 5.5. P and Q are 100 cm³ beakers acting as voltameters; one contains approximately 0.05M copper(II) sulphate solution and copper electrodes, the other contains approximately 0.05M silver nitrate solution and silver electrodes.

R is a rheostat and should be used to keep the current to such a value that the current density at either cathode is about 10 milliamps cm⁻². With the suggested electrodes, about 100 milliamps are required since the area immersed is roughly 10 cm².

The copper and silver cathodes, which must be clean and dry, are weighed. After connecting them in the circuit, pass the current for about one hour. Then remove the two cathodes and wash them by

dipping in distilled water, then in alcohol and finally in acetone. Weigh them when they are completely dry.

The silver deposit is crystalline and may not adhere well to the cathode. The greatest care must be taken that as little as possible is lost in the washing. By observing this precaution, accurate results can be obtained.

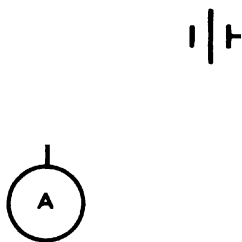


Figure 5.5

Calculate the increase in weight of both cathodes and compare them with one another, and with the ratio of the atomic weights of the metals.

Experiment E5.8

How to compare the weights of the same metal formed when carrying different quantities of electricity

APPARATUS

Each pair of pupils will need:

Page from *Laboratory Investigations* (experiment 16.5b)

Source of 6 volts d.c.

Rheostat

Ammeter (0.5 amp maximum)

Two beakers, 100 cm³

Tripod and gauze

Bunsen burner and asbestos square

Thermometer, -10° to $+110^{\circ}\text{C}$

Four copper foil electrodes and two supports

Connecting wire

Paper tissues

Fine steel wool

Access to balance

Approximately 0.5M copper(II) sulphate solution

Aqueous solution containing 100 g sodium chloride and 1 g sodium hydroxide per litre

PROCEDURE

Set up a similar circuit to the one used for experiment E5.7. P is again a 100 cm³ beaker containing 0.5M copper sulphate solution, in which are placed two copper electrodes. Q, however, contains an electrolyte having 100 g/l of sodium chloride and 1 g/l of sodium hydroxide, which should be maintained at 80°C, in which are placed two copper electrodes and a thermometer.

The rheostat R should be controlled so that the current density is kept at about 10–20 milliamps cm⁻².

Clean both anodes thoroughly with steel wool and paper tissues and weigh them before placing in their respective voltameters. A red-orange precipitate of copper(I) oxide appears immediately around the anode in the voltameter having the alkaline sodium chloride electrolyte. While the current is passing this anode should be moved about from time to time to stop copper(I) oxide from adhering to it.

After about fifteen minutes stop the flow of electricity, remove the anodes, and dry them with paper tissues. The anode from the alkaline sodium chloride should be rubbed hard to remove any adhering copper(I) oxide but the other anode merely needs blotting. Weigh them when dry.

The decrease in weight of the anodes can now be calculated, and compared; one will be found to be very nearly twice that of the other.

Other electrolysis experiments

Experiment E5.9

Demonstrations of the movement of ions during electrolysis

A. A simple pupil experiment

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 16.6b)

Microscope slide or watch-glass
Filter paper strips cut to fit the slide
Two lengths of connecting wire, fitted with crocodile clips
Source of about 20 volts d.c.
Small crystal of potassium permanganate

PROCEDURE

Place a strip of filter paper along the microscope slide or watch-glass using the crocodile clips to clamp it at each end and to connect it to the source of about 20 volts d.c. Just moisten the filter paper with tap water (sufficient for the water to spread out as far as the crocodile clips and no more) and drop a small crystal of potassium permanganate onto the middle of the paper. After a few minutes the colour of the permanganate will be seen to be moving towards one of the crocodile clips.

The experiment can be repeated, using small crystals of other coloured, water soluble compounds.

B. *Teacher demonstration*

APPARATUS

The teacher will need:

Buchner funnel, flask, and filter pump
Wide U-tube
Stand and clamp
Supply of about 20 volts d.c.
Two lengths of connecting wire fitted with crocodile clips
Pipette
Two carbon electrodes
2M hydrochloric acid
1M copper(II) sulphate solution } or copper(II) chromate
1M potassium chromate solution }
Urea

PROCEDURE

The teacher may demonstrate the movement of ions during electrolysis in a convincing manner using the following method.

If copper(II) chromate cannot be bought, prepare some by adding 100 cm³ of 1M copper(II) sulphate solution to an equal volume of 1M potassium chromate solution. Filter the insoluble copper(II) chromate using a Buchner funnel and filter flask and pump, and wash the precipitate with distilled water.

Dissolve the solid copper(II) chromate in the minimum quantity of 2M hydrochloric acid, and then dissolve as much urea as possible in the solution, in order to increase its density.

Next fill one-third of a wide U-tube with 2M hydrochloric acid and run the copper(II) chromate solution in very slowly and carefully from a pipette, delivering it at the bottom of the U-tube so that it forms a separate layer with a clear layer of hydrochloric acid above it on both sides of the U-tube. Withdraw the pipette carefully to avoid mixing.

Insert a carbon electrode into each arm of the U-tube, so as to dip into the dilute hydrochloric acid, and connect to a source of about 20 volts d.c. After five to ten minutes a development of a green colour on the cathode and orange on the anode side is observed. After about thirty minutes, the blue copper(II) ion and the orange dichromate ion boundaries are clearly seen.

c. *Teacher demonstration*

APPARATUS

The teacher will need:

Tripod and gauze

Bunsen burner and asbestos square

Beaker, 250 cm³

Stand and clamp

Glass stirring rod

U-tube, 1.0-1.5 cm diameter, 10 cm long

Two carbon electrodes

Source of d.c. about 50 volts (a H.T. battery is suitable)

Two lengths of connecting wire fitted with crocodile clips

Potassium nitrate

Agar (powdered)

Bromothymol blue solution

PROCEDURE

Add about 5 g of potassium nitrate to 100 cm³ of water in a 250 cm³ beaker, place on a gauze on a tripod above a lighted Bunsen burner, and bring to the boil. Add about 3 g of powdered agar to the boiling solution, stirring well and keeping the flame very low to keep the solution just boiling but not burning. When all the agar has dissolved, add sufficient bromothymol blue solution to make the solution quite highly coloured (green). The amount should be enough to make the solution quite definitely coloured when viewed through the narrow U-tube. Pour this liquid into the U-tube until it is 2-3 cm from the top of each limb, clamp the tube upright, and leave it until the contents have set. When

this has happened, and the contents are cold, fill the space above the gel with more potassium nitrate solution (without agar). Place the two carbon electrodes so that one dips into each limb of the U-tube; avoid dipping them into the gel, but be sure that they are dipping into the solution. Do not use corks to support the electrodes because gases are evolved.

Connect the electrodes to a source of d.c. of about 50 volts; the potential should be about 3 volts per cm of gel and should be adjusted accordingly if a different size of U-tube has to be used. Leave the electrical circuit switched on until a convenient amount of ionic movement becomes visible. The H^+ movement shows as a yellow band travelling down from the cathode, and the OH^- movement as a blue band from the anode. A movement of several cm will be obtained in about twenty minutes.

This experiment, under these conditions, is not suited to use at 2 volts overnight (for example) because the mobilities and diffusion rates are so high that there is an inadequate length of gel.

D. *Teacher demonstration*

APPARATUS

The teacher will need:

Tripod and gauze

Bunsen burner and asbestos square

Stand and clamp

Beaker, 250 cm³

Glass stirring rod

U-tube, 1.0–1.5 cm diameter, 10 cm long

Two carbon electrodes

Source of d.c. about 50 volts (a dry battery is suitable)

Two lengths of connecting wire fitted with crocodile clips

Potassium nitrate

Potassium hexacyanoferrate(III) (potassium ferricyanide)

Agar, powdered

Phenolphthalein solution

Sodium hydroxide solution

Iron(II) sulphate

Dilute sulphuric acid

PROCEDURE

Carry out this experiment in a similar manner to that described for experiment C, using as before about 5 g of potassium nitrate in 100 cm³ of water, but also adding one or two small crystals of potassium

hexacyanoferrate(III). Disperse about 3 g of agar as before, followed by 10–15 drops of phenolphthalein indicator solution and a few drops of sodium hydroxide solution, just sufficient to give the solution a definite red colour. When the solution has set to a gel in the U-tube, fill up the remaining portion of each limb with a strong solution of iron(II) sulphate in dilute sulphuric acid.

Connect the electrodes as before. In this case the H^+ movement is seen by the removal of the phenolphthalein colour, and it is followed much more slowly by Fe^{2+} ions forming Prussian blue. Diffusion of the ions occurs at the cathode side and provides a simple visible check on the extent of this.

Typical results:

15 min	H^+ 4.5 cm	diffusion 0.5 cm
	Fe^{2+} 0.8 cm	diffusion 0.1 cm
60 min	H^+ 13 cm	diffusion 1 cm
	Fe^{2+} 1.5 cm	diffusion 0.1 cm

Experiment E5.10

Are different cations discharged preferentially?

APPARATUS

Each pupil or pair of pupils will need:

Source of 3–4 volts d.c.

Two platinum electrodes 1 cm square with platinum leads sealed through glass tubes, the whole supported in a cork so that the electrodes are about 2 cm apart.

Beaker, 100 cm³

Beaker, 5 cm³

Two or three test-tubes, 75 × 12 mm

Teat pipette

Bunsen burner and asbestos square

Test-tube holder

Two connecting wires fitted with crocodile clips

Access to centrifuge

0.5 molar solutions of copper(II) sulphate, iron(II) sulphate, and zinc sulphate.

4M solutions of nitric acid and ammonium hydroxide

0.1 per cent copper(II) sulphate solution

Ammonium thiocyanate solution (15.2 g in 100 cm³ of water)

Ammonium mercuri-thiocyanate solution (2.7 g of mercury(II) chloride and 3 g of ammonium thiocyanate in 100 cm³ of water)

Distilled water

PROCEDURE

The electrolytes used in this experiment are copper(II) sulphate, iron(II) sulphate, and zinc sulphate solutions. Using these, it is possible to find out that copper is deposited in preference to iron or zinc. Using other electrolytes may not give clear results as many metals do not give adherent coatings to the platinum electrodes.

Pour the electrolyte, or equal volumes of two electrolytes, into the 100 cm³ beaker. Place the electrodes in the solution and electrolyse it for one to two minutes using 3-4 volts d.c. Remove the electrodes and carefully rinse them free from any electrolyte. Dip the cathode into 2 cm³ of hot 4M nitric acid in a 5 cm³ beaker. Test the resulting solution for the deposited metals as follows.

Copper

Using a teat pipette, transfer 5 drops to a 75 × 12 mm test-tube. Add 4M ammonium hydroxide solution drop by drop until it is in excess. A deep blue colour indicates the presence of copper.

Zinc

Using a teat pipette, transfer 5 drops to a 75 × 12 mm test-tube. Add 1 cm³ of distilled water, followed by one drop of 0.1 per cent copper(II) sulphate solution and 6 drops of ammonium mercuri-thiocyanate solution. A violet precipitate indicates the presence of zinc.

Iron

Using a teat pipette, transfer 5 drops to a 75 × 12 mm test-tube. Boil the solution, then allow it to cool and dilute it considerably with distilled water. Add one drop of ammonium thiocyanate solution. A blood-red colour indicates the presence of iron.

Zinc in the presence of an excess of copper

Using a teat pipette, transfer 5 drops to a 75 × 12 mm test-tube. Add 8 drops of mercuri-thiocyanate solution. A yellow precipitate is given by copper alone. In the presence of zinc the precipitate is of a darker hue. Comparison with a solution containing copper alone is helpful here. On standing it is often possible to see the violet precipitate above the yellow one as the yellow one settles out. Alternatively, dilute the 5 drops with distilled water and precipitate the copper with hydrogen sulphide. Centrifuge off the copper sulphide so formed. Boil the residual solution to remove hydrogen sulphide. Add one drop of 0.1 per cent copper(II) sulphate solution and 6 drops of ammonium mercuri-thiocyanate solution. A violet precipitate indicates the presence of zinc.

Iron in the presence of an excess of copper
Proceed as described for iron alone.

Note: With nitric acid and thiocyanates, particularly in hot solution, some hydrogen cyanide may be evolved. Care should be taken to keep solutions cool and dilute, and to use the minimum volume of ammonium thiocyanate or mercuri-thiocyanate solutions.

Experiment E5.11

How does the resistance of an electrolyte solution vary with the distance between the electrodes?

APPARATUS

Source of 6 volts d.c.

Ammeter, 0-5 amps

Three pieces of connecting wire

Special apparatus as shown in the diagram

Approximately M copper(II) sulphate solution

PROCEDURE

Set up the apparatus as shown in figure 5.6. Fill the tube with approximately molar copper(II) sulphate solution and set the electrodes at the maximum distance apart. Switch on the current and note the ammeter reading.

Move the electrodes 3 cm closer together and again note the ammeter reading. Repeat the operation of moving the electrodes together, taking successive ammeter readings, and plot a graph of resistance (i.e. the voltage divided by the current in amps) against the distance between the electrodes.

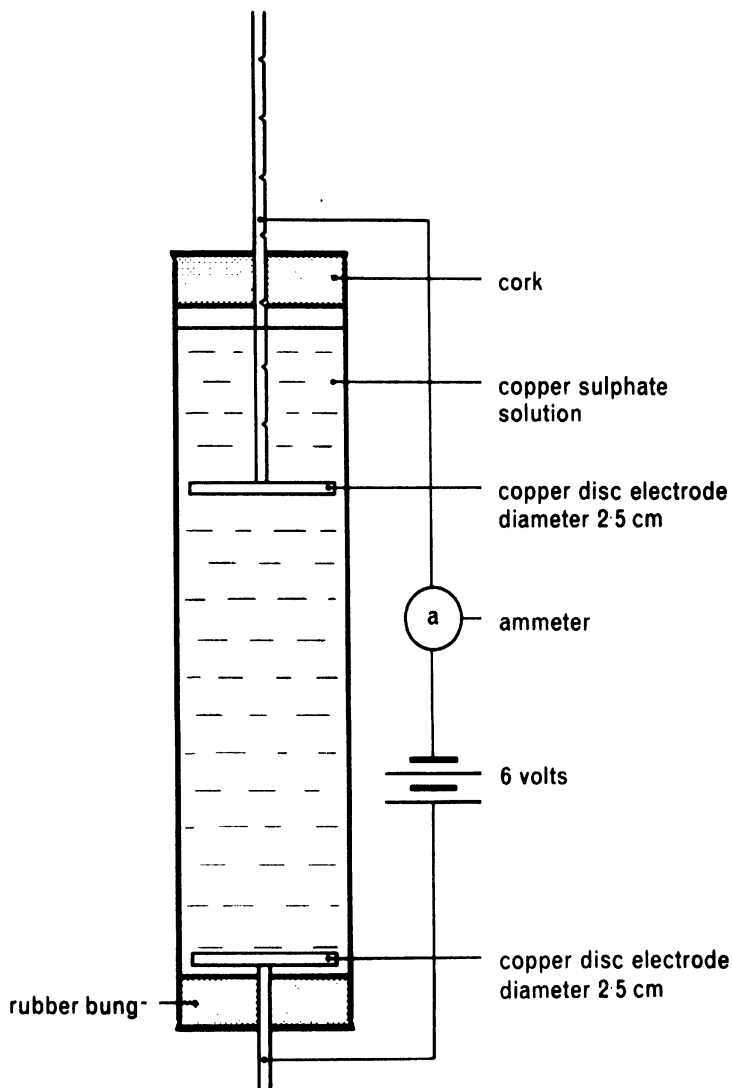


Figure 5.6

Experiment E5.12

How does the resistance of an electrolyte solution vary with concentration of the electrolyte?

APPARATUS

Source of 6 volts d.c.

Ammeter, 0-5 amps

Three pieces of connecting wire

Special apparatus as shown in the diagram

Strong solution of copper(II) sulphate

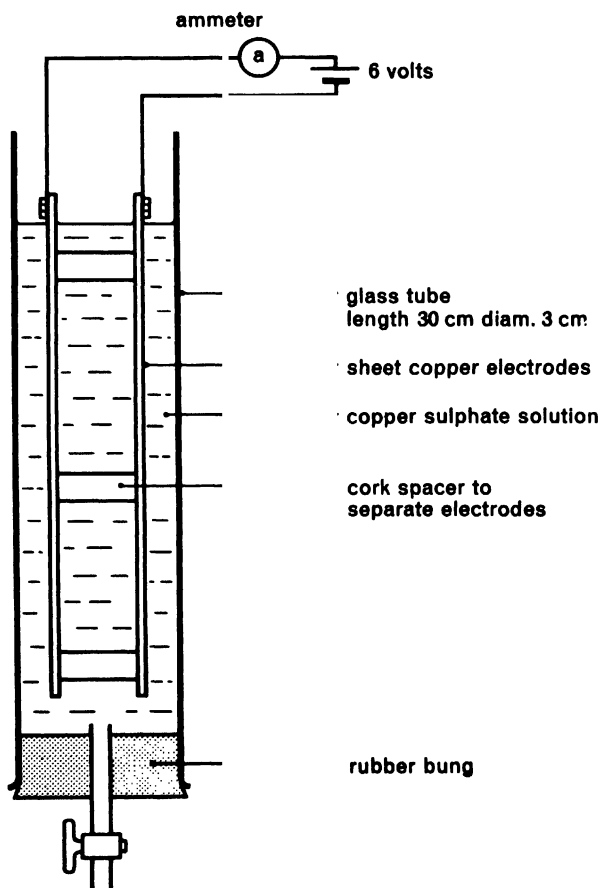


Figure 5.7

PROCEDURE

Set up the apparatus as shown in figure 5.7, and fill the cell up to the mark at the top of the tube with a strong solution of copper(II) sulphate.

Switch on the current and note the ammeter reading, then switch the current off again. Run out the solution and dilute it with an equal volume of water. Stir the mixture well and fill the cell with it.

Switch on the current again, and note the ammeter reading. Repeat the operation several times, thus measuring the current at various dilutions.

Plot a graph of resistance (i.e. the voltage divided by the current in amps) against dilution.

Experiment E5.13

How does the resistance of an electrolyte solution vary with the nature of the electrolyte (strong or weak)?

APPARATUS

Source of 6 volts d.c.

Ammeter, 0-5 amps

Five pieces of connecting wire

Reversing switch

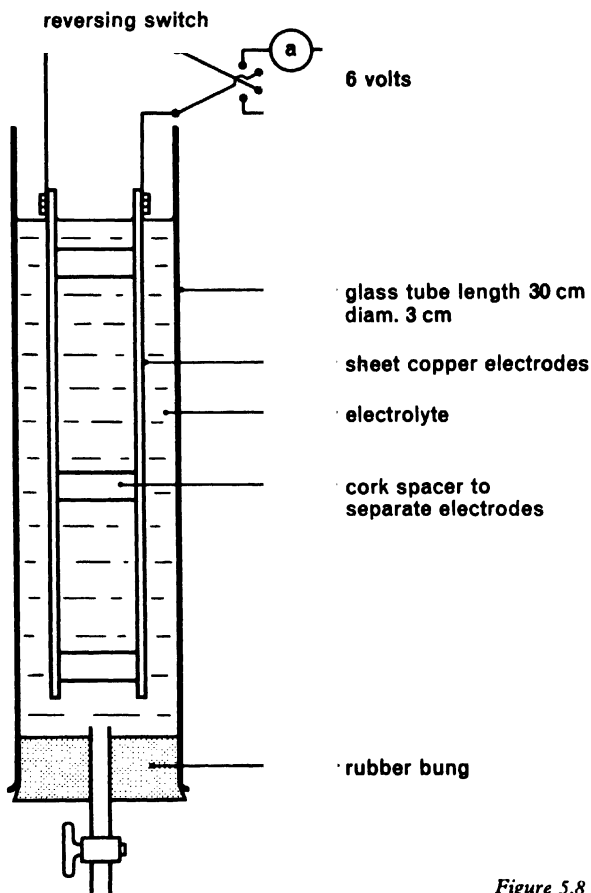
Special apparatus as shown in the diagram

Molar solutions of hydrochloric acid, sodium sulphate, ammonium hydroxide, acetic acid, etc.

PROCEDURE

Set up the apparatus as shown in figure 5.8. Cover the electrodes with molar hydrochloric acid, switch on the current, and note the ammeter reading. Reverse the direction of the current using the reversing switch and again note the ammeter reading.

Repeat these operations using molar solutions of other electrolytes, making sure that the apparatus is thoroughly rinsed out between each use.

*Figure 5.8*

It will be seen that electrolytes can be grouped roughly into two classes: those which allow a lot of current through, and those which do not allow much.

Experiment E5.14

How does the resistance of an electrolyte solution vary with the size of the electrode?

APPARATUS

Source of 6 volts d.c.

Ammeter, 0-5 amps

Three pieces of connecting wire

Special apparatus as shown in the diagram

Approximately molar copper(II) sulphate solution

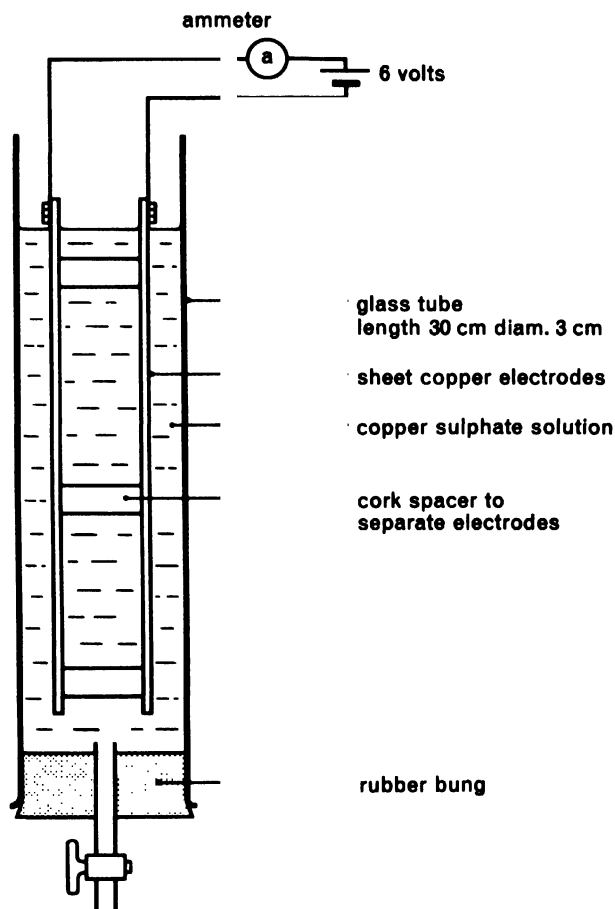


Figure 5.9

PROCEDURE

Set up the apparatus as shown in figure 5.9, measuring the electrodes carefully. Fill the tube up to the top of the electrodes with approximately molar copper(II) sulphate solution, switch on the current, and note the ammeter reading. Run out 20 cm³ of solution and again note the ammeter reading. Measure the level of the solution on the electrodes, and calculate how much of them is immersed.

Continue to run out successive 20 cm³ portions, taking the ammeter readings and measuring the amount of electrode immersed, until the electrodes are no longer in contact with any solution.

Plot a graph of resistance (i.e. voltage divided by the current in amps) against the area of electrode immersed.

Chapter 6

Elements, their classification, and their differences from compounds

Introduction

The idea of elements should not be forced on the pupils in a dogmatic way, but allowed to grow in their minds as their chemical experience increases. The first three experiments described in this chapter are of a type that will lead to the idea.

Using the results of these experiments the pupils can be helped to get the idea of an element by attempting to classify the substances that they have met into:

- a. Those that are *decomposed* into other substances by the action of heat or electricity.
- b. Those that are *formed* by these processes, and do not further decompose.

The rôle of elements in compound formation can then be made clear by experiments such as E6.4 and E6.5. All these experiments are suitable for use at an early stage in the pupils' progress.

At a more advanced level, attempts can be made to classify elements according to their chemical behaviour.

As a first step to this classification a division into metals and non-metals can be attempted. Experiment E6.7 suggests a way of comparing some of the properties of metals and non-metals; for another difference pupils may be reminded of the result of experiment E3.8 (if they have already done this) in which it was seen that metals when burnt yield basic oxides, whereas non-metals give acidic oxides.

Family relationships between elements can then be investigated, and experiments E6.8–E6.11 suggest ways by which these comparisons can be made. Varying degrees of reactivity of different elements can be investigated, and many more comparisons studied, using experiments given in Chapter 7.

As soon as the idea of atomic weights has been introduced, the Periodic Table can be used, and the relationship between it and the previous attempts at classification noted.

References

The ideas developed in this theme are discussed in the *Handbook for Teachers*, Chapter 3, *Making a start: the route to elements and the Periodic Table*.

Two methods of approach to the teaching of the theme of 'elements' are given in the Sample Scheme, Part I, *Basic Course*:

Stage I A, Topic 5: 'The elements'

Stage I B, Topic 7: 'The elements'

The periodic classification of the elements is used in:

Stage II, Topic 13: 'Looking at the elements in the light of the Periodic Table'

The classification of the elements is dealt with in more detail in the *Course of Options*, Option 13, 'Periodicity and Atomic Structure'.

List of experiments

The idea of elements

- E6.1 Decomposition of compounds on heating
- E6.2 Decomposition of compounds by electricity
- E6.3 To find out if lead is an element

The chemical combination of elements to form compounds

- E6.4 The reaction between iron and sulphur
- E6.5 The element is still present, and may be recovered from its compound
- E6.6 Differences between elements and compounds

The classification of elements

- E6.7 The physical differences between metals and non-metals
- E6.8 Comparing the properties of the alkali metals

- E6.9 Comparing the properties of the heavy metals
- E6.10 Comparing the properties of carbon and silicon
- E6.11 The halogens
- E6.12 The alkaline-earth metals
- E6.13 Chemical behaviour of aluminium

The idea of elements

Experiment E6.1

Decomposition of compounds on heating

Details of how to carry out an experiment on the action of heat on substances are given in experiment E1.1. If the particular point which it is wished to bring out is that of elements as the ultimate products of thermal decomposition, some other decomposable substances might be added to the list given in that experiment.

Examples include:

Mercury(II) oxide

This is best dealt with according to the details given in experiment E3.6. It is inadvisable to have a whole class heating quantities of this substance in an open tube, as mercury vapour is very poisonous.

Wood

This substance on strong heating in the absence of air decomposes to yield carbon. A little sawdust may be heated in a test-tube; alternatively the more elaborate method of experiment E10.2 can be employed. Pupils can be reminded of burnt toast, which is largely carbon.

Further experiments on carbon compounds which are relevant include experiments E10.1 and E10.3.

A mixture of cerussite and carbon

On heating this mixture a sample of lead is obtained by the reduction of lead carbonate. Details are given in experiment E2.23, and this mixture should certainly be heated if the theme of this chapter is being followed, and it is intended to do experiment E6.3.

Experiment E6.2

Decomposition of compounds by electricity

A simple experiment obtaining copper from copper sulphate solution is

experiment E2.25. A similar one obtaining zinc from zinc sulphate solution is given later in this chapter (experiment E6.5).

Other experiments are included in Chapter 5, and illustrate the decomposition of compounds both molten and in solution. The electrolysis of fused lead bromide (experiment E5.5) should be done if this theme is being followed, and it is intended to do experiment E6.3.

Experiment E6.3

To find out if lead is an element

Lead is one of the substances obtained as a result of the last two experiments and is therefore likely to be an element. In this experiment it is submitted to the action of heat and electricity and found not to change; this provides further evidence in favour of its being an element.

APPARATUS

Each pupil or pair of pupils will need:

Hard-glass test-tube, 100×16 mm

Test-tube holder

Bunsen burner and asbestos square

Two strips of lead foil, about 2×1 cm

Source of 2-3 volts d.c.

Torch-bulb in holder

Three pieces of connecting wire

PROCEDURE

Heat one of the strips of lead in the test-tube. It will melt and on cooling return to its former state, and it is not decomposed by heating. This can be confirmed by weighing the tube and contents before and after heating.

Test the electrical conductivity of the lead in the manner described in experiment E5.1. It will be found to conduct, but not to be changed chemically by the passage of the current.

The chemical combination of elements to form compounds

Experiment E6.4

The reaction between iron and sulphur

APPARATUS

Each pupil, or pair of pupils, will need:

Tongs

Asbestos paper strip, about 7×2 cm

Sulphur

Iron filings

PROCEDURE

Mix together a measure of sulphur with a measure of iron filings, and heat a small quantity of the mixture on a piece of asbestos paper. Note the evolution of heat, and that the reaction, once started, continues unaided.

Note: Some samples of iron filings may be found not to react with sulphur under the conditions of this experiment, and the teacher should therefore try out his supply in advance. Iron metal powder reduced by hydrogen (British Drug Houses Ltd) is satisfactory.

This experiment is extended to the reaction of other metals with sulphur in experiment E7.9.

Another experiment suitable to illustrate the formation of compounds from elements is the reaction between magnesium and oxygen, experiment E1.3 part (a).

Experiment E6.5

The element is still present, and may be recovered from its compound

There are a number of possible experiments in which an element can be converted into one of its compounds, which can be seen to be noticeably different in form, and subsequently recovered from the compound. Some of these are now suggested.

Experiment a. Mercury is converted into mercury(II) oxide, and then recovered from the oxide

Owing to the poisonous nature of mercury and its compounds, this experiment must be performed by the teacher.

Mercury can be converted into mercury(II) oxide using the method described in experiment E3.7.

The element can then be recovered as described in experiment E3.6.

Experiment b. Carbon is burnt to form carbon dioxide, and then recovered from the oxide

APPARATUS

Each pupil or pair of pupils will need:

Hard-glass test-tube, 150×25 mm, with cork to fit

Test-tube rack

Combustion spoon

Bunsen burner and asbestos square

Tongs

Small piece of wood charcoal

Magnesium ribbon (about 5 cm)

The teacher will need:

Cylinder of oxygen

Length of rubber tubing to lead oxygen from cylinder to pupil's test-tube

PROCEDURE

Fill a test-tube with oxygen, put a cork in it, and stand it in a test-tube rack. Place a small piece of wood charcoal in a combustion spoon, heat it to redness in a Bunsen burner flame and insert it into the test-tube of oxygen. It will be seen to glow strongly, and carbon dioxide will be formed. When the glowing ceases, remove the combustion spoon and replace the cork.

Hold a 5 cm length of magnesium ribbon in a pair of tongs in the Bunsen burner flame until it catches fire. Quickly transfer it, still held in the tongs, to the test-tube of carbon dioxide. The magnesium will continue to burn in this gas. When burning has finished, remove the tongs and replace the cork. Examine the contents of the test-tube. Black specks of carbon can be seen; if they are not clear, the addition of a little dilute sulphuric acid will remove the magnesium oxide and any unburnt magnesium and make the carbon more obvious.

Experiment c. Zinc is converted to zinc sulphate, and then recovered from this compound using an electrical method

APPARATUS

Each pupil or pair of pupils will need:

Two beakers, 100 cm³

Two carbon electrodes

Source of 10-12 volts d.c.

Two pieces of connecting wire fitted with crocodile clips

Stand and clamp

Filter funnel and paper
Zinc, granulated
Dilute sulphuric acid
Copper(II) sulphate solution

PROCEDURE

Place a few pieces of granulated zinc in a 100 cm³ beaker, cover them with dilute sulphuric acid, and add a few drops of copper(II) sulphate solution. The zinc will be converted steadily into zinc sulphate solution. When the reaction has ceased (no more bubbles of hydrogen are evolved) pour the contents of the beaker through a filter paper held in a filter funnel supported by a stand and clamp, and collect the filtrate in a second beaker.

Evaporate this solution to obtain colourless crystals of zinc sulphate, obviously different in appearance from zinc.

To recover the zinc from the zinc sulphate, dissolve the crystals in water, place the two carbon electrodes in the solution, making sure that they do not touch, and connect them to a source of about 10–12 volts d.c. The zinc will rapidly form on one of the electrodes (the cathode) and care should be taken to see that the long strands of zinc which grow towards the other electrode do not join the two together.

Experiment d. Copper is converted into copper nitrate; this is then heated until it has decomposed to the oxide, and the metal is then recovered from this oxide

APPARATUS

Each pupil or pair of pupils will need:

Hard-glass test-tube, 150 × 25 mm
Hard-glass test-tube, 125 × 16 mm, with a small hole blown at the end, bung and straight glass delivery tube to fit, and rubber connecting tube to join to gas supply
Bunsen burner
Stand and clamp
Spatula
Copper metal turnings
Concentrated nitric acid

PROCEDURE

Place a few copper turnings in a 150 × 25 mm hard-glass test-tube, support it by means of a stand and clamp and place it in a fume cupboard. Cautiously add concentrated nitric acid a little at a time until no

further reaction takes place (no more brown fumes of nitrogen dioxide are evolved and no copper remains). Gently heat the green solution by means of a Bunsen burner until the water and any excess of nitric acid have been boiled off. The blue solid which remains is copper(II) nitrate.

Continue heating until the blue colour turns black (copper(II) oxide) and there is no further evolution of brown fumes.

Scrape out the copper(II) oxide with a spatula and place it in a 125×16 mm test-tube with a small hole blown at the end. Fit the tube with a bung carrying a short glass delivery tube and connect this to the town gas supply. Pass a small steady stream of town gas through the apparatus. As soon as it is certain that all the air has been displaced from the test-tube, but *NOT* before, the town gas should be ignited at the small hole (about 10 seconds of gas flow will ensure this). Heat the contents of the tube with a colourless Bunsen burner flame about 5 cm high. After a short time a red glow will spread through the copper(II) oxide and it will change from black to the pink colour of copper. Remove the burner, but do not turn the gas supply through the tube off until it is cool, otherwise air may enter and reoxidize the copper.

Experiment E6.6

The differences between elements and compounds

Experiment a.

In this experiment, the differences between elements and the compounds that they form are investigated; hydrogen, chlorine, and hydrogen chloride are used as examples to illustrate these differences.

Part 1. The differences between hydrogen and chlorine

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B8.1)

Four test-tubes, 150×25 mm, fitted with corks

Test-tube rack

Shallow beaker or small trough

Source of hydrogen (cylinder or standard preparation, see experiment E4.3)

Source of chlorine (standard preparation, see experiment E6.8)

Wood splint

Indicator paper

PROCEDURE

Fill three test-tubes with hydrogen from whatever source is available and cork them up. Open one under water in the beaker or trough to find

out whether the gas is soluble. Hold the second *beneath* an empty test-tube for a few seconds so that the hydrogen passes from the lower to the upper test-tube. Apply a light to both to find out where the hydrogen is. Do a similar experiment with the third test-tube of hydrogen but hold it *above* an empty test-tube. A lighted splint applied to the mouth of each shows that the hydrogen has not passed downwards to the lower test-tube. Be careful not to hold the test-tubes together for too long as some hydrogen may enter the lower tube by diffusion.

Next, fill three test-tubes with chlorine, and repeat the experiments on solubility and density. The presence of the chlorine can be detected by the use of damp indicator paper instead of a flame. Compare the results obtained.

Part 2. The formation of hydrogen chloride, and an examination of some of its properties

This experiment **must** be done by the teacher.

APPARATUS

Plastic safety screen

Electrodes and 6-volt battery for testing for conductivity

Indicator paper

Special delivery tube

Beaker, 100 cm³ or test-tube, 150 × 25 mm

Hydrogen cylinder

Three gas jars, with covers, full of chlorine (for preparation, see experiment E6.8)

Trough of water

hydrogen



Figure 6.1

PROCEDURE

Burn the hydrogen at the end of a delivery tube about 25 cm long, bent at the end into a U-shape as shown in figure 6.1. Partly remove the cover from a gas jar of chlorine and lower the flame into it. Note the colour of the flame and the nature of the gas formed. Test this by blowing damp air across the top of the jar and observing the fumes produced. Also test it with damp indicator paper. Repeat the experiment and invert the gas jar containing the hydrogen chloride over water. Notice that the water rises well up into the jar. Burn the hydrogen in the third gas jar, add a few cm³ of water, shake, and test the resulting solution with indicator paper and for electrical conductivity.

Experiment b.

The differences could also be brought out using, say, hydrogen, oxygen, and water.

*Part 1. The differences between hydrogen and oxygen***APPARATUS**

Each pupil or pair of pupils will need:

Four test-tubes, 150 × 25 mm, fitted with corks

Test-tube rack

Wood splints

The teacher will need:

Cylinder of hydrogen (or standard preparation, see experiment E4.3)

Cylinder of oxygen

Rubber tubing to connect cylinders to pupils' test-tubes

PROCEDURE

Fill two test-tubes with oxygen and two with hydrogen. Place a lighted splint in one test-tube of each gas; in oxygen the splint will flare up but in hydrogen the gas itself will catch fire. Next place a glowing splint in one test-tube of each gas; in oxygen the splint will be rekindled; in hydrogen the gas may catch fire but the splint will go out.

Part 2. The formation of water, and an examination of some of its properties

This is described in experiment E4.4.

Experiment c.

The differences between elements and their compounds can be brought out in a more general way using some metals, sulphur, chlorine, oxygen, and the compounds formed between these elements.

Part 1. The differences between metals and non-metals

Some of these differences can be seen by doing experiment E6.7.

*Part 2. The formation of metal sulphides, chlorides, and oxides and an examination of some of their properties**Formation of some sulphides*

This experiment should be done by the teacher.

APPARATUS

Bunsen burner and asbestos square

Tongs

Spatula

Asbestos paper strips, 7×2 cm approximately

Sulphur powder

Zinc dust

Iron filings

Copper powder

PROCEDURE

Mix a spatula measure of sulphur with a spatula measure of each metal powder in turn. Place a small quantity of each mixture on a piece of asbestos paper, folded so that its cross section is V-shaped. Hold the asbestos paper with a pair of tongs, and heat the mixture with a Bunsen burner. Note the evolution of heat in each case; the reaction with zinc is the most vigorous.

Formation of some chlorides

This experiment should be done by the teacher.

APPARATUS

Combustion spoon suitable for use in gas jars

Bunsen burner and asbestos square

Tongs

Four gas jars of chlorine

Steel wool

Sodium

'Dutch metal'

Phosphorus

PROCEDURE

Heat each element in turn and plunge it into a jar of chlorine. Use very small quantities, particularly of the sodium and phosphorus, the size of which should be smaller than a rice grain. The steel wool can be held in tongs, heated to redness and at once transferred to the chlorine. The

Dutch metal should be dry and need not be heated beforehand. The sodium and phosphorus should be melted in a combustion spoon and then held in the chlorine. In each case point out the evolution of heat as the element combines with chlorine. Note that the sodium gives a coloured product owing to the attack of the spoon by the chlorine.

Formation of some oxides

This is described in experiment E3.1.

Finding out some properties of sulphides, chlorides, and oxides

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B8.2c)

Six test-tubes, 100 × 16 mm, in a rack

Universal Indicator paper

Electrode holder with pair of steel electrodes

6-volt battery and 6-volt bulb in holder

Three lengths of connecting wire fitted with crocodile clips

Distilled water

CHEMICALS

Chlorides:

Sodium, magnesium, zinc, iron(II), and copper(II) chlorides, phosphorus trichloride, and carbon tetrachloride.

Sulphides:

Zinc, iron(II), copper(II), sodium, and calcium sulphides, carbon disulphide.

Oxides:

Magnesium, zinc, iron(III), calcium, and copper(II) oxides, phosphorus pentoxide.

The pupils should *not* be allowed access to the bottles of carbon tetrachloride, phosphorus trichloride, or carbon disulphide, as these liquids are too poisonous or dangerous for them to handle at this stage. If he likes, the teacher can give some pupils one drop of each of the liquids in test-tubes, but no more.

PROCEDURE

Test each of the substances mentioned in the list above for solubility. Note any heat changes on the addition of water, and test any resulting solution for pH and for electrical conductivity.

Use very small quantities, one drop in the case of the liquids and half a measure of the solids will be sufficient. Add sufficient distilled water in each case to fill the test-tube about one-third full.

The classification of elements

Experiment E6.7

The physical differences between metals and non-metals

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A5.1a)

Bunsen burner and asbestos square

6-volt bulb and bulb holder

Source of approximately 6 volts d.c.

Three lengths of connecting wire, fitted with crocodile clips

Carbon, sulphur, copper, aluminium, lead, and other metal rods as available, and of similar diameter about 15 cm long. Alternatively, pieces of metal foil of approximately the same thickness and about 6×2 cm

PROCEDURE

1. Try to put the elements in approximate order of density by weighing the samples and estimating their sizes.
2. Test the elements for brittleness by hitting the samples with a hard object such as the base of a Bunsen burner. Divide the elements into those which are easily broken up and those which are not.
3. Test for thermal conductivity by holding one end of a rod or foil of each element in a Bunsen burner flame and comparing the time taken for the other end to become too hot to hold. (Do not test the sulphur in this way.)
4. Test for electrical conductivity by using the 6-volt supply, bulb, and bulb holder and connecting the circuit across the sample with crocodile clips. The bulb will light up for all the samples except sulphur. Carbon will conduct electricity if it is in the form of graphite.

Experiment E6.8

Comparing the properties of the alkali metals

Owing to the danger involved in handling the more reactive alkali metals, the teacher should show the pupils the properties of sodium and potassium. The pupils can then find out if lithium, the least reactive of the alkali metals, has similar properties.

Part 1. To be done by the teacher – the burning of sodium and potassium, and their reaction with chlorine

APPARATUS

Chlorine generator (set up in the fume cupboard) made from a filter flask, 100 cm³, fitted with a bung carrying a tap funnel and delivery tube

Stand and clamp

Two gas jars and covers

Two combustion spoons for use in gas jars

Bunsen burner and asbestos square

Filter paper

Asbestos paper

Concentrated hydrochloric acid

Potassium permanganate

Vaseline

Sodium

Potassium

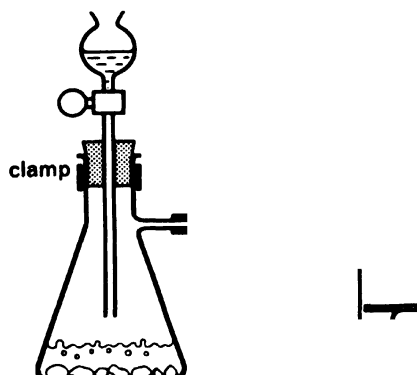


Figure 6.2

PROCEDURE

WARNING: Chlorine gas is extremely dangerous if inhaled. Also care must be taken to avoid the risk of pieces of burning sodium and potassium flying about the room and to use only very small pieces of these metals.

a. To obtain several gas jars of chlorine. This part of the experiment must be carried out in a well-ventilated fume cupboard. Chlorine sufficiently pure for this experiment can be obtained by dropping concentrated hydrochloric acid onto potassium permanganate crystals, using a filter flask as the generator as shown in figure 6.2. Chlorine is denser than air and if you stand the gas jar in front of a white background the greenish-yellow colour of the gas will enable you to see when the jar is full. It should be sealed with a vaselined cover.

b. The action of chlorine on the alkali metals. Remove a small piece of sodium (a cube of side 3–4 mm is suitable) from the bottle and free it from the protective paraffin oil under which it is kept by pressing it gently between filter papers. Cut away any surface coating of oxide, etc. and place the piece in a combustion spoon. Heat the spoon in a Bunsen burner flame carefully and as soon as the sodium shows signs of burning, plunge the spoon into the jar of chlorine. The sodium will continue to 'burn', white clouds of sodium chloride being formed. The product may be contaminated by the chlorides of metals present in the combustion spoon; iron for example will give a reddish colour. Contamination may be kept to a minimum by placing a piece of asbestos paper between the sodium and the combustion spoon.

Repeat the experiment using potassium, which is more reactive than sodium.

Note: Small pieces of alkali metals which may be left over should not be returned to the stock bottle, and must on no account be put into sinks or rubbish boxes. They can be dealt with by putting them into an excess of industrial methylated spirits, which may be safely poured away after the metals have finished reacting.

Part 2. To be done by the teacher – the action of water on sodium and potassium

This is described in experiment E4.2.

Part 3. To be done by the pupils – to find out if the properties of lithium are similar to those of sodium and potassium

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 13.2)

Beaker, 100 cm³

Tongs and piece of broken crucible or crucible lid;
or tripod, pipeclay triangle, and piece of asbestos paper

Universal Indicator solution

Bunsen burner and asbestos square

Two small pieces of lithium

PROCEDURE

Heat a piece of lithium about the size of a rice-grain on a piece of broken crucible, or an old crucible lid held by a pair of tongs, until it catches fire and burns. Alternatively use a piece of asbestos paper supported on a pipe-clay triangle and tripod. The reaction should be compared with that obtained by the teacher in Part 1 of the experiment (in which sodium and potassium were ignited before being lowered into gas jars of chlorine).

Take a second piece of lithium about the size of a rice-grain and put it on some water in a beaker. After the reaction has finished put a few drops of Universal Indicator into the water and compare the result with that obtained by the teacher for sodium and potassium.

Finally, the teacher may demonstrate the action of chlorine on heated lithium in the same manner as was used for sodium and potassium earlier.

Experiment E6.9

Comparing the properties of the heavy metals

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 13.4)

Six hard-glass test-tubes, 100 × 16 mm

Samples of:

Sodium carbonate

Copper carbonate

Nickel carbonate

Iron(II) carbonate

Cobalt carbonate

Manganese carbonate

Approx. 2M hydrochloric acid

Copper foil

Nickel foil

Iron wire

PROCEDURE

Place a spatula measure of each of the carbonates in a test-tube and add half a test-tube of the hydrochloric acid. Observe the reaction and note the colour of the resulting solution.

Test the metal samples for strength and ductility, comparing their physical properties with those of the alkali metals. Try the action of cold and hot water on these metals.

Further tests may be devised, and the *Book of Data* used to make a comparison of the physical properties of the heavy metals.

Experiment E6.10**Comparing the properties of carbon and silicon**

The removal of oxygen from carbon dioxide using magnesium was described in experiment E6.5, experiment (b). This can be compared with the reaction between magnesium and silicon dioxide as now described, and the similarities noted.

Some of the properties of the silicon made in this experiment, such as its appearance, solubility in water and the action on it of acids and alkalis, can then be compared with those of a sample of charcoal.

This experiment **must** be done by the teacher.

Caution: As explosions have been known to occur during this experiment owing to the presence of water, make sure that the sand and tube are absolutely dry.

APPARATUS

Plastic safety screen

Hard-glass test-tube, 100×16 mm

Test-tube holder

Bunsen burner and asbestos square

Beaker, 100 cm³ or 250 cm³

Watch-glass big enough to rest on top of the beaker

Tripod and gauze

Funnel and quick filter paper

Funnel stand

Magnesium powder

Sand

2M hydrochloric acid

PROCEDURE

First make an intimate mixture of magnesium powder (1 part by volume) and dry purified sand (2 parts by volume). About 2-3 g of the mixture are required.

Place the mixture in a hard-glass test-tube, and clamp it horizontally. Then heat the mixture at the end nearest the mouth of the tube with a Bunsen burner. As the reaction proceeds and the mixture glows, follow the glow with the flame to the bottom of the tube. A sample of silicon can be obtained as follows: when the tube is cool, shake its contents onto an asbestos square for examination and then transfer them to a beaker containing about 20 cm³ of 2M hydrochloric acid. This will react with the magnesium oxide and any magnesium silicide that may be present; the latter will form gaseous hydrides of silicon and a few harmless explosions may occur as they ignite on contact with the air.

Place the beaker on a tripod and gauze and heat the contents just to boiling-point with a Bunsen burner. Filter the hot mixture through a quick filter paper, and wash the silicon on the filter paper with a little hot hydrochloric acid, followed by a quantity of water. The paper plus silicon can then be transferred to a watch-glass and dried.

Experiment E6.11**The halogens****Experiment a.**

This experiment should be done by the teacher.

APPARATUS

- (1) Gas jar of chlorine
- (2) 500 or 1000 cm³ round-bottomed flask, stoppered and containing 3 or 4 drops of bromine
- (3) 500 cm³ round-bottomed flask containing 5-6 g of solid iodine

PROCEDURE

Into (1) sprinkle a little dry, powdered antimony.

Into (2) drop a little tin-foil and heat gently if no immediate reaction occurs.

To (3) add a small globule of mercury and warm gently.

Note the reactions between the halogens and metals.

Experiment b.**APPARATUS**

Each pupil or pair of pupils will need:

Four test-tubes, 125×16 mm, in rack

Silver nitrate solution

A measure each of potassium fluoride, chloride, bromide, and iodide

Concentrated sulphuric acid

Litmus paper

Chlorine water

Bromine water

Carbon tetrachloride

Strong ammonia solution

PROCEDURE

1. Make 5 cm^3 of a dilute solution of each potassium salt, add a few drops of silver nitrate solution to each and note the colours of the precipitates. Allow each to settle, decant off the clear liquid, and add $3\text{--}4 \text{ cm}^3$ of strong ammonia solution. Which precipitates dissolve?

2. Add (cautiously) 1–2 drops of concentrated sulphuric acid to strong solutions of each of the potassium salts in turn. Test the action of any liberated gases on moist litmus paper.

3. Add chlorine water to a solution of potassium bromide and add bromine water to a potassium iodide solution. Add an equal volume of carbon tetrachloride to each solution and shake. Note the characteristic colours of the carbon tetrachloride layer.

The colours of the silver halides, and their solubility in water and concentrated ammonia solution are as follows:

<i>Compound</i>	<i>Colour</i>	<i>Solubility in water</i>	<i>Solubility in concentrated ammonia solution</i>
AgF	White	Large	—
AgCl	White	$1.5 \times 10^{-3} \text{ g/l}$	Soluble
AgBr	Creamy	$1.4 \times 10^{-4} \text{ g/l}$	Slightly soluble
AgI	Yellow	$2.5 \times 10^{-6} \text{ g/l}$	Insoluble

Experiment E6.12

The alkaline-earth metals

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Three test-tubes, 125×16 mm, in rack

Magnesium ribbon (about 30 cm) and emery cloth to clean it

Small pieces of calcium and barium
 Magnesium sulphate solution
 Strontium chloride solution
 Strontium sulphate solution
 Calcium sulphate solution
 Barium chloride solution
 2M sodium hydroxide solution
 2M hydrochloric acid

PROCEDURE

1. Drop a piece of bright clean magnesium ribbon into each of three tubes and cover with distilled water. Heat the water in one tube and pour a few drops of dilute hydrochloric acid into another. Compare the rates of the reactions in the three tubes.
2. Treat small pieces of calcium and barium with cold water in test-tubes. Test the gases evolved for inflammability.
3. Add sodium hydroxide solution to a solution of magnesium sulphate and treat the precipitate with dilute hydrochloric acid.
4. Add strontium chloride solution to a saturated solution of calcium sulphate, and add barium chloride solution to a saturated solution of strontium sulphate.

The solubilities of the hydroxides and sulphates of these metals are as follows:

	<i>Solubility of hydroxide</i>	<i>Solubility of sulphate</i>
Magnesium	0.0003 mol/l	3.1 mol/l
Calcium	0.022 mol/l	0.015 mol/l
Strontium	0.066 mol/l	0.0015 mol/l
Barium	0.233 mol/l	0.000011 mol/l

Experiment E6.13

Chemical behaviour of aluminium

APPARATUS

Each pupil or pair of pupils will need:

Six test-tubes, 125 × 16 mm, in rack
 Small pieces of aluminium foil
 Aluminium chloride solution
 Mercuric chloride solution
 2M hydrochloric acid

2M sodium hydroxide solution

2M ammonia solution

Cotton wool

Sodium chloride

Magnesium chloride

Universal Indicator solution

PROCEDURE

1. Add small pieces of aluminium foil to:

- a) distilled water
- b) 2M hydrochloric acid
- c) sodium hydroxide solution

in test-tubes, warming gently. Add ammonia solution to tube (b)

2. Hold a piece of aluminium foil with tongs, dip it in mercuric chloride solution, and immerse it in hot water. (*Care: mercuric chloride is very poisonous.*)

3. Prepare aluminium hydroxide by adding ammonia solution to a solution of aluminium chloride. Divide the precipitate into two parts. Add dilute hydrochloric acid to one part and sodium hydroxide solution to the other.

4. Examine the acidity of solutions of sodium chloride, magnesium chloride and aluminium chloride in water, using Universal Indicator solution.

A solution of sodium chloride is seen to be neutral, one of magnesium chloride acidic, and one of aluminium chloride more acidic.

Chapter 7

Reactivity series

Introduction

The idea that elements can be arranged in an order of reactivity has been introduced in other chapters, notably in experiment E3.1, 'What happens when metals are burnt?' and experiment E4.2, 'What happens when metals are placed in water?' The experiments in this chapter develop this theme.

These experiments are for the most part concerned with the metallic elements and begin with the burning of some metals in oxygen (an amplified version of experiment E3.1) and a study of the reactions of these metals with water and steam (an amplified version of experiment E4.2).

The reactions with water and steam are of course concerned with the *displacement of hydrogen from water by metals*, and suggest two questions:

- Can this displacement be reversed, that is, can hydrogen displace metals from oxides to form water?* This question is answered in experiment E7.4.

- Can the field be widened, that is, can some metals displace other metals from their oxides?* This question is answered in experiment E7.5.

From these experiments the metals can be arranged in a reactivity series.

The fundamental nature of the reactivity series can then be tested by finding out what happens when metals react with acids, chlorine and sulphur; and the elements carbon and hydrogen can be introduced into the series.

A displacement series can be constructed for the metals using experiment E7.10, and the idea extended to non-metals by means of experiment E7.11.

The results of these experiments can be compared with those obtained by means of electrolysis (experiment E5.10, 'Are different cations discharged preferentially?').

References

This theme is introduced in the Sample Scheme, Part I, *Basic Course*, in two ways at the following points:

Stage 1 A, Topic 6: 'Competition among the elements'

Stage 1 B, Topic 10: 'Competition among the elements'

List of experiments

- E7.1 What happens when elements are burned in oxygen?
- E7.2 What happens when metals are placed in water?
- E7.3 What happens when metals are heated in steam?
- E7.4 Can hydrogen be used to remove the oxygen from metal oxides?
- E7.5 Can metals be used to remove the oxygen from the oxides of other metals?
- E7.6 Where does carbon come in the reactivity series?
- E7.7 What happens when metals are placed in acids?
- E7.8 What happens when metals are heated with chlorine?
- E7.9 What happens when metals are heated with sulphur?
- E7.10 How to make a displacement series for metals
- E7.11 Can a displacement series be made for non-metals?

Experiment E7.1

What happens when elements are burned in oxygen?

This is an advanced version of experiment E3.1, and is designed to bring out the idea of a reactivity series. Although mostly concerned with metals, three non-metallic elements are included for comparison. The experiment demands careful observation and recording.

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Combustion spoon to fit a gas jar

Gas jar and lid

Spatula

Universal Indicator solution or litmus solution

Samples of elements mentioned in the table below, in the amounts and states given there

The teacher will need:

Cylinder of oxygen

Rubber tubing to connect oxygen supply to pupils' gas jars

PROCEDURE

Fill the gas jar with oxygen from a cylinder. Take a sample of one of the elements that are provided, and place it in a combustion spoon. Special care should be taken with sodium and phosphorus; sodium must not be touched at all, and the red form of phosphorus must be used – not the yellow.

Hold the spoon in the flame of a Bunsen burner until the sample of element has caught fire, or is red hot, and then place it quickly in the gas jar. As soon as it has finished burning, remove the combustion spoon (with care, in case the element catches fire again on contact with the air) and replace the lid of the gas jar. Note what you have seen. Now place a few cm^3 of distilled water in the gas jar, shake well and note what happens. Add a few drops of Universal Indicator solution, or litmus solution, and note the result.

Repeat the experiment for each of the elements provided.

The pupils should be encouraged to record their results in tabular form; an example of such a table is now given.

RESULTS

<i>Element</i>	<i>Amount and State</i>	<i>Appearance of reaction</i>	<i>Appearance of product</i>	<i>Solubility in distilled water</i>	<i>Reaction of Solution with Universal Indicator Litmus</i>
Sodium	2 mm cube	Brilliant yellow flame	White marks on sides of gas jar	Dissolves completely	Violet (pH 11.0) Blue
Calcium	1 cm long turning scraped	Vigorous orange flame	White solid formed	Forms a white suspension	Blue-violet (pH 10.0) Blue
Magnesium	10 cm ribbon	Brilliant white flame	White powder mainly on spoon	Forms a white suspension	Blue-violet (pH 10.0) Bluish purple
Zinc	Zinc dust half filling the spoon or thin strips of foil	Strong greenish-yellow flame	Yellow powder which is white on cooling	Forms a white suspension	Yellowish green (pH 8.0) Bluish purple
Tin	Foil cut in thin strips	Small yellow flame	White powder	Forms a white suspension	Yellowish green (pH 8.0-7.0) Purple
Iron	Reduced iron powder, spoon half-full	Yellow glow	Black lump in spoon	Lump remains unchanged	Yellowish green (pH 7.0) Purple
Copper	Strips of foil	Momentary glow	Blackish appearance on foil	No visible change	Yellowish green (pH 7.0) Purple
Carbon	Powdered wood carbon, spoon half-full	Red glow	Nothing to see	No visible change but gas jar lid difficult to remove	Yellow (pH 6.0) Dull red
Phosphorus	Red phosphorus, spoon half-full	Yellow flame	White smoke	Smoke dissolves; gas jar lid difficult to remove	Red (pH 5.0) Red
Sulphur	Flowers of sulphur, spoon half-full	Bright mauve flame	Nothing to see	No visible change but gas jar lid difficult to remove	Red (pH 4.0) Red

Experiment E7.2

What happens when metals are placed in water?

The idea of a reactivity series can also be brought out using the action of metals on water. Details of how to examine these reactions are given in experiment E4.2, and that experiment can be extended to other metals. In addition to sodium and potassium, lithium can be treated as in part (1), and additionally aluminium, zinc, tin, iron, and lead as in part (3). In the latter case, only magnesium will give appreciable quantities of gas, and this result will lead into the next experiment, in which the action of steam on the metals is examined.

Experiment E7.3

What happens when metals are heated in steam?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A7.3)

Stand and clamp

Bunsen burner and asbestos square

Hard-glass test-tube, 125×16 mm, fitted with bung and delivery tube

Four test-tubes, 150×25 mm, and corks

Small trough

Teat pipette

Spatula

Asbestos wool

Iron filings

Zinc powder or filings

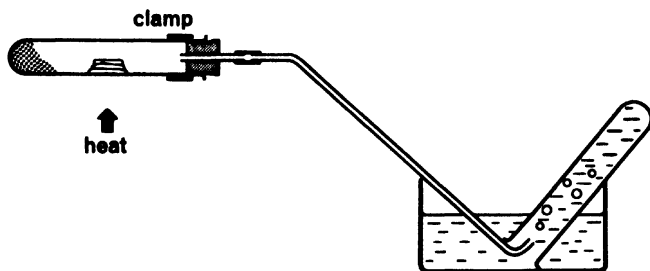


Figure 7.1

PROCEDURE

Caution: Warn the pupils about the danger of 'sucking back' before allowing them to start the experiment.

Place some loosely packed asbestos wool in a 125×16 mm hard-glass test-tube to a depth of about 3 cm. Add by teat pipette as much water as the asbestos will absorb (about 2 cm^3 is sufficient). Spread about one spatula measure of zinc or iron filings in the test-tube, at the position indicated in the diagram. Then assemble the rest of the apparatus.

Heat the part of the tube holding the metal, by means of a Bunsen burner, gently at first. Do not heat the asbestos directly. By moving the flame backwards and forwards, boil the water in the asbestos and keep the metal hot. In this way a gentle flow of steam is passed over the heated metal.

Collect the hydrogen by the displacement of water from the 150×25 mm test-tubes which are inserted in the trough. Try the effect of igniting (a) a full test-tube of the hydrogen, and (b) a test-tube containing half hydrogen and half air.

In addition to zinc and iron, other metals can be examined in a similar manner; they must be metals that do not react with cold water, and examples include aluminium, nickel, tin, and copper.

Experiment E7.4

Can hydrogen be used to remove the oxygen from metal oxides?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B10.1b)

Hard-glass test-tube, 125×16 mm, with a hole blown near the closed end, or a special combustion tube

One-hole cork or bung to fit test-tube, carrying glass delivery tube

Rubber connecting tube to connect town gas supply to test-tube

U-tube or calcium chloride tube containing silica gel or anhydrous calcium chloride

Bunsen burner and asbestos square

Copper(II) oxide, previously dried

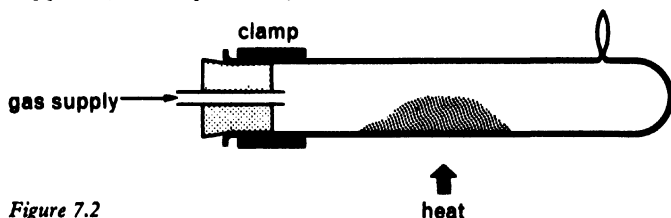


Figure 7.2

PROCEDURE

In this experiment dry town gas is used as a source of supply of hydrogen. Set up the apparatus as shown in the diagram. Turn on the supply of town gas and when it is certain that all the air has been displaced from the apparatus (about ten seconds rapid flow of gas will ensure this) light the gas as it escapes from the small hole in the test-tube. As soon as it is lit adjust the flame to a height of about two cm.

Heat the copper oxide gently and, as soon as the exothermic reaction starts, take the burner away.

The copper(II) oxide will turn pink, showing that it has lost its oxygen; and if the heating has been moderate enough, water will be seen condensed at the closed end of the test-tube. As soon as the tube is cool, turn off the gas supply; do not do this while the tube is still hot, or the copper will reoxidize. The pink residue, which consists of a sintered powder, can be shown to be a metal by touching it with the two leads from a bulb and battery.

This experiment can be repeated using samples of zinc oxide, iron(III) oxide and lead(II) oxide. In this way the position of hydrogen in the reactivity series can be found.

Experiment E7.5

Can metals be used to remove the oxygen from the oxides of other metals?

A. This experiment **must** be demonstrated by the teacher.

APPARATUS

Plastic safety screen

Small crucible or piece of asbestos paper

Tripod

Pipe-clay triangle (if crucible is used)

Bunsen burner and asbestos square

Beaker, 100 cm³

Filter funnel and paper

Stand and clamp

Spatula

2M hydrochloric acid

Magnesium powder

Copper(II) oxide or lead(II) oxide

Magnesium oxide

PROCEDURE

Heat about one spatula measure of a mixture of equal parts of magnesium powder and dry copper(II) oxide – or lead(II) oxide – in an open crucible or on a piece of asbestos paper. After a few moments a violent reaction occurs. In view of this, the teacher should place the Bunsen burner under the mixture and stand back until the reaction is over.

The pupils will expect to see some copper at the end! Very little remains, so repeat the experiment but moderate the reaction by mixing in some magnesium oxide. Then, either dissolve away the magnesium oxide at the end with 2M hydrochloric acid and filter off the metal residue, or do a control experiment with magnesium powder only. The reaction with copper oxide is seen to be much more vigorous and the residue is very different.

B. This experiment **must** be done by the teacher.

APPARATUS

Plastic safety screen

Silica crucible

Tin of sand

Asbestos squares

Spatula

Taper

Aluminium powder

Iron(III) oxide (3 to 5 g is sufficient; thoroughly dried by heating in an evaporating dish over a Bunsen burner)

Barium peroxide

Magnesium powder and ribbon

PROCEDURE

Mix together about equal volumes of dry aluminium powder and thoroughly dried iron(III) oxide (see above). Place the mixture in a silica crucible and stand this in a tin filled with sand. Place a spatula full of a mixture of barium peroxide and magnesium powder on top of the first mixture and insert a piece of freshly scraped magnesium ribbon through this pile to act as a fuse. Light the magnesium ribbon with a taper fixed into the end of a long glass tube and stand well back. *An extremely vigorous reaction takes place*, quantities of light and smoke are emitted, and a bead of iron can be emptied out when the reaction has subsided.

This is known as the thermit reaction.

After seeing this experiment the pupils can try a few examples for themselves. *Do not allow them to heat any of the oxides with sodium, magnesium, or calcium as these reactions can be dangerous.*

c. An experiment for pupils

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations*

Asbestos paper strips, approximately 7×2 cm

Bunsen burner and asbestos square

Tongs

Iron filings

Zinc oxide

Copper(II) oxide

Lead(II) oxide

PROCEDURE

Mix together a little of one of the oxides with an equal volume of iron filings, and heat the mixture on a strip of asbestos paper. If a reaction occurs a glow will spread through the mass. Repeat with the other oxides.

Experiment E7.6

Where does carbon come in the reactivity series?

Part 1. A competition between carbon and copper

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B10.1a)

Two hard-glass test-tubes, 100×16 mm

Test-tube holder

Bunsen burner and asbestos square

Teat pipette

Spatula

Lime water

Copper(II) oxide

Carbon (dry powdered wood charcoal)

PROCEDURE

Place a spatula measure of copper(II) oxide and a spatula measure of carbon (powdered wood charcoal is suitable) in a test-tube and mix them well by shaking. Heat the test-tube for some time in a near vertical position so as to ensure that any carbon dioxide formed does not 'fall

out'. Then allow the test-tube to cool, withdraw a sample of the gas above the heated solid by means of a teat pipette, and bubble through a *small* quantity of lime water in another test-tube.

Carbon dioxide will be detected.

Part 2. A competition between carbon and lead

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A6.2)

Asbestos paper strip, approximately 7×2 cm

Bunsen burner and asbestos square

Tongs

Watch-glass

Spatula

Magnesium oxide

Lead(II) oxide

Carbon (dry powdered wood charcoal)

PROCEDURE

Mix together a little lead(II) oxide with an equal volume of powdered charcoal on a watch-glass and transfer to a piece of asbestos paper folded so that its cross-section is V-shaped. Hold the asbestos paper with a pair of tongs, and heat the mixture with a Bunsen burner.

After a short time beads of molten lead appear. A bead can be shown to have one property of lead by washing it and rubbing it on paper with the fingernail. Lead marks the paper. Samples of lead beads on asbestos paper may be stuck into laboratory books with Sellotape.

Part 3. A competition between carbon and magnesium

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A6.2)

Gas jar and cover or corked hard-glass test-tube, 150×25 mm

Bunsen burner and asbestos square

Tongs

Magnesium ribbon, approximately 5 cm

The teacher will need a supply of carbon dioxide.

PROCEDURE

Fill a gas jar or test-tube with carbon dioxide and seal it carefully with a greased cover or cork. Place the gas sample near a Bunsen burner. If the test-tube is used support it in a rack during this experiment.

Hold a piece of magnesium ribbon about 5 cm long by means of a pair of tongs and place one end in a Bunsen burner flame. As soon as it ignites plunge it quickly into the carbon dioxide; it will continue to burn for some few moments. As soon as the magnesium has finished burning, examine the contents of the gas jar or test-tube for black specks of carbon and for white magnesium oxide.

*Part 4. A competition between carbon and iron***APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment A9.1)

Bunsen burner and asbestos square

Hard-glass test-tube, 75 × 12 mm

Test-tube holder

Magnet

Spatula

Iron(III) oxide

Powdered wood charcoal

1M sulphuric acid

PROCEDURE

Mix together a spatula measure of iron(III) oxide with a rather greater amount of powdered wood charcoal. Heat the mixture strongly in a hard-glass test-tube and allow it to cool. A black magnetic product is obtained which evolves hydrogen on treatment with molar sulphuric acid.

Note: Some samples of iron(III) oxide are magnetic and some become magnetic on heating. The magnet test alone is therefore not reliable evidence that iron is present.

Experiment E7.7

What happens when metals are placed in acids?

APPARATUS

Each pupil or pair of pupils will need:

Several test-tubes, 100 × 16 mm

Test-tube rack

Samples of the following metals:

Magnesium, about 10 cm of ribbon, freshly cleaned

Aluminium

Zinc

Iron

Tin about 5 mm square of foil, freshly cleaned

Lead

Copper

3M hydrochloric acid

3M sulphuric acid

PROCEDURE

Place an approximately 5 cm depth of one of the acids into each of several test-tubes, and place a piece of a different metal foil in each. Note the evolution of hydrogen, and compare the different rates at which the bubbles are formed. Repeat using the other acid.

Pupils can be told to record their observations in tabular form; a typical result is now given.

<i>Metal</i>	<i>3M hydrochloric acid</i>	<i>3M sulphuric acid</i>
Magnesium	Very rapid	Rapid
Aluminium	Slight	None
Zinc	Moderate	Slight
Iron	Very slight	Very slight
Tin	None	None
Lead	None	None
Copper	None	None

Experiment E7.8

What happens when metals are heated with chlorine?

This experiment **must** be done in a well-ventilated fume cupboard

APPARATUS

Each pupil or pair of pupils will need:

Round-bottomed flask, 250 cm³, fitted with tap funnel and right angle delivery tube

Two conical flasks 100 cm³, fitted with tubes to act as wash bottles

Long combustion tube fitted with one-holed stoppers and tubes

Short lengths of rubber tubing for connections

Two Bunsen burners with flame spreaders

T-piece so that both burners can be run off one gas tap

Length of PVC tube

Three stands and clamps

Powdered potassium permanganate

Concentrated hydrochloric acid
Concentrated sulphuric acid

Metals:

Calcium turnings
Magnesium ribbon
Aluminium foil
Copper turnings
Iron wire
Zinc in granulated form
Tin in granulated form
Lead foil
Sodium

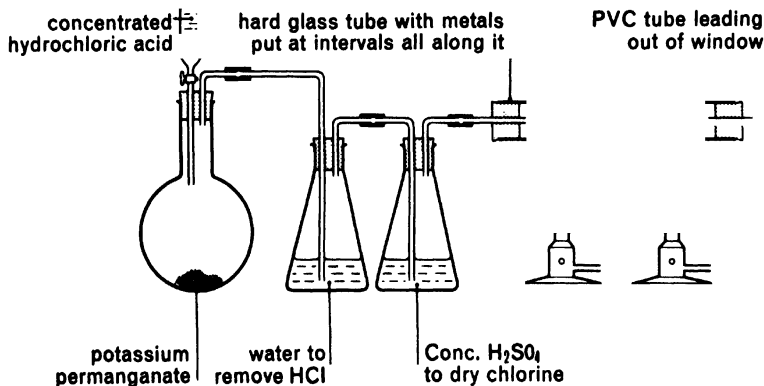


Figure 7.3

PROCEDURE

Arrange the metals in the combustion tube with lead, tin, zinc, and aluminium nearest the chlorine generator. Calcium (rubbed clean with a piece of emery cloth) and sodium should be placed furthest away from the generator. Add the concentrated hydrochloric acid drop by drop to the potassium permanganate so that chlorine is made and passes through the apparatus. As soon as all the air has been displaced, gently heat the metals by means of two Bunsen burners with flame spreaders, and note the order in which the metals react.

Experiment E7.9

What happens when metals are heated with sulphur?

Experiments with sulphur show a similar order of reactivity to that shown in reaction with oxygen.

Caution. The reactions between sulphur and some elements (e.g. magnesium) are extremely vigorous and should not be attempted even as a demonstration. No experiments other than those given below should be attempted.

Part 1

This experiment **must** be done by the teacher. An alternative method (also a teacher experiment) is given in experiment E6.6c.

APPARATUS

Tongs

Test-tube, 150×25 mm

Bunsen burner and asbestos square

Tripod

Asbestos paper

Zinc dust

Copper foil

Powdered sulphur

PROCEDURE

Zinc

Carefully mix together equal quantities of zinc dust and powdered sulphur, having about as much of the mixture as can be piled on a six-pence. Place the mixture on a piece of asbestos paper on a tripod, and heat it by placing a Bunsen burner underneath. Stand well back: the reaction is vigorous.

Copper

Place a small quantity of sulphur in a 150×25 mm test-tube and heat it until it is boiling. By means of a pair of tongs, hold some bright copper foil in the sulphur vapour.

The copper will rapidly acquire a black coating of copper sulphide, which may be removed if desired by scraping with a pen-knife in order to expose fresh copper.

Part 2. Pupils' experiment**APPARATUS**

Each pupil or pair of pupils will need:

Asbestos paper strip, 75×25 mm

Bunsen burner and asbestos square

Iron metal powder reduced by hydrogen

Powdered roll sulphur

PROCEDURE

Carefully mix together a small quantity of the iron and sulphur powders and place a measure of the mixture on one end of a piece of asbestos paper. Hold the paper in a Bunsen burner flame, and withdraw it as soon as a red glow begins to spread through the mixture. The reaction will be seen to complete itself without further heating.

Experiment E7.10

How to make a displacement series for metals

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B10.3)

Six test-tubes, 100×16 mm, in test-tube rack

Samples of the following metals: a suitable form for each metal is indicated, but this choice is not essential:

Copper 1 cm square pieces of foil

Iron 3 cm iron wire or nail

Lead 1 cm square pieces of foil

Magnesium 3 cm lengths of ribbon

Tin 1 cm square pieces of foil

Zinc 1 cm square pieces of foil

Samples of the following solutions, of approximately molar strength:

Copper(II) nitrate

Lead nitrate

Tin(II) chloride

Iron(II) sulphate

Magnesium sulphate

Zinc sulphate

PROCEDURE

Take samples of the metals copper, iron, lead, magnesium, tin, and zinc and place a small piece of each metal in a little copper(II) nitrate solution, to see if the metal replaces the copper in the solution. (Ensure

that the metal samples are free from grease and that they are not handled more than is necessary.) If it does, a copper coating will be seen on the surface of the metal, but several minutes may be needed before the coating is clearly visible. If no action appears to have taken place, remove the sample from the solution and gently scrape the surface to see if there is any deposit.

The experiment should then be repeated but this time placing a sample of each metal in iron(II) sulphate solution; then lead nitrate solution, magnesium sulphate solution, tin(II) chloride solution, and zinc sulphate solution may be used.

Draw up a list of the metals such that any metal will displace all those below it in the list from solutions of their salts.

The observed displacement series for this list of metals is: magnesium, zinc, iron, tin, lead, copper.

Experiment E7.11

Can a displacement series be made for non-metals?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B10.4)

Four test-tubes, 100×16 mm, and test-tube rack

Polythene bag, one-hole cork or bung, and 10 cm length of glass tubing
1M solutions of potassium sulphide, potassium chloride, potassium bromide and potassium iodide

Chlorine water

The teacher will need:

Cylinder of oxygen

PROCEDURE

Collect half a test-tube full of each of the following 1M solutions and place them in a test-tube rack:

Potassium sulphide

Potassium chloride

Potassium bromide

Potassium iodide

Bubble a little oxygen through each to find out if any change takes place. This may be done directly from a cylinder of the gas. With a large class it may be convenient for the teacher to inflate a small polythene bag from an oxygen cylinder for each pupil, who can then squeeze out the

oxygen through glass tubing held in a cork and bubble it through each solution.

The experiment can then be repeated using the same solutions but with chlorine in place of oxygen. *Owing to the poisonous nature of chlorine and the difficulty of providing adequate fume cupboard facilities, chlorine water should be provided.* Each pupil should add a little chlorine water to the solutions. Displacement of bromine and iodine will be observed.

The displaced bromine may itself be used to displace iodine.

For a more detailed experiment on these lines see experiment E9.4.

Chapter 8

Acidity – alkalinity

Introduction

In the Sample Scheme the subject of acidity-alkalinity is introduced as a single concept, and the pH scale as a means of numbering positions on a scale of acidity-alkalinity. The experiments in this chapter are written from that point of view.

Experiments E8.1–E8.5 are purely introductory and give an indication of the types of substances that display acidity-alkalinity. The remaining experiments are concerned with attempts to explain the concept and to show its usefulness.

References

Basic ideas of which pupils should be aware after a modern treatment of acidity-alkalinity are briefly discussed in the *Handbook for Teachers*, Chapter 2. How these ideas can be used in a teaching scheme is shown in the introduction to Topic 20 in Stage II of the *Basic Course*, and elaborated in the *Course of Options*, Option 10, 'Acidity-alkalinity'.

List of experiments

- E8.1 An introduction to acidity-alkalinity
- E8.2 How to prepare solutions of some coloured substances from natural sources
- E8.3 How to cure acidity
- E8.4 Acidity-alkalinity in products of burning
- E8.5 The behaviour of metals when placed in acidic solutions
- E8.6 Acidity-alkalinity in compounds
- E8.7 The part that water plays in acidity-alkalinity

- E8.8 How do acids and alkalis react?
- E8.9 Preparing some salts
- E8.10 How does dilution affect the pH of solutions?
- E8.11 How does the addition of acid to alkali affect the pH of solutions?
- E8.12 Using titrations to solve problems
- E8.13 Conductimetric titrations

Experiment E8.1

An introduction to acidity-alkalinity

APPARATUS

Six test-tubes, 100 × 16 mm, and rack

Teat pipette

Supply of small acid drops

Proprietary stomach powder (or sodium hydrogen carbonate)

Lemon juice

Vinegar

Citric acid

Calcium hydroxide (slaked lime)

Extract of plant material prepared as described in experiment E8.2 (or pupils' own extract)

Various acidic and alkaline solutions as available, including 0.1M hydrochloric acid and 0.1M sodium bicarbonate solution.

Universal Indicator – B.D.H. or, if not available, Yamada's Indicator which is made by dissolving 5 mg of thymol blue, 12.5 mg of methyl red, 100 mg of phenolphthalein and 50 mg of bromothymol blue in 100 cm³ of ethanol, adding 0.05M sodium hydroxide solution from a burette until the colour is green, and making up to 200 cm³ with distilled water. This indicator changes colour as shown below:

pH	4	5	6	7	8	9	10
Colour:	red	orange	yellow	green	blue	indigo	violet

Use two drops to every 10 cm³ of solution to be tested.

PROCEDURE

Give each pupil a small acid drop to suck. The pupils can taste the 'acid' taste. Now give a little 'stomach powder' or bicarbonate of soda

to each of them in their cupped hand and tell them to pick up a little on the tip of their tongue. The acidic taste will go, at least for a short time. The acidity has been removed.

Now they can be more scientific about it. If they partly dissolve some of the acid drop in a little water in a test-tube and try adding samples of the coloured extracts to the acid drop solution, do the extracts change colour? What happens when stomach powder is added now? At this stage the teacher may introduce his own coloured substance as: 'The processed extract of lichen, called litmus, which has been used for centuries for this purpose.' The pupils will see that the change they experienced by taste can now be seen in changes in the colour of certain substances.

Another way is to put a large drop of extract on a piece of filter paper and put drops of the solution under test at the edge. This gives sharp colour changes.

From the acid drop, it is an easy step to lemon juice, vinegar, and other 'acid' substances. What is the effect of adding the bicarbonate? From seeing the effect it will become apparent that these 'acidic' substances are not all the same. Here we need something which will 'indicate' not only whether a substance is acidic but also how acidic it is. Introduce a Universal Indicator.

Now they can take a series of solutions and see what happens when the Universal Indicator is added to each solution. How are we to specify the different acidities of the solutions? It would be clumsy continually to refer to the colour of the indicator. We therefore use a scale of numbers – the pH scale. The pupils may be told that there is a quantitative definition which determines this scale, but for the time being it will be used simply as a way of expressing acidity and alkalinity, alkalinity being at this stage simply the property of any solution which has a pH greater than 7. The 'acidity' of a solution of pH 7, which is the acidity of pure water, is called a neutral solution.

Finally, the Universal Indicator may be used to find the pH of such common materials as toothpaste, lemonade, and tap water. A chart may be made showing the pH of these common substances.

Experiment E8.2

How to prepare solutions of some coloured substances from natural sources

APPARATUS

The following will be needed:

Pestle and mortar
Round-bottomed flask equipped with reflux condenser
Pumice stone or pieces of broken porcelain
Stand and clamp
Water bath
Tripod
Bunsen burner and asbestos square
Filter funnel and filter papers
Bottle to store solution obtained
Industrial methylated spirits
Plant material (see Procedure)

PROCEDURE

Flowers, fruits and vegetables which make good indicators are:

Delphinium flowers
Blackcurrants
Rose petals of various colours
Red cabbage leaves
Blackberries
Beetroot

Almost all coloured plant material is suitable, with the exception of yellow flowers such as daffodils and dandelions.

First crush the plant material with a pestle and mortar and then grind it up thoroughly with a mixture of equal volumes of industrial methylated spirits and water. You will require a quantity of this mixture at least ten times the weight of the plant material being used.

Transfer the material to a round-bottomed flask (which should not be more than half filled) and add two or three pieces of broken porcelain or pumice stone to make it boil evenly. Insert a condenser vertically in the flask, and reflux the contents gently over a boiling water-bath until it can be seen that the solid parts of the mixture have gone 'white'. This should take about twenty minutes.

Allow to cool, and then filter to obtain the coloured extract.

Experiment E8.3**How to cure acidity****APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B2.2)

Beaker, 100 cm³

Measuring cylinder, 25 cm³

Glass rod or robust -10° to $+110^{\circ}\text{C}$ thermometer

Spatula

Vinegar

Slaked lime

Ground limestone

Small pieces of Universal Indicator paper

PROCEDURE

In this experiment Universal Indicator paper is used to follow the change of pH when portions of slaked lime are added to vinegar.

Place 10 cm³ of vinegar in a beaker and dilute it with an equal volume of water. Withdraw a drop of solution by means of a stirring rod and place it on a small piece of Universal Indicator paper. Compare the colour with the indicator chart and note the corresponding pH.

Add a spatula measure of slaked lime to the vinegar, stir, and test the resulting solution for its pH as before. Avoid collecting particles of slaked lime when withdrawing drops from the solution. It is best to rinse the stirring rod with water and dry it on a cloth between each sampling.

Continue to add spatula measures of slaked lime, testing the solution for pH after each addition, until the colour of the indicator no longer changes. Plot a graph of pH against the numbers of spatula measures of slaked lime added. Even if the pupils have not been introduced to graphs in their mathematics lessons, they can 'make a chart' of the results.

Repeat the experiment with ground limestone and compare the results obtained with those of the first experiment.

If thermometers are used as stirrers, they can follow the temperature changes throughout the reaction (a 4°C rise is typical).

Note: There may be small variations in the acidic content of different vinegars. It is advisable to check the amount of slaked lime needed

before the lesson and adjust the strength of the vinegar accordingly. About six spatula measures of slaked lime are a convenient number for the pupils to have to use; more may make the experiment tedious.

Vinegar substitute. 5 cm³ of 2M acetic acid require about seven spatula measures and produce a temperature rise of approximately 4°C.

Ammonia as an alternative to slaked lime. 10 cm³ of vinegar require five 1 cm³ portions of 4M ammonium hydroxide solution added by teat pipette, giving a temperature rise of about 4°C.

Experiment E8.4

Acidity-alkalinity in products of burning

How to find the acidity-alkalinity of the products formed when various elements are burned in oxygen is described in experiment E3.8.

Experiment E8.5

The behaviour of metals when placed in acidic solution

The effects which acidic solutions have on metals can be found using experiment E7.7.

Experiment E8.6

Acidity-alkalinity in compounds

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 20.1)

Four test-tubes, 100 × 16 mm

Spatula

Teat pipette

Indicator paper (very carefully dried in a desiccator)

Dry glacial acetic acid (add acetic anhydride to ensure that it is dry)

Tartaric acid crystals

Magnesium ribbon, 2 cm lengths

Bismuth nitrate crystals

Aluminium chloride

Boiled distilled water

PROCEDURE

Test the glacial acetic acid and tartaric acid crystals with *dry* indicator paper and magnesium ribbon. No acidity is shown. Now add a little of each 'acid' to about 10 cm³ of water in a test-tube and test again with indicator paper and magnesium ribbon. This time acidity is shown.

Add two drops of Universal Indicator to two test-tubes, each about one-third full of distilled water. Drop a crystal of bismuth nitrate into one and a little aluminium chloride into the other. The indicator will change colour showing that the solution has become acidic. Add magnesium ribbon to each. A very slow reaction takes place with the bismuth nitrate solution and a rather faster one with the aluminium chloride solution.

Experiment E8.7

The part that water plays in acidity-alkalinity

APPARATUS

The teacher will need:

Gas generator for supplying dry hydrogen chloride, made from a filter flask, 100 cm³, fitted with a bung carrying a tap funnel and right-angle tube

Corked flask for drying toluene

Concentrated sulphuric acid

Sodium chloride

Wash bottle

Calcium chloride, anhydrous

Toluene

2M hydrochloric acid, labelled 'Solution of hydrogen chloride in water'

Access to fume cupboard

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations*

Forceps or tongs

Six test-tubes, 100 × 16 mm, in rack

Test-tube, 150 × 25 mm, corked

Conical flask, 100 cm³, corked, for collecting solution in toluene

Beaker 100 cm³, for collecting solution in water

Two teat pipettes

6-volt bulb and holder

6-volt dry battery or alternative source of 6 V d.c.

Two steel electrodes

Three connecting wires fitted with crocodile clips

Paper tissues or cloth

Universal Indicator paper, stored in central desiccator

Marble chips

Magnesium ribbon, 2 cm lengths

Hydrogen chloride solutions in toluene and water

Distilled water

PROCEDURE

The teacher should first prepare a saturated solution of dry hydrogen chloride in dry toluene: it is advisable to do this in a fume cupboard. To ensure that the toluene is absolutely dry, it should stand overnight in a corked flask in contact with anhydrous calcium chloride. Hydrogen chloride is made by dropping concentrated sulphuric acid from a tap funnel onto sodium chloride contained in a filter flask. The gas should be dried by bubbling through concentrated sulphuric acid in a wash bottle before passing it through the dry toluene. It is advisable to keep the toluene solution of hydrogen chloride corked until immediately before use. 2M hydrochloric acid can be used as the solution of hydrogen chloride in water.

The pupils should take a supply of each solution and test it as follows:

1. Dip in each solution a piece of dry Universal Indicator paper. The paper should be stored in a desiccator until immediately before it is required and be handled in use by forceps or tongs to prevent contamination by moisture. Little change is observed with the toluene solution but there is an acid reaction with the aqueous solution.

2. To about 2 cm³ of each solution in separate dry 100 × 16 mm test-tubes add a small marble chip.

3. Repeat (2) using a small piece of magnesium ribbon

Effervescence occurs with the aqueous solutions in (2) and (3) but not with the toluene solution.

4. Immerse two steel electrodes in a portion of solution in a test-tube and connect to a 6-volt dry battery, using a bulb in the circuit. Electricity is conducted by the aqueous solution but not by the toluene solution. Note: the electrodes should be placed in the toluene solution first to avoid transferring any water to the toluene; alternatively the electrodes must be dried carefully with paper tissues or a cloth before being dipped into the toluene solution.

5. Transfer about 10 cm³ of the toluene solution to a corked 150 × 25 mm test-tube and shake with the same volume of distilled water. Pour off the upper layer of toluene and test portions of the aqueous layer with indicator paper, marble chips, magnesium ribbon, and for electrical conductivity as described above. From these tests it will be clear that some, at least, of the hydrogen chloride has passed into the aqueous layer and that, in doing so, it has developed different properties.

Experiment E8.8**How do acids and alkalis react?**

This experiment may be done by the teacher, or by pupils working in pairs.

APPARATUS

Test-tube, 100×16 mm

Beaker, 100 cm^3

Two carbon electrodes in holder

Three lengths of connecting wire, fitted with crocodile clips

12-volt bulb in bulb holder

Source of about 12 volts a.c. (d.c. may be used provided that the current is only switched on briefly after each addition from the burette)

Glass rod

Burette and stand

Measuring cylinder, 25 cm^3

Phenolphthalein solution

0.1M barium hydroxide solution

1M sulphuric acid solution

Distilled water

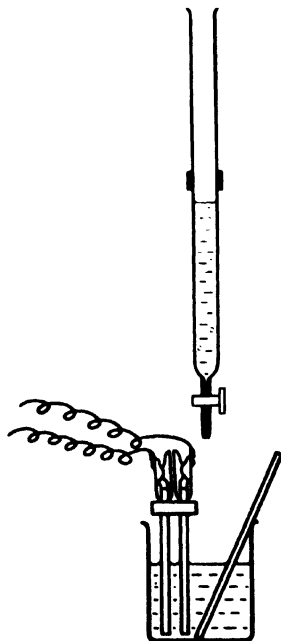


Figure 8.1

PROCEDURE

As a preliminary step mix together about equal volumes of sulphuric acid and barium hydroxide solutions in a 100×16 mm test-tube to observe what happens. Test each of the solutions for conductivity.

In order to find the ratio in which the barium and sulphate ions react, place about 50 cm³ of the barium hydroxide solution, measured in a measuring cylinder, into a 100 cm³ beaker. (*Note: barium hydroxide is very poisonous and the solution must not be sucked up into a pipette.*) Then place two electrodes in the solution and connect them in series with a 12 volt lamp and source of about 12 volts a.c. Add a few drops of phenolphthalein to colour the solution.

Now set up a burette containing the sulphuric acid solution above the beaker and run in the sulphuric acid solution slowly, stirring the contents of the beaker with a glass rod.

At the 'end-point', when equivalent amounts of sulphuric acid and barium hydroxide are present, the light will go out and the indicator will be decolorized. If more of the acid is added, the lamp will be seen to light again.

Experiment E8.9

Preparing some salts

Preparation of copper sulphate from copper oxide

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigation* (experiment 20.4a)

Beaker, 100 cm³

Tripod and gauze

Bunsen burner and asbestos square

Filter funnel, paper and stand

Crystallizing dish

Spatula

2M sulphuric acid

Copper(II) oxide powder

Blotting paper

PROCEDURE

Place sufficient 2M sulphuric acid solution in a 100 cm³ beaker to fill it to a depth of about 1 cm. Place the beaker on a gauze on a tripod and heat it by means of the Bunsen burner until it is nearly boiling. Stop heating and add copper(II) oxide, a spatula measure at a time, until no more will react, i.e. until on further heating for about a minute after addition, a quantity of black solid is still present.

Filter the hot suspension through a fluted filter paper into a crystallizing dish, acidifying the solution with one or two drops of 2M sulphuric acid, and allow to cool.

When cool (or possibly after two or three days left covered with a filter paper) blue crystals of copper(II) sulphate will be seen. Decant the surplus liquid, remove the crystals with a spatula and dry them by pressing on blotting paper.

Preparation of magnesium sulphate from magnesium carbonate

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 20.4b)

Beaker, 100 cm³

Tripod and gauze

Bunsen burner and asbestos square

Filter funnel, paper and stand

Crystallizing dish

Spatula

2M sulphuric acid

Magnesium carbonate powder

Blotting paper

PROCEDURE

Place enough 2M sulphuric acid solution in a 100 cm³ beaker to fill it to a depth of about 1 cm. Add magnesium carbonate powder, two or three spatula measures at a time, until no more will react, i.e. until on addition of more magnesium carbonate there is no further effervescence and a quantity of white solid is still present.

Then bring the suspension to the boil and filter hot through a filter paper into a crystallizing dish. (Boiling before filtering will ensure that any magnesium sulphate which has crystallized prematurely is brought back into solution, and that both filtering and evaporation take place more rapidly.) Add one or two drops of 2M sulphuric acid, evaporate until the solution is concentrated enough to crystallize on cooling, and then allow to cool.

When cool, colourless crystals of magnesium sulphate will be seen. Decant the surplus liquid and remove the crystals with a spatula, and dry them by pressing on blotting paper.

Preparation of lead(II) chloride from lead(II) nitrate solution

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 20.4c)

Test-tube, 100 × 16 mm

Teat pipette
Glass rod
Bunsen burner and asbestos square
Beaker, 100 cm³
Measuring cylinder, 25 cm³
Access to centrifuge
1M lead(II) nitrate solution (5 cm³)
2M sodium chloride solution (5 cm³)
Distilled water

PROCEDURE

To 5 cm³ of molar lead(II) nitrate solution in a test-tube add 5 cm³ of 2M sodium chloride solution and shake well. Centrifuge the suspension, and pour away the clear liquid. Wash the precipitate of lead(II) chloride by placing a few cm³ of distilled water in the test-tube, stirring the precipitate around in it with a glass rod, and then centrifuging again. The clear liquid is discarded.

If desired, recrystallize the lead(II) chloride by adding it to about 50 cm³ of distilled water in a beaker. Stir the precipitate around in it with a glass rod and heat the suspension until the lead(II) chloride has all dissolved. On cooling the beaker, crystals of lead(II) chloride will rapidly form.

Preparation of ammonium sulphate from ammonia solution

APPARATUS

Each pupil or pair of pupils will need:
Page from *Laboratory Investigations* (experiment 22.5)
Evaporating basin or 100 cm³ beaker
Glass rod
Bunsen burner and asbestos square
Tripod and gauze
Microscope slide
Filter flask, funnel, filter paper, and pump
Approximately 1M sulphuric acid
Approximately 2M ammonia solution

PROCEDURE

Place about 20 cm³ of molar sulphuric acid in an evaporating basin or 100 cm³ beaker. Add 2M ammonium hydroxide solution slowly, about 5 cm³ at a time, stirring the acid with a glass rod. The addition of ammonia should be stopped as soon as the mixture has acquired a definite smell of ammonia.

Evaporate the solution until it is ready to crystallize. This point may be found easily by withdrawing a drop of the hot liquid on a glass rod and placing it on a microscope slide; the drop will cool quickly and if crystals are obtained in this drop the liquid as a whole will crystallize on cooling.

When the whole solution has cooled the crystals can be filtered off using a filter pump and dried on the filter paper.

Experiment E8.10

How does dilution affect the pH of solutions?

APPARATUS

Each pupil or pair of pupils will need:

Six test-tubes, 100×16 mm

Teat pipette, graduated, or burette

Universal Indicator solution

0.1M hydrochloric acid

0.1M oxalic acid

0.1M acetic acid

Distilled water

PROCEDURE

Start with a 0.1M solution of hydrochloric acid. Prepare some 0.01M acid by diluting 1 cm^3 with 9 cm^3 of distilled water. From this prepare 10 cm^3 of 0.001M acid, and continue the dilution in order to prepare acids of molarities $M \times 10^{-4}$, $M \times 10^{-5}$, and $M \times 10^{-6}$. Place the solutions in test-tubes in a rack. Add three drops of Universal Indicator to each and note the colours.

Now use the 0.1M solutions of oxalic acid and acetic acid. To 9 cm^3 of each in test-tubes, add three drops of Universal Indicator solution. It will be clear that the colours produced differ from the colour of the indicator in the 0.1M solution of hydrochloric acid. They correspond to the colours in the more dilute hydrochloric acid solution. They are, in fact, 'weaker' acids – hydrochloric acid is a 'stronger' acid. 0.1M solutions of all these acids have the same *concentration*, but different 'strengths'. The strength of an acid is the extent to which its acidic nature is developed.

If it is assumed that the colour of the indicator is determined by the hydrogen ion concentration, it follows that the hydrogen ion concentrations in equi-molar solutions of the three acids differ markedly.

Experiment E8.11

How does the addition of acid to alkali affect the pH of solutions?

APPARATUS

Each pair of pupils will need:

Stand and clamp

Burette and pipette

Conical flasks

Approximately 0.1M solutions of hydrochloric acid, sodium hydroxide, acetic acid, and ammonia

Universal Indicator solution

Methyl orange, phenolphthalein, and bromophenol blue indicator solutions

This experiment attempts to find out what happens to the degree of acidity during a titration by drawing titration curves. These could be drawn for a series of acids and alkalis; a strong alkali with a weak acid and a strong acid, and a weak alkali with a weak acid and a strong acid. A class could work in pairs for this experiment, the various pairs being given different alkalis and acids to investigate.

PROCEDURE

Take a certain volume of alkali, and add a few drops of Universal Indicator solution. Note the change in pH as the appropriate acid is added in volumes of 1.0 cm³ (or 0.1 cm³) at a time.

Experiment E8.12

Using titrations to solve problems

There are a number of problems that can be solved using titration methods, such as finding the number of gram-molecules of water associated with each gram-molecule of sodium carbonate in washing soda crystals, or finding the molarity of a solution of an acid by titration with an alkali solution of known molarity. Examples of these problems are given in many textbooks of practical chemistry.

Experiment E8.13

Conductimetric titrations

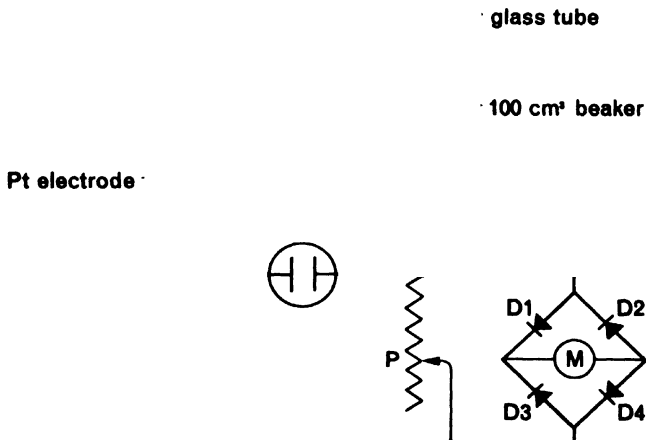
INTRODUCTORY NOTE

To make a conductivity meter

For this experiment the apparatus described below is very suitable. (Quite useful results can be obtained, however, by using an a.c. milliammeter or multirange meter such as an Avometer.) It consists of a 0-50 μ A (or 0-100 μ A) meter (M) in a rectifying bridge of four ger-

manium diodes (D) (GEX 34 is a suitable type), shunted by a 50-ohm wire-wound potentiometer (P). A low voltage a.c. supply is obtained by the use of a small bell transformer (T) or valve-heater transformer. Three to five volts would be suitable. These components may be mounted as shown in a small aluminium box. An electrode system (E) should be constructed as shown in the diagram. Bright platinum electrodes are quite suitable. Alternatively, the support wires from an old electric lamp bulb may be used as electrodes. Efficient stirring is necessary, and may be produced by means of a magnetic stirrer, a motor-driven stirrer, or even by arranging to pump air through the solution. Reasonable results may be obtained if the experimenter blows air through the solution by means of a glass tube.

Figure 8.2



Note: By using a.c. at 50 c/s electrolysis of the solution is practically avoided. Before measurement, the current passing to the meter is rectified by the bridge of four GEX 34 germanium diodes, and as the voltage is constant, the current is inversely proportional to the resistance of the circuit, and hence is proportional to the conductance. During an experiment only the conductance of the solution varies, and so the meter reading gives a direct indication of this conductance.

During a titration the conductance should first fall linearly with the volume of the titrant added until the end-point is reached, and then rise linearly again. However in practice it will be found that non-linear variations occur at the very beginning, at the end-point, and when about

two equivalents of titrant have been added. There will nevertheless be two straight-line portions on the graph obtained, and if these are produced their point of intersection will give the required end-point.

Experiment 1. To find out how acidity-alkalinity is related to hydrogen ions and hydroxide ions

APPARATUS

'Conductivity titration' apparatus (see introductory note)

Burette

Beaker, 100 cm³

Pipette

Mechanical stirrer or glass rod

Universal Indicator solution

Approximately 0.5M sulphuric acid

Approximately 0.05M sodium hydroxide solution

PROCEDURE

This experiment may be carried out either as a demonstration by the teacher, or as individual work by the pupils. Fill the burette with 0.5M sulphuric acid. Place the electrodes in the 100 cm³ beaker, and connect the apparatus to the mains supply, but not yet switched on. Arrange a stirrer so as not to interfere with the electrode. (Air blowing is not satisfactory for this experiment; if a mechanical stirrer is not available the experimenter must stir the solution with a glass rod.) Rotate the potentiometer knob to give zero resistance, and pipette into the beaker 50 cm³ of 0.05M sodium hydroxide solution. The reason for the difference in concentration is to reduce the volume change which takes place during the experiment; obviously this will have a disturbing effect on the conductance. Add 0.5 cm³ of Universal Indicator. Make sure that the electrodes are completely immersed, and then switch on the current. Rotate the potentiometer knob until the meter reading is 50 μ A. Start the stirrer, and when conditions are stable use the potentiometer to bring the reading back to 50 μ A. (It may be necessary to stop stirring as the meter is read.) Now run in the sulphuric acid in portions of 0.5 cm³ noting the meter reading, and the colour of the indicator, each time, until about 10 cm³ have been added. It helps to form a picture of what is happening to plot a graph showing the variation of current with volume of solution added while the results are obtained.

It is then obvious that the solution becomes less conductive as the titration proceeds, until the indicator shows that a mid-point on the acidity scale has been reached. From this point the conductance increases again.

Experiment 2. To investigate the changes in conductivity during titrations using weak acids and bases

APPARATUS

As in the previous experiment, with the addition of approximately 1.0M acetic acid and 0.1M ammonia solution.

PROCEDURE

The following titrations may be performed:

- (a) 1.0M acetic acid in burette; 0.05M sodium hydroxide in the beaker.
- (b) 0.5M sulphuric acid in burette; 0.1M ammonia solution in the beaker.

In experiment (a) the conductance falls to the end-point, but does not rise again. Why? From what we have learnt in previous experiments this can only be due to the absence of 'mobile' ions. Then what has happened to the hydrogen ions which presumably continue to be added to the solution after one mole of acid has been added (per mole of alkali)? The explanation is that the acetic acid is only slightly ionized and addition of acid produces very few hydrogen ions.

Careful pupils may be able to note that the indicator does not change colour so rapidly after the end-point; this shows once again the dependence of the properties which we call 'acidity' on the presence of hydrogen ions.

In experiment (b) the result may be forecast – the conductance hardly alters until half a mole of sulphuric acid has been added per mole of alkali, then it increases. In this experiment therefore it is necessary to start with a low value of meter reading. It is useful to start with the potentiometer setting at which the previous experiment finished. The actual end-point may be difficult to detect, and if this method is being used to determine concentration, it would be better to place 1M ammonia solution in the burette, and 0.05M sulphuric acid in the beaker.

It is possible to titrate a weak acid and a weak base by this method, having, for example, 1M acetic acid in the burette and 0.1M ammonia solution in the beaker. It is again necessary to start with a low value of meter reading. If this is not done, the current will rise to so high a value that it will be necessary to reset the potentiometer to bring the reading back to zero, thus making it difficult to plot a graph.

Chapter 9

Electron transfer

Introduction

The reactions investigated in the experiments in this chapter involve the transfer of electrons from one atom to another. They are thus closely linked with electrical processes, for the electrode reaction in electrolysis (Chapter 5) and in cells (Chapter 17) are both reactions involving electron transfer.

References

The subject of electron transfer, and its relation to the terms oxidation and reduction, is discussed in the *Handbook for Teachers*, Chapter 2.

In the Sample Scheme, Part I, *Basic Course*, oxidation and reduction are not considered formally, and the words are introduced solely to indicate giving oxygen, and taking oxygen away, respectively. Oxidation-reduction is discussed in detail for the first time in the *Course of Options*, Option 12, 'Atoms into ions', where it is dealt with solely in terms of electron loss and gain.

List of experiments

- E9.1 Exchange of electrons between ions and metal atoms
- E9.2 Examining the changes of iron(II) to iron(III) ions
- E9.3 Electron transfer from a distance
- E9.4 Electron transfer reactions of chlorine, bromine, and iodine
- E9.5 An investigation of some redox reactions

Experiment E9.1

Exchange of electrons between ions and metal atoms

The exchange of electrons between ions and metal atoms can be studied by placing various metals into solutions of other metal salts. Experiment E7.10 gives details of a suitable procedure.

Experiment E9.2**Examining the changes of iron(II) to iron(III) ions****APPARATUS**

Each pupil or pair of pupils will need:

Test-tubes in rack

Conical flask, 100 cm³

Iron(II) sulphate

Ammonium iron(III) sulphate

Zinc metal granulated

Access to solutions of:

Bromine water

Potassium hexacyanoferrate(III) (ferricyanide)

Potassium thiocyanate

PROCEDURE

The pupils should start with a knowledge of the tests for iron(II) and iron(III) ions; these should be introduced or revised. In this experiment potassium hexacyanoferrate(III) is used as a test for iron(II) ions, and potassium thiocyanate as a test for iron(III) ions. It is as well to point out that Fe^{3+} is nearly always present with Fe^{2+} , so that the *deepening* of the colour of the thiocyanate is the sign of the conversion of Fe^{2+} to Fe^{3+} .

The solutions of the iron(II) and iron(III) salts should each be made in the presence of dilute sulphuric acid.

Test some of the iron(II) sulphate solution with thiocyanate solution. Then add some bromine water gradually to another sample of the solution containing iron(II) ions; the bromine is decolorized but this reaction may take a little time to start. Any excess of bromine can be driven off by warming. Test the final solution with thiocyanate; a considerable increase in the amount of Fe^{3+} will be seen.

Place a little of the ammonium iron(III) sulphate solution in the conical flask, acidify it with dilute sulphuric acid, add a few pieces of granulated zinc, and boil the contents vigorously for five minutes. Pour off some of the solution and test it with thiocyanate and hexacyanoferrate(III). The former reagent will probably show that there is still some iron(III) present, but the colour should be weaker than that with the original solution; the latter reagent will show a marked increase in the amount of iron(II).

Experiment E9.3**Electron transfer from a distance**

This experiment works quite well as a demonstration, so long as the group is not too large, but it could be adapted to be done by a class if apparatus were available.

APPARATUS

U-tube, about 10 cm high, diam. 1.5 cm (or a larger one is better if the experiment is to be a demonstration)

Two electrodes, consisting of 3 cm of platinum wire sealed through glass, about 2 cm of wire external, passing through corks with slits.

Two teat pipettes

High resistance voltmeter

Milliammeter

Two lengths of connecting wire, fitted with crocodile clips.

SOLUTIONS

10 per cent potassium iodide solution

Weak bromine water

0.02M potassium permanganate solution

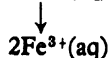
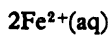
2M sulphuric acid

Iron(II) sulphate (concentrated solution, acidified)

0.02M potassium thiocyanate solution

0.02M potassium dichromate solution

a. Fill the U-tube to within about 5 cm of the top with 2M sulphuric acid solution and clamp it vertically. Then to one limb add bromine water and to the other iron(II) sulphate solution (acidified with a little sulphuric acid) very carefully with a teat pipette so that a layer of each, about 2 cm in depth rests on the acid solution. Insert the corks with the electrodes so that the platinum wires are immersed in the two upper layers. (It does not matter if they touch the sulphuric acid layer.) Connect the electrodes to a voltmeter and note the reading. Now replace the voltmeter with a milliammeter and note that a current passes. If the pupils have done experiment E9.2 they will have already met the reaction between iron(II) ions and bromine. Here the reactants are separated, but electron transfer takes place through the milliammeter and the same reaction occurs:

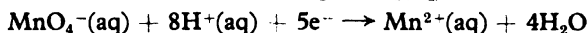
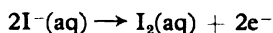


After about a minute, remove the electrodes and test for iron(III) ions in the iron(II) solution. If the current is left running for long enough, the bromine water will be decolorized.

b. Now set up another similar cell, using the permanganate solution instead of the bromine water. Iron(III) ions are again produced, showing that the permanganate, like the bromine, is a 'sink' for electrons.

c. This time use a solution of potassium iodide instead of the iron(II) sulphate, and again use permanganate in the other limb of the U-tube. Connect the electrodes to the voltmeter; a steady voltage (about 0.6 volt) is shown. Replace the voltmeter by the milliammeter and plunge the electrodes in quickly. A maximum of about 100 milliamp is obtained which quickly falls off. After about 10 seconds, move the wire in the potassium iodide limb quickly sideways; a fine line of free iodine is seen. This procedure emphasizes that the chemical reaction is taking place at the surface of the platinum wire. Now connect the electrodes directly; after some few minutes a distinct yellow colour is seen in the solution.

The two ionic reactions may be written:



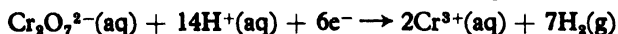
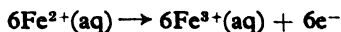
(There will not, in fact, be any appreciable diminution of colour of the permanganate in this experiment.)

d. The experiment can be repeated using, in the left-hand limb, freshly made concentrated iron(II) sulphate solution to which a few drops of potassium thiocyanate solution have been added (there should only be a very slight pink colour) and, in the right-hand limb, dilute potassium dichromate solution acidified with sulphuric acid.

The values both for the potential difference and for the current are smaller than in (*c*) (about 0.2 volt and 20 milliamp respectively). (The values quoted for the currents are those obtained in a particular experiment – they will of course vary with the dimensions of the electrodes and of the apparatus.)

After leaving for some five minutes, a really distinct red colour, visible at the back of the class, is seen.

Ionic reactions:



Experiment E9.4**Electron transfer reactions of chlorine, bromine, and iodine****APPARATUS**

Each pupil or pair of pupils will need:

Test-tubes

Dilute sulphuric acid

Sodium hypochlorite solution

Bromine water

Iodine solution

Potassium bromide solution

Potassium iodide solution

Carbon tetrachloride

Solution of sulphur dioxide

Solution of potassium permanganate

This is an experiment involving only test-tube reactions, and it is important that the purpose of *each stage* is fully understood.

1. The differences between the appearance of chlorine, bromine, and iodine in solution in carbon tetrachloride are first examined. The 'chlorine solution' is made by adding about 1 cm³ of dilute sulphuric acid to 2–3 cm³ of sodium hypochlorite solution.

Add about 1 cm³ of carbon tetrachloride to 2–3 cm³ of each halogen solution, shake the mixtures, and allow to settle. Examine the colour of the carbon tetrachloride layer in each case.

2. Add about 1 cm³ of chlorine solution separately to an excess of potassium bromide and potassium iodide solutions, followed by about 1 cm³ of carbon tetrachloride. Shake the mixtures and allow to settle, then examine the colour of the carbon tetrachloride layer in each case. Add about 1 cm³ of bromine water to an excess of potassium iodide solution, followed by carbon tetrachloride, and shake and examine as before.

It will be seen that the halogens can be arranged in a series:

Cl₂ oxidizes Br⁻ to Br₂. Cl₂(aq) + 2Br⁻(aq) → Br₂(aq) + 2Cl⁻(aq)

Cl₂ oxidizes I⁻ to I₂. Cl₂(aq) + 2I⁻(aq) → I₂(aq) + 2Cl⁻(aq)

Br₂ oxidizes I⁻ to I₂. Br₂(aq) + 2I⁻(aq) → I₂(aq) + 2Br⁻(aq)

3. Potassium permanganate was used in experiment E9.3 to provide a 'sink' for electrons released by iodide ions becoming iodine. Find out whether a dilute acidified solution of potassium permanganate will

oxidize a solution of potassium iodide to give a solution of iodine. (Only small quantities of permanganate should be added so that the iodide remains in excess.) Find out if the permanganate will also oxidize a bromide and a chloride in solution.

Experiment E9.5

An investigation of some redox reactions

APPARATUS

Each pupil, or pair of pupils, will need:

Six test-tubes, 100×16 mm, in rack

Two teat pipettes (with 1 cm^3 graduation)

Access to the necessary reagents. These will vary with the plans made by the teacher. For the examples given below the following reagents are required: Solutions which are 0.2M with respect to the following ions:

Iron(III), (chloride, nitrate, sulphate, or alum)

Iron(II), (sulphate or nitrate)

Zinc, (sulphate or nitrate)

Chromium(III), (sulphate or alum)

Lead(II), (acetate)

Silver, (nitrate)

Barium, (chloride or nitrate)

Iodide, (potassium salt)

Bromide, (potassium salt)

Permanganate, (potassium salt)

Hypochlorite, (Domestos or other commercial sodium hypochlorite solution suitably diluted)

Iodate, (potassium salt)

Hydrogen peroxide solution

Sulphur dioxide solution

1M sodium hydroxide solution

1M hydrochloric acid

Zinc filings

Starch solution

The notes which follow merely outline the sort of problem that can be presented to pupils. Where possible, and this is frequently the case, the work should be on a semi-quantitative basis, e.g. relative volumes of solutions used to complete a reaction should be estimated and used to check the relative numbers of different species shown in the equation predicted. The use of solutions of known molarity makes this a simple matter.

1. Would you expect a reaction to take place between iron(III) ions and metallic zinc? If so, work out the equation for the reaction. Obtain the solutions needed to test your conclusions and do this experimentally as completely as you can.

Notes: a. Cork the test-tube when shaking a finely divided solid with a liquid.

b. Solutions containing iron(III) and iron(II) may be distinguished by their reactions with hydroxide ions (sodium hydroxide solution). Try this test with the solutions provided to see what to look for.

2. Suggest a reaction in which chromium(III) would be oxidized to chromate ions. Would the presence of either acid or alkali be necessary? Write the equation for the reaction you suggest. Test your conclusions by experiments.

Note: Chromate ions combine with lead ions in dilute acetic acid solution to give a yellow precipitate of lead chromate.

3. Can silver ions be reduced to silver by iron(II) ions? If you think they can, give your reasons, and suggest an equation for the reaction. Test your conclusions by using solutions of silver nitrate and iron(II) sulphate, using the tests described in note (b) above.

4. Investigate the reaction between permanganate ions and sulphurous acid, using potassium permanganate solution and sulphur dioxide solution. Work out the equation for the reaction and name the ions formed.

Notes: a. Add potassium permanganate solution (1 cm³ at a time) to 2 cm³ sulphur dioxide solution until no further change appears to take place.

b. Sulphate ions, in solution, react with barium ions (barium chloride or nitrate solution) to produce a white precipitate of barium sulphate.

5. Hypochlorite ions are said to react with lead hydroxide to produce lead(IV) oxide. Using your table of E° values (see *Book of Data*) as a guide, find whether this reaction is likely to happen. Test your conclusions by experiments.

Notes: a. Precipitate lead hydroxide by adding sufficient M sodium hydroxide solution to 2 cm³ of 0.2M lead acetate solution just to complete the reaction. You should be able to work out how much is needed.

b. Is the reaction fast? If not, try to make it go more quickly.

6. When iodide ions are oxidized, iodine is formed. Is hydrogen peroxide a suitable oxidizing agent for this purpose? Investigate the reaction, using potassium iodide solution and hydrogen peroxide solution. Try the effect of adding:

a. Hydroxide ions (sodium hydroxide solution)

b. Hydrogen ions (hydrochloric acid)

to the potassium iodide solution before you add the hydrogen peroxide solution. You should know how to test for iodine.

7. Use your table of E° values to find a reagent which will oxidize bromide ions to bromine. Write the equation for the reaction you suggest. Check your conclusions experimentally.

8. Would you expect a reaction to take place between iodate ions and iodide ions in aqueous solution? If so, what is the equation? Under what conditions would you expect the reaction to go reasonably quickly? Test your answers by experiment.

9. Choose two electrode systems and work out the equation for the reaction responsible for the potential difference that can be detected between them. Show your report to your teacher and ask permission to investigate the reaction experimentally.

Do your experimental observations agree with your predictions? If not, suggest reasons for any difference.

Chapter 10

Carbon compounds

Introduction

Experiments involving carbon compounds are collected together in this chapter for convenience, but it is not suggested that any rigid boundary should be drawn between the behaviour of this element and that of the others.

The theme can be begun, and the element introduced, at an early stage by experiments on the thermal decomposition of a number of carbon compounds.

At a later stage groups of experiments can be undertaken to find out what sorts of substances can be obtained from various naturally occurring sources of carbon compounds. Experiments are suggested starting from oil, starch, and coal tar.

Reference

One way of treating this theme is described in the Sample Scheme, Part I, *Basic Course*, Stage II, Topic 21: 'Breaking down and building up large molecules.'

List of experiments

- E10.1 To find out what happens when carbon compounds are heated
- E10.2 What is the effect of heat on wood?
- E10.3 What is the effect of heat on coal?
- E10.4 How carbon and hydrogen can be identified in compounds
- E10.5 How nitrogen can be identified in compounds
- E10.6 The fractional distillation of crude oil
- E10.7 The cracking of oil

- E10.8 A comparison of alkanes and alkenes
- E10.9 The hydrolysis of starch
- E10.10 Making ethanol by fermentation
- E10.11 The dehydration of ethanol
- E10.12 The hydration of alkenes
- E10.13 The oxidation of ethanol
- E10.14 What happens when alcohols and acids are mixed?
- E10.15 The saponification of esters
- E10.16 The saponification of fats
- E10.17 How detergents are made

Experiment E10.1

To find out what happens when carbon compounds are heated

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 13.5a)

Piece of broken crucible or a crucible lid

Tongs

Bunsen burner and asbestos square

Teat pipette

Hard-glass test-tube, 100 × 16 mm

Test-tube holder

Lime water

Samples of compounds from plants or animals such as sugar, starch, fat (e.g. butter or lard), rice, custard powder, dried leaves or grass, coal

PROCEDURE

Heat a very small sample of some of the substances on a piece of broken crucible or a crucible lid.

It will be seen that a black substance (carbon) is obtained in each case. If this is further heated in a hard-glass test-tube, some carbon dioxide will be obtained. Test with lime water.

Experiment E10.2

What is the effect of heat on wood?

APPARATUS

Each pupil or pair of pupils will need:

Side-arm test-tube, 125 × 16 mm

Hard-glass test-tube, 100 × 16 mm

Rubber bungs and delivery tube (see diagram)

(Note: It is advisable to keep a number of sets of this apparatus in the laboratory for use from year to year as they are difficult to clean afterwards.)

Stand and clamp

Bunsen burner and asbestos square

Small pieces of dry softwood



Figure 10.1

PROCEDURE

Set up the apparatus as shown in the diagram, placing a few small pieces of dry softwood in the horizontal hard-glass test-tube, and about 2 cm depth of water in the vertical side-arm test-tube.

Heat the wood steadily, gently at first but gradually increasing the temperature until the test-tube is just red-hot. The burner must be moved about so as to drive off any liquid in the horizontal test-tube.

Drops of tar will collect in the vertical test-tube and 'pyroligneous acid' will dissolve in the water, which can be tested with indicator paper. If a light is applied to the side-arm of the vertical test-tube, the gas being given off will catch fire. After heating the wood will leave a residue of wood charcoal.

Experiment E10.3

What is the effect of heat on coal?

The experiment can be carried out using the apparatus and procedure of experiment E10.2. Place a few small lumps of coal in the horizontal test-tube; on heating, coal tar will collect in the vertical test-tube, ammonium compounds will dissolve in the water, coal gas (also inflammable) will be driven off, and a residue looking like smokeless fuel will be formed in the horizontal test-tube.

See *Laboratory Investigations* (experiment 13.5b)

Experiment E10.4

How carbon and hydrogen can be identified in compounds

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 13.6)

Hard-glass test-tube, 100×16 mm

Hard-glass test-tube, 75×12 mm

Teat pipette

Spatula

Test-tube holder

Bunsen burner and asbestos square

Dry copper(II) oxide

Cobalt chloride paper

Lime water

Selection of carbon compounds (the same materials may be used as in experiment E10.1)

PROCEDURE

Put a spatula measure of copper(II) oxide in a 100×16 mm test-tube, add about half as much of the carbon compound, and then add another spatula measure of copper(II) oxide. Have ready a teat pipette and a 75×12 mm test-tube containing about 1 cm depth of lime water. Heat the test-tube containing the organic substance with a small hot flame.

After heating for a minute or two, holding the test-tube at an angle of about 45° to the vertical, remove some of the gas from the test-tube using the teat pipette and then expel it through the lime water in the small test-tube. It will be seen that the lime water turns milky.

In many cases drops of moisture will be noticed condensing on the upper, cooler part of the test-tube. These may be tested using cobalt chloride paper and will be found to be water.

Experiment E10.5**How nitrogen can be identified in compounds**

The following experiments are two ways of obtaining ammonia from some carbon compounds. A knowledge of the fact that ammonia contains nitrogen is assumed.

Experiment 1. An alkaline gas obtained from proteins**APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 22.1b)

Hard-glass test-tube, 100 × 16 mm

Test-tube holder

Drying tube filled with activated charcoal and plugged with cotton wool

Cork or bung to fit test-tube and drying tube

Bunsen burner and asbestos square

Indicator paper

Concentrated hydrochloric acid

Soda lime granules

Gelatin

Milk powder

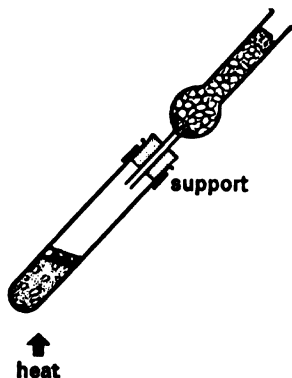


Figure 10.2

PROCEDURE

Mix together two or three spatula measures of gelatin or milk powder with an equal volume of soda lime in a 100 × 16 mm test-tube. Add a further two or three spatula measures of soda lime on top of the mixture. Fit the drying tube onto the test-tube as in the diagram. Heat the mixture until a gas is evolved. Test the gas with indicator paper (it is alkaline) and show that it fumes with hydrogen chloride.

Ammonia can be detected by its smell from time to time.

Experiment 2. Ammonia from a protein

This experiment **must** be done by the teacher.

APPARATUS

Kjeldahl flask

Drying tube filled with activated charcoal and plugged with cotton wool

Cork or bung to fit flask and drying tube

Bunsen burner and asbestos square

Tripod and gauze

Stand and clamp

Measuring cylinder, 25 cm³

Pumice stone or pieces of broken porcelain

Egg albumin

Concentrated sulphuric acid

Potassium hydrogen sulphate

30 per cent sodium hydroxide solution

Indicator paper

Access to fume cupboard

PROCEDURE

Do this experiment in a fume cupboard

Put 5 g of egg albumin into the Kjeldahl flask and cautiously add 15 cm³ of concentrated sulphuric acid in which a spatula measure of potassium hydrogen sulphate has been dissolved. Boil in a fume cupboard for about ten minutes. Allow to cool and then add the residue very carefully to a beaker containing about 10 cm³ of water. Return the solution to the flask and add sufficient 30 per cent sodium hydroxide to make the solution strongly alkaline. Put some pumice stone or pieces of broken porcelain in the flask, fit the drying tube on the top and place a wad of damp cotton wool on the open end of the drying tube. After five minutes' boiling the cotton-wool wad will be saturated with ammonia. Pass it round the class for the pupils to smell.

Experiment E10.6

The fractional distillation of crude oil

A simple experiment in which some of the components of crude oil are separated by distillation is given as experiment E2.13.

Experiment E10.7**The cracking of oil****APPARATUS**

Each pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.4)

Four test-tubes, 150×25 mm

Hard-glass test-tube, 125×16 mm, fitted with cork and delivery tube

Bunsen burner and asbestos square

Asbestos wool

Broken pieces of porous pot

Stand and clamp

Teat pipette

Medicinal paraffin

Dilute bromine water

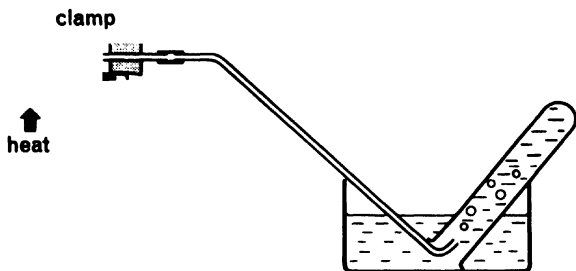


Figure 10.3

PROCEDURE

In this experiment the vapour from medicinal paraffin is passed over pieces of porous pot which are strongly heated.

Set up the apparatus shown in the diagram, and carry out the experiment using a similar technique to that of experiment E10.11, but using medicinal paraffin instead of ethanol.

The gas that collects, after the air has been expelled, contains alkenes and will be found to have a characteristic smell, to burn with a yellow flame, and to decolorize weak bromine water.

Experiment E10.8**A comparison of alkanes and alkenes****APPARATUS**

Each pupil or pair of pupils will need:

Two test-tubes, 100×16 mm

Test-tube rack

Teat pipette

Acidified potassium permanganate solution

Bromine water

Samples of alkanes and alkenes (e.g. cyclo-hexane and cyclo-hexene)

The teacher will need:

Bromine

Concentrated sulphuric acid

PROCEDURE

Carry out the following reactions, and compare the results obtained with the alkanes and the alkenes. Reactions (1) and (2) are suitable for pupils; the remainder are best done by the teacher, unless the pupils have sufficient experience to handle bromine and concentrated sulphuric acid.

1. Place 2 cm^3 of acidified potassium permanganate solution in each of two test-tubes. Add two drops of the alkane to one, and two drops of the alkene to the other, and shake both tubes well. (Alkenes are oxidized by potassium permanganate in acid solution, and the colour will go.)

2. Repeat reaction (1), but using 2 cm^3 of bromine water instead of the potassium permanganate. (Alkenes react with bromine, and so the colour will go.)

3. Place two drops of alkane in one test-tube and two drops of alkene in the other. Using a teat pipette add one drop of bromine to each. (There is a vigorous reaction in the case of the alkene.)

4. Repeat reaction (3), but adding one drop of concentrated sulphuric acid instead of the bromine. (Alkenes react vigorously with sulphuric acid, and darken in colour; alkanes do not react.)

Experiment E10.9

The hydrolysis of starch

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.1)

Test-tube, 150×25 mm

Test-tube, 125×16 mm, with side-arm, fitted with bung and delivery tube to make a 'cold finger' condenser

Two lengths of rubber tubing

Test-tubes, 100×16 mm

Stand and clamp
Tripod and gauze
Bunsen burner and asbestos square
Teat pipette
Rectangle of Whatman No. 1 filter paper to form a cylinder inside a gas jar
Two clips, or needle and thread
Gas jar and cover
Litmus or Universal Indicator paper

In addition the pupils will need to have access to:

Two or more sets of apparatus for concentrating solutions under reduced pressure using filter pumps.

One or more plastic troughs or shallow beakers to contain the solution of the locating agent selected.

CHEMICALS

Approximately 2M hydrochloric acid
Approximately 2M sodium hydroxide solution
1 per cent starch solution (20 cm³ per pupil)
Fehling's solution
Iodine in potassium iodide solution
1 per cent glucose solution
1 per cent maltose solution
Propan-2-ol
Acetic acid (glacial)

LOCATING AGENTS

Either, aniline, acetone, diphenylamine, phosphoric acid
or, *p*-anisidine hydrochloride, *n*-butanol
or, *m*-phenylene diamine, tin(II) chloride, ethanol
or, saturated aqueous aniline oxalate solution.

PROCEDURE

Hydrolysis by dilute acid

Add ten drops of 2M hydrochloric acid to about 10 cm³ of freshly prepared 1 per cent starch solution in a 150 × 25 mm test-tube. After this has been boiled for a few minutes, using a side-arm test-tube as a cold finger condenser to reduce evaporation, remove five drops of the solution and make them alkaline by the addition of five drops of 2M sodium hydroxide solution before checking the presence of a reducing sugar by Fehling's test. When the test is positive some of the starch has been hydrolysed. Complete hydrolysis can be brought about by further boiling, checked by removing one drop of the hydrolysed solution and

adding iodine in potassium iodide solution; absence of a blue colour indicates that all the glucose chains in the starch have been broken.

The unused hydrolysed solutions should be just neutralized with 2M sodium hydroxide solution and evaporated under reduced pressure using a filter pump, to obtain a more concentrated solution for chromatography. This may be done individually or the hydrolysates from the whole class collected, neutralized, and then concentrated.

Hydrolysis by enzyme action

Take about 10 cm³ of the freshly prepared 1 per cent starch solution and mix with it about 1 cm³ of saliva. Allow the mixture to stand at room temperature and remove samples at intervals of a few minutes to test for hydrolysis with Fehling's and iodine solutions. When hydrolysis is complete concentrate the solution by evaporation under reduced pressure.

Identification of the hydrolysis products by paper chromatography

Rectangles of Whatman No. 1 paper should be available so that when shaped into a cylinder the edges can be fastened together but will not overlap. Rule a line in pencil about 2 cm from the bottom of the paper. The cylinders should stand freely inside the gas jar without touching the sides. The edges of the cylinder may be fastened by paper clips, sewn with two small thread loops or, more conveniently, fastened by the special tongued plastic clips produced for the purpose.

Spot the paper with aqueous 1 per cent solutions of glucose and maltose, concentrated acid- and enzyme-hydrolysed solutions at marked intervals along the pencilled line. Clean capillary tubes must be used for this and the size of the spot should be about 5 mm diameter, after spreading. When the solution spots have dried, place the cylinder, spots downward, in the gas jar containing about 1 cm depth of solvent and put the cover-glass on the gas jar. Allow the chromatogram to run overnight or for about eighteen hours. Dry the paper in the air and identify sugars with one of the locating agents suggested.

Solvent System

Propan-2-ol	3 vol.
Acetic acid (glacial)	1 vol.
Water	1 vol.

Locating agents

1. Dip quickly into a freshly prepared mixture of the following solutions:

2 per cent aniline in acetone 5 vols.

2 per cent diphenylamine in acetone 5 vols.

85 per cent phosphoric acid 1 vol.

and air dry before heating in an oven at 100°C for 2-3 minutes or warming cautiously above gauze heated by a Bunsen flame. Different colours appear for the various sugars:

Glucose – greenish fading to greenish-brown after an hour or so.

Maltose – bluish-grey.

The position on the paper of the glucose and maltose spots should be compared with those of the *two* hydrolysates.

2. Dip in freshly prepared solution made as follows:

m-Phenylene diamine 0.5 g

Tin(II) chloride 1.2 g

Acetic acid 20 cm³

Ethanol 80 cm³

Air dry the dipped paper and heat in an oven at 100°C for five minutes, or above a gauze heated by a Bunsen flame, taking care to avoid scorching the paper.

3. Alternatively the procedure in (2) may be followed using *p*-anisidine hydrochloride in *n*-butanol as the locating agent.

4. Dip the dried paper in saturated aqueous aniline oxalate solution and heat cautiously some distance above a gauze heated by a Bunsen burner or before an electric fire until brown spots appear.

Experiment E10.10

Making ethanol by fermentation

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.2)

Conical flask, 100 cm³, with cork or bung and delivery tube

Test-tubes, 100 × 16 mm

Spatula

Stand and clamp

Glucose

Yeast

Lime water

For the distillation the teacher will need:

Round-bottomed flask, 500 cm³

Fractionating column packed with glass rod or tube

Thermometer, -10 to $+110^{\circ}\text{C}$
Bunsen burner and asbestos square
Tripod and gauze
Receiving flask or beaker
Two stands and clamps

PROCEDURE

Make about 50 cm^3 of an approximately 10 per cent aqueous solution of glucose in a conical flask and add to it a spatula measure of yeast. The conical flask should be fitted with a cork or bung carrying a delivery tube bent through two right angles and dipping under a little lime water in a test-tube. The whole apparatus should be left in a warm place, preferably near a radiator, for 3–7 days.

At the end of this time the ethanol formed in the flask can be separated by distillation. Remove the test-tube containing lime water, noticing that a precipitate has formed (if there is no precipitate, boil to decompose the calcium hydrogen carbonate), and replace it with a clean test-tube.

To get a reasonable sample of ethanol it is best for the teacher to pool the results and distil all the fermented products in a large flask with a fractionating column. It is advisable to decant or filter the solution for distillation from any solid residue, to prevent excessive frothing during the distillation. The first few cm^3 to come over will contain enough ethanol to burn.

Experiment E10.11

The dehydration of ethanol

APPARATUS.

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.3)

Three hard-glass test-tubes, $150 \times 25\text{ mm}$, and corks

Hard-glass test-tube, $125 \times 16\text{ mm}$, fitted with cork and delivery tube

Broken pieces of porous pot

Teat pipette

Asbestos wool

Stand and clamp

Trough

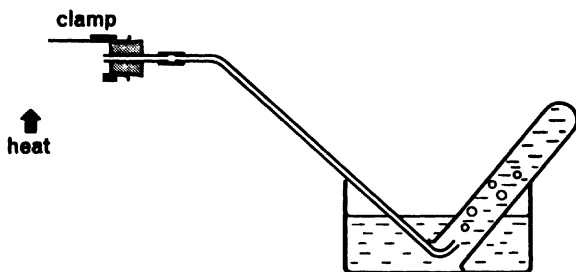
Bunsen burner and asbestos square

Ethanol (industrial methylated spirits)

Bromine water

Potassium permanganate solution

2M sulphuric acid

*Figure 10.4***PROCEDURE**

Set the apparatus up as shown in the diagram. Put asbestos wool, loosely packed, to a depth of about 2 cm in the 125 × 16 mm test-tube and drop 2 cm³ of ethanol onto it with a test pipette. Fill up the rest of the test-tube with broken porous pot. Heat the porous pot strongly with the test-tube clamped horizontally and the delivery tube attached. Do not heat the asbestos directly. After air has been expelled from the apparatus, collect the ethylene gas which is evolved in the three 150 × 25 mm test-tubes and perform the following tests.

1. Burn the gas in one test-tube. If ethylene is present it will burn with a smoky luminous flame.
2. Add a little bromine water to a test-tube of ethylene and shake it. Ethylene will react with the bromine to form 1,2-dibromoethane and the solution will become colourless.
3. Add a little dilute potassium permanganate solution, acidified with 2M sulphuric acid, to a test-tube of ethylene and shake. The colour will go as the permanganate oxidizes the ethylene.

Experiment E10.12**The hydration of alkenes****APPARATUS**

Each pupil or pair of pupils will need:

Beaker, 250 cm³

Test-tube, 150 × 25 mm

Pentene

Ice

75 per cent sulphuric acid

The teacher will need:

Separating funnel, 250 cm³

Test-tube rack and test-tubes

Distillation apparatus, with 0–200° or 0–250°C thermometer

Conical flask, 250 cm³

Anhydrous potassium carbonate

Bromine water

Acidified potassium permanganate solution

Sodium

PROCEDURE

This experiment is conveniently carried out partly by the class and partly by the teacher.

Pupils' part

Cool 5 cm³ of pentene in a 150 × 25 mm test-tube in an ice bath. Slowly add, with shaking, an equal volume of 75 per cent sulphuric acid, keeping the test-tube in the ice bath. Shake the mixture until it becomes homogeneous (this will take about five minutes) and then allow it to stand for a further five minutes in the ice bath. Carefully add an equal volume of cold water, whereupon the mixture will separate into two layers, one of pentanol and the other of dilute sulphuric acid.

Teacher's part

Gather all the products together and separate the pentanol, using a separating funnel. Wash the product with a little water, and dry it by allowing it to stand over anhydrous potassium carbonate. (Place 20–30 g of the solid in a conical flask, tip in the pentanol, and leave to stand for at least twenty minutes.) Set up a distillation apparatus, filter the dried pentanol and transfer it to the distillation flask. Distil, collecting the fraction boiling between 110° and 140°C; contrast this with the low boiling point of the pentene used.

Compare the product with the starting material in their reactions with bromine water, acidified potassium permanganate solution and sodium, and in their flammability and type of flame.

Experiment E10.13

The oxidation of ethanol

APPARATUS

Each pupil or pair of pupils will need:

Test-tube, 150 × 25 mm

Teat pipette

Acidified sodium dichromate solution (sodium dichromate 25 g, water 75 cm³, concentrated sulphuric acid 10 cm³; each pupil requires 5 cm³)

Ethanol**Bunsen burner and asbestos square****PROCEDURE**

Place about 5 cm³ of acidified sodium dichromate solution in a 150 × 25 mm test-tube and, using a teat pipette, add a few drops of ethanol. If the mixture is kept cool under a tap the sweet smell of acetaldehyde can at first be detected but after a time this becomes supplemented by a sharp acidic smell – acetic acid. When the reaction has subsided the mixture can be warmed gently, when the smell of acetic acid will become more noticeable.

Experiment E10.14

What happens when alcohols and acids are mixed?

APPARATUS

Each pupil or pair of pupils will need:

Four test-tubes, 100 × 16 mm, and corks to fit

Test-tube, 75 × 10 mm

Beaker, 250 cm³

Tripod and gauze

Teat pipette

Bunsen burner and asbestos square

Butanol

Acetic acid glacial

Concentrated sulphuric acid

Silica gel (self-indicating)

In addition as many other alcohols and carboxylic acids should be available as time and stocks allow

PROCEDURE

1. Place 10 cm³ of butanol in one test-tube, 10 cm³ of acetic acid glacial in another, and a mixture of 6 cm³ of butanol and 4 cm³ of acetic acid glacial in a third. Add four drops of concentrated sulphuric acid and a small piece of blue silica gel to each, cork lightly and allow to stand in a beaker of nearly-boiling water. Shake and inspect from time to time.

2. Place five drops of glacial acetic acid and five drops of ethanol in a 75 × 10 mm test-tube and add three drops of concentrated sulphuric acid. Warm gently and smell, and then pour the product into a 100 × 16 mm test-tube one-third filled with water and note what happens.

3. Repeat (2) using acetic acid with other alcohols, and also using ethanol with other acids. Describe the differences in words and see if you could use this reaction as a test to identify an unknown alcohol.

Experiment E10.15**The saponification of esters****APPARATUS**

Each pupil or pair of pupils will need:

Hard-glass test-tube, 150×25 mm

Cork to fit test-tube, carrying a 25 cm length of 10–12 mm diameter glass tubing to act as an air condenser

Cork to fit test-tube, carrying a right-angled delivery tube

Test-tube, 100×16 mm

Stand and clamp

Bunsen burner and asbestos square

Pumice stone or piece of broken porcelain

Ethyl acetate

2M sodium hydroxide solution

2M sulphuric acid

PROCEDURE

Place 2 cm^3 of ethyl acetate and 10 cm^3 of 2M sodium hydroxide solution in a 150×25 mm hard-glass test-tube, and support it by means of a stand and clamp above a Bunsen burner. Place the air condenser in the test-tube and boil the contents very gently until the ethyl acetate layer has disappeared.

Replace the air condenser with the delivery tube, incline the test-tube at an angle of 45° to the vertical, and support the 100×16 mm test-tube so as to collect the material coming out of the delivery tube. Heat the mixture in the larger test-tube so that some of it distils over into the smaller one. The first 2 cm^3 will contain most of the ethanol formed, and this can be detected by smell and inflammability. The residue obtained after most of the mixture has been distilled will be sodium acetate, which on acidifying with sulphuric acid will give the smell of vinegar.

Experiment E.10.16**The saponification of fats**

The breakdown of castor oil by (1) sodium hydroxide, and (2) sulphuric acid

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.5)

Four test-tubes, 100×16 mm, and rack

Test-tube, 150×25 mm

Teat pipette
Two beakers, 100 cm³
Tripod and gauze
Bunsen burner and asbestos square
Glass rod
Filter flask, funnel, filter papers, and pump
Spatula
Castor oil
5M sodium hydroxide solution
Common salt
Concentrated sulphuric acid

PROCEDURE

1. Place about 2 cm³ of castor oil in a beaker, with about 10 cm³ of 5M sodium hydroxide solution. (The pupils should be warned not to get this on their skins or on the bench.) Warm the beaker and stir the contents with a glass rod until it is boiling. Boil it gently for a few minutes, stirring throughout.

Add about 10 cm³ of distilled water and six spatula measures of salt; boil gently and stir for 2 to 3 minutes. Let the mixture cool, stir to break up any large pieces of solid and filter this off. Wash the solid residue in the funnel with a little distilled water and then allow to dry. Shake a small quantity of the solid with water in a test-tube, when it should lather, showing the formation of soap.

2. Use a teat pipette to add carefully about 2 cm³ of concentrated sulphuric acid to 1 cm³ of castor oil in a 100 × 16 mm test-tube, and stir the mixture with a glass rod. Some charring occurs and the reaction mixture becomes very hot and more viscous. Transfer the product to about 3 cm depth of distilled water in a 150 × 25 mm test-tube and stir to remove the excess of acid. Decant the water and discard it but wash the solid product again with two more portions of distilled water.

Shake a small quantity of the solid product with water in a test-tube and note the lathering effect produced.

Experiment E10.17

How detergents are made

For the preparation of a common soapless detergent, see the Unilever Laboratory Experiment, *The preparation of sodium alkylbenzene sulphonate*, (obtainable from Unilever Ltd, Education Division, Unilever House, Blackfriars E.C.4).

Chapter 11

Raw materials and industry

Introduction

This set of experiments has the object of making the pupil aware of the chemical nature of familiar substances, and of their usefulness. It is also intended to emphasize the practical value of chemistry in the world.

References

The usefulness of raw materials, and the ways in which they are exploited, are described in the *Handbook for Teachers*, Chapter 23, 'Man's conquest of materials'. A number of additional experiments illustrating industrial processes are given in *Tested Experiments for use with Chemistry for Grammar Schools* (Murray).

List of experiments

- E11.1 Electrolysis of fused sodium hydroxide
- E11.2 Electrolysis of sodium chloride solution
- E11.3 Electrolysis of sodium chloride solution with the mixing of the products
- E11.4 Electrolysis of sodium chloride solution using a mercury cathode
- E11.5 Nickel and zinc plating
- E11.6 Regeneration of cellulose
- E11.7 Experiments with rubber
- E11.8 Experiments with silicones
- E11.9 Experiments on glassmaking

E11.10 Production and curing of casein

E11.11 Analysis of a mineral

E11.12 Costing of a chemical preparation

For further experiments illustrating industrial processes see:

E10.6 The fractional distillation of crude oil

E10.7 The cracking of oil

E13.20 The polymerization of styrene (three methods)

E13.21 Production of urea-, phenol-, and resorcinol-formaldehyde resins

E13.22 Preparation of nylon

E13.23 Breaking down and building up Perspex

Experiment E11.1

Electrolysis of fused sodium hydroxide

This experiment should be done by the teacher

APPARATUS

Sheathed steel electrode (see diagram)

Nickel crucible

Source of 6 volts d.c.

Two pieces of connecting wire

Rubber tubing to connect sheath to gas supply

Tripod and pipe-clay triangle

Bunsen burner and asbestos square

Stand and clamp

Sodium hydroxide (solid)

PROCEDURE

Set up the apparatus as shown in figure 11.1 and fuse the sodium hydroxide in the crucible. (Take care.) Introduce the sheathed electrode and pass town gas through it. Ignite the issuing gas.

Switch on the current and adjust the potential to 6 volts. After twenty minutes switch off the current but still allow the gas to flow over the electrode until it is cool. Remove the electrode carefully and examine its appearance.

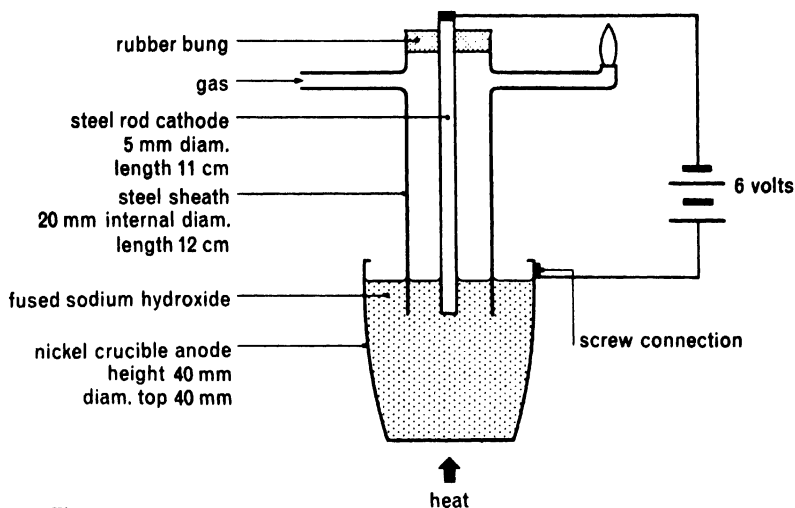


Figure 11.1

Experiment E11.2**Electrolysis of sodium chloride solution****APPARATUS**

Each pupil or pair of pupils will need:

Porous pot and electrode assembly (see diagram)

Large beaker

Test-tube

Source of 6-8 volts d.c.

Two pieces of connecting wire

2M sodium chloride solution

Potassium iodide solution

Starch solution

Phenolphthalein or litmus solution

PROCEDURE

Set up the apparatus as shown in figure 11.2

Steep the cell in approximately 2M sodium chloride solution until it is saturated, and then fill it with the sodium chloride solution and set it up as shown. Apply a potential of 6-8 volts across the electrodes.

After five minutes notice the colour of the solution in the test-tube. To this solution add one drop of freshly prepared starch solution. Notice

the colour change. Add phenolphthalein or litmus solution to the beaker. Notice the colour change.

Figure 11.2



6-8 volts

- rubber bung
- porous pot
diam. 2 cm (inter.)
length 15-20 cm
- copper wire cathode
- 5 mm diam. carbon anode
- 2M NaCl solution
- rubber bung
- water

acidified potassium
iodide solution

Experiment E11.3

Electrolysis of sodium chloride solution with the mixing of the products

APPARATUS

Each pupil or pair of pupils will need:

Porous pot and electrode assembly in steam jacket (see diagram)

Source of 6-8 volts d.c.

Two pieces of connecting wire

Steam generator and connection tubing

Beaker, 250 cm³

Tripod and gauze

Bunsen burner and asbestos square

2M sodium chloride solution

Potassium iodide solution

Starch solution

Access to fume cupboard

PROCEDURE

Set up the apparatus as shown in figure 11.3

Steep the cell in 2M sodium chloride solution until it is saturated, fill it with the sodium chloride solution, and set it up as shown. Pass steam through the steam jacket and apply a potential of 6-8 volts across the electrodes.

After fifteen minutes switch off the current and transfer the solution from the steam jacket to a beaker. Boil the solution in a fume cupboard until no more chlorine is evolved and then allow it to cool.

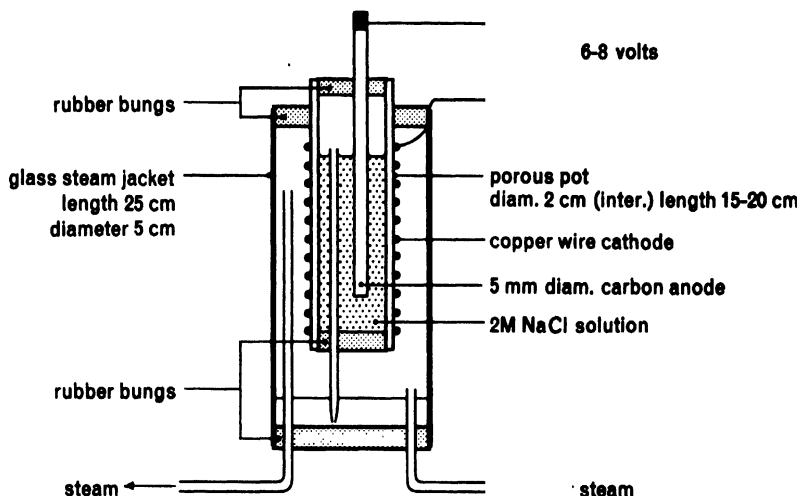


Figure 11.3

To the cooled solution, which is a mixture of sodium chlorate and sodium chloride, add acidified potassium iodide solution and notice the colour change. Add to this solution one drop of freshly prepared starch solution and again notice the colour change.

Experiment E11.4

Electrolysis of sodium chloride solution using a mercury cathode

APPARATUS

Each pupil or pair of pupils will need:

Source of 4 volts d.c.

Two pieces of connecting wire

Test-tube, 150 × 25 mm

Electrode assembly (see diagram)

Beaker, 100 cm³

Iron stirring rod

Stand and clamp

Mercury

2M sodium chloride solution

Access to fume cupboard

PROCEDURE

Place about 1 cm depth of mercury in a 150 × 25 mm test-tube and fill it to two-thirds with 2M sodium chloride solution. Insert the electrode assembly as shown in figure 11.4, making sure that the platinum wire lead actually dips into the mercury. Support the apparatus by means of a stand and clamp and place it in a fume cupboard. Connect a source of 4 volts d.c. to the electrodes, having the mercury as the cathode. Allow the current to flow for about half an hour, and then remove the electrode assembly. Carefully tip away as much as possible of the sodium chloride solution, into a beaker (not straight down the sink in case any mercury comes out). Transfer the mercury (now sodium amalgam) to a beaker, cover its surface with a dilute solution of sodium chloride, and thoroughly stir the two liquids together with an iron rod. Test the resulting solution, which contains sodium hydroxide, with red litmus paper.

After a thorough washing the mercury can be returned to stock.

chlorine ←
outlet



4 volts

rubber bung

5 mm diam.
carbon anode

2 M NaCl solution

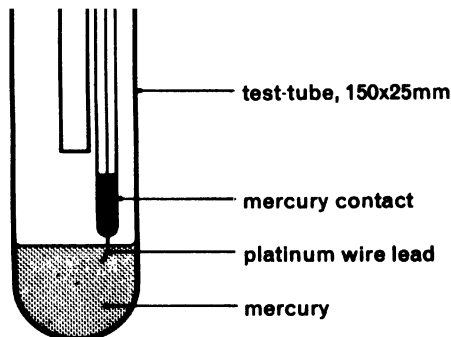


Figure 11.4

Experiment E11.5**Nickel and zinc plating**

Instructions for carrying out nickel and zinc plating were given in experiment E5.4.

Experiment E11.6**Regeneration of cellulose****APPARATUS**

Each pupil or pair of pupils will need:

Beaker, 100 cm³

Hypodermic syringe

Test-tube, 150 × 25 mm

Measuring cylinder, 25 cm³

Glass stirring rod

Cotton wool

Copper carbonate

Concentrated ammonia solution diluted with an equal volume of water

1M sulphuric acid

PROCEDURE

Place a small piece (not more than 0.25 g) of cotton wool in a test-tube and cover it with a layer of about 0.5 g of copper carbonate. Add the ammonia solution (about 25 cm³ is suitable), stirring the carbonate and the cotton wool until they have dissolved. Put the resulting solution in the hypodermic syringe and squirt it into some dilute sulphuric acid in a beaker. A thread of cellulose will be reprecipitated. The blue colour will gradually disappear from the thread.

Experiment E11.7**Experiments with rubber****APPARATUS**

Each pupil or pair of pupils will need:

Beaker, 100 cm³

Glass rod

Bunsen burner and asbestos square

Test tubes

Glass sheet

Latex (from Educational Section, Dunlop Rubber Co.)

Dilute acetic acid

Benzene

2½ per cent solution of sulphur monochloride in carbon disulphide

Samples of vulcanized, unvulcanized, and synthetic rubber sheets

1. Coagulation of latex

Add a small quantity of dilute acetic acid to latex in a beaker. Stir with a glass rod. Rubber will mass around the rod and may be withdrawn as a wet spongy mass.

A kit showing the source and materials in the initial production of rubber can be obtained from the Education Section of the Dunlop Rubber Co.

Vulcanizing is usually carried out by heating the coagulated rubber with 1–3 per cent sulphur. This cannot easily be done in the laboratory, but there is a cold cure process which is sometimes used commercially for thin rubber films.

2. Vulcanization ('cold cure' process)

Make a thin film by spreading a small quantity of natural-rubber latex onto a piece of glass and allowing it to dry; then strip the film and immerse it for one or two minutes in a 2½ per cent solution of sulphur monochloride in carbon disulphide. You will find that the rubber is now vulcanized without any further treatment.

This 'cold-cure' has been a commercial process for vulcanizing the rubber-proofing on fabrics since the early days of the industry; it was invented in 1846 by Alexander Parkes, who also founded the plastics industry.

3. Effects of vulcanization

- a. Try stretching pieces of vulcanized, unvulcanized, and synthetic rubber. Notice the differences between them.
- b. Warm gently *above* a small flame, strips of each type. Notice the effect.
- c. Place small squares of each type in benzene and leave for several hours. Note the differences in the swelling.

Experiment E11.8

Experiments with silicones

Experiment a. Effect of temperature

APPARATUS

Each pupil or pair of pupils will need:

Two soft-glass test-tubes, 125 × 16 mm

Silicone grease

Vaseline

PROCEDURE

Prepare sealed tubes made from soft-glass test-tubes containing silicone grease (e.g. silicone stopcock grease) and an organic grease such as Vaseline. Immersion for some time in very hot water will show the organic grease melting and the silicone grease unchanged. The tubes can be retained and used over and over again.

Experiment b. Water-repellency of silicones**APPARATUS**

Each pupil or pair of pupils will need:

Silicone fluid, MS 1107
Cotton wool, treated and untreated
Beaker
Red ink
Sellotape
Brick, treated and untreated
Paper and cloth, treated and untreated
Test-tubes, treated and untreated

A very thin silicone film is sufficient. An excess, as indicated by an oily appearance, is unnecessary and undesirable. The article or material to be treated is dipped in, or filled with, a 1–3 per cent solution of MS 1107 in a solvent such as carbon tetrachloride, xylene, toluene, petroleum spirit, acetone or ethyl acetate. After the surface is thoroughly wetted, the excess of solution is drained off, and the article air-dried and baked for 1–2 hours at 100°C or 15 minutes at 150°C. Glass surfaces in particular must be clean and grease-free before treatment.

PROCEDURE

- a. Cotton wool treated with MS 1107 may be dropped onto water. It will not become saturated and sink as will untreated cotton-wool.
- b. Thermal insulating brick treated with MS 1107 is non-absorbent and will float on water.
- c. Treated and untreated cotton cloth may be sprayed with a red ink solution and then washed off with water. The untreated cloth will be stained whereas the treated will not.
- d. Sellotape will not stick to treated paper, but it will stick to untreated paper.
- e. Glass test-tubes may be treated with MS 1107. The water repellency may be seen by comparing menisci of treated and untreated tubes, and by emptying the tubes and noting the amounts of residual water.

Experiment c. Reduction of surface friction**APPARATUS**

Each pupil or pair of pupils will need:

Glass plate, treated on one side with MS 1107

Coin

PROCEDURE

A coin will slip on a glass plate treated with MS 1107 tilted at an angle that will not cause it to slip on untreated glass.

Experiment d. To show antifoaming properties**APPARATUS**

Each pupil or pair of pupils will need:

Measuring cylinder, 500 cm³

Teepol

Antifoam RD

PROCEDURE

A foaming solution may be prepared by shaking Teepol with water in a measuring cylinder, and the effect of various additions of a silicone antifoaming material (e.g. Antifoam RD) noted. Additions of the order of 200 parts per million are usually effective.

AVAILABILITY OF SILICONE LIQUIDS AND TREATED PAPER AND CLOTH

Silicone fluids and silicone-treated paper and cloth are available from Messrs Hopkin and Williams Ltd, Chadwell Heath, Essex. Paper and cloth can be obtained free on request.

Experiment E11.9**Experiments on glassmaking****1. MAKING GLASSES**

No proportions are given below – make your own experiments, starting with roughly equal quantities of each constituent and then doubling them one at a time.

A. Crown glass

Mixtures of soda ash, quicklime, and precipitated silica, in varying proportions, are finely ground in small glass mortars and then heated on pieces of porcelain (bits of broken evaporating basins) over roaring Bunsen burners. The smelt becomes uniform in half an hour with a really good burner. It is then allowed to cool.

B. Flint glass

The mixtures for this glass contain litharge (prepared by ignition of lead nitrate) in addition to soda ash, lime, and silica. The finely-ground mixtures are treated as above. It is plainly to be seen that the presence of lead oxide makes the melt more easily fusible.

C. Faraday's glass

A mixture of 5.1 g of lead nitrate, 0.8 g of silica, and 1.4 g of crystalline boric acid is treated as above. The melt is seen to be readily fusible.

2. DETERMINATION OF DENSITIES

Small 'blobs' of each kind of glass are lifted from the melt on platinum wires, and then weighed in air and water. The specific gravities are calculated and compared.

3. ROUGH DETERMINATION OF REFRACTIVE INDICES

The 'blobs' of glass are dropped into small test-tubes containing solutions of carbon disulphide in ethanol, the refractive indices of which range from about 1.36 to 1.63. A 'blob' becomes almost invisible in a solution which has the same refractive index. Unfortunately, there is no readily available liquid with anything like the high refractive index of Faraday's glass.

4. THE COLOURING EFFECT OF SMALL AMOUNTS OF METAL OXIDES

Minute amounts of metal oxides can be picked up on blobs of molten glass (made more easily fusible by the addition of borax) heated in a roaring Bunsen flame on loops of platinum wire. The blob with its adhering oxide is dipped into powdered borax and reheated in the hot flame. When the writhing and swelling have subsided, the blob is allowed to cool and then observed against a bright light.

Intense colours are imparted to the glass beads by oxides of:

- a. Iron – green or brown
- b. Nickel – reddish brown
- c. Cobalt – blue
- d. Manganese – purplish

5. TO SHOW THE PRESENCE OF FREE ALKALI

Small pieces of crown glass and flint glass are ground very finely in agate mortars and moistened with phenolphthalein solution. Crown glass gives a bright purple colour and flint glass a paler purple, but the Faraday glass gives no colour.

6. TO SHOW THE TARNISHING EFFECT OF HYDROGEN SULPHIDE
Powdered crown glass, flint glass, and Faraday's glass are sprinkled inside wet pyrex test-tubes which are then filled with hydrogen sulphide and corked. In a few days some of the glass powders are dark grey instead of white. Which look as white as they did originally?

Experiment E.11.10

Production and curing of casein

APPARATUS

Each pupil or pair of pupils will need:

Milk ($\frac{1}{2}$ -pint bottle)

2M acetic acid

Formaldehyde solution, 40 per cent

Beaker, 600 cm³

Bunsen burner and asbestos square

Tripod and gauze

Thermometer, -10° to $+110^{\circ}\text{C}$

Measuring cylinders, 250 cm³ and 25 cm³

PROCEDURE

Shake a bottle of milk and allow it to stand. (This must be done the day before.) Carefully pour off the top layer and put about 100 cm³ of the lower layer (separated milk) into the large beaker. Warm the separated milk to 50°C and add 2M acetic acid dropwise until no further casein seems to be precipitated. When no further action occurs, remove the lump of casein and press it free from whey with the fingers. Knead it in hot water until it assumes an elastic nature. Wipe it dry and break it in two. Keep one piece as a control and drop the other into 40 per cent formaldehyde solution. Examine it after leaving it to stand for a day or two. Dry it and compare the properties of the two pieces. The treated casein should be much harder and bony.

Experiment E11.11

Analysis of a mineral

A detailed experiment and discussion of the analysis of a mineral is given in the Sample Scheme, Part II, *Course of Options*, Option 11, 'Analysis with a purpose'.

Experiment E11.12

Costing of a chemical preparation

A detailed experiment and discussion of the costing of a chemical preparation is given in the Sample Scheme, Part II, *Course of Options*, Option 8, 'The Chemical Industry'.

Chapter 12

Using gram-atoms; formulae and equations

Introduction

The idea that weighing substances before and after chemical reactions will provide useful information can be established early in a chemistry course (for example by use of experiment E1.2).

As soon as atomic masses have been introduced the value of quantitative experiments becomes much greater. The ideas of the gram-atom and gram-formula can be established at once, and the meanings of these terms can be made clearer by doing experiments E12.1–E12.4.

The remaining experiments in this chapter are concerned with methods by which reacting weights can be found, and used

- a. To find the formula of a compound.
- b. To find the relative number of molecules involved in a chemical reaction.

In addition to the methods described in this chapter, various electrical experiments can be done to find out the quantity of electricity required to liberate one gram-atom of various elements, and the results used to calculate the charges on the ions. These are given in Chapter 5, experiments E5.6, E5.7, and E5.8.

References

This theme is discussed in the *Handbook for Teachers*, Chapter 5, 'Gram particles and quantitative work'. In the Sample Scheme, Part I, *Basic Course*, a way of introducing the subject is given in Stage II, Topics 11.3 and 11.4, and this is elaborated in Topic 14.9.

List of experiments

- E12.1 Measurement of the volumes occupied by 1 gram-atom of various elements

- E12.2 Measurement of the volumes occupied by 1 gram-formula of various salts
- E12.3 Comparison of the volumes occupied by 1 gram-molecule of various liquids
- E12.4 Measurement of the volumes occupied by 1 gram-molecule of various gases
- E12.5 Comparison of the total weight of products of a reaction with the total weight of the reactants
- E12.6 To find out how many gram-atoms of copper combine with 1 gram-atom of oxygen
- E12.7 To find out how many gram-atoms of magnesium combine with 1 gram-atom of oxygen
- E12.8 To find out how many gram-atoms of hydrogen combine with 1 gram-atom of oxygen
- E12.9 To find out how many gram-atoms of zinc combine with 1 gram-atom of iodine
- E12.10 To find out how many gram-atoms of mercury combine with 1 gram-atom of chlorine
- E12.11 To find out how many gram-atoms of nitrogen combine with 1 gram-atom of hydrogen
- E12.12 To find out how many gram-molecules of water are associated with 1 gram-formula of a salt
- E12.13 To find out how many gram-molecules of ammonia are associated with 1 gram-formula of a salt
- E12.14 To find out how many gram-atoms of copper are made when 1 gram-atom of iron is added to copper(II) sulphate solution
- E12.15 To find the relative number of ions involved in a reaction by measuring the height of the precipitate obtained
- E12.16 To find the relative number of ions involved in a reaction by a conductivity method

- E12.17 To find the reacting quantities of hydrochloric acid and sodium carbonate – rough method
- E12.18 To find out how much carbon dioxide is evolved by the reaction between hydrochloric acid and a known quantity of sodium carbonate (syringe method)
- E12.19 To find out how much hydrogen is evolved by the reaction between hydrochloric acid and a known quantity of magnesium (syringe method)
- E12.20 To find out how much nitrogen monoxide will react with a known volume of oxygen
- E12.21 To find the simplest formula for a hydrocarbon
- E12.22 What is the formula of hydrogen chloride?
- E12.23 To find out how many gram-molecules of hydrogen chloride react with 1 gram-molecule of ammonia

Experiment E12.1

Measurement of the volumes occupied by 1 gram-atom of various elements

APPARATUS

Each pupil or pair of pupils will need:

Measuring cylinder, 25 cm³

Acetone

Samples of elements, in fairly large chips

PROCEDURE

1. *Magnesium*

Place approximately 15 cm³ of acetone in a dry 25 cm³ measuring cylinder and note the volume. Weigh accurately about 6 g (about 0.25 gram-atom) of magnesium turnings and place them in the measuring cylinder. Stir the suspension to remove air bubbles and note the new volume.

The difference in volume is the volume of that fraction of a gram-atom of magnesium that was weighed.

2. *Heavy metals*

A suitable choice of size of cylinder and volume of liquid used will depend upon the size of the chips of metal that are available. For the

heavy metals about 0.5 gram-atom is a more suitable amount to weigh, and water is a satisfactory liquid to use; the procedure is the same as that described for magnesium.

3. *Non-metals*

A similar procedure can be followed for solid non-metals, but it should be noted that fine powders are not suitable; flowers of sulphur, for example, should be avoided and chips of roll sulphur used instead.

Experiment E12.2

Measurement of the volumes occupied by 1 gram-formula of various salts

APPARATUS

Each pupil or pair of pupils will need:

Measuring cylinder, 25 cm³

Acetone or paraffin

Samples of compounds to be examined

PROCEDURE

The procedure to be followed is exactly that of experiment E12.1. As an example, the volumes of 1 gram-formula of the alkali metal halides can be compared in the following manner.

Place approximately 12 cm³ of acetone in a dry 25 cm³ measuring cylinder and read the volume as accurately as possible. Weigh out 0.25 gram-formula of the salt and place it in the measuring cylinder. Stir well to release air bubbles and read the total volume.

For this experiment it is essential to use granular materials. Fine powders cannot be handled in small cylinders, in small volumes of liquid. Lumps of crystals should be broken up to release trapped air. The volume occupied by substances that do not dissolve in water are best found using water as the liquid; for water-soluble compounds either acetone or paraffin can be employed.

Experiment E12.3

Comparison of the volumes occupied by 1 gram-molecule of various liquids

APPARATUS

Each pupil or pair of pupils will need:

Three measuring cylinders, 100 cm³

Samples of liquids mentioned below

PROCEDURE

To compare the volumes occupied by 1 gram-molecule of various liquids, place a measuring cylinder on a rough balance and pour into it sufficient of one liquid to increase its weight by the weight of 1 gram-molecule of that liquid. Repeat with a second liquid, using a second measuring cylinder.

Suitable liquids for comparison are:

Water, methanol, ethanol

Benzene, toluene, mixture of xylenes

Dichloromethane, trichloromethane (chloroform), and carbon tetrachloride

Experiment E12.4

Measurement of the volumes occupied by 1 gram-molecule of various gases

The volume occupied by 1 gram-molecule of a gas can be found by using the method of experiment E2.17. That experiment gives the weight of a certain volume of gas; from this the volume of the gram-molecular weight is easily calculated.

Experiment E12.5

Comparison of the total weight of products of a reaction with the total weight of the reactants

APPARATUS

Each pupil or pair of pupils will need:

Conical flask, 250 cm³, and cork to fit

Test-tube, 75 × 10 mm, with rim

20 cm length of cotton

Access to balance

Access to solutions of:

magnesium sulphate and barium chloride,

silver nitrate and potassium chromate,

lead acetate and potassium iodide, etc

PROCEDURE

The solutions listed above are grouped in pairs. Take a conical flask and place sufficient of one of the solutions in it just to cover the bottom. Tie a piece of cotton round the open end of a 75 × 10 mm test-tube and half-fill it with the other solution of the pair. Support the test-tube in the conical flask by means of the cotton and a cork as shown in the diagram. Weigh the whole apparatus.

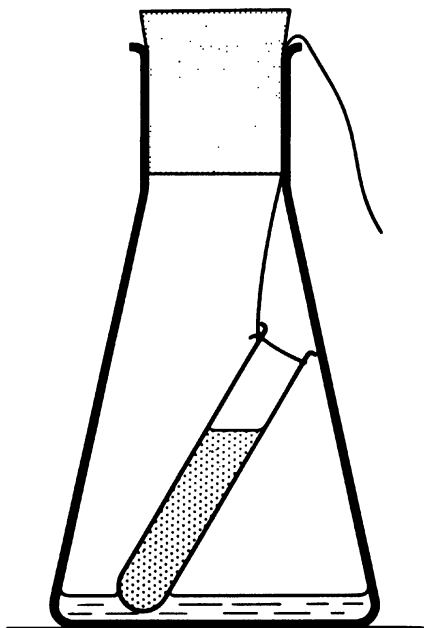


Figure 12.1

Tilt the flask so that the two solutions mix. A visible change will take place, and the cork will ensure that all the products are retained. Re-weigh the whole apparatus; no change in weight should be detected.

Experiment E12.6

To find out how many gram-atoms of copper combine with 1 gram-atom of oxygen

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 11.4a)

Hard-glass test-tube, 125×16 mm, with small hole near the closed end

Length of rubber tubing with means of connection to the test-tube

Bunsen burner and asbestos square

Stand and clamp

Pure dry copper(II) oxide (about 2 g), *analytical grade*

Access to two gas taps, and balance

PROCEDURE

In this experiment, town gas is used as a source of hydrogen for the reduction of copper(II) oxide. Take a hard-glass test-tube having a

small hole near the closed end and weigh it. Introduce 2-3 spatula measures of copper(II) oxide (see note below) and weigh the tube and contents. Set up the tube as illustrated in the diagram, and pass a small steady stream of town gas through the apparatus. As soon as it is certain that all the air has been displaced from the test-tube, but NOT before, ignite the town gas at the small hole (gas flow for about ten seconds will ensure this).

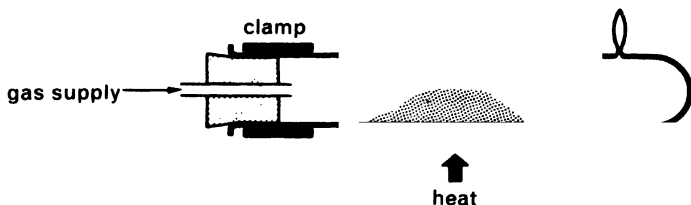


Figure 12.2

Heat the contents of the test-tube with a colourless Bunsen burner flame about 3 cm high (stronger heating will tend to 'crack' the hydrocarbons in the gas and cause a black deposit of carbon to be formed on the copper) and after a short time a red glow will be seen to spread through the copper oxide, and it will change from black to the pink colour of copper. When reduction is complete, remove the burner and leave the apparatus to cool; but it is important to keep a small stream of town gas passing through the apparatus until it is quite cold, otherwise air will enter and oxidize the copper.

When the apparatus is quite cold, switch off the gas supply, take the apparatus apart and reweigh the tube and contents.

Note: 1. It is important to use analytical grade copper oxide. 'Technical' copper oxide contains appreciable quantities of copper and use of it will give poor results. For the best results heat the copper oxide before the lesson in an open dish at 300-400°C for several minutes to drive off water vapour, and allow to cool in a desiccator.

2. It is advisable when discussing the results to take the class average. This gives a reasonably satisfactory result which the class will accept.

TREATMENT OF RESULTS

The pupils will have the following weighings:

Wt of test-tube empty = W_1 g

Wt of test-tube and copper oxide = W_2 g

Wt of test-tube and copper = W_3 g

$(W_3 - W_1)$ gives the weight of copper
and
 $(W_2 - W_3)$ gives the weight of oxygen
with which it combines

To help the pupils understand the calculation (see *Laboratory Investigations*) get them to work out the calculations like this:

1. ... g of oxygen was combined with ... g of copper.
2. How many g of copper were combined with 1 g of oxygen?
3. How many g of copper were combined with 1 gram-atom of oxygen? (Remember that 1 gram-atom of oxygen weighs 16 g)
4. How many gram-atoms of copper is this?

Hence we can write that the formula of copper oxide is CuO .

Experiment E12.7

To find out how many gram-atoms of magnesium combine with 1 gram-atom of oxygen

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 11.4b)

Crucible and lid

Tongs

Pipe-clay triangle

Bunsen burner and asbestos square

Tripod

Magnesium wire or ribbon

Access to balance

PROCEDURE

Take a crucible and lid and weigh it. Then take about 15 cm of magnesium wire (or 30 cm of magnesium ribbon), scrape the surface so that it is free from oxide, and make it into a tight coil. Place this in the crucible, put the lid on, and weigh the whole. A disc of dried asbestos wool will protect the bottom of the crucible from the burning magnesium. If this is used, it must be weighed with the crucible and treated as part of it.

Place the crucible on a pipe-clay triangle on a tripod, and the tripod on an asbestos square to protect the bench from reflected heat, and heat by means of a Bunsen burner. The flame should be a low one to start with, and should be raised gradually until the magnesium ignites, but should never be too big or the loss of magnesium oxide will be excessive. Remove the Bunsen and raise the crucible lid for a few moments at a

time with a pair of tongs so that air may be admitted. Take care to see that as little smoke as possible escapes.

As soon as the magnesium ceases to flare up when the crucible lid is removed, and appears to have finished burning, remove the crucible lid and heat the crucible strongly to make sure that combustion is complete. Then take away the flame and allow the crucible to cool. Reweigh, not forgetting the lid. Repeat the heating, raising and lowering of the lid, cooling and reweighing, in order to be sure that all the magnesium has burned.

TREATMENT OF RESULTS

The pupil will have the following weighings:

Wt of crucible and lid, empty $= W_1$ g

Wt of crucible, lid, and magnesium $= W_2$ g

Wt of crucible, lid, and magnesium oxide $= W_3$ g

$(W_2 - W_1)$ gives the weight of magnesium

$(W_3 - W_2)$ gives the weight of oxygen that combines with it

Get the pupils to work out the calculation in the way described for experiment E12.6. One gram-atom of magnesium is found to combine with 1 gram-atom of oxygen; hence the formula is found to be MgO .

Experiment E12.8

To find out how many gram-atoms of hydrogen combine with 1 gram-atom of oxygen

This experiment **must** be done by the teacher.

APPARATUS

Plastic safety screen

Combustion tube, about 15 cm

Two test-tubes, with side-arm, 125×16 mm, fitted with bungs and delivery tubes

Calcium chloride tube, bung and jet

Glass wool

Access to balance

Cylinder of hydrogen (or other source)

Concentrated sulphuric acid

Copper(II) oxide, wire form, 25–30 g

Three stands and clamps

Test-tube, 125×16 mm

PROCEDURE

Wire-form copper(II) oxide is reduced in a stream of hydrogen (town gas will *not* do for this experiment) and the water formed is absorbed. The loss in weight of the copper(II) oxide is the weight of oxygen in the water; the gain in weight of the whole apparatus is the weight of hydrogen in the water. The apparatus should first be dried in an oven and the anhydrous calcium chloride must be freshly roasted.

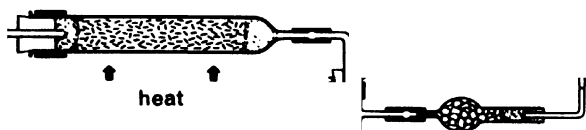


Figure 12.3

Care and attention to detail are required if a reliable answer is to be obtained.

The apparatus is illustrated in the diagram. It consists of a piece of combustion tubing shaped as shown and connected to a test-tube with side-arm. This holds concentrated sulphuric acid and in turn leads to a calcium chloride tube.

Place 25–30 g of pure dry wire-form copper(II) oxide in the combustion tube, secure it in place with tufts of glass wool, and weigh it (the weight of the copper oxide alone is not required). Then connect the combustion tube to the rest of the apparatus and weigh the whole apparatus.

Pass hydrogen, preferably from a cylinder, through a side-arm test-tube containing concentrated sulphuric acid to dry it, and then through the apparatus. (*Caution:* careful regulation of the hydrogen pressure is necessary.) It is most important to test the issuing gas before heating the apparatus. Take samples of the gas in a test-tube and set fire to them at some distance from the apparatus. When a sample burns evenly and not explosively all the air has been displaced. If the apparatus is heated before this, an explosion may take place. When it is certain that all the air has been displaced, heat the combustion tube by means of a medium-sized Bunsen flame until the copper oxide is reduced to copper.

It is important to reduce *all* the oxide and to continue the heating until no more moisture comes out of the combustion tube. The apparatus is then left to cool and when quite cold the gas supply is stopped.

The whole apparatus is then reweighed, and finally the combustion tube is detached, and it and its contents are reweighed.

Note: It is most important that both the hydrogen and the copper oxide are quite dry. Before use in this experiment, the copper oxide should be heated to 300–400°C for several minutes, and allowed to cool in a desiccator.

TREATMENT OF RESULTS

The following weighings will be obtained:

Wt of combustion tube and copper oxide = W_1 g

Wt of combustion tube and copper = W_2 g

Wt of whole apparatus at start of experiment = W_3 g

Wt of whole apparatus at end of experiment = W_4 g

$(W_1 - W_2)$ gives the weight of oxygen in the water formed

$(W_4 - W_3)$ gives the weight of hydrogen in the water formed

So

$(W_4 - W_3)$ g of hydrogen combine with $(W_1 - W_2)$ g of oxygen

Get the pupils to work out the calculations in the way described for experiment E12.6. Two gram-atoms of hydrogen are found to combine with 1 gram-atom of oxygen. Hence the formula of water is found to be H_2O .

Experiment E12.9

To find out how many gram-atoms of zinc combine with 1 gram-atom of iodine

In this experiment a weighed quantity of iodine is added to a known excess of zinc. The resulting zinc iodide is dissolved in alcohol and removed. The excess of zinc remaining is then dried and weighed. The amount used in the formation of the iodide is found by subtracting the residual weight from the weight first taken.

APPARATUS

Each pupil or pair of pupils will need:

Hard-glass test-tube, 100 × 16 mm

Tripod and gauze

Bunsen burner and asbestos square

Zinc powder

Iodine

Ethanol (industrial methylated spirits)

Access to centrifuge and balance

PROCEDURE

Weigh a dry hard-glass test-tube (100×16 mm, or whatever size will fit the centrifuges available). Put in it approximately 0.5 g of zinc powder and reweigh. Add approximately 1.0 g of iodine and weigh again. Although these elements have a great affinity for one another, no visible reaction takes place at this stage.

Cautiously add ethanol drop by drop until no further reaction takes place. This should result in a total of not more than 1–2 cm³ of ethanol being added. Shake the tube gently until the colour of the dissolved iodine disappears. At this point the reaction is complete.

Centrifuge the tube and contents for about thirty seconds, and pour off the supernatant liquid. Wash the residual zinc by adding a few drops of ethanol, agitating vigorously, centrifuging, and pouring off the supernatant liquid.

Dry the tube and contents by laying it on a gauze, on a tripod, warmed by a Bunsen burner flame not more than 3 cm high. This avoids overheating the tube and contents, which can cause particles of zinc to fly out suddenly. Allow the tube to cool and reweigh it. Obtain, by subtraction of the appropriate weighing results:

- a. the weight of zinc combining
- b. the weight of iodine combining

and hence calculate how many gram-atoms of zinc combine with 1 gram-atom of iodine.

Experiment E12.10

To find out how many gram-atoms of mercury combine with 1 gram-atom of chlorine

This experiment should be done by the teacher.

APPARATUS

Beaker, 100 cm³

Water bath or large beaker

Filter paper

Bunsen burner, tripod, gauze, and asbestos square

Measuring cylinder, 25 cm³

Glass rod

Access to balance

Mercury(II) chloride

Hypophosphorous acid solution (about 50 per cent)

Acetone

PROCEDURE

Weigh accurately about 5 g of mercury(II) chloride into a previously weighed 100 cm³ beaker. Heat on a water bath with about 30 cm³ of distilled water and add about 10 cm³ of hypophosphorous acid. The mercury(II) chloride is soon reduced to the metal, which on further heating and stirring, collects into one or more globules. When this stage has been reached, wash the metal by decantation with water, and acetone in succession – the last few drops being removed by filter paper. Allow to stand for two or three minutes and then weigh.

TREATMENT OF RESULTS

The following weighings will be obtained:

Wt of beaker empty	= W_1 g
Wt of beaker and mercury(II) chloride	= W_2 g
Wt of beaker and mercury	= W_3 g
Wt of mercury(II) chloride taken	= $(W_2 - W_1)$ g
Wt of mercury formed	= $(W_3 - W_1)$ g

One gram-atom of mercury is found to combine with 2 gram-atoms of chlorine, and hence the formula of mercury(II) chloride is found to be HgCl_2 .

Experiment E12.11

To find out how many gram-atoms of nitrogen combine with 1 gram-atom of hydrogen

This experiment should be done by the teacher.

APPARATUS

Three syringes, 100 cm³ glass

Three syringe holders and stands, or special syringe bench

Two three-way stopcocks with capillary tubing

Two combustion tubes, 15 cm long, 7 mm in diameter (ordinary hard glass is not satisfactory; transparent silica tubing or a high-silica glass is necessary)

Four pieces of hard-glass rod about 2 cm long and of diameter slightly smaller than the internal diameter of the tubes

Thick-walled rubber connecting tubing

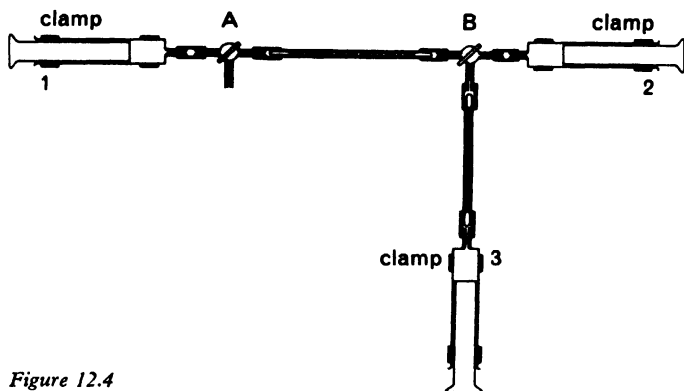
Apparatus for producing dry ammonia:

Hard-glass test-tube, 150 × 25 mm

T-piece and bung for test-tube

Drying tube containing calcium oxide

Bunsen burner and asbestos square

Hydrogen and nitrogen cylinders**Iron wool****Copper(II) oxide, wire-form****0.880 ammonia solution****Indicator paper***Figure 12.4***PROCEDURE**

Set up the apparatus in the following order: syringe No. 1, three-way stopcock (A), tube containing iron wool (one piece of glass rod at each end to keep the wool in place), three-way stopcock (B) attached to (i) syringe No. 2 and (ii) tube containing copper oxide (one piece of glass rod at each end to keep the copper oxide in place), syringe No. 3.

Make sure that the iron wool is freshly reduced to remove any trace of oxide by heating in a stream of hydrogen from a cylinder and flush the whole apparatus with nitrogen from a cylinder to drive out all air. Fill syringe No. 1 with 40 cm³ dry ammonia through stopcock (A) by gently warming about 10 cm³ of 0.880 ammonia solution in the test-tube with drying tube attached. Heat the combustion tube containing iron wool very strongly with a roaring Bunsen burner (the iron wool must be red hot). Have stopcock (B) set so that syringes No. 1 and No. 2 are connected and pass the ammonia backwards and forwards over the heated iron wool from syringe No. 1 to syringe No. 2. Do this until no further increase in volume takes place. This may require several passes. Now cool the combustion tube with a damp cloth, pass all the gas into syringe No. 1 and read the increase in volume. The volume of gases (now a mixture of hydrogen and nitrogen) should have nearly doubled. Pass the gases into syringe No. 2 and turn stopcock (B) so that syringes No. 2 and No. 3 are connected. Heat the combustion tube containing copper oxide and pass the gases from syringe No. 2 over the copper

oxide into syringe No. 3 and back until there is no further decrease in volume. The hydrogen will reduce the copper oxide and form water. The volume should be reduced to just over a quarter of the volume of the mixture of gases. Hence the result is
 2 volumes ammonia $\rightarrow$ 1 volume nitrogen + 3 volumes hydrogen.

CAUSES OF ERROR

1. It is most important that the apparatus should be dry. Otherwise some ammonia will dissolve in the water and the volume will more than double when the gas is passed over heated iron wool.
2. If there is (a) residual air in the apparatus, or (b) iron oxide on the wool, some of the hydrogen will react with this and the result will be low.
3. In the second part of the experiment water is produced. This means that the nitrogen remaining is saturated with water vapour whereas all the other volumes were measured dry. The result is therefore rather high.

Specimen result

40 cm³ of ammonia

79.5 cm³ of nitrogen and hydrogen

22 cm³ of nitrogen

The experiment shows that 1 volume of nitrogen and 3 volumes of hydrogen go to make up 2 volumes of ammonia. Assuming the formulae of nitrogen and hydrogen we can write



The formula of ammonia is thus established.

Experiment E12.12

To find out how many gram-molecules of water are associated with 1 gram-formula of a salt

APPARATUS

Each pupil or pair of pupils will need:

Evaporating basin, 50 cm³

Tripod and gauze

Bunsen burner and asbestos square

Copper(II) sulphate, finely-ground hydrated crystals

Access to balance

PROCEDURE

Weigh an evaporating basin, then place in it about 1.5-2.0 g of finely ground copper(II) sulphate crystals and reweigh. Warm the evaporating

basin and contents gently until no further change takes place (about five minutes will be required).

Allow the basin to cool and reweigh it. Repeat the heating for a few minutes, cool and reweigh. If the last two weights are not the same, repeat the heating and weighing a second time. Obtain, by subtraction of the appropriate weighing results:

a. the weight of the hydrated crystals

b. the weight of the water driven off

and hence calculate how many gram-molecules of water are associated with 1 gram-formula of copper(II) sulphate hydrated crystals.

Other suitable salts include barium chloride, magnesium sulphate, and zinc sulphate.

Experiment E12.13

To find out how many gram-molecules of ammonia are associated with 1 gram-formula of a salt

APPARATUS

Each pupil or pair of pupils will need:

Wide-mouthed bottle, 250 cm³, with air-tight lid

Crucible

Tongs

Paper tissues

Copper(II) sulphate, anhydrous

0.880 ammonia solution

Access to fume cupboard and balance

PROCEDURE

Weigh the crucible. Cover the bottom of the crucible with not more than 2–3 mm depth of anhydrous copper(II) sulphate. Assuming that the bottom of the crucible has a diameter of 2 cm, this should mean 0.3–0.5 g of the solid. Reweigh the crucible.

Place 0.880 ammonia solution in the wide-mouthed bottle to a depth of about 0.5 cm. Warning: the inhalation of the vapour can give a severe shock if unexpected, so use a fume cupboard. Using tongs, carefully lower the crucible into the bottle, and fit the lid on the bottle. Reaction begins immediately, as shown by the colour change, and is complete after about forty-five minutes.

Remove the crucible from the bottle using tongs, wipe the outside with a paper tissue, and reweigh. Expose the material to the vapour of ammonia for another five minutes, remove and clean the crucible, and

weigh it again. There should be no further increase in weight; if there is, repeat the cycle of operations until two successive weighings are the same.

Obtain:

- a. the weight of anhydrous copper(II) sulphate taken
 - b. the weight of ammonia which has been absorbed
- and hence calculate the number of gram-molecules of ammonia associated with 1 gram-formula of anhydrous copper(II) sulphate.

Other suitable salts include copper(II) chloride, and cobalt(II) chloride.

Experiment E12.14

To find out how many gram-atoms of copper are made when 1 gram-atom of iron is added to copper(II) sulphate solution

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 17.2)

Two test-tubes, 100×16 mm, in rack

Test-tube holder

Glass rod

Teat pipette

Beaker, 100 cm^3

Spatula

Bunsen burner, tripod, gauze, and asbestos square

Access to balance

Access to centrifuge – optional

Iron filings

Copper(II) sulphate, small crystals

Acetone

Distilled water

PROCEDURE

Weigh accurately about 0.5 g of iron filings in a 100×16 mm test-tube of known weight. Take approximately 2.5 g of copper(II) sulphate crystals in a second test-tube and dissolve them in about a third of a test-tube of distilled water by heating it cautiously almost to boiling point. Add the hot solution in two separate portions to the test-tube containing the iron filings, ensuring thorough mixing between solution and iron for a complete reaction to occur. The heat of reaction raises the mixture to boiling point and the reaction is complete in about a minute. Stand the test-tube for the copper to settle, or use a centrifuge if this is available, and remove the solution by teat pipette. Wash the copper with distilled water and finally with acetone. After removing the bulk of

the acetone the remainder may be driven off by standing in a beaker of nearly boiling water for a few minutes.

When all the acetone has evaporated, and only dry copper remains, weigh the test-tube and contents again to find the weight of copper displaced by the iron.

TREATMENT OF RESULTS

The weight of 1 gram-atom of iron is 56 and that of copper is 63.5.

Suppose 1.5 g of iron displaced x g of copper

Then 1 g of iron would displace $\frac{2}{3}x$ g of copper

56 g of iron would displace $\frac{2}{3}x \times 56$ g of copper

This weight of copper is then compared with the value 63.5 from which it will be seen that 1 gram-atom of iron displaces 1 gram-atom of copper.

Experiment E12.15

To find the relative number of ions involved in a reaction by measuring the height of the precipitate formed

APPARATUS

Each pair of pupils will need:

Page from *Laboratory Investigations* (experiment 17.1)

Two burettes and stands or 5 cm³ graduated pipettes

Six test-tubes, 100 × 16 mm, in rack

Glass rod

Ruler

Teat pipette

Watch-glass

Beaker, 100 cm³

Bunsen burner, tripod, gauze, and asbestos square

Access to centrifuge

Solutions selected from

1. M lead(II) nitrate and M potassium iodide
2. M barium chloride and M sodium carbonate
3. M barium chloride and M potassium chromate

PROCEDURE

Each pair of pupils is provided with molar solutions necessary for the reaction selected for investigation. As a preliminary step they should mix together approximately equal volumes of each solution in a test-tube to observe what happens.

1. Reaction between lead(II) nitrate and potassium iodide

Let us assume that the lead(II) nitrate-potassium iodide reaction has been selected. In order to find out the ratio in which the ions of the lead nitrate and potassium iodide react the pupils should take 5 cm³ volumes of the molar potassium iodide solution and add increasing volumes of molar lead(II) nitrate solution to the separate samples. This may be done by delivery from two burettes, one for each solution; a pair of burettes would be shared by two pairs of pupils if necessary. Alternatively, each pair of pupils may be provided with a 5 cm³ pipette for the potassium iodide solution and a graduated pipette for use with the lead(II) nitrate solution.

The test-tubes used must be of uniform internal diameter, and some selection may be necessary to ensure that this is so.

Each pair of pupils should deliver 5 cm³ of potassium iodide solution into each test-tube and then add 1.0 cm³ of lead(II) nitrate solution to the first tube, 1.5 cm³ to the second, 2.0 cm³ to the third, and so on. Each test-tube should be given an equal amount of stirring and then centrifuged for an equal length of time. Find out how much centrifuging is needed by a trial run on the test-tube containing most precipitate. Drops of ethanol added to a test-tube of precipitate before centrifuging help to give a cleaner precipitation in the case of lead(II) iodide.

When precipitation is complete the test-tubes should be placed in the rack and the height of precipitate measured in each. The pupils should then plot a graph of the heights measured against the volumes of lead(II) nitrate solution added. They will see that the amount of precipitate increases with increasing amounts of lead(II) nitrate solution until 2.5 cm³ is reached; further amounts of lead(II) nitrate cause no further increase in precipitate.

To find out what remains in solution, the pupils should mix 2.5 cm³ of lead(II) nitrate solution with 5 cm³ of potassium iodide solution, stir well, and centrifuge. The clear liquid should be removed using a teat pipette, and placed on a watch-glass supported over a beaker of water on a tripod and gauze. Boil the water in the beaker until the contents of the watch-glass have evaporated to dryness and examine the solid residue.

2. Reaction between barium chloride and sodium carbonate

Add 5 cm³ volumes of molar barium chloride solution to 125 × 16 mm test-tubes selected for uniform internal diameter and heat in a beaker of boiling water. Run suitable volumes of molar sodium carbonate, such as 2.0, 3.0, 4.0, 5.0, 6.0, and 7.0 cm³, into the hot solutions of barium

chloride, stir them and stand the test-tubes in the beaker of hot water until the precipitates have settled. After about five minutes remove the test-tubes from the hot water and measure the heights of the precipitates if a graph is to be plotted. The precipitates will have settled sufficiently for one to see that 5 cm³ of molar barium chloride react with 5 cm³ of molar sodium carbonate. Slight irregularities may occur in the heights of the larger precipitates and should cavities appear on standing, the contents of all test-tubes should be stirred once more and the precipitates allowed to settle more uniformly.

3. *Reaction between barium chloride and potassium chromate*

This reaction may be investigated in a similar manner to (2) the molar potassium chromate being added to hot molar barium chloride solution. The precipitate however takes much longer to settle in this case, but it will be noted that the colour of the supernatant liquid changes after 5 cm³ of potassium chromate have been added to 5 cm³ of barium chloride, indicating an excess of potassium chromate solution.

The results of these experiments are not always satisfactory as small variations in the particle size of the precipitates affect their volumes. An alternative means of finding at which stage the reaction is complete is to test the supernatant liquid for each of the reactants in turn. The test-tube without excess of either reactant is the one in which the reaction is complete.

Experiment E12.16

To find the relative number of ions involved in a reaction by a conductivity method

The relative number of ions involved in an acid-alkali reaction can be found using a method involving the measurement of the electrical conductivity of mixtures of the reactants. This is described in experiment E8.8.

Experiment E12.17

To find the reacting quantities of hydrochloric acid and sodium carbonate – rough method

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 17.3a)

Burette and stand

Conical flask, 100 cm³

Measuring cylinder

About 10 cm³ of 1.0M sodium carbonate solution

About 20 cm³ of 1.0M hydrochloric acid

PROCEDURE

Place 5 cm³ of the sodium carbonate solution in a 100 cm³ conical flask and add the acid solution 1 cm³ at a time using the burette. After each addition, the solution should be well shaken. Bubbles of carbon dioxide appear. Note the stage at which the further addition of acid produces no more evolution of gas.

Experiment E12.18

To find out how much carbon dioxide is evolved by the reaction between hydrochloric acid and a known quantity of sodium carbonate (syringe method)

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigation*: (experiment 17.3b)

Syringe, 100 cm³ glass, or 50 cm³ plastic

Test-tube, 150 × 25 mm, with bung and right-angle glass tube

Test-tube, 75 × 10 mm

Spatula

Stand and syringe holder

Rubber connecting tubing

Measuring cylinder, 25 cm³

Access to balance

Anhydrous sodium carbonate

5M hydrochloric acid

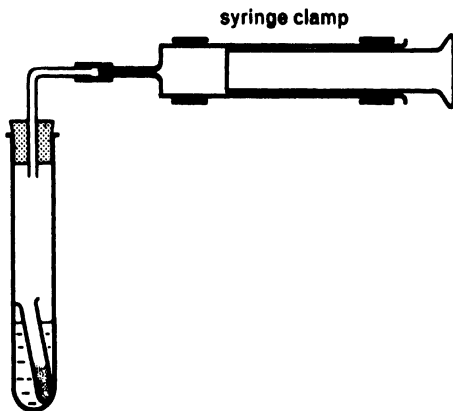


Figure 12.5

PROCEDURE

Weigh 0.002 gram-molecule (i.e. 0.212 g) of anhydrous sodium carbonate in a small test-tube or about two-thirds of this quantity if a 50 cm³ plastic syringe is used. Pour about 15 cm³ of 5M hydrochloric acid into a 150 × 25 mm test-tube. If any of the carbon dioxide evolved dissolves in the hydrochloric acid solution it will not be measured. To prevent this put two spatula measures of sodium carbonate into the hydrochloric acid to saturate it with carbon dioxide before starting the experiment. Make sure that all of this sodium carbonate has reacted and that none is left on the walls of the test-tube. Hold the test-tube nearly vertically and place the small test-tube with the carbonate in the large test-tube so that the acid does not come over the top onto the carbonate. Place the rubber bung in the test-tube mouth (note that this causes the syringe to open by about 1 cm³) and tip the acid into the carbonate by turning the test-tube towards the horizontal position. The carbon dioxide evolved will push the syringe piston out. Ensure that pressure in the test-tube is atmospheric by twisting the syringe piston lightly backwards and forwards. A manometer may be used but for this experiment 'feeling' the pressure is adequate.

Experiment E12.19

To find out how much hydrogen is evolved by the reaction between hydrochloric acid and a known quantity of magnesium (syringe method)

APPARATUS

Each pupil or pair of pupils will need:

Stand and syringe clamp

Syringe, 100 cm³ glass, or 50 cm³ plastic

Right-angle delivery tube and bung to fit test-tube

Test-tube, 150 × 25 mm

Thick-walled rubber connecting tubing

2M hydrochloric acid

Magnesium ribbon

PROCEDURE

Place about 2 cm depth of hydrochloric acid in the 150 × 25 mm test-tube and, tilting it to one side, place a small piece of magnesium ribbon (about 0.024 g) on the side so that it does not fall into the acid. Without disturbing the magnesium place the bung in the test-tube thus connecting it to the syringe. Now turn the test-tube so that it is vertical, allowing the magnesium to fall into the acid. The evolution of hydrogen will push the syringe plunger out. After the reaction is over make sure

that the syringe is at atmospheric pressure by turning the plunger lightly and then read the volume of hydrogen evolved.

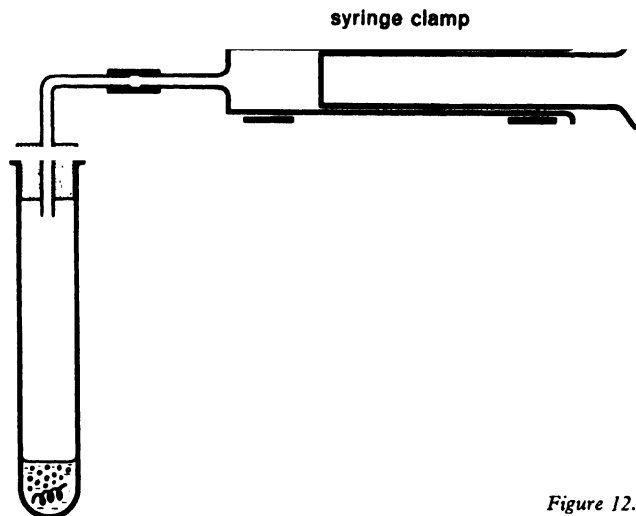


Figure 12.6

Experiment E12.20

To find out how much nitrogen monoxide will react with a known volume of oxygen

Part 1. Method for use if no syringes are available

APPARATUS

Each pupil or pair of pupils will need:

Plastic trough

Measuring cylinder, 100 cm³

Stand and clamp

Test-tube, 150 × 16 mm

The teacher will need:

Cylinder of oxygen

Supply of nitrogen monoxide (for a preparation of the pure gas see, for example, Fowles, *Lecture Experiments in Chemistry*, 6th edition, p. 314. (Bell.))

PROCEDURE

Set up the apparatus as shown in Figure 12.7, having the opening of the inverted measuring cylinder at least 1 cm below the surface of the water to prevent accidental entry of air.

Place the test-tube in the trough, fill it with water and then, by displacement, with oxygen (position 1 in the figure). Transfer the oxygen to the measuring cylinder (position 2 in the figure).

Repeat the procedure with nitrogen monoxide, adding the first few bubbles slowly, and then answer the following questions:

- At what moment does the colour begin to develop?
- What new substance is formed?
- Why does the colour disappear at the same time as the water level begins to rise?
- What is the water level in the measuring cylinder after one test-tube of nitrogen monoxide has been added?
- How much more nitrogen monoxide do you think it will be necessary to add before the level rises to the top of the measuring cylinder?

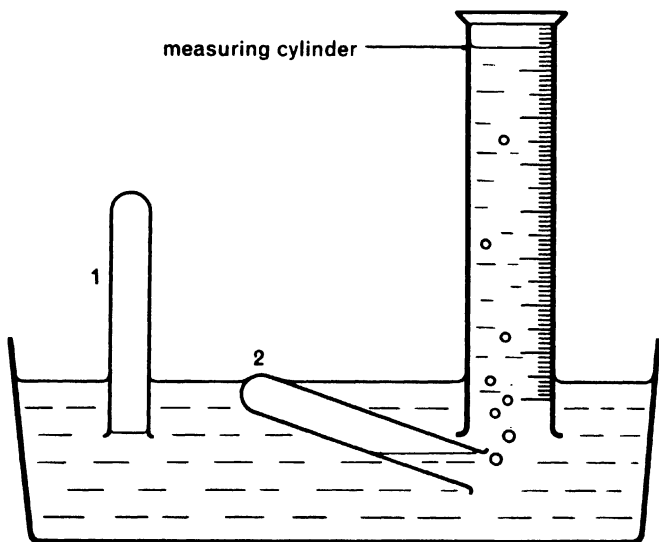


Figure 12.7

Confirm your prediction by filling the test-tube with nitrogen monoxide once again and slowly discharging it into the cylinder. If care has been taken to exclude air from the system, the volume of residual gas should be negligible.

Calculate the ratio (a) of reacting volumes, and (b) of reacting gram-molecules of nitrogen monoxide and oxygen, and from these deduce an equation for the reaction.

Part 2. Syringe method

This experiment should be demonstrated by the teacher.

APPARATUS

Two syringes, 100 cm³ glass

One three-way stopcock with capillary tubing

Apparatus for producing pure nitrogen monoxide

Rubber connecting tubing

Two stands and syringe holders

Oxygen cylinder (or apparatus for producing oxygen)

syringe clamp

syringe clamp



Figure 12.8

PROCEDURE

Set up the apparatus as shown in the diagram. Fill one syringe with 40 cm³ of pure nitrogen monoxide through the three-way stopcock 'washing out' the syringe two or three times with nitrogen monoxide first. Fill the other syringe with 50 cm³ of oxygen through the three-way stopcock, washing it out first with oxygen as before. Now turn the three-way stopcock so that the two syringes are connected, inject 5 cm³ of oxygen into the nitrogen monoxide and turn the stopcock to disconnect the syringes. The volume will expand and then contract as the gases react. Brown fumes of nitrogen dioxide will be formed. Measure the total volume of both gases and write it on the board. It will of course be less than the original volume of $40 + 50 = 90$ cm³. Repeat this process until there is no further contraction in volume when oxygen is added. Note that it sometimes takes two or three minutes for the gases to reach their final volume. Draw a graph on the board of total volume versus volume of oxygen added. It will be seen that contraction ceased after 20 cm³ oxygen had been added to 40 cm³ nitrogen monoxide.

Experiment E12.21

To find the simplest formula for a hydrocarbon

40–50 mg of the hydrocarbon are oxidized by copper(II) oxide. The volume of carbon dioxide is measured. From the volume of carbon dioxide (corrected to s.t.p.) the weight of carbon in the original sample is calculated. The weight of hydrogen is found by subtraction.

APPARATUS

Hard-glass test-tube, 125 × 16 mm

Syringe, 100 cm³

Direct-reading balance
Copper(II) oxide (dry)
Asbestos wool

PROCEDURE

A hard-glass test-tube is mounted vertically on the pan of a direct-reading balance. (A small block of expanded polystyrene or wood, with a hole provided for the test-tube, may be used.) Add 40–50 mg of the hydrocarbon to the test-tube, taking care to ensure that the sample is placed at the bottom and that none gets onto the walls of the test-tube. Fill the test-tube with dry copper(II) oxide to within 3 cm of the mouth and tap it gently to ensure firm packing. Clamp the tube at an angle of 45° as near to the mouth as possible. Insert a small plug of asbestos wool, fairly loosely packed, into the tube. (This helps to prevent the copper(II) oxide being blown or washed into the syringe.) Support the syringe loosely in a clamp and attach it to the tube via a rubber bung. Take the initial reading. If reasonable results are to be obtained, it is necessary to start the heating as soon as possible after weighing the fraction.

Heat the test-tube near the top of the copper(II) oxide filling, gently for the first few seconds and then with a near-roaring 7 cm flame. Care must be taken to heat the tube from all sides. Some of the fraction will vaporize fairly quickly and is oxidized as it passes over the hot copper(II) oxide. The carbon dioxide produced tends to push the copper(II) oxide up the tube, but this may be counteracted by tapping the tube from time to time with the barrel of the Bunsen burner. The position of the hot zone may be gradually moved to the bottom of the tube.

The rate of movement should be such as to ensure a slow and steady evolution of carbon dioxide (1 cm^3 per 2 sec). When all the contents of the test-tube have been heated and the reading on the syringe is constant, leave the tube to cool to room temperature and then note the final reading on the syringe. The room temperature and pressure will be required.

The simplest formula for the hydrocarbon can then be worked out as shown below, where figures obtained in a trial experiment are used.

Volume of carbon dioxide	$= 66 \text{ cm}^3$
Weight of sample	$= 0.040 \text{ g} = 40 \text{ mg}$
Volume of 1 gram-molecule of carbon dioxide at 764 mm and 20°C	$= 23.7 \text{ litres}$

$$\begin{array}{rcl}
 23,700 \text{ cm}^3 (\text{CO}_2) \text{ at } 20^\circ\text{C}/764 \text{ mm weigh} & 44 \text{ g} & \\
 66 \text{ cm}^3 \quad \text{''} \quad \text{''} \quad \text{''} \quad \text{''} \quad \text{''} & \frac{44 \times 66}{23,700} \text{ g} & \\
 & = \frac{44 \times 66}{23.7} \text{ mg} &
 \end{array}$$

Every 44 g of carbon dioxide contains 12 g of carbon

$$\begin{aligned}
 \text{Wt of carbon} &= \frac{12 \times 44 \times 66}{44 \times 23.7} \text{ mg} \\
 &= 33.4 \text{ mg}
 \end{aligned}$$

33.4 mg of carbon were combined with $(40 - 33.4) = 6.6$ mg of hydrogen

So 12 g of carbon are combined with $\frac{6.6 \times 12}{33.4}$ g of hydrogen

This suggests a ratio for the numbers of carbon and hydrogen atoms of 3 : 7. The molecular formula might therefore be C_3H_7 , C_6H_{14} , etc. The next step is to find out which it is.

Having found this, the simplest formula, the actual molecular formula can then be found using, for example, the method of Victor Meyer. Details of this are given in textbooks of physical chemistry.

Experiment E12.22

What is the formula of hydrogen chloride?

This experiment **must** be done by the teacher

APPARATUS

Plastic safety screen

Syringe bench with stands for syringes and pipette

Two syringes, 100 cm³ glass

Three three-way stopcocks with capillary tubing

Manometer containing light oil

Orsat pipette

Transparent silica-glass tube, 15 cm $\times$ 7 mm o.d.

Glass wool

Platinum spiral made of about 5 cm of approximately 0.2 mm diameter wire pulled out to about 1.5 cm length

Bunsen burner

Thick-walled rubber connection tubing

Hydrogen cylinder

Source of chlorine

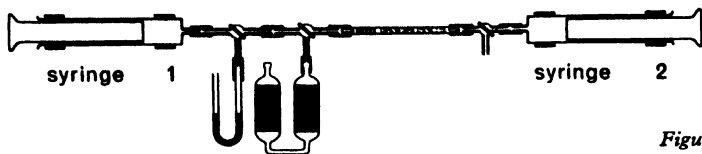


Figure 12.9

PROCEDURE

Fill 10 cm of the 15 cm $\times$ 7 mm tube with glass wool, packed fairly tightly but not so tightly that gases cannot freely pass through it. Place the platinum spiral in one end, and set up the apparatus on the syringe bench as shown in the diagram. The apparatus *must* be dry except for the Orsat pipette, which is half filled with water.

Fill syringe No. 1 with hydrogen from the cylinder through the three-way stopcock; flush the apparatus out several times before leaving 50 cm³ of the gas in syringe No. 1.

Turn the three-way stopcock so that syringe No. 2 is connected to the atmosphere and the rest of the apparatus is sealed off. Fill syringe No. 2 with 20 cm³ dry chlorine, flushing out two or three times first. It is wise to do this in a fume cupboard. Now turn the three-way stopcock so that the two syringes are connected to each other, but sealed from the atmosphere. Push the hydrogen from syringe No. 1 into syringe No. 2 so that a mixture of hydrogen and chlorine is obtained. **This mixture is potentially explosive and must not be exposed to direct sunlight.** Cover the syringe and stopcock with a cloth and place a safety screen between the apparatus and the class. Heat the silica glass tube under the platinum spiral with a medium flame until it glows red hot and then pass the mixture of hydrogen and chlorine very slowly from syringe No. 2 through the tube to syringe No. 1. The glass wool will prevent any possibility of the reaction 'blowing back' into syringe No. 2. Most of the chlorine will combine with hydrogen during the first pass but to ensure completion of the reaction the tube should be cooled, the gases returned to syringe No. 2, and two more passes made over the heated platinum spiral. Finally allow the tube to cool (a damp cloth may be used) and measure the volume of the resultant mixture of hydrogen chloride and hydrogen. The volume will be found to have contracted slightly owing to the absorption of hydrogen chloride by the small amount of water which will inevitably remain in the apparatus. To find the volume of hydrogen chloride in the mixture turn the three-way stopcock over the Orsat pipette so that syringe No. 1 is connected to the Orsat pipette, and 'pump' the gases carefully in and out until there is no further reduction in volume. The hydrogen chloride dissolves readily in the water and its volume is equal to the reduction of volume

of the gases measured. Use the manometer for the final reading to ensure that the gases are at atmosphere pressure. The volume of hydrogen chloride is usually a little less than the theoretical 40 cm^3 .

Experiment E12.23

To find out how many gram-molecules of hydrogen chloride react with 1 gram-molecule of ammonia

APPARATUS

Each pupil or pair of pupils will need:

Two syringes, 100 cm^3 glass, or 50 cm^3 plastic

One three-way stopcock with capillary tubing

Rubber connection tubing

Two stands and syringe holders

Indicator paper

Apparatus for producing dry hydrogen chloride

Apparatus for producing dry ammonia

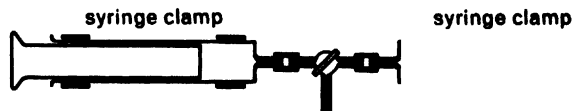


Figure 12.10

PROCEDURE

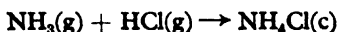
Set up the apparatus shown in the diagram. Fill one syringe with 40 cm^3 of dry ammonia through the three-way stopcock, flushing the syringe out with the gas two or three times first. Similarly, fill the other syringe with 50 cm^3 of dry hydrogen chloride. Turn the three-way stopcock so as to connect the two syringes but isolate them from the atmosphere. Push the hydrogen chloride through the stopcock into the ammonia. The gases react, forming a white powder, ammonium chloride. There remains 10 cm^3 of gas that has not reacted. Show that it is hydrogen chloride by passing it over damp indicator paper.

The result of this experiment is that 40 cm^3 of the ammonia reacts with 40 cm^3 of hydrogen chloride.

Using Avogadro's hypothesis we can write the left-hand side of the equation thus:



Given the formula of ammonium chloride, the full equation becomes:



Chapter 13

Structure

Introduction

This chapter contains experiments designed to give an indication of the way in which atoms are arranged in matter, and how the different arrangements give rise to different properties. The experiments are divided into six groups.

Group 1. Existence of particles, consists of experiments suggesting that matter is made up of particles, and setting an upper limit to the size of these particles.

Group 2. Orderly arrangement of particles, illustrates a number of the properties of crystals, and aims to show that solid substances have a regular arrangement of particles.

Group 3. Structures of elements, contains experiments by which allotropes can be made and compared, and the influence of structure on properties of elements noted.

Group 4. Structures of compounds, serves, by the making of models, as an introduction to this much more complicated field, and leads to

Group 5. Giant molecules. In this group there are experiments in which some substances which have an extended structure can be prepared, and their properties noted. Finally there are experiments on materials which have the biggest particles of all, namely,

Group 6. Colloids.

References

A full discussion on the teaching of structure is given in the *Handbook for Teachers*, Part IV, *Structural Chemistry*. One way of introducing the subject is given in the Sample Scheme, Part I, *Basic Course*, Stage II at Topic 11, 'Atoms in chemistry', and Topic 14, 'Finding out how atoms are arranged in elements'. The ideas are then used in later topics.

List of experiments**Existence of particles**

E13.1 Diffusion experiments

E13.2 The size of particles

Orderly arrangement of particles

E13.3 Watching crystals grow

E13.4 The effect of rate of crystallization on size of crystal

E13.5 Growing crystals of metals

E13.6 Crystallization from the melt

E13.7 Isomorphism

E13.8 Oriented overgrowths

E13.9 The shape and cleavage of crystals

E13.10 Making a model of a crystal structure

E13.11 Measuring the angles between crystal faces

E13.12 Looking at crystals through crossed polaroids

E13.13 Bubble-raft illustration of crystal faults

Structures of elements

E13.14 How to make different structures (allotropes) of sulphur

E13.15 How to make a model of the sulphur molecule

E13.16 The different structures (allotropes) of carbon are chemically identical

E13.17 How to make models of the carbon structures

Structures of compounds

E13.18 How to make a model of the structure of magnesium oxide

E13.19 How to make a model of the structure of ice

Giant molecules

- E13.20 The polymerization of styrene (three methods)
- E13.21 Production of urea-, phenol-, and resorcinol-formaldehyde resins
- E13.22 Preparation of nylon
- E13.23 Breaking down and building up Perspex
- E13.24 The relation between viscosity and molecular weight
- E13.25 How to make models of some large structures

Colloids

- E13.26 How to make some sols
- E13.27 The Tyndall effect
- E13.28 Brownian motion
- E13.29 Dialysis
- E13.30 Electrophoresis
- E13.31 The colloidal nature of clay
- E13.32 How to make some gels
- E13.33 Emulsions

Existence of particles**Experiment E13.1****Diffusion experiments**

- A. This experiment is best done by the teacher.

APPARATUS

Potassium permanganate solution or ammonium dichromate crystals
Small wide-mouthed bottle
Large beaker

PROCEDURE

Fill a small bottle with a strong solution of potassium permanganate and put it in the middle of a large glass beaker. Carefully run water into

the beaker until it is nearly full. Leave it until the next week, when the colour of the permanganate will have diffused throughout the water.

Alternatively drop a small crystal of ammonium dichromate into a beaker of water. Within ten to fifteen minutes the colour will spread throughout the water.

B. This experiment is best done by the teacher.

APPARATUS

One piece of glass tubing, approx. 2 cm diameter and 1 metre long

Two stands and clamps

Cotton wool

Tongs or forceps

Two corks to fit glass tube

0.880 ammonia solution

Concentrated hydrochloric acid

PROCEDURE

Support the piece of glass tubing horizontally by means of two stands. Have ready two small plugs of cotton wool and two corks to fit the tube.

Dip one piece of cotton wool in 0.880 ammonia solution and the other in concentrated hydrochloric acid. Allow the surplus liquid to drain off and place one in one end of the tube and the other in the other end. Close the ends of the tube with the corks to prevent unnecessary fumes in the room. After a short length of time a white cloud of ammonium chloride will be seen at the point at which the two gases have met after diffusing through the air towards each other. This is best viewed against a dark background.

c. This experiment is best done by the teacher.

APPARATUS

Two gas jars and covers

Vaseline

Teat pipette

Bromine

White cardboard (or other material) for background

PROCEDURE

Place a few drops of liquid bromine into a gas jar. Invert a second jar over the first, making sure that the joint between them is gas-tight by lightly smearing the top of one of the jars with Vaseline. Notice the absence of colour in the upper jar.

Leave the jars for a time. Notice the eventual appearance of the colour of bromine in the upper jar, and how long it takes to come. It is best viewed against a white background.

D. This experiment should be done by the teacher.

APPARATUS

Two gas jars and covers
Bunsen burner and splints
Supply of hydrogen

PROCEDURE

Fill a gas jar with hydrogen and invert over it a gas jar of air. After a few moments separate the jars, and apply a flame to the upper one. There will be a loud bang, as the hydrogen ignites. This demonstrates that hydrogen is a light gas and will rise into a jar containing air. Repeat the experiment, but this time have the hydrogen in the upper jar, inverted over a jar of air.

If the jars are left for about five minutes some hydrogen will be carried into the lower jar by diffusion and its presence can be detected by ignition.

E. Pupils' experiment

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 11.1)

Two gas jars and covers
Lime water
Supply of carbon dioxide

PROCEDURE

Fill a gas jar with carbon dioxide and invert it over a gas jar full of air. After a few moments separate the jars, place a few cm³ of lime water in the lower one, and shake it. The lime water will turn milky, indicating the presence of carbon dioxide. This demonstrates that carbon dioxide is a heavy gas and will fall into a jar containing air. Now repeat the experiment but this time have the carbon dioxide in the lower jar, and invert a jar of air over it. If the jars are left for about five minutes some carbon dioxide will be carried into the upper jar by diffusion and its presence can be detected using lime water.

Experiment E13.2**The size of particles**

Experiment a. Progressive dilution demonstrates that, if matter is particulate, the size of the particles must be very small

APPARATUS

Each pupil or pair of pupils will need:

One test-tube, 100×16 mm

Beaker, 100 cm^3

Crystal of potassium permanganate

White paper as background

PROCEDURE

Place a crystal of potassium permanganate in a test-tube, add about 1 cm of water and with your thumb over the end shake the tube vigorously until the whole of the crystal has dissolved. If the test-tube is now filled with water the solution will be diluted about ten times. If this solution is emptied into a 100 cm^3 beaker and the beaker is filled up, dilution by a further factor of 10 will be achieved. Fill a test-tube with this solution from the beaker and throw the rest away. Dilution of the solution can then be repeated until the colour is so pale that it is only just visible.

Experiment b. An estimate of the size of the particles**1. Pupils' experiment****APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 11.2)

Flat dish or tray, made of plastic (Polythene) or a painted 'tin' tray, at least 25 cm square, preferably larger

Powdered talc ('Baby powder'), or flowers of sulphur

Camphorated oil

Glass rod or tube drawn out to a solid 'point'

Small burette

PROCEDURE

Fill the tray with water to a depth of at least 0.5 cm and sprinkle the surface with powder. Pour the oil into the burette and run it out drop by drop. Count fifty drops and read off their volume on the burette. One drop is allowed to fall on a piece of plastic, say the lid of a bottle. The point of the glass rod is then dipped in this drop of oil and touched on the surface of the water in the tray. The oil spreads out, pushing the

powder to one side. A rough measurement is made of the area over which it has spread. It now remains to estimate what fraction of the oil drop was removed on the glass point. This may be done approximately by using the glass point to remove successive fractions from the drop until it has been used up.

An estimate of the order of magnitude of the thickness of the oil film on the water can thus be made. It will come to about 10^{-7} cm.

2. Teacher's experiment

A more accurate estimate of the thickness of an oil film can be made as follows.

APPARATUS

Large funnel, at least 15 cm diameter (or flat tray)

Stand and clamp to support the funnel

Length of rubber tubing

Teat pipette with fine point

Powdered talc, lycopodium powder, or flowers of sulphur in muslin bag

Solution of 0.1 g per litre stearic acid in petroleum ether (b.p. $40-60^{\circ}\text{C}$) (a few cm^3 only will be required). Alternatively use a solution of palmitic or oleic acid containing 0.1 g per litre. The solution may be prepared by measuring out 0.1 cm^3 of the acid.

Measuring cylinder, 25 cm^3

Reel of cotton thread or unspun silk

Vaseline or candle wax

Rule, half-metre size

Glass rod

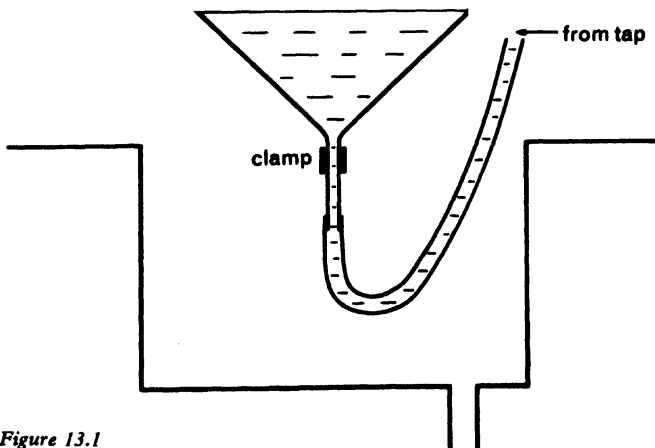


Figure 13.1

*First method***PROCEDURE**

In this experiment, which is best done by the teacher, with two pupils helping, the 'oil' is a solution of stearic (or palmitic or oleic) acid in petroleum ether containing 0.1 g per litre of the acid. When dropped on to water this 'oil' spreads out rapidly, the solvent evaporates, and a monomolecular layer of acid remains. If the surface of the water has first been dusted with talc, or with flowers of sulphur, this will be pushed aside by the spreading drop and thus the area covered by the drop will be made more visible.

A good way of obtaining a clean water surface, and of renewing it quickly for a repeat experiment, is to hold a large funnel in a ring stand over the sink and connect the stem of the funnel to the water tap by a piece of rubber tubing, as shown in the diagram. Fill the funnel. A fresh water surface is obtained merely by turning the tap on again for a few moments. An alternative method is to use a large flat dish or plastic tray, or even the sink if it can be made scrupulously clean.

Cover the clean surface of the water with powdered talc or flowers of sulphur from a muslin bag, or by shaking a little lycopodium powder over it. With the aid of a teat pipette drop one drop of the solution of acid onto the centre of the water surface. Note that spreading occurs. If the size of the spread-out drop is small compared with the area of water surface available, add a further drop or two, but remember to count how many have been added.

Now make the following measurements:

1. Make an estimate of the area of surface film formed.
2. Obtain the volume of one drop of the solution of acid by allowing 1 cm³ to drop from the pipette into a measuring cylinder, counting the number of drops.

Next calculate the thickness of the acid layer. Assuming that matter is particulate, this will show that the particles of which it is composed must be of this order of size or smaller.

SAMPLE CALCULATION

Suppose that in the above experiment one drop was found to spread out to an area of 25 cm². (It is not necessary to measure this area accurately. If the drop forms an irregular circle, about 5 cm across, call it a square to the first approximation and take its area to be 25 cm².) Suppose also that the pipette, or a burette, shows that there are 20 drops in a cm³. The solution contains 0.1 g per litre of stearic (or other) fatty acid. The density of stearic acid is 0.94 and we can take it as 1 to

the first approximation. The calculation is made easier for the class if you say that the solution contains 0.1 cm^3 of stearic acid per litre in the first case.

All that need go on the board then is:

1000 cm^3 of solution contain 0.1 cm^3 acid

$\therefore 1 \text{ cm}^3$ „ „ „ $\frac{0.1}{1000} \text{ cm}^3$ acid

1 drop „ „ „ $\frac{0.1}{20 \times 1000} \text{ cm}^3$ acid

Let the thickness of the drop when it is spread out on the surface of the water be $t \text{ cm}$. Then the volume of the drop is the thickness times the area

$$\text{i.e. } t \times 25 \text{ cm}^2$$

We can equate the two expressions for the volume

$$t \times 25 = \frac{0.1}{20 \times 1000}$$

$$\therefore t = \frac{0.1}{25 \times 20 \times 1000} \text{ cm}$$

$$t = 2 \times 10^{-7} \text{ cm}$$

or 20 Ångstrom units

Second method

PROCEDURE

Another way of performing the experiment is to float a loop of cotton or unspun silk on the water and to count the number of drops of fatty acid solution required to fill the loop with a monolayer of oil.

Make a loop from a piece of cotton or unspun silk thread about 30 cm long by tying the ends together. Cut off the loose ends near the knot. Slightly grease the thread by drawing it through fingers greased with a trace of Vaseline or candle wax. Float the loop on the water. Add drops of the ether solution from the teat pipette one at a time until the loop is taut. When the loop is filled by a solid film of fatty acid molecules, it can be pushed across the surface of the water by a glass rod, so after each drop has been added push gently against the cotton loop with a glass rod. It will be found that the loop can be dented at first, but after a certain number of drops have been added the loop will not dent but will move across the surface as a solid raft.

At this point it may be assumed that a monolayer fills the area enclosed by the loop and the thickness of the layer can be calculated. This gives an upper limit to the size of the particles. The calculation is similar to the one given for the first method.

Orderly arrangement of particles

Experiment E13.3

Watching crystals grow

A. This experiment should be done by the teacher.

APPARATUS

Micro-projector and screen

Microscope slides

Test-tubes, 100×16 mm

Teat pipettes

Spatula

Beaker, 250 cm³

Bunsen burner, tripod, gauze, and asbestos square

Warm concentrated solutions in the test-tubes of:

potassium nitrate

potassium chloride

potassium dichromate

ammonium chloride

potassium chlorate

sodium hydrogen sulphate

potassium bromide

copper sulphate

PROCEDURE

Before the lesson prepare in test-tubes about 5 cm³ of warm concentrated solutions selected from the materials above. The solutions should be sufficiently concentrated to be saturated at room temperature and form good crystals readily when placed on the microscope slide, but not so concentrated that they crystallize prematurely. When suitable solutions have been obtained, they should be kept warm in a beaker of hot water until required for the projection.

Set up the micro-projector and screen, place two or three drops of a warm concentrated solution from a teat pipette on the microscope slide and project immediately. As the solution cools, crystals are deposited and their growth observed. Select solutions so that a variety of crystal form and colour is investigated.

B. Pupils' experiment**APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.1)

Test-tube, 100×16 mm

Beaker, 100 cm^3

Two microscope slides

Teat pipette

Bunsen burner, tripod, gauze, and asbestos square

Spatula

Acetamide or naphthalene

Acetone

PROCEDURE

Add two spatula measures of acetamide (or naphthalene) to 3 cm^3 of acetone in the test-tube. The test-tube must not be heated directly over the Bunsen flame since acetone is very volatile and inflammable. To dissolve the acetamide (or naphthalene), warm the test-tube in a beaker one-quarter filled with water and supported by gauze and tripod over the Bunsen burner.

Use the teat pipette to place about five drops of the warm solution on each of the microscope slides and leave for the acetone to evaporate. If no crystallization has occurred within about a minute, touch the drop with the end of the dry teat pipette to seed the super-saturated solution, whereupon crystallization occurs immediately.

Experiment E13.4

The effect of rate of crystallization on size of crystal

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 100×16 mm, with corks to fit

Beaker, 250 cm^3

Conical flask, 250 cm^3

Thermometer, -10° to $+110^\circ\text{C}$

Potassium nitrate

PROCEDURE

Into each of two test-tubes weigh out 5 g of potassium nitrate. Add to each 10 cm^3 of water. Heat a beaker of water to 35° , and stand the two test-tubes in the water until the contents of both are also at 35° . Put a cork in each after stirring until the contents are completely dissolved. Now stand one of the test-tubes in a conical flask to protect it from

draughts, and do not move it at all. Cool the other test-tube under the tap. What is observed in each case? Leave the first tube for some hours or until the next lesson. Decant the water, and remove the crystals. Draw them. Note the different effects of rapid and slow cooling.

Experiment E13.5

Growing crystals of metals

1. Lead

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.3)

Conical flask, 100 cm³, or test-tube, 150 × 25 mm

0.1 to 0.5M lead acetate solution

Strip of zinc foil

PROCEDURE

Crystals of lead can be grown in an attractive manner by placing some approximately 0.1 to 0.5M lead acetate solution in a 100 cm³ conical flask or 150 × 25 mm test-tube, having the flask or test-tube about half full. Bend a strip of zinc foil over the neck of the flask or tube in such a way that it dips into the solution. If this arrangement is left undisturbed for several hours crystals of lead will grow outwards from the zinc.

2. Silver

APPARATUS

Each pupil or pair of pupils will need:

Test-tube, 100 × 16 mm

15 cm length of thick copper wire, about 22 s.w.g., or strip of foil

Silver nitrate solution, about 0.1M

Mercury

PROCEDURE

Silver crystals can be obtained as follows. Fill two-thirds of a test-tube with the silver nitrate solution. Shape about 15 cm of thick copper wire into a helix, by winding it around a pencil, but leave one end of the wire straight for about 5 cm. Pull the coil slightly open so that each turn is separated from its neighbour and then place it in the silver nitrate solution with the straight part bent over the top of the test-tube to hold the coil and prevent it from moving about. A cork should be placed on the test-tube to prevent dust from entering and the whole left for several hours. Crystals of silver will be seen to start developing within a few minutes.

As an alternative method, needle-like crystals of silver can be obtained by placing a drop of mercury in the bottom of a test-tube containing 0.1M silver nitrate.

Experiment E13.6

Crystallization from the melt

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 100×16 mm, with cork carrying a thermometer -10° to $+110^\circ\text{C}$

Beaker, 250 cm^3

Conical flask, 250 cm^3

One or two of the following:

t-butyl alcohol (m.p. 25°C)

phenyl salicylate (m.p. 42°C)

p-toluidine (m.p. 43°C)

p-nitrotoluene (m.p. 52°C)

p-bromophenol (m.p. 64°C)

naphthalene (m.p. 80°C)

m-dinitrobenzene (m.p. 90°C)

PROCEDURE

Crystallization may be used as a method of purification. Fill a test-tube to a depth of 2–3 cm with one of the chemicals listed above. Warm in a beaker of water to a temperature about 10° above the melting point, stir, insert a stopper carrying a thermometer, and stand in a conical flask to cool. Read the temperature every minute, and draw a cooling curve. Observe the process of freezing, and the formation of crystals, especially at first before they become massed together.

Experiment E13.7

Isomorphism

APPARATUS

Each pupil or pair of pupils will need:

Six test-tubes, 125×25 mm, with stoppers, in a rack

Beaker, 100 cm^3

Potassium chromium(III) sulphate (chrome alum)

Potassium aluminium sulphate (potash alum)

PROCEDURE

Prepare a rack of six test-tubes with stoppers, and label them A, B, C, D, E, F. Weigh out 40 g of chrome alum into a 100 cm^3 beaker, add

40 cm³ of water, and warm to 50°, stirring to dissolve the alum. The temperature should never be allowed to exceed 60° otherwise this substance does not crystallize. If the alum will not dissolve completely decant into another beaker, and keep at 50° until you are ready. In another beaker weigh out 14 g of potash alum and dissolve in 40 cm³ of hot water. Measure the hot solutions into the labelled tubes as follows:

Tube	A	B	C	D	E	F
Chrome alum	10	8	6	4	2	0 cm ³
Potash alum	0	2	4	6	8	10 cm ³

(This is best done by marking the tubes beforehand to show the appropriate levels for the volumes of solutions required.)

Stopper each tube and leave to cool slowly in a place free from draughts. After some hours filter and observe the crystals. In some cases crystals may not appear. It will then be necessary to leave these solutions to evaporate slowly at room temperature.

Experiment E13.8

Oriented overgrowths

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 100 × 16 mm, and corks to fit

Beaker, 250 cm³

Thermometer, -10° to 110°C

Single-edge razor blade

Cotton thread

Microscope slide

Teat pipette

Low-power microscope or hand lens

Crystal of calcite

Sodium nitrate

2M hydrochloric acid

PROCEDURE

Dissolve 11 g of sodium nitrate in 10 cm³ of water in a test-tube by warming and stirring. Stand it in a beaker of water at 35°C and keep it at that temperature until ready.

Cleave a rhomb of calcite with a razor blade and, without touching it with the fingers but by using forceps, tie a loop of fine thread round it. Immerse momentarily in 2M hydrochloric acid, and wash with distilled water immediately. Suspend the rhomb in the sodium nitrate

solution, and clip the thread to the lip of the test-tube by inserting a cork. Now allow the solution to cool slowly by standing the test-tube in a conical flask. Crystallization of the sodium nitrate begins on the rhomb of calcite. The sodium nitrate crystals form along the cleavage planes of the calcite. It may be found that the calcite is completely surrounded by a single crystal of the nitrate.

Discuss with the class the inference that can be drawn about the structure of NaNO_3 and CaCO_3 . This experiment may also be performed as follows:

Place a small cleaved rhomb of calcite on a microscope slide with a freshly cleaved face uppermost. Etch the face of the calcite with a drop of hydrochloric acid and wash with distilled water. Cover the crystal with saturated sodium nitrate solution, and set aside to crystallize, protected from dust. Examine the crystals with a lens or low-power microscope. Most of the sodium nitrate crystals should be aligned along the cleavage directions of the calcite.

Experiment E13.9

The shape and cleavage of crystals

APPARATUS

Each pupil or pair of pupils will need:

Crystals of sodium chloride for cleaving (or crystals of calcite, nickel sulphate, alum etc.)

Small hammer or heavy object

Single-edge razor blade

PROCEDURE

Look at some well-developed crystals of sodium chloride, calcite, nickel sulphate, or alum. These may have to be purchased or grown beforehand. A practical guide to growing crystals will be found in the Background Book, 'Growing Crystals'. Try to split a crystal with a single-edge razor blade. This is best done by placing the blade vertically upon it and giving the upper, reinforced, edge of the blade a smart tap with a small hammer. Does it cleave equally well in all directions? How might the cleavage directions be related to its structure?

Experiment E13.10

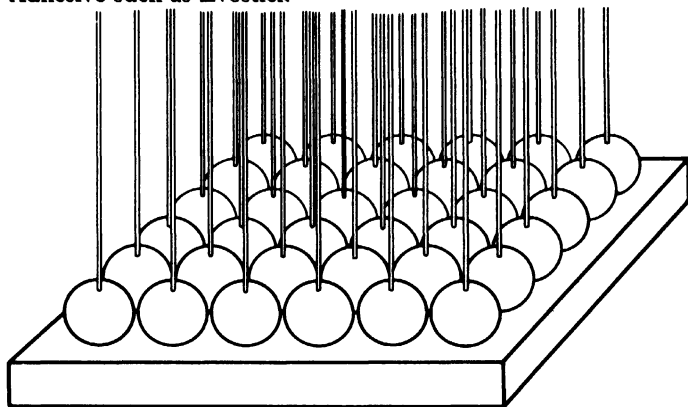
Making a model of a crystal structure

APPARATUS

The teacher will need:

Expanded polystyrene spheres 110, $\frac{3}{4}$ in or 1 in diam. and 110, $1\frac{1}{2}$ in diameter

Lengths of $\frac{1}{8}$ in dowling or plastic rod, totalling about 55 ft
 Two wooden or polystyrene base-boards, 9 in $\times$ 9 in approximately
 $\frac{1}{8}$ in drill
 Crystals of sodium chloride
 Single-edge razor blade
 Small hammer or heavy object
 Adhesive such as Evostick



PROCEDURE

Build a model of a sodium chloride or potassium chloride crystal, using expanded polystyrene spheres. The ionic radii and sphere diameters suggested are:.

Sodium: 0.95 Å
 $\frac{3}{4}$ in sphere

Potassium: 1.33 Å
 1 in sphere

Chloride: 1.81 Å
 1½ in sphere

The cube side should be at least 6 ions (108 of each kind of ion), if the model is required for X-ray diffraction experiments later. Opportunity could be taken to construct a caesium chloride structure at the same time (50 caesium and 108 chloride spheres or two caesium and three chloride layers).

Caesium: 1.69 Å
 $1\frac{1}{4}$ or $1\frac{1}{2}$ in spheres

For the NaCl or KCl structure, bore a $\frac{1}{8}$ in hole diametrically through each ball, and then string them on lengths of $\frac{1}{8}$ in dowling obtainable from a model shop. Metal supports should not be used, but plastic is suitable. A spot of adhesive at the top and bottom will hold the row in position. The rows may now be inserted in holes bored in a wooden or polystyrene base as shown in figure 13.2, when judicious use of adhesive

will hold the rows together. (It should be emphasized that the rods are aids to making the model and not representations of bonds.)

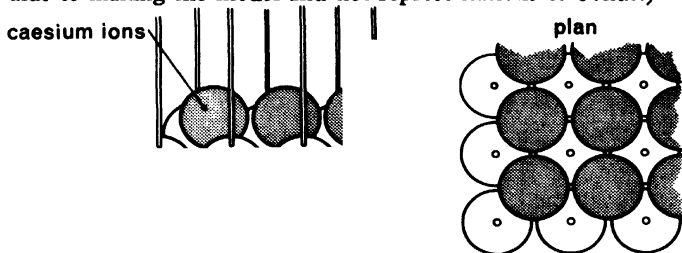
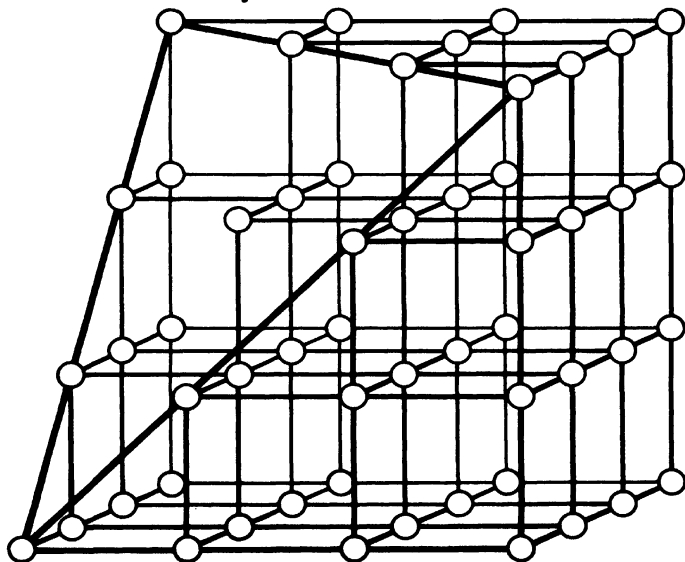


Figure 13.3

For the CsCl structure only the chloride ions need be drilled, and the bottom layer should be pegged to the base as shown in figure 13.3. The caesium ions may be placed in position with adhesive, and then the next layer of chloride ions slid down the dowels.

Examine the NaCl lattice. Ask the pupils if they think there are other possible ways of cleaving a salt crystal than parallel to a cube face. Try to cleave one in a direction parallel to an octahedral face. Suggest why it does not cleave easily in this direction.



sodium chloride (showing the direction of cleavage)

Figure 13.4

Experiment E13.11**Measuring the angles between crystal faces****APPARATUS**

Each pupil or pair of pupils will need:

Torch bulb, bulb-holder, and battery

Stand with clamp and ring

Sheet of thick card, wood, or plastic

Plain paper

Microscope slide

Protractor

Copper(II) sulphate, sodium thiosulphate, calcite, sodium nitrate, common salt, etc.

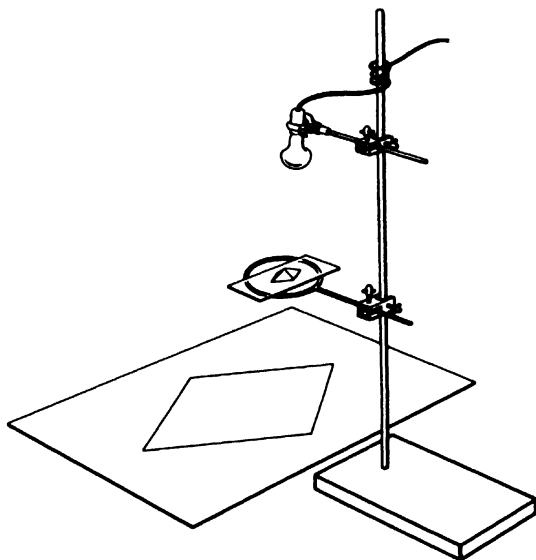


Figure 13.5

PROCEDURE

Set up the apparatus shown and screen it from direct light with sheets of cardboard. Collect very small crystals of salt, calcite, copper(II) sulphate, sodium thiosulphate, etc. Place one on the microscope slide, and cast a well-defined shadow on the card. The relative heights of lamp and crystal should be altered until a clear shadow is obtained. Make sure that the crystal is in the centre of the beam.

Now draw the outline of the crystal and measure its angles by means of a protractor. Repeat for different crystals of one compound, trying various sizes.

Grow some crystals of common salt, sodium nitrate, etc., on a microscope slide and examine them also in this way.

Experiment E13.12

Looking at crystals through crossed polaroids

APPARATUS

Six microscope slides

Two pieces of polaroid, 1 in $\times$ 1 in

Two blocks of wood, cork or styrofoam, 1 in $\times$ $\frac{1}{4}$ in $\times$ $\frac{1}{4}$ in

Hand lens or low-power microscope

Sodium nitrate, sodium chloride, etc.

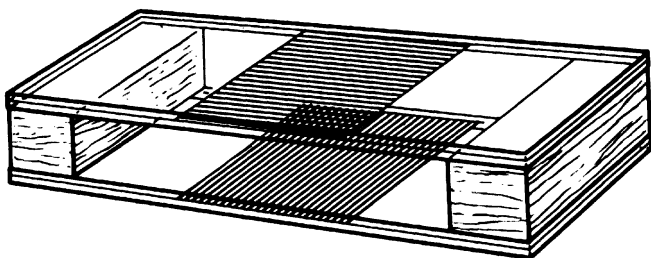


Figure 13.6

PROCEDURE

A simple polariscope can be made using crossed polaroids as shown. Place one square of polaroid centrally between two microscope slides, and cement them together. Use a second pair of slides to hold the other piece of polaroid, which must be 'crossed' with respect to the print piece. The pairs of slides are then cemented to the $\frac{1}{4}$ in spacers. A crystal placed on a microscope slide can be examined by inserting it in the gap between the two polaroids. There is enough space to rotate the slice between the crossed polaroids. Examine the appearance of the crystal through the top polaroid with a hand lens or, better, with a low-power microscope. In some cases, instead of the extinction of light, a coloured image is observed. This is characteristic of certain crystals and although the effects are too complex for full discussion at this stage, they may be noted as being connected with the structure of crystals. (Reference: Most textbooks on Crystallography or Mineralogy, e.g. *Crystals and Light* by E. A. Wood.)

Using the simple polariscope examine both (1) small crystals of sodium nitrate and (2) a drop of sodium nitrate solution, on microscope slides. We may ask why the optical activity of the sodium nitrate crystal is lost when it dissolves. Obviously the ions, though still present, are no longer arranged in the right sort of way. This experiment may be repeated with other substances.

Experiment E13.13

Bubble-raft illustration of crystal faults

APPARATUS

Each pupil or pair of pupils will need:

Beaker, 100 cm³

Petri dish and rectangular glass cover-plate

Glass tube drawn out to very fine jet

Small quantity of a solution of 10 cm³ of Teepol in about a litre of water

Air reservoir (described below), or delivery tube from gas tap

PROCEDURE

Place a quantity of the solution of Teepol in water in a Petri dish. Then lead a steady stream of gas into the solution so as to blow a large number of similarly sized bubbles. It is difficult to blow into a tube sufficiently steadily to obtain equal-sized bubbles but a steady flow of air can be obtained as follows. Have a large flask or bottle equipped with a cork carrying two short delivery tubes. Connect with rubber tubing one delivery tube to the water tap and the other to a piece of glass tubing drawn out to a fine jet. A screw-clip on the rubber tubing is needed to control the flow.

When the water tap is turned on gently a steady stream of air will be displaced. It is important to obtain bubbles no larger than 2–3 mm in diameter. They may be fixed by means of a glass plate covering the dish just above the surface of the liquid. Alternatively use town gas from the gas taps. *In this case make sure that the laboratory is well ventilated and that the gas taps are turned off as soon as the raft has been made.*

A device which is alternative, or complementary, to a bubble raft and which may be made for homework is as follows.

MATERIALS

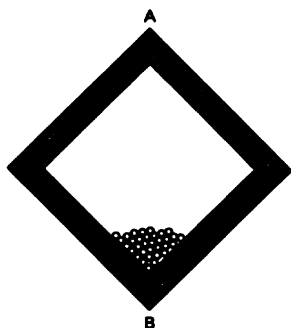
Two glass cover-slips 2 × 2 in sold in photographic shops for colour slides

Lead shot or silver balls used for cake decoration

Thick cardboard (corrugated suitable)

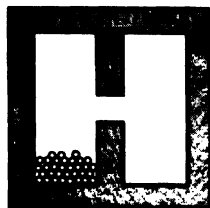
Sellotape

SOLID-LIQUID-GAS MODEL



Hold vertically between A and B using thumb and first finger. Gentle random motion is obtained by shaking, and tilting A backwards towards the horizontal.

DIFFUSION OF GASES MODEL



30 to 40 small spheres are placed in the model preferably selecting one of different colour from the remainder. Random motion is produced by gentle shaking in a horizontal plane.

Figure 13.7

Structures of elements

Experiment E13.14

How to make different structures (allotropes) of sulphur

Note: In these experiments *powdered roll sulphur* should be used. 'Flowers of sulphur' is not suitable.

1. Rhombic sulphur

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.5)

Test-tube, 100 × 16 mm

Watch-glass

Spatula

Filter paper

Powdered roll sulphur or pestle, mortar, and small lump of roll sulphur

Access to carbon disulphide, which should be kept in a fume cupboard.

PROCEDURE

Powder a piece of roll sulphur about the size of a small pea, or take a spatula measure of powdered roll sulphur, and add it to about 2 cm depth of carbon disulphide in a test-tube. Agitate the mixture (but do *not* warm it) and when most of the sulphur has dissolved decant the

solution onto a watch-glass in a fume cupboard. Cover this with a piece of filter paper and leave to evaporate slowly. Rhombic crystals will be seen after ten or twenty minutes.

Note: Carbon disulphide is dangerously inflammable, extremely toxic, highly volatile, and possesses an unpleasant smell. Its use should be carefully controlled.

2. Monoclinic sulphur from solution

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.5)

Hard-glass test-tube, 150×25 mm

Spatula

Bunsen burner and asbestos square

Powdered roll sulphur or pestle, mortar, and a small lump of roll sulphur

Xylene (toluene is a suitable alternative but is a little more volatile; olive oil may also be used)

PROCEDURE

Powder a piece of roll sulphur about the size of a small pea, or else take a spatula measure of powdered roll sulphur, and add it to about 3 cm depth of xylene in the test-tube. The test-tube and contents should then be warmed over a *low* Bunsen burner flame until the sulphur has all dissolved. The liquid should NOT be allowed to boil.

A little more sulphur may be added until a saturated solution is made, that is, until a few grains of sulphur remain undissolved. The solution should now be left to cool.

Caution: Xylene is inflammable, but is not very volatile and the vapour is dense and tends to stay in the test-tube, provided a low Bunsen burner flame is used. In the event of fire, turn the burner out at once, and cover the mouth of the test-tube with a piece of card to extinguish the flames. If the test-tube breaks, use sand, not water, to cover the flames.

3. Monoclinic sulphur from liquid sulphur

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.5)

Hard-glass test-tube, 100×16 mm

Test-tube holder

Powdered roll sulphur
Paper clip
Filter papers
Tongs
Bunsen burner and asbestos square

PROCEDURE

Place sufficient powdered roll sulphur in a test-tube to fill it to a depth of about 6 cm. Fold together two thicknesses of filter paper as though to put in a filter funnel, fasten with a paper-clip, and hold the cone of filter paper with the tongs over an asbestos square.

Heat the test-tube containing the sulphur gently over a low Bunsen burner flame until it has all just melted to a pale amber coloured liquid. (Deep red liquids produced by too strong a heating should be allowed to cool.) Pour the amber liquid into the filter paper and allow it to cool.

As soon as a crust has formed over the liquid, take off the paper clip, open the paper, and break open the cone-shaped piece of sulphur. Surplus liquid will drain away (take care! this is very hot) revealing crystals of sulphur growing from the sides of the cone towards the middle.

4. Plastic sulphur

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 14.5)

Test-tube, 100 × 16 mm

Beaker, 100 cm³

Test-tube holder

Bunsen burner and asbestos square

Powdered roll sulphur

PROCEDURE

Place sufficient powdered roll sulphur in a test-tube to fill it to a depth of about 3 cm, and fill a 100 cm³ beaker with water. Hold the test-tube with a test-tube holder and heat the tube until the sulphur first melts, and finally boils. As soon as it is boiling pour the liquid swiftly into the water.

Plastic sulphur will form under the water, which rapidly cools the boiling liquid form. After it has cooled sufficiently to touch, it may be removed and examined.

Experiment E13.15**How to make a model of the sulphur molecule****APPARATUS**

Each pupil or pair of pupils will need:

- Page from *Laboratory Investigations* (experiment 14.5)
- Eight expanded polystyrene spheres ($\frac{1}{2}$ to 1 inch diam.)
- Pipe cleaners, approximately 15 cm long
- Access to wire cutters
- Dividers, or instrument with similar point
- Protractor for use with 1 inch diameter spheres

PROCEDURE

Put two marks on the equators of each sphere, 105° apart. Pierce the spheres at these marks, and using short lengths of pipe cleaner, join eight spheres together in a ring structure so that four of the spheres are below in the form of a square, and the other four are in a square above them, at 45° to the first square.

Experiment E13.16

The different structures (allotropes) of carbon are chemically identical

A. Pupils' experiment**APPARATUS**

Each pupil or pair of pupils will need:

- Page from *Laboratory Investigations* (experiment 14.6)
- Two hard-glass test-tubes, 100×16 mm
- Test-tube holder
- Spatula
- Teat pipette
- Samples of charcoal, graphite (and industrial diamond if available)
- Copper(II) oxide powder
- Lime water

PROCEDURE

Mix a spatula measure of the carbon sample with three times its volume of copper(II) oxide and heat the mixture strongly in a test-tube. Extract a sample of the gas in the test-tube with a teat pipette and expel it under lime water in another test-tube. The lime water will go milky, showing that carbon dioxide is present.

B. This experiment should be done by the teacher.

APPARATUS

Two syringes, 100 cm³ glass

Silica glass tube, 15 cm × 7 mm o.d.

Two hard-glass rods, approximately 5 cm long, to fit inside the tube, leaving about 1 mm free

Manometer containing light oil

T-piece with tap

Three-way stopcock with capillary tubing

Absorption pipette

Syringe bench with stands for two syringes and absorption pipette

Glass wool

Bunsen burner

Access to four-place balance (preferably automatic)

Dry charcoal

Graphite

Industrial diamonds if available

5M potassium hydroxide solution (enough to half fill the absorption pipette)

Oxygen cylinder (or alternative source of oxygen)

Grease for one syringe

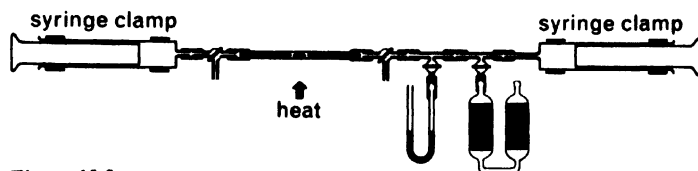


Figure 13.8

PROCEDURE

Set up the apparatus as shown in the diagram. The syringe nearest the absorption pipette must be greased. Weigh a piece of the carbon sample, about 0.024 g, in the silica glass tube. In the case of charcoal or graphite fill the ends of the tube with glass rods to prevent the sample moving out of the tube when gas is passed over it. If industrial diamonds are used, plug the ends of the tube with glass wool. It is most important to ensure that no pieces of diamond or diamond dust enter the syringes, as they may cause considerable damage.

Graphite and diamond. Fill one syringe with 50 cm³ of oxygen through the three-way stopcock.

Charcoal. Fill one syringe with 50 cm³ of oxygen and draw in 50 cm³ of air to dilute. Charcoal sometimes gives minor explosions with pure oxygen.

Heat the carbon sample strongly and push the gas backwards and forwards across it slowly, having your hands on both syringes to prevent any excess pressure causing a leakage. When the carbon has burnt away, leaving only a residue of ash, cool the tube down with a damp cloth and measure the total volume of gases. It should remain unchanged. The explanation, which should be discussed with the pupils, is that each molecule of oxygen is replaced by one of carbon dioxide which occupies the same volume. To find the volume of carbon dioxide draw all the gases into the syringe nearest the absorption pipette, turn the stopcock so as to isolate the syringe and pipette from the rest of the apparatus and connect the pipette with the syringe. The manometer tap should be closed at this stage. Pump the gases in and out of the absorption pipette until no further diminution of volume takes place, using the manometer to check that the gases are being measured at room pressure. The volume of carbon dioxide is the volume of gas absorbed. Finally weigh the silica tube again to find out how much of the sample has been burnt.

Experiment E13.17

How to make models of the carbon structures

APPARATUS

Each pupil or pair of pupils will need:

Pipe cleaners

Access to wire cutters

Dividers or other instrument with a point

For graphite: Thirteen expanded polystyrene spheres, 1 inch diam.

For diamond: Fourteen expanded polystyrene spheres, 1 inch diam.

PROCEDURE

Graphite

Mark the spheres at 120° round the equator. Pierce and join the spheres together with short lengths of pipe cleaner in rings of six carbon atoms. With thirteen spheres, one layer containing three rings can be made.

Diamond

Mark the spheres tetrahedrally. Pierce and join together with short lengths of pipe cleaner. The unit cell requires fourteen spheres.

Structures of compounds

Experiment E13.18

How to make a model of the structure of magnesium oxide

APPARATUS

For each model is needed:

Fourteen polystyrene spheres (say $1\frac{1}{2}$ inch diam.)

Thirteen polystyrene spheres (say $\frac{3}{4}$ inch diam.)

Pipe cleaners

Wire cutters

Dividers, or instrument with similar point

PROCEDURE

The radii of the magnesium and oxygen ions are in the ratio of about 1:2 ($Mg^{2+} = 0.66 \text{ \AA}$, $O^{2-} = 1.40 \text{ \AA}$). The arrangement is like that of common salt, i.e. a face-centred cubic structure with respect to both magnesium and oxygen particles. Start by marking out all the spheres with four points at 90° round an equator. Make one face of the cube by joining five $1\frac{1}{2}$ in spheres and four $\frac{3}{4}$ in spheres so that four of the larger ones are at the corners of a square with the fifth in its centre, and the four smaller ones are in the middle of each side of the square. Lay this on the bench and add a second layer consisting of four large spheres and five small ones, this time with the small ones at the corners and the large ones in the middle of the sides of the square. Finally, add a third layer identical with the first.

Experiment E13.19

How to make a model of the structure of ice

A useful jig to hold the spheres in the correct positions may be constructed by drilling holes in a sheet of hardboard using dimensions given below.

APPARATUS

Hardboard jig

Twenty-nine polystyrene spheres 2 inch diam.

Evostick or pipe cleaner

JIG CONSTRUCTION

Draw an equilateral triangle of side 9.9 in on hardboard, and drop a perpendicular from an apex.

Construct circles, diameter 1.3 in, on the sides of the triangle as well as at the apices, the distance between centres being 3.3 in. Along a perpendicular 5.7 in from the apex make the centre for one further circle diameter 1.3 in.

Cut out the circles and use the jig for making the ice structure. Place one ball in each hole in the jig (nos. 1–10 in figure 13.9(a)). Rest six more balls above them (nos. 11–16). Stick or connect at their points of contact all balls except nos. 1, 7 and 9. Lift off the resulting assembly of 13 balls. Repeat this operation making a second assembly of 13 balls (figure 13.9(b)). Stick the two assemblies together so that the following pairs of balls are in contact: 11/24, 12/26, 13/25, 14/29, 15/28, 16/27.

Note that balls 1, 7, 9 are for support only.

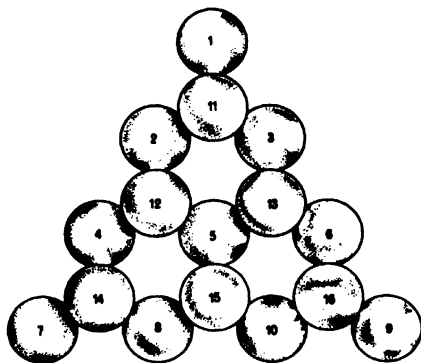


Figure 13.9a

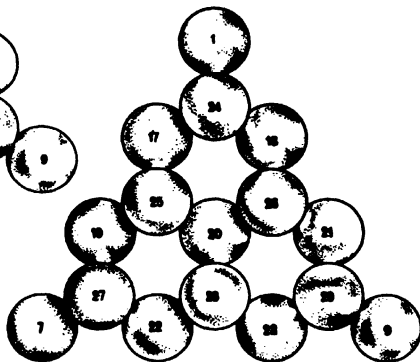


Figure 13.9b

The balls represent oxygen atoms in the ice structure. For the sake of simplicity hydrogen atoms are not represented. Oxygen atom 11 is joined through hydrogen atoms to four other oxygen atoms, three in the plane below (1, 2, 3) and one in the plane above (24) in a tetrahedral arrangement. This applies to all oxygen atoms in the interior of the ice crystal. Each oxygen atom has four hydrogen atoms as fairly close neighbours; two of its own which are strongly attached but two more (belonging to two other oxygen atoms) which are at a greater distance, held by the weak hydrogen bonding. Thus the whole structure is one large molecule, made of many water molecules joined together by hydrogen bonds.

This is an 'open' structure and has relatively large empty spaces in it. The roominess of the ice structure explains the expansion which takes place when water freezes.

Giant molecules**Experiment E13.20**

The polymerization of styrene

Method (a). Using kerosine as catalyst

(The use of benzoyl peroxide as a catalyst is to be avoided, as this substance may decompose violently if dry.)

APPARATUS

Each pupil or pair of pupils will require:

Styrene

Kerosine

Flask, 100 cm³, with reflux air condenser

Beaker, 250 cm³

Methanol

Spatula

PROCEDURE

Mix equal volumes (say 10 cm³) of styrene and kerosine and reflux the mixture under an air condenser for 45 to 60 minutes. The temperature will be about 150°C.

When it is cool, pour the mixture into about five times its own volume of methanol. The polymer appears as a white solid and hardens to a wax when worked with a spatula under the surface of a fresh lot of methanol.

Method (b). Using tin(IV) bromide as catalyst

APPARATUS

Each pupil or pair of pupils will require:

Spatula

Hard-glass test-tube, 150 × 25 mm, with air condenser, and cork to fit
Beaker, 250 cm³

Bunsen burner and asbestos square

Tripod and gauze

Approximately 30 cm length of glass tubing 5-6 mm internal diameter

Sticky tape

Retort stand and clamp

Tin(IV) bromide

1, 2-dibromoethane

Styrene

PROCEDURE

Put about 0.5 g of tin(IV) bromide in a 150 × 25 mm test-tube. Add about 1 cm depth of 1, 2-dibromoethane and shake to dissolve the tin(IV) bromide (pour off the clear layer into a second test-tube if all the tin(IV) bromide does not dissolve). Add about 1 cm depth of styrene and shake to mix. Fit the test-tube with a cork and air condenser and stand in a beaker of boiling water for 20 minutes. (Stage A.)

Take a length of glass tubing, 5-6 mm internal diameter, and seal it at one end. Pour about 10 cm³ of the mixed reactants from stage A above into the tube placed vertically. Allow to cool and mark the level with sticky tape. Note the decrease in volume as the polymerization increases. (This may take several days.)

Method (c). Emulsion polymerization**APPARATUS**

Each pupil or pair of pupils will require:

Mechanical stirrer

Bunsen burner and asbestos square

Tripod and gauze

Water bath

Thermometer, -10° to +110°C

Wide-necked flask (to accept stirrer), 500 cm³

Separating funnel, 50 cm³

Distilled water

Measuring cylinders, 250 cm³ and 25 cm³

Styrene

2M sodium hydroxide solution

Detergent

Potassium persulphate or ammonium persulphate

Sodium metabisulphite

PROCEDURE

Take 20 cm³ of styrene and shake in a separating funnel with 20 cm³ of 2M sodium hydroxide solution to remove inhibitor. Run off the aqueous layer and shake the styrene with distilled water to wash it and run off the aqueous layer again. Place the styrene in the 500 cm³ flask and add 200 cm³ of distilled water, 1 g of detergent such as sodium lauryl sulphate and 1 g of a water-soluble initiator, potassium or

ammonium persulphate. Also add 0.5 g of sodium metabisulphite. Heat the water bath to about 40°C, stirring the contents of the flask thoroughly all the time. A polymer latex will form within half an hour.

Experiment E13.21

Production of urea-, phenol-, and resorcinol-formaldehyde resins

APPARATUS

Each pupil or pair of pupils will need:

Asbestos square

Measuring cylinders, 100 cm³, and 10 cm³

Stirring rod

Beakers, 1 litre, 250 cm³, and 100 cm³

Spatula

Filter papers

Concentrated sulphuric acid

Concentrated hydrochloric acid

Formaldehyde, 40 per cent

Resorcinol

Phenol

Urea

PROCEDURE

This experiment should be done in a fume cupboard. It can be done in the open if a litre beaker is inverted over the smaller beaker until the fumes subside.

1. Place the 250 cm³ on the asbestos square, pour in 45 cm³ of formaldehyde solution and add 20 g of resorcinol. Mix thoroughly by stirring, add 3 cm³ of concentrated hydrochloric acid and stir again. Wash the solid thoroughly with water and remove it from the beaker.

2. Repeat the above experiment with phenol as an alternative. A few cm³ of concentrated sulphuric acid should be added carefully and the residue kept until condensation is complete (which may take several days).

Warning: Phenol has considerable blistering action on flesh.

3. Place 20 cm³ of 40 per cent formaldehyde solution in a 100 cm³ beaker and add about 10 g of urea with stirring until a saturated solution is obtained. Add a few drops of concentrated sulphuric acid. The mixture hardens quite suddenly. Dry the residue with filter paper, after washing it with water.

Experiment E13.22**Preparation of nylon****APPARATUS**

Each pupil or pair of pupils will need:

Beaker, 100 cm³

5 per cent solution of hexamethylene diamine in water

Either 5 per cent solution of adipyl chloride in carbon tetrachloride

or 5 per cent solution of sebacoyl chloride in carbon tetrachloride

Glass rod

PROCEDURE

It is suggested that half the class prepare 66 nylon and the rest the 610 variety. They can then compare results. Place a few cm³ of the adipyl chloride solution (or sebacoyl chloride for the 610 nylon) in the beaker and carefully add the hexamethylene diamine solution so as to form a separate layer. Pull out a little of the solid produced with a glass rod and draw it out by lifting the rod and winding the thread onto it. After some of the thread has been obtained the mixture can be stirred to get a lump of nylon.

Experiment E13.23**Breaking down and building up Perspex****APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 21.6)

Hard-glass test-tube, 125 × 16 mm, with cork and delivery tube

Test-tube, 100 × 16 mm

Beaker, 100 cm³

Bunsen burner and asbestos square

Tripod and gauze

Stand and clamp

Access to oven

Chips of Perspex (about 5 g)

Lauroyl peroxide (about 0.02 g)

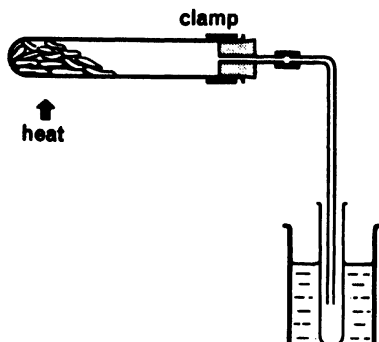


Figure 13.10

PROCEDURE

Caution. This experiment *must* be done in a very well-ventilated laboratory as poisonous fumes are evolved during the heating of Perspex.

Place a few chips of Perspex in the test-tube fitted with a cork and delivery tube. Heat the tube with a non-roaring flame and collect the distillate in a test-tube, which should be cooled by standing it in a beaker of water.

The liquid distillate contains the monomer which may be polymerized by heating it in a test-tube standing in a beaker of warm water at about 50°C. Add a small quantity (about 0.2 per cent) of lauroyl peroxide to catalyse the polymerization. This occurs in about two hours but may be speeded up by increasing the concentration of the catalyst.

Experiment E13.24

The relation between viscosity and molecular weight

APPARATUS

Each pair of pupils should have access to the following, but one or two sets would probably be adequate. Alternatively this could be done as a demonstration experiment.

Three tubes approx. 70 cm long and 5–7 mm internal diam., sealed at one end

Silicone liquids (obtainable from Hopkin & Williams) as follows:
MS200/3, 100, 100000

Three short lengths of glass rod and rubber tube for sealing

Three retort stands

PROCEDURE

The three liquids are put into separate tubes allowing an air space of about 2 cm at the top. The pieces of glass rod can then be fixed to the open ends.

If the tubes are now inverted, the air bubbles will slowly rise through the liquid. The time each takes to rise a certain distance can be measured and compared with the molecular weights. In the MS 200 liquids, the ratio is approx. 1 : 50 : 220.

It is a good idea to have three lengths of string in the proportions of the relative chain lengths (approx. 6 : 330 : 1450).

Experiment E13.25

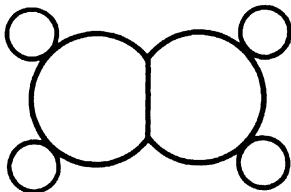
How to make models of some large structures

Spheres of expanded polystyrene are the most convenient type to use. They can be joined by 1 in lengths of pipe cleaners after piercing with a paper clip or pin. Tangential contact can be used except in obvious cases of double-bonding. Estimation of bond angles is usually adequate after one or two have been made and errors of a few degrees are easily absorbed. If time permits, it is well worth colouring the spheres and in some cases this is essential, as the same sized spheres are representing several different types of atoms. A spot of paint can be used as an alternative but is not so attractive visually.

As some of the models are large, 2 cm spheres can be used for carbon, oxygen and nitrogen, 1 cm for hydrogen and 2.5 cm for silicon. The instructions given below are merely intended as guidance and are certainly not comprehensive.

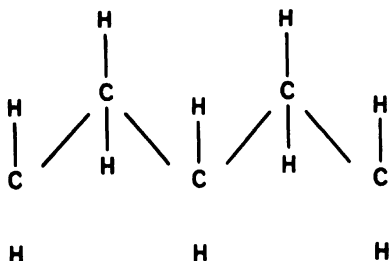
Model 1: Ethylene

Cut a small slice from each carbon atom before assembly to indicate 'double-bonding'.



Model 2: Polythene

A small part of the chain can be represented by, say, eight carbon atoms arranged in a 'straight' chain, though they can be arranged at random if desired.

**Model 3: Styrene**

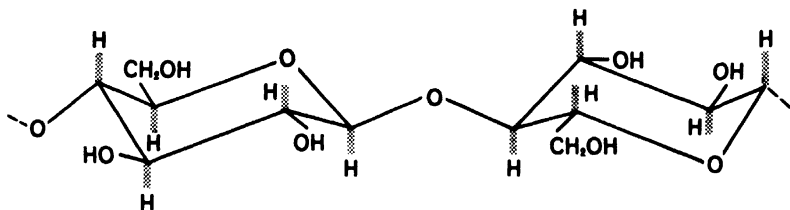
As ethylene, but one hydrogen atom is replaced by a benzene ring. The hexagon is made as in graphite but one hydrogen is added to each.

Model 4: Polystyrene

As polythene, but replace every other hydrogen by a 2 cm wide disc cut from a 5 cm sphere, which gives some idea of the effect of the large benzene rings.

Model 5: Cellulose unit

The shape can be deduced from the following diagram.

**Model 6: Nylon 66 and 610**

This can easily be built up from the formula, remembering that the 'CO' represents 'C = O'.

Model 7: Linear polymer

Take about 15–20 feet of plastic covered garden wire. Every 1 cm or so bend it at approx. 108° in random fashion. This can be done very quickly as the more haphazard the effect the better. Repeat with several other 'molecules' preferably using different colour wire and weave them in between the others so that a coiled ball is obtained.

Models 8 and 9: Minor crosslinking

There is no need to make a separate model. The crosslinks can be represented by a few paper clips joining pairs or chains. A piece of sticky tape wound round each clip will hold it in place.

To represent minor crosslinking in another form use a piece of chicken netting about 22 cm by 15 cm. Cut out most of the wires connecting the longer sides, leaving just two or three crosslinks. The horizontal 'chains' can be painted differently from the crosslinks but this is not essential.

Model 10: Extensive crosslinking

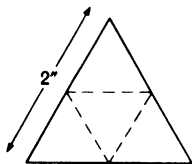
Extensive crosslinking is represented by a similar piece of netting without the middle cut away. These two models show the relative rigidities well.

Model 11: SiO_4 unit

As diamond, but replace carbons by silicons and place oxygen spheres between each pair of silicons.

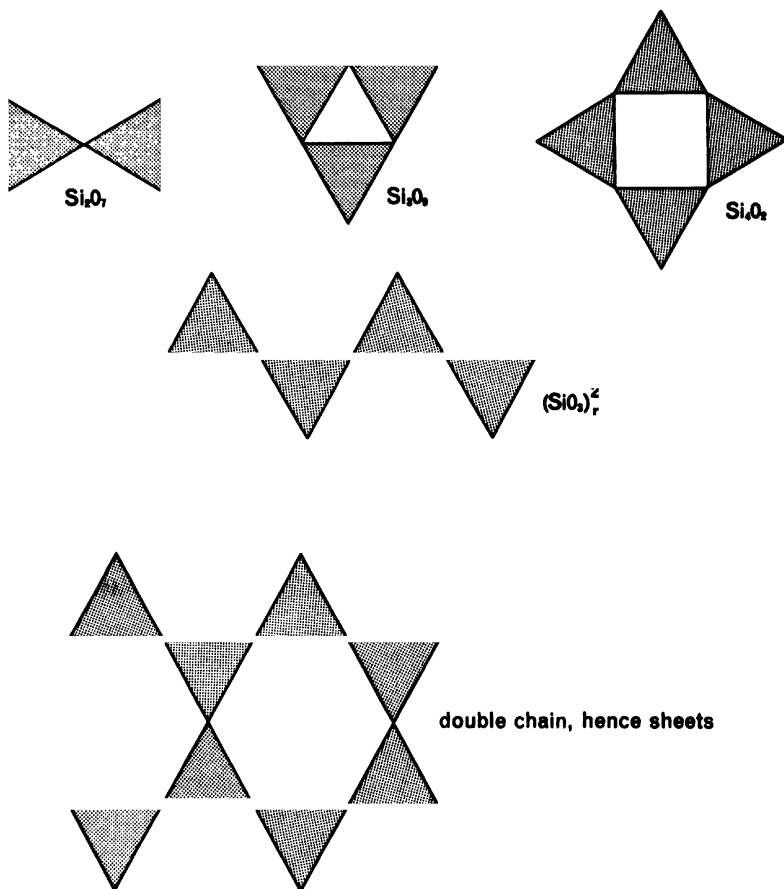
Model 12: Silicates

The above tetrahedra can be represented in bulk by cardboard tetrahedra made as follows: (a junior form will make dozens in half an hour and with great enthusiasm).

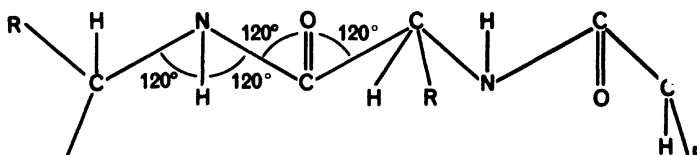


Bisect each side of triangle. Fold along dotted lines and join with adhesive tape.

Some possible arrangements are represented by the shaded triangles overleaf:

**Model 13: Peptide link**

As in the following diagram. Slice small piece off spheres for double bonds and treat all angles as about 110° except those shown. Use 4 cm spheres for R.



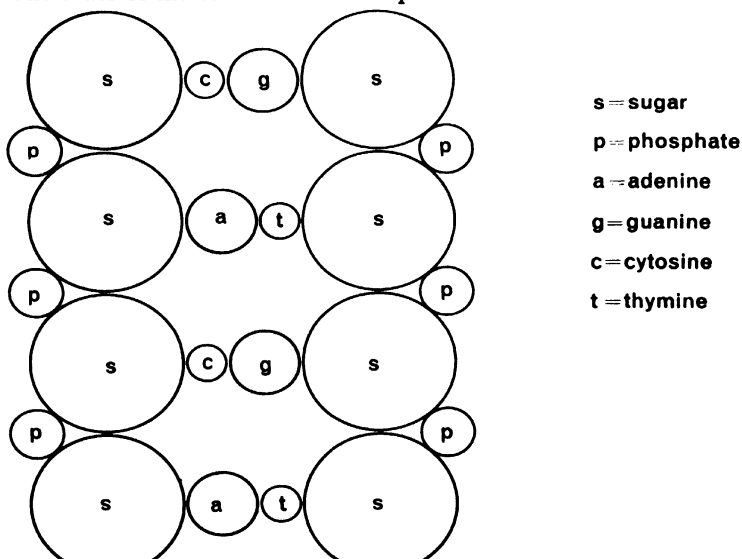
Model 14: DNA

This is a model which appears very complex at the start but as one proceeds, the pattern becomes automatic and if coloured, the result is most spectacular. It is of course only a representation and the scale is very approximate. The ribose units are represented by 2.5 cm spheres, the phosphates by 1 cm spheres and the bases by 2 cm and 1 cm spheres.

The model is started by taking two lengths of thick copper wire about 75 cm long. Twist each in the form of a helix (each with about three turns). Clamp the two wires at one end and fix a 'sugar' sphere onto each of the other ends. Join these two spheres by a large and small 'base' sphere. The bottom is now fixed and 'sugar' spheres (previously pierced) are slid onto each wire. Each pair is joined as the first pair but the large-small spheres are alternated. Continue until you reach the top of the wire. 'Phosphate' spheres are then stuck on the outside between each adjoining pair of spheres. There should be ten 'sugar' spheres to the turn. When finished the model can be placed over the rod of a retort stand and any imperfections can usually be noticed by irregularities in the pattern and can easily be corrected.

The sugar spheres are left white, the phosphates coloured blue, adenine maroon, guanine grey, cytosine yellow and thymine green but these colours are arbitrary.

The order of the bases has been simplified.



Colloids

Experiment E13.26

How to make some sols

Part 1. Gold sols

APPARATUS

The teacher will need:

Clean glass bottle

0.1 per cent solution of gold chloride

Solution of yellow phosphorus in carbon disulphide (a few drops only)

Each pupil or pair of pupils will need:

Six test-tubes, 100×16 mm, in rack

Teat pipette

Bunsen burner and asbestos square

0.1 per cent solution of gold chloride

Solutions of a variety of reducing agents, such as: phenylhydrazine hydrochloride, formaldehyde, tannic acid, hydrazine sulphate, hydroxy-ammonium chloride, tin(II) chloride

PROCEDURE

As a start, Faraday's gold sol could be made as a demonstration. Add 1 cm^3 of the gold chloride solution to 100 cm^3 of distilled water in a very clean glass bottle. Make a solution of yellow phosphorus in carbon disulphide and stir a few drops of this into the gold solution. The colour changes to deep red as the gold ions are reduced and the metal appears in particles of colloidal size. If the particles are smaller, the colour is a brownish-red, and if larger, the colours are purple, violet, or even blue.

The pupils can try the effect of various reducing agents by placing about 1 cm^3 of the gold chloride solution in a small test-tube and adding a few drops of reducing agent from a teat pipette. The effect on particle size of heat, and of dilution, should also be explored. The more concentrated the reducing agent, the smaller the particle size produced. As mentioned above, the colour gives some indication of particle size.

Part 2. Investigation of a familiar 'precipitate'

APPARATUS

Each pupil or pair of pupils will need:

Teat pipette

Test-tube, 150 × 25 mm

1 litre flask or beaker

Solid iron(III) chloride

PROCEDURE

Prepare a small quantity of a concentrated solution of iron(III) chloride by dissolving about a measure in three times its volume of water. Boil a little in a test-tube. What happens as the boiling proceeds? A reddish-brown precipitate of iron(III) hydroxide is formed as a result of hydrolysis and the boiling off of the other product, hydrogen chloride.

Now heat about 500 cm³ of distilled water nearly to boiling point in the 1 litre flask. Using a teat pipette, add the concentrated iron(III) chloride solution a few drops at a time. A beautiful cherry-red set of hydrated iron(III) oxide is produced.

Experiment E13.27

The Tyndall effect

APPARATUS

Each pupil or pair of pupils will need:

Wash bottle or side-arm test-tube

Two flasks, 250 cm³

Filter funnel and stand

Filter paper

Cellophane

Light source, such as 12-volt or mains bulb

Lens for producing convergent beam

Sources of hydrogen sulphide and sulphur dioxide

PROCEDURE

Make sure that the hydrogen sulphide is free from acid spray by passing it through water in a wash bottle. Pass the clean gas into about 100 cm³ of distilled water in one of the flasks for a minute or so. Pass sulphur dioxide into a similar quantity of water in the other flask for about a minute and then add this solution to the other, a little at a time, until there is only a faint smell of hydrogen sulphide. A yellow sol of sulphur is produced. Pass a beam of light through it and view it at right angles to the direction of the beam.

Attempt to filter the solution. Will it pass through (a) filter paper, and (b) Cellophane?

Keep the sol, protected from dust, for a few days. Note any change in colour or appearance.

Examine it periodically for Brownian motion. See the next experiment.

Experiment E13.28

Brownian motion

APPARATUS

Each pupil or pair of pupils will need:

Sulphur sol from experiment E13.27

Microscope with 4 mm objective

Microscope slide and cover slip

Horizontal beam of light

Colloidal graphite (Aquadag)

PROCEDURE

To observe Brownian motion, colloidal graphite, available commercially as Aquadag, is a suitable substance. Put a very small drop on a microscope slide and stir distilled water into it so that it is almost colourless. Place a cover slip over it, put it on the stage of the microscope, illuminate it from the side, i.e. with the light beam aimed parallel with the top of the slide, and observe it with a 4 mm objective. It may take a little practice to see the moving particles.

This technique can be used to study the 'ageing' of the particles of the sulphur sol prepared in experiment E13.27.

Experiment E13.29

Dialysis

APPARATUS

Each pupil or pair of pupils will need:

Iron(III) hydroxide sol from experiment E13.26

Square of Cellophane, side 8–10 cm

Shallow dish or beaker, 100 cm³

Laboratory solutions of sodium hydroxide and silver nitrate

Universal Indicator paper

PROCEDURE

Place the red sol in a Cellophane bag suspended in a shallow dish of distilled water. The water remains colourless, but the presence of chloride ions can be shown by removing a few drops and testing with silver nitrate solution. The water can also be tested with Universal Indicator paper. There may also be some iron ions and these could be

detected by withdrawing a few drops and testing with sodium hydroxide solution. The reversible nature of the above reaction could be discussed, in addition to the mechanism of dialysis.

The water can be changed every five minutes and the test repeated. By using the process of dialysis in this way, the sol may be largely freed from electrolyte.

Experiment E13.30

Electrophoresis

APPARATUS

Each pupil or pair of pupils will need:

U-tube

Platinum or copper electrodes

Teat pipette

Distilled water, preferably de-ionized

Source of 24–30 volt d.c.

Sols of clay, iron(III) hydroxide or gold

PROCEDURE

Make sure that the U-tube has been thoroughly cleaned. Clamp it vertically and almost half-fill it with the sol. With a very clean teat pipette, slowly run the de-ionized water down the side of each limb of the U-tube to a depth of about 3 cm, so that it forms a separate layer above the colloid. Immerse the electrodes in the distilled water near the top. Mark the level of the colloid-water junctions with a small strip of gummed paper. Apply the d.c. voltage and see whether there is any movement of either boundary. Continue the observations for at least half an hour. The rates of movement of the boundary can be measured, perhaps under more than one voltage. The velocity should be expressed in cm/sec for a potential gradient of 1 volt/cm.

The movement observed in this experiment is called ‘electrophoresis’ and is good evidence for the theory that the particles of sol are charged relative to the water.

Experiment E13.31

The colloidal nature of clay

Crumble a piece of clay into a beaker of water and let it settle overnight.

Decant the colloidal solution from the sediment through filter paper and use some of the filtrate for the following experiments.

1. Shine a beam of light through it. Is there a Tyndall effect?
2. Try the effect of adding a little lime water. Does the colloidal solution precipitate out?
3. Use some of the colloidal solution in the experiment on electrophoresis, experiment E13.30.

Experiment E13.32

How to make some gels

Experiment a. Preparation of a gel of gelatin

APPARATUS

Supply of gelatin

Beaker, 250 cm³

PROCEDURE

Dissolve about 2 g of gelatin in about 100 cm³ of distilled water by warming the water nearly to boiling and stirring the gelatin in until it has dissolved. The solution should set to a jelly when cold.

Experiment b. Preparation of silica gel

APPARATUS

Burette

Beaker, 100 cm³

1 litre of sodium silicate solution made by adding sodium silicate to distilled water until the specific gravity is about 1.06 (this stock solution will be useful for other experiments with silica gel)

1M hydrochloric acid

Phenolphthalein solution

PROCEDURE

Put the acid in the burette and about 20 cm³ of the sodium silicate solution in the beaker. Add a few drops of indicator. Run in the acid, stirring occasionally, until the pink colour of the indicator just disappears. The silica formed in the reaction soon sets to a gel.

Experiment c. The properties of gels

A number of tests can be carried out on the gels designed to answer the following questions:

1. Are they elastic solids, i.e. if slightly deformed, do they return to their original shape?
2. Do they 'flow' like liquids, i.e. if an irregular piece is left in a test-tube does it eventually fill the bottom of the tube?
3. What happens when they are warmed and cooled again?

4. What happens when they are diluted with increasing amounts of water, warmed and cooled again?
5. What happens when a narrow beam of light is shone through the gel?

Discussion of the results of these tests leads to the conclusion that gels behave in some ways like solids and in others like liquids. This justifies the use of a new term – the colloidal state. The fact that a beam of light is visible when viewed at right angles to the beam, suggests the presence of large particles, larger than those in true solutions. If the solutions are of suitable concentrations, gels are formed. One model for gels consists of a loose framework of the particles of the dispersed substance within the solvent. If the concentration is not high enough, the framework cannot form. If the gel is heated, thermal energy transferred to the particles enables them to break the weak bonds holding them together and the framework is disrupted – the gel ‘melts’. It may reform on cooling.

Pupils may be familiar with the silica gel used as a desiccant and sometimes kept in balance cases. How is this related to the silica gel just prepared? Experiments can be done on the latter in which the solvent is removed by warming. Provided the temperature does not rise too high (this could be a matter for investigation) the residue will re-absorb water. This behaviour favours the ‘framework’ model. This model suggests that large molecules, like gelatin, starch etc., form gels more easily than smaller ones. This is so, yet gel formation is not restricted to large molecules, as subsequent experiments will show. In the gel described below it seems as if ionic structure can be ‘opened out’ to form a gel, the solvent presumably penetrating the lattice.

Experiment d. Preparation of a gel of sodium chloride

APPARATUS

Small bottle with glass stopper

Sodium salicylate

Thionyl chloride

PROCEDURE

Thoroughly dry the bottle and 15 g of sodium salicylate in a hot-air oven. Put the sodium salicylate in the bottle, and transfer it to a fume cupboard. Here add, a little at a time, 20 g of thionyl chloride, shaking the bottle after each addition. Sulphur dioxide is evolved and the reaction takes a few days to complete. A very beautiful greenish-yellow gel, coloured blue and lilac in reflected light, is formed.

Experiment E13.33**Emulsions**

In this experiment a comparison is made of the effectiveness of a number of oils and emulsifying agents in forming an emulsion.

APPARATUS

Test-tubes

Soap solution

Some detergents

Benzene

Some oils, such as medicinal paraffin, linseed, cooking, and engine oil

PROCEDURE

About 1 cm³ of the oil is vigorously shaken with about 1 cm³ of water and allowed to stand. The times taken for the various oils to form a separate layer are measured. Each group of pupils could use a different oil.

One or two drops of soap solution are added to each test-tube and the whole is vigorously shaken again. The times taken to form separate layers can be compared with those needed without the emulsifying agent. The experiment can then be repeated using a few drops of a detergent instead of soap.

Part II

How fast do the changes take place, and can their speeds be altered?

Chapter 14

Rates of reaction and catalysis

Introduction

Once the 'cataloguing' of chemical changes has been developed, some attempt can be made to understand the principles which govern these changes. These may be arrived at by answering the questions:

1. How fast does the reaction go?
2. How far does the reaction go?
3. How does energy interact with matter?

In this chapter experiments are described which enable an answer to be found to the first of these questions. The existence of different rates of reactions is something with which the pupils will be familiar from the start of their chemical investigations; different rates of thermal decomposition, for example, provide an introduction to the subject.

The experiments are grouped according to their objects, which are to find out what factors govern the rate of a reaction. These are indicated on the list of contents.

References

The teaching of the subject of rates of reaction is discussed in the *Handbook for Teachers*, Part VI, Chapters 20 and 21. One way of putting those ideas into practice is described in the Sample Scheme, Part I, *Basic Course*, Topic 18.

List of experiments

The existence of rates

- E14.1 Reactions happen at different rates

Dependence of rate on surface area of contact

- E14.2 How does the fineness of division of reactants affect the rate of reaction?

Dependence of rate on relative proportions of reactants

E.14.3 Study of the Bunsen burner flame

E14.4 A town gas-air explosion

Dependence of rate on concentration

E14.5 How does the burning of elements in oxygen differ from their burning in air?

E14.6 What is the effect of concentration on the reaction between hydrochloric acid and sodium thiosulphate?

E14.7 The iodine clock

E14.8 The reaction between hydrochloric acid and marble

Dependence of rate on temperature

E14.9 What is the effect of temperature on the reaction between hydrochloric acid and sodium thiosulphate?

Dependence of rate on voltage

E14.10 How does the applied voltage affect the rate of electrolysis?

Dependence of rate on catalysts

E14.11 The reaction between zinc and sulphuric acid catalysed by copper

E14.12 The thermal decomposition of potassium chlorate catalysed by copper(II) oxide

E14.13 The reaction between hydrogen and oxygen catalysed by platinum

E14.14 The decomposition of hydrogen peroxide catalysed by manganese dioxide

E14.15 The decomposition of nitric acid catalysed by glass

E14.16 Water as a catalyst

E14.17 An illustration of the intermediate compound theory

The existence of rates

Experiment E14.1

Reactions happen at different rates

Several experiments which lead to the idea that different reactions proceed at a different rate have been given in earlier chapters. Suitable examples to introduce this concept include:

Experiment E1.1	To find out what happens when substances are heated
Experiment E3.8	What happens when elements are burnt in oxygen?
Experiment E7.7	What happens when metals are placed in acids?

Dependence of rate on surface area of contact

Experiment E14.2

How does the fineness of division of reactants affect the rate of reaction?

Part 1. The reaction between magnesium and oxygen

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Tongs

Matches

Magnesium wire, ribbon, and powder

PROCEDURE

Before any magnesium is burnt it is essential that the following precautions are carefully read and observed.

1. Magnesium burns with an intensely bright white flame and looking directly at this flame for long can damage the eyes. Do not therefore look directly at the flame except through a piece of coloured glass or sun spectacles.

2. Care must be taken to protect the bench surface, as bits of burning metal may fall off from the ribbon. Always work over a bench which is protected by an asbestos square. If a piece of burning magnesium does fall onto the asbestos let it burn itself out; do **not** try to put it out by pouring on water.

3. Neither the ribbon nor the wire should be held in the fingers while they are being heated; a pair of tongs must be used.

Take about a 5 cm length of the magnesium wire and holding one end with the tongs place the other in a Bunsen flame. Repeat the experiment with a 5 cm piece of magnesium ribbon. Which is more easily lit? Can you explain the difference?

Take another 5 cm piece of ribbon and with one end held in the tongs try to light the other end with an ordinary match, a process which will be found difficult if at all possible. Now take another piece of ribbon and make, with a pair of scissors, an incision in one end of the ribbon about half an inch long running down the middle of the ribbon. Try lighting this cut end with a match. Why does it light more easily?

Take a pinch of magnesium powder on a spatula or pen-knife blade and holding it just above a Bunsen flame gently shake the powder off. Does it burn easily?

Try to relate the ease with which magnesium burns with the particle size of the specimen burnt.

Note the appearance of the product of the combustion of magnesium. What do you think it might be?

Part 2. The reaction between hydrochloric acid and calcium carbonate

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 150 × 16 mm

Powdered chalk

Marble chips

Dilute hydrochloric acid

PROCEDURE

Marble and powdered chalk both consist of the same chemical substance, calcium carbonate, but differ in their particle size.

Take about 0.5 cm depth of powdered chalk in a 150 × 16 mm test-tube and shake it up with a 3 cm depth of water. Pour about a 2 cm depth of dilute hydrochloric acid into a second test-tube. Hold both tubes over the sink and, keeping your head well clear, pour the dilute acid onto the suspension of powdered chalk. Note the vigorous reaction, and rinse off any acid which may have overflowed onto your hands.

Repeat the experiment using a lump of marble in place of the powdered chalk, but adding the water and acid as before.

Does the rate of the reaction depend on the particle size?

Dependence of rate on relative proportions of reactants

Experiment E14.3

Study of the Bunsen burner flame

APPARATUS

Each pupil or pair of pupils will need:

Bunsen burner and asbestos square

Tongs

Piece of fine iron wire

Piece of white porcelain, e.g. a crucible lid

Copper wire gauze

PROCEDURE

Light a Bunsen burner, keeping its air-hole closed. Notice the colour of the flame and then, with the aid of tongs, hold a piece of iron wire in the flame. Does it glow red-hot or not? Put the wire down on the asbestos square and again using the tongs, wave a piece of white porcelain through the upper part of the flame. Examine it carefully after a few minutes.

Next move the collar of the Bunsen burner around so that the air-hole is open, but not so much that the flame is roaring. Notice the colour of the flame and repeat the experiments with the piece of iron wire, and the piece of porcelain.

From these experiments and observations, answer the following questions:

1. Which flame do you think is the hotter? What evidence can you bring to support your view?
2. The two elements which occur most abundantly in the gases which make up town gas are hydrogen and carbon. From your observation of the white porcelain in each case, what do you conclude to be the main difference in the chemical reactions taking place in each flame?

Now, with the air-hole wide open, slowly turn the gas supply down until there is a 'strike-back'. This occurs when the gas burns at the jet at the

base of the burner. Why has this happened? (The products of combustion when a Bunsen burner has struck back are poisonous and therefore a burner should not be left alight in this condition for very long. To cure the strike-back turn the gas supply full on and then strike the rubber gas-supply tube a sharp blow with your hand. If that does not work, turn the burner off and relight it again with the air-hole closed.)

Finally, hold a piece of copper wire gauze with a pair of tongs and place it about 3 cm above the top of the Bunsen burner in a horizontal position. Turn on the gas supply with the air-hole closed and then apply a lighted match to the top of the gauze. What happens? Now light the Bunsen in the normal way and lower the gauze still in a horizontal position onto the flame. How does this experiment compare with the previous one? After a little while the flame will be seen to pass through the gauze. Why does this occur?

One can easily underestimate what can be brought out about factors which influence the rate of a reaction by this series of simple experiments. The following points can be mentioned in a class discussion after the experiments have been done.

1. The reaction between town gas and air needs heat to start it, but once started it yields heat which ensures that it will continue.
2. The products of the reaction and also the amount of heat evolved depend on the proportion of the reactants. The use of the iron wire provides a rough estimate of the relative temperatures of the two flames.
3. Normally the combustion proceeds quietly, but by varying the composition of the burning gas an explosive mixture can be formed giving rise to the 'strike back'.
4. If heat is removed from the gas-air mixture by the copper wire gauze, no reaction occurs.

Experiment E14.4

A town gas-air explosion

This experiment should be carried out by the teacher

APPARATUS

Rubber tubing

Tripod

A tin can, volume 250–500 cm³, with a firmly-fitting press-on lid, having a hole of diameter about 0.5 cm in the lid, and one of about 2 cm in the base.

PROCEDURE

Fill the tin with town gas by placing a tube leading from the gas supply into the hole in the base and passing gas in while covering the smaller hole with a finger. This filling operation, which takes about thirty seconds, should be performed in a fume cupboard.

Place the tin on a tripod on the demonstration bench, and apply a light to the hole in the lid. This will cause the escaping gas to burn with a characteristic yellow flame. This flame becomes smaller and more blue in colour as the gas is burnt away and air is drawn in through the larger hole to replace it. Finally, after a couple of minutes, there is created an explosive gas-air mixture inside the tin and there is a loud detonation causing the tin lid to be ejected into the air, probably hitting the ceiling.

Dependence of rate on concentration**Experiment E14.5**

How does the burning of elements in oxygen differ from their burning in air?

This experiment can be done using the methods of experiments E3.1 and E3.8, and comparing the results.

It will be seen that substances burn more brilliantly in pure oxygen than they do in oxygen diluted with four times its volume of nitrogen.

Experiment E14.6

What is the effect of concentration on the reaction between hydrochloric acid and sodium thiosulphate?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 18.2)

Conical flask, 100 cm³

Measuring cylinder, 25 cm³

Beaker, 100 cm³

Stop clock or watch with seconds hand

Sheet of white paper

2M hydrochloric acid

Sodium thiosulphate solution containing 40 g/l; about 200 cm³

PROCEDURE

The reaction to be examined is that between sodium thiosulphate solution and hydrochloric acid.

Place 50 cm³ of the thiosulphate solution in the conical flask. Add 5 cm³ of the 2M hydrochloric acid to the solution in the flask, at the same time starting the stop clock. Swirl the flask once or twice and then place it over a piece of paper with a cross marked on it. Look down vertically onto the cross and take the time when the cross disappears.

Repeat the experiment using 40, 30, 20, and 10 cm³ of the thiosulphate solution made up each time to 50 cm³ with water. If time allows, the remaining solution could be used to take an intermediate reading, using say 15 cm³.

Plot the following graphs:

1. Concentration of thiosulphate solution against time. The concentration may be measured as the volume of original solution taken or as the weight of thiosulphate present in 50 cm³ of diluted solution.
2. Concentration of thiosulphate solution against 1/time, since the reciprocal of time is a measure of the rate of the reaction.

Experiment E14.7

The iodine clock

The dependence of reaction rate on concentration can be illustrated very well using the 'iodine clock' experiment. Details of this can be found in Fowles, *Lecture Experiments in Chemistry*. 6th edn., (Bell).

Experiment E14.8

The reaction between hydrochloric acid and marble

This experiment should be done by the teacher.

APPARATUS

Three conical flasks, 100 cm³

Stop clock

Measuring cylinder, 100 cm³

Direct-reading balance

Graph paper

Cotton wool

Marble chips, medium size

2M hydrochloric acid

PROCEDURE

The course of the reaction is followed by observing the weight change as the carbon dioxide is evolved. For this a direct-reading balance is preferable, and the experiment will therefore probably have to be

demonstrated by the teacher. The pupils may help with weighing, timing, and recording results.

Put 40 cm³ of 2M hydrochloric acid in a 100 cm³ conical flask; add 20 g of marble chips, and place a loose plug of cotton wool in the neck of the flask to prevent any acid spray from damaging the balance. Weigh the whole apparatus, and at the moment at which the weight is determined start the stop clock.

Note the weight every one or every half-minute. (Alternatively note the time taken to lose successive weights of 0.10 g.) Continue for ten to fifteen minutes.

Plot a graph showing the loss of weight (that is, the extent of the reaction) against time. The slope of the graph gives the rate of the reaction at the time represented by that point. Find out what is the slope of the graph in the middle section of the curve.

Now repeat the experiment with:

1. 20 g of the same size of marble chips and 40 cm³ of 1M hydrochloric acid, made by diluting the 2M acid with an equal volume of water.
2. 20 g of marble crushed into pieces of much smaller size.

Compare the slopes of the three graphs in their central sections.

The experiments could be extended by using another concentration of acid, e.g. 0.5M.

Dependence of rate on temperature

Experiment E14.9

What is the effect of temperature on the reaction between hydrochloric acid and sodium thiosulphate?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 18.3)

Conical flask, 100 cm³

Measuring cylinder, 25 cm³

Beaker, 100 cm³

Stop clock or watch with seconds hand

Thermometer, -10° to +110°C

Bunsen burner, tripod, gauze, and asbestos square

Sheet of white paper

2M hydrochloric acid

Sodium thiosulphate solution containing 40 g/l; about 60 cm³

PROCEDURE

Measure out 10 cm³ of the thiosulphate solution into the conical flask, add 40 cm³ of water and warm the solution gently, if necessary, until the temperature is about 20°C. Now add 5 cm³ of the 2M hydrochloric acid, note the temperature of the mixture, start the stop clock, and swirl the mixture. Note the time of disappearance of the mark on the paper as in experiment E14.6.

Repeat the experiment four more times, heating the thiosulphate solution before the addition of hydrochloric acid to a little over 30°, 40°, 50°, and 60°C so that the reaction can be started as near to these temperatures as possible.

Plot the following graphs:

1. The time taken for the mark to disappear against the temperature.
2. The reciprocal of the time taken for the mark to disappear against the initial temperature.

Dependence of rate on voltage**Experiment E14.10**

How does the applied voltage affect the rate of electrolysis?

APPARATUS

Each pupil or pair of pupils will need:

Water voltameter of the Hofmann type

Source of 20 volts d.c.

Rheostat

Voltmeter, 0–20 volts

Stop watch

Five pieces of connecting wire

Dilute sulphuric acid

Non-availability of apparatus may well require this experiment to be performed by the teacher.

PROCEDURE

Set up the voltameter in the usual fashion, filling it with dilute sulphuric acid. Should there be no suitable apparatus available, one can be improvised using a large beaker with two graduated tubes inverted into it. The electrodes must be inert, preferably of platinum.

The rheostat is placed in series and the voltmeter (20 volt) in parallel with the voltmeter.

Start the apparatus working making sure that a suitable setting of the rheostat is chosen so that the current flowing through the apparatus is not excessive. (If the teacher is doing the experiment he may, for his own convenience, like to put an ammeter in series with the apparatus, but it should not be in too prominent a position as its readings are not required by the class.)

Note the voltmeter reading and measure the time taken to collect 1 cm^3 of hydrogen gas at the cathode. Alter the rheostat setting and note the new potential difference across the apparatus and the time taken for a further 1 cm^3 of hydrogen to be evolved. Repeat with several different rheostat settings and plot a graph of rate of evolution (cm^3 per minute) against potential difference.

In general it is more important to measure the current than the potential in a voltmeter experiment. In this particular experiment we are trying to show how a reaction rate is influenced by the applied conditions and strictly speaking it is the potential difference maintained across the apparatus which is the variable applied condition, and not the current flowing through, although with the resistance of the voltmeter being approximately constant the two factors are proportional to each other.

It is wiser to measure the gas evolved at the cathode rather than at the anode both because (a) the rate of evolution is faster at the cathode and (b) hydrogen is a good deal less soluble in water than oxygen.

Dependence of rate on catalysts

Experiment E14.11

The reaction between zinc and sulphuric acid catalysed by copper

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, $100 \times 16 \text{ mm}$

Zinc, granulated

Copper turnings

Dilute sulphuric acid

Copper(II) sulphate solution

PROCEDURE

Place a few pieces of granulated zinc in a test-tube and cover them with about 5 cm depth of dilute sulphuric acid. Note carefully the rate

at which hydrogen gas bubbles are formed on the zinc. The reaction proceeds according to this equation: $\text{Zn} + \text{H}_2\text{SO}_4 \rightarrow \text{ZnSO}_4 + \text{H}_2$.

Now add a few fine copper turnings so that they lie in the tube in contact with the zinc. How is the rate of production of hydrogen affected by the addition of the copper?

Repeat the experiment in another tube with a fresh lot of acid and zinc. Now add about 1 cm³ of the copper(II) sulphate solution to the mixture. Shake and note the change in the rate of gas evolution. What happens to the blue colour of the copper(II) sulphate? Pour away the acid and carefully examine the pieces of granulated zinc. What do you think might have happened to them and to the copper(II) sulphate solution?

By a *catalyst* is meant a substance which affects the rate of a reaction, but is itself chemically unchanged by the reaction. Do you think that the copper(II) sulphate solution can be called a catalyst under this definition?

Experiment E14.12

The thermal decomposition of potassium chlorate catalysed by copper(II) oxide

This experiment should be done by the teacher.

APPARATUS

Two hard-glass test-tubes, 125 × 16 mm

Two stands and clamps

Bunsen burner and asbestos square

Potassium chlorate

Copper(II) oxide

Splint

PROCEDURE

Take about 1 cm depth of potassium chlorate in the two test-tubes. To one add a little dry copper oxide and mix it up well. Clamp the test-tubes side by side and warm them both equally with a medium-sized, non-roaring Bunsen flame. Hold a glowing splint at the mouth of the tubes. The splint held in the mouth of the tube containing the catalyst will ignite well before the other.

Experiment E14.13

The reaction between hydrogen and oxygen catalysed by platinum

This experiment **must** be done by the teacher

APPARATUS

Plastic safety screen

Two gas jars and covers

Tongs

Bunsen burner and asbestos square

Cylinder of hydrogen, with delivery tube (or other source of the gas)

Some platinized asbestos

PROCEDURE

Before the lesson, heat a tuft of platinized asbestos in a Bunsen flame for a few seconds to ensure that it is dry, and return it to the bottle.

Fill a gas jar with hydrogen and close it with a cover. Hold the edge of the tuft of platinized asbestos with tongs; remove the gas jar cover, and hold the tuft over the mouth. As the hydrogen rises out of the jar and mixes with the air the tuft glows red and ignites the remaining hydrogen, resulting in the usual gentle explosion.

Experiment E14.14

The decomposition of hydrogen peroxide catalysed
by manganese dioxide

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 18.4c)

Conical flask, 100 cm³ wide necked, with bung and delivery tube

Measuring cylinder, 25 cm³

Stop clock or watch with seconds hand

Graph paper

Thermometer, -10° to $+110^{\circ}\text{C}$

Spatula

Bunsen burner and asbestos square

Tripod and gauze

Teat pipette with 1 cm³ graduation

Either *a*

Syringe, 100 cm³ glass or 50 cm³ plastic

Syringe holder and stand

Or *b*

Burette, 50 cm³

Stand and clamp

Trough

Delivery tube

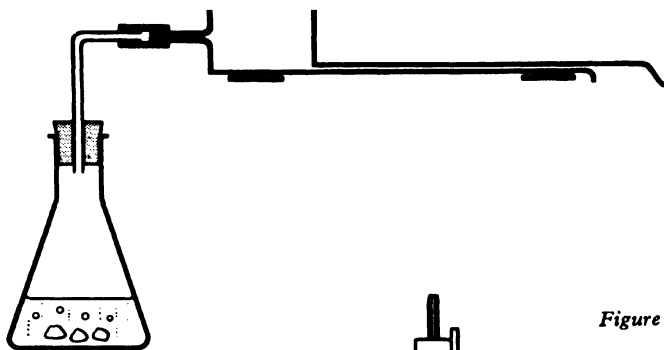
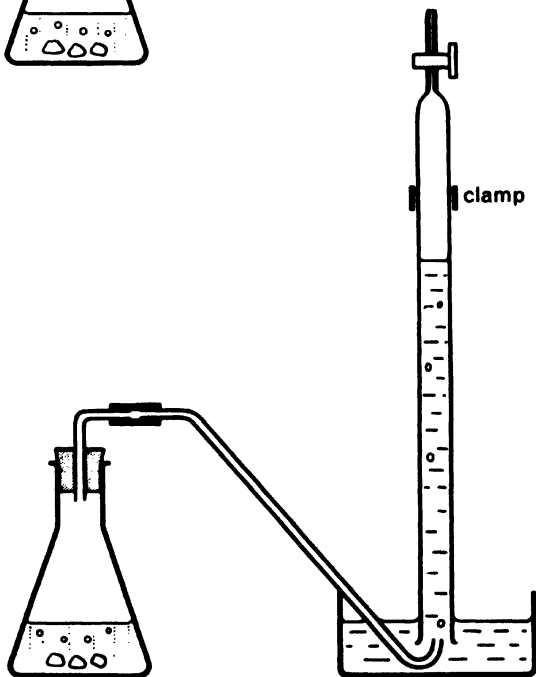
20-volume hydrogen peroxide

Manganese dioxide

PROCEDURE

As a start tell the pupils to add 2 cm³ of the solution of hydrogen peroxide to 48 cm³ of water in the flask. About half a spatula measure of manganese dioxide is then put in and the bung immediately inserted. A stop clock is started and the volume of oxygen that collects is noted at regular intervals. A graph should be plotted showing the volume of oxygen produced against the time. The slope of this graph is, of course, a measure of the rate of reaction.

syringe clamp

*Figure 14.1a**Figure 14.1b*

The pupils can then investigate the effects on the rate of reaction of changing, in turn, the concentration of the peroxide, the quantity of manganese dioxide, and the temperature.

After each experiment, a graph should be plotted.

If time is available the effect of biological catalysts (enzymes) in the blood and in potatoes on the decomposition of hydrogen peroxide may also be investigated.

Experiment E14.15

The decomposition of nitric acid catalysed by glass

This experiment should be done by the teacher.

APPARATUS

Test-tube, 125 × 16 mm, fitted with a bung carrying a delivery tube. The bung can be of cork (which will be destroyed in one experiment) or of asbestos

Bunsen burner and asbestos square

Stand and clamp

Trough

Test-tubes, 150 × 25 mm, to act as gas jars

Broken glass in small pieces, or glass wool

98 per cent nitric acid

PROCEDURE

Place 1–2 cm³ of 98 per cent nitric acid in the 125 × 16 mm test-tube and closely pack the space above with broken glass, or loosely fix a plug of glass wool several cm long in the space. Close the tube with the bung carrying the delivery tube, and clamp it so that the delivery tube is beneath the surface of some water in a trough. Heat the glass or glass wool with a small flame which is just non-luminous, occasionally flicking for a moment at the acid. Collect the misty bubbles of oxygen and test. Brown fumes are more noticeable after the acid vapour has passed over some divided glass; red heat is not required. If ordinary laboratory concentrated acid (68 per cent) is used, stronger heating may be necessary. Sucking-back can be troublesome, and a careful watch must be kept.

The acid can be distilled with much less decomposition in an all-glass apparatus, showing the need for a finely-divided catalyst.

The experiment is based on a suggestion of G. Fowles, *School Science Review*, 147, 302.

Experiment E14.16**Water as a catalyst**

This experiment should be done by the teacher

*Part 1. The reaction between sulphur dioxide and hydrogen sulphide***APPARATUS**

Sulphur dioxide cylinder

Hydrogen sulphide generator

Wash-bottle containing concentrated sulphuric acid

Calcium chloride tube, filled

Two gas jars

Rubber connection tubing

PROCEDURE

Fill one gas jar with sulphur dioxide which has been dried by passing the gas through concentrated sulphuric acid, and then fill another gas jar with hydrogen sulphide which has been dried by passing it through a calcium chloride tube. Invert the jar containing the sulphur dioxide over the hydrogen sulphide jar and remove both lids. No observable reaction takes place. If water (5-10 drops) is added to the lower jar and the upper one is rapidly replaced, it will be noticed that a deposit of sulphur is rapidly produced on the sides of the jars.

A similar result can be obtained by using ammonia and hydrogen chloride as the two reacting gases.

*Part 2. The reaction between aluminium and iodine***APPARATUS**

Pestle and mortar

Teat pipette

Tin lid

Asbestos square

Aluminium powder

Iodine

PROCEDURE

Finely grind about 5 g of iodine crystals in the mortar and then carefully mix with about the same amount of aluminium powder. Place this mixture as a mound on the tin lid, on an asbestos square, and carefully add one drop of water from the teat pipette. There is an immediate and vigorous reaction. and iodine fumes are evolved.

Caution: Iodine fumes are toxic, and this experiment should be done in a fume cupboard.

Experiment E14.17**An illustration of the intermediate compound theory**

Although the intermediate compound theory of catalysis is widely employed to explain these phenomena, it proves to be difficult to give any practical evidence in support of this theory.

The experiment described here involves the oxidation of potassium sodium tartrate (Rochelle salt) with hydrogen peroxide, using a cobalt salt as a catalyst. A green intermediate compound is formed, but finally decomposes to produce the original pink cobalt salt.

APPARATUS

Each pupil or pair of pupils will need:

Beaker, 250 cm³

Tripod and gauze

Bunsen burner and asbestos square

Measuring cylinder, 25 cm³

Glass stirring rod

Potassium sodium tartrate

20-volume hydrogen peroxide solution

Cobalt(II) chloride

PROCEDURE

Place in the beaker a solution of potassium sodium tartrate containing about 3 g of the salt in about 50 cm³ of distilled water. Warm this solution to about 70°C and then add 20 cm³ of 20-volume hydrogen peroxide solution. There will be little visible reaction at this stage.

Now add a little of the cobalt salt so that the solution has a definite pink colour; about 0.1 to 0.2 g of the salt will suffice. After an induction period of a few seconds the pink colour fades and is replaced by a deep green and a mixture of oxygen and carbon dioxide is rapidly evolved. Within a few minutes the reaction ceases and the original pink colour is restored.

Variations in the concentration of the reactants and or the temperature will alter both the induction period and the duration of the whole experiment. Cooling the reaction mixture while it is at the green stage will have the effect of 'freezing the equilibrium', and the intermediate compound can be examined.

Part III

How far do the changes go,
and can the final proportions
be adjusted?

Chapter 15

Equilibria

Introduction

This chapter contains experiments in which reversible reactions are investigated, and the conditions which affect the final proportions of materials are discovered.

References

A full discussion of the subject of equilibrium is given in the *Handbook for Teachers*, Part VI, 'How fast, and how far?' One way of presenting elementary ideas on the subject is outlined in the Sample Scheme, Part 1, *Basic Course*, Stage II, at Topic 19: 'How far? The idea of dynamic equilibrium.'

List of experiments

- E15.1 Some examples of reactions that 'go both ways'
- E15.2 How does iodine distribute itself between potassium iodide solution and chloroform?
- E15.3 The hydrolysis of bismuth(III) chloride
- E15.4 The reaction between silver nitrate and iron(II) sulphate
- E15.5 The reversible change from tetra-aquocopper(II) ions to tetra-amminecopper(II) ions
- E15.6 The thermal dissociation of dinitrogen tetroxide
- E15.7 The reaction between iron(III) ions and thiocyanate ions
- E15.8 Investigating the equilibrium between a solute and its saturated solution using a radioactive tracer
- E15.9 Investigating the equilibrium between rhombic and monoclinic sulphur

Experiment E15.1

Some examples of reactions that 'go both ways'

A. Pupils' experiment**APPARATUS**

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 19.1a)

Beaker, 100 cm³

White tile or sheet of white paper

Three Petri dishes

Two teat pipettes

Bromine water

2M sulphuric acid

2M sodium hydroxide solution

0.1M potassium chromate solution

Litmus solution

Boiled distilled water (pH 7)

Bromine water may be prepared in front of the class by shaking up 2 or 3 cm³ of bromine with about 200 cm³ of distilled water in a stoppered bottle.

PROCEDURE

1. Put 10 cm³ of bromine water in a 100 cm³ beaker and stand it on a white tile or sheet of white paper. By means of a teat pipette, add alkali and acid alternately to the bromine water and see it go colourless with alkali and the colour return with the acid.

2. Repeat experiment (1) with a solution of potassium chromate. In both cases look for an intermediate stage.

3. Place a little dilute acid in one Petri dish, a little sodium hydroxide solution in the second, and distilled water in the third. Use a teat pipette to add 1 cm³ of indicator to each of the first two dishes and 2 cm³ to the third dish. If the first two dishes are placed one on top of the other over a piece of white paper and viewed from above, the colour should be that of the water in the third dish. This will illustrate the fact that the neutral colour is an equal mixture of the colours of the indicator in acid or alkaline solution.

B. This experiment should be done by the teacher.

APPARATUS

U-tube

Cork or bung to fit U-tube, carrying a right-angle delivery tube

Apparatus to produce chlorine and connection tubing to the U-tube

Access to fume cupboard

Iodine crystals

PROCEDURE

Place a few small crystals of iodine in the bottom of the U-tube and then pass chlorine through it. The experiment should be performed in a fume cupboard. Tell the pupils to watch carefully. They will see the iodine turn into a brown liquid (iodine monochloride) with a brown vapour above it. The bottom of the U tube gets hot. On passing more chlorine through the tube yellow crystals (iodine trichloride) appear on the walls of the U-tube. Now detach the chlorine supply and remove the U-tube from the fume cupboard. Tip the U-tube slowly until it is almost upside down and then turn it upright again. The yellow crystals disappear and the brown vapour can be seen again. Pass more chlorine through the U-tube and the yellow crystals reappear. The experiment may be repeated several times.

Experiment E15.2

How does iodine distribute itself between potassium iodide solution and chloroform?

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 19.2a)

Two test-tubes, 100 × 16 mm

Teat pipette

Spatula

Chloroform

Approximately 1M potassium iodide solution

Iodine crystals

PROCEDURE

Take two very small crystals of iodine of the same size. Pour 2 cm depth of chloroform into one test-tube and a similar quantity of potassium iodide solution into the other. Add one crystal of iodine to each test-tube and shake the test-tubes until the iodine dissolves. The solution of iodine in chloroform will be purple and of iodine in potassium iodide will be brown. (There is no need to explain that the iodine in potassium iodide is largely present as I_3^- (aq) ions. As far as the pupils are concerned it has simply dissolved.) Now add an equal volume of potassium iodide to the chloroform solution and of chloroform to the potassium iodide solution. The potassium iodide will become slightly brown and

the chloroform slightly purple. Shake both tubes gently. In each case the added solvents will darken. Now shake both tubes vigorously. The density of colour of each solvent in both tubes will be seen to be the same; equilibrium has been reached. The equilibrium can be disturbed again by removing the top layer (potassium iodide) from one test-tube with a teat pipette and adding fresh potassium iodide solution. On shaking some of the iodine is transferred from the chloroform to the potassium iodide solution. If this procedure is repeated two or three times, most of the iodine will be transferred from the chloroform.

Experiment E15.3

The hydrolysis of bismuth(III) chloride

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 19.2b)

One test-tube, 100×16 mm

Four test-tubes, 150×25 mm, in a rack

Glass rod

Spatula

Two teat pipettes

Bismuth(III) chloride

Concentrated hydrochloric acid

PROCEDURE

Place a spatula measure of bismuth(III) chloride in a 100×16 mm test-tube and dissolve it in about 1 cm^3 of concentrated hydrochloric acid. Pour a little of the solution into a 150×25 mm test-tube two-thirds full of water. A white precipitate of bismuth oxychloride will appear. To study the equilibrium more carefully fill the other three test-tubes two-thirds full of water and add five drops of concentrated hydrochloric acid to the first test-tube, ten to the second, and fifteen to the third, and stir. Now add five drops of the bismuth(III) chloride solution to each test-tube and note the different densities of the precipitate of bismuth oxychloride. Note too that the precipitates form progressively more slowly.

Experiment E15.4

The reaction between silver nitrate and iron(II) sulphate

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 19.2c)

Two test-tubes

0.1M solution of silver nitrate

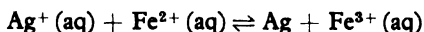
0.1M solution of iron(II) sulphate

Freshly prepared potassium hexacyanoferrate(III) (ferricyanide) solution

Freshly prepared potassium thiocyanate solution

PROCEDURE

Investigate the reaction between silver nitrate and iron(II) sulphate by mixing equal volumes of the two solutions together and examining the results. Silver is deposited and the iron(II) ions are converted into iron(III) ions according to the equation:



The reaction may be reversed by decanting off the solution from the deposited silver and adding an excess of iron(III) sulphate solution. The silver will dissolve.

In both of these reactions the presence of iron(II) and iron(III) ions should be confirmed by the following tests.

Iron(II) ions give a deep blue colour with potassium hexacyanoferrate(III) (potassium ferricyanide) and iron(III) ions give a red colour with potassium thiocyanate.

Experiment E15.5

The reversible change from tetra-aquocopper(II) ions to tetra-amminecopper(II) ions

APPARATUS

Each pupil or pair of pupils will need:

Two test-tubes, 100 × 16 mm

Teat pipette

1M copper(II) sulphate solution

2M ammonia solution

1M sulphuric acid

Litmus solution

PROCEDURE

Take 1–2 cm³ of 1M copper(II) sulphate solution in a test-tube and add a drop of ammonia solution; shake well. Continue to add drops of ammonia solution until no further change occurs (guidance may be needed here). Record the number of drops of ammonia solution used (N). Now add dilute sulphuric acid drop by drop, shaking after each addition and count the number of drops needed to restore the original colour of the solution.

Take N drops of the ammonia solution in a clean test-tube, add one drop of litmus solution and dilute sulphuric acid drop by drop, shaking after each addition, until the solution is red. Count the number of drops of acid required, and compare with that found in the first experiment.

Experiment E15.6

The thermal dissociation of dinitrogen tetroxide

APPARATUS

Each pupil or pair of pupils will need:

Two identical round-bottomed flasks, 100 cm³, with one-holed rubber bungs to fit

Glass tubing bent as shown in the diagram

Mercury

Thermometer, -10° to $+110^{\circ}\text{C}$

Two troughs, one containing water at about the boiling point of dinitrogen tetroxide (22°C) and the other containing water at about 35°C

Hard-glass test-tube with rubber bung and delivery tube

Powdered lead nitrate

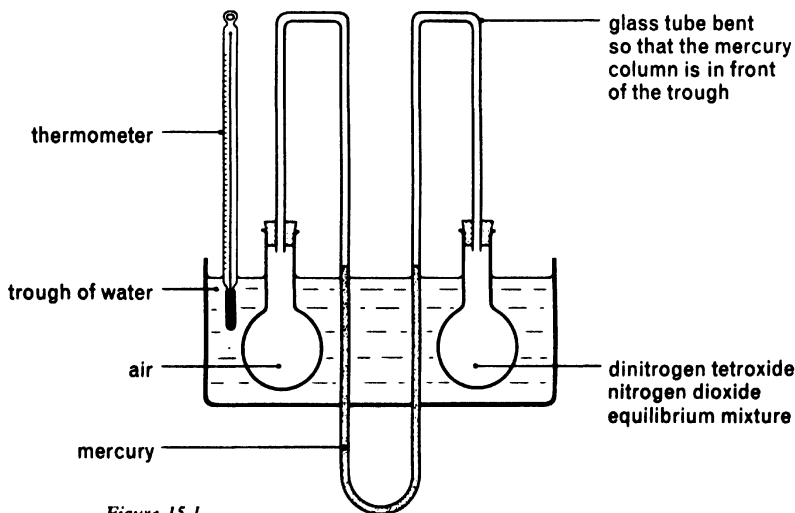


Figure 15.1

PROCEDURE

Set up the apparatus as shown in figure 15.1, preferably in a fume cupboard. The flasks are immersed in a trough with water at about 22°C . Disconnect one of the flasks and flush out the air with a current of nitrogen dioxide, generated by heating the lead nitrate in the hard-glass

test-tube. It is particularly important that all the apparatus is carefully dried beforehand.

Allow the flask which contains the dinitrogen tetroxide, as it now mainly is, to cool and then firmly cork the flasks to the tube containing the mercury. Note the positions of the mercury levels when they are stationary and then transfer the apparatus to the other trough. Leave until the mercury levels are again stationary. For the temperatures stated it will be found that the pressure in the flask containing the dinitrogen tetroxide is about 6 cm of mercury more than in the flask containing air.

The flask should now be returned to the original trough, when it will be found that the mercury levels go back to their original values. The flask containing dinitrogen tetroxide should be flushed out with a current of dry air until the brown colour of the original contents is no longer discernible and the experiment repeated with air in both flasks. This time when the apparatus is placed in the trough of warmer water there is no perceptible change in the mercury levels. The increase in pressure in the first experiment can only be explained by there being an increase in the number of gas molecules as a result of the temperature rise. This strongly supports the view that the dinitrogen tetroxide undergoes thermal dissociation as its temperature rises.

The experiment can be repeated at different temperatures, but they must not be much higher than 35° otherwise the bungs may be blown out.

Experiment E15.7

The reaction between iron(III) ions and thiocyanate ions

An experiment investigating this equilibrium is given in *Tested Experiments for use with Chemistry for Grammar Schools* (Murray), Experiment 57, 'Study of a reversible reaction'.

Experiment E15.8

Investigating the equilibrium between a solute and its saturated solution using a radioactive tracer

This experiment **must** be done by the teacher.

Reference should be made to the precautions to be taken when handling radioactive substances.

APPARATUS

Beaker, 100 cm³

Stand and clamp

Two test-tubes, 100×16 mm
Teat pipette
Mechanical stirrer or shaker
Bunsen burner and asbestos square
Tripod and gauze
Scaler
G.M. liquid counter
Centrifuge and test-tubes, 100×16 mm
Thorium nitrate solution 0.1M (5 per cent)
Lead nitrate solution 0.2M (5 per cent)
2M hydrochloric acid
Acetone
Distilled water

PROCEDURE

The equilibrium between solid lead chloride and its solution is followed by using a radioactive tracer.

Before starting the experiment make a count of background radiation. Make a saturated solution of lead chloride by adding just enough 2M hydrochloric acid to $5\text{--}10\text{ cm}^3$ of 0.2M lead nitrate solution to precipitate most of the lead. Shake well, centrifuge off the solid, transfer 5 cm^3 of the solution to the G.M. tube, and count it over a period of a few minutes. It should give no more than a background count.

To about 10 cm^3 of the 0.1M thorium nitrate solution in a beaker add 5 cm^3 of the lead nitrate and precipitate lead chloride by the addition of hydrochloric acid. This precipitate will contain the radioactive isotope, lead 212. Centrifuge the precipitate; add it to the saturated lead chloride solution prepared above and shake and stir mechanically for about twenty minutes. Centrifuge off the precipitate and count the clear liquid over a period of a few minutes. The liquid will now be active, due, not to solution of lead chloride, for the solution is already saturated, but to the interchange of lead ions between the solution and the solid as indicated by the radioactive tracer. The count rate will be found to be several times greater than that obtained with the original saturated lead chloride solution.

Experiment E15.9

Investigating the equilibrium between rhombic and monoclinic sulphur

APPARATUS

Each pupil or pair of pupils will need:

Dilatometer, made from a test-tube (125×16 mm for example), fitted

with a waxed cork carrying a 25 cm length of glass tubing of diameter 1–2 mm

Thermometer, 0° to 200°C

Large test-tube, fitted with drilled cork and stirrer (see diagram)

Bunsen burner and asbestos square

Stand and clamp

Ruler to use as a scale

Freshly-prepared samples of rhombic and monoclinic sulphur (for preparation see experiment E13.14)

Dilute sulphuric acid (about 5 M)

Glycerol

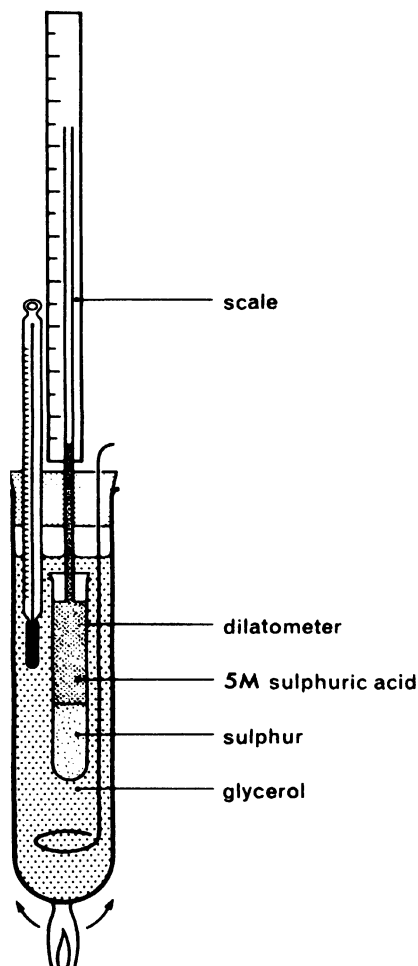


Figure 15.2

PROCEDURE

Without powdering them, place some crystals of rhombic sulphur in a 125×16 mm test-tube until it is one-third full, add a few small particles of monoclinic sulphur, shake the mixture, and add dilute sulphuric acid until the tube is two-thirds full. Dislodge any air bubbles from the sulphur, fill the tube with dilute sulphuric acid, and fit a waxed cork with a tube of diameter 1–2 mm, so that the end of the tube is flush with the base of the cork. Adjust the cork so that the acid goes part of the way up the tube.

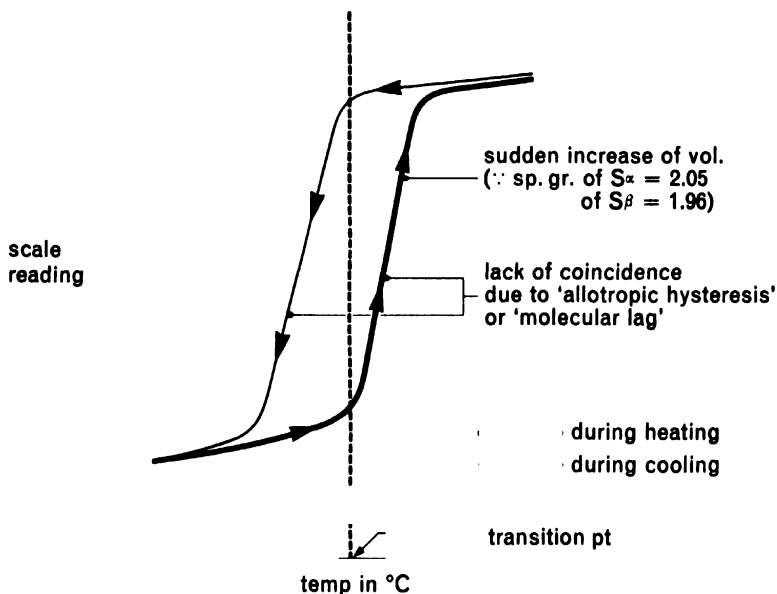


Figure 15.3

Place this dilatometer, with a thermometer and a stirrer, in a large test-tube containing glycerol. Fit a scale to the vertical tube and heat the apparatus gently. Plot a graph of readings on the scale against temperature, for a temperature range from 85 to 110°C during heating, and for the same range, as the dilatometer cools. The wax on the cork melts during the heating, but even so the cork is little affected by the acid; a rubber bung becomes charred. The transition point can be estimated from the graph (see figure 15.3). (See Bowden, S. T., *School Science Review*, 82, pp. 804–5.)

Part IV

What energy changes accompany the chemical change, and what is their significance?

Chapter 16

Energy

Introduction

The interaction of energy and matter is obvious from the earliest stages of chemical investigation, and many experiments in earlier chapters can be used, incidentally, as illustrations of processes in which energy is evolved or absorbed. Examples include the combustion of elements (experiment E3.1); the combination of metals with sulphur (experiment E7.9); the reaction of an acid with an alkali (experiment E8.8), and many others.

After an introduction to the subject in experiment E16.1, the experiments in this chapter are mostly concerned with the quantitative aspect of the energy-matter relationship – and are principally designed to measure the amount of heat evolved during a chemical change.

The reversibility of the process is brought out by such experimental results as:

1. The need to supply heat in order to boil a liquid or melt a solid, which is given back when the vapour condenses or the liquid freezes (illustrated during distillation, for example, experiment E2.11, and again in this chapter by experiments E16.3 and E16.4).
2. The need to supply heat in order to make anhydrous copper(II) sulphate from the hydrated crystals, which is given back when the products are mixed (see experiment E1.4).

Quantitative measurements of the amount of heat evolved in various reactions are described in experiments E16.5–E16.10.

References

The interaction of energy and matter is discussed in the *Handbook for Teachers*, Part V, 'Energy changes'. Its presentation in the Sample Scheme, Part 1, *Basic Course*, begins in Topic 15, 'Solids, liquids, and gases', and is continued in Topic 23, 'Chemicals and energy', both topics being in Stage II of the course. The subject is elaborated in the

Course of Options, Option 5, 'Chemical changes and the production of electrical energy'.

List of experiments

- E16.1 Some exothermic and endothermic processes
- E16.2 To find the effect of pressure on the boiling point of water
- E16.3 To compare the amounts of heat required to warm and to boil liquids
- E16.4 To find out what happens when benzene is frozen
- E16.5 To measure the heat of combustion of a liquid
- E16.6 To measure the heat of combustion of charcoal, using Thiemann's apparatus
- E16.7 To measure the heat evolved in some reactions
- E16.8 To measure ΔH and ΔG for a copper-silver cell
- E16.9 To measure the heat evolved when powdered metals react with an acid
- E16.10 To measure the rise in temperature when one metal displaces another from its salt

Experiment E16.1

Some exothermic and endothermic processes

This experiment should be done by the teacher, but a pupil could take the temperatures.

APPARATUS

Four beakers, 2 litre

Four thermometers, -5° to $+50^{\circ} \times 0.1^{\circ}\text{C}$

Five measuring cylinders, 500 cm³

Concentrated sulphuric acid

2M sodium hydroxide solution

2M hydrochloric acid

2M calcium chloride solution

2M potassium carbonate solution

Ammonium nitrate on a sheet of paper, 80 g

PROCEDURE

1. Take the temperature of 1 litre of water in the 2-litre beaker. Add 53.5 cm³ (1 g-mol) of concentrated sulphuric acid to the water and stir well. Take the temperature of the mixture. The temperature *rises* about 17°C.

2. Repeat the procedure of 1 in another 2-litre beaker but replace sulphuric acid by 80 g (1 g-formula) of ammonium nitrate crystals. *Fine* crystals should be bought as they dissolve more quickly. (It is **not safe** to grind ammonium nitrate in a mortar with a pestle.) The temperature *drops* about 5°C.

3. Pour 500 cm³ of 2M sodium hydroxide solution into a 2-litre beaker. Take the temperature. Add 500 cm³ of 2M hydrochloric acid. Stir well. Take the final temperature. The temperature rises 13–13.5°C.

4. Repeat the procedure of 3 using 2M solutions of calcium chloride and potassium carbonate.

Experiment E16.2

To find the effect of pressure on the boiling point of water

This experiment should be done by the teacher.

APPARATUS

Flask, round-bottomed, 500 cm³, fitted with a two-hole bung

Winchester bottle, fitted with a three-hole bung and three glass delivery tubes

Manometer, mercury filled, with mm scale

Thermometer, –10° to +110°C

Condenser and rubber tubing

Bunsen burner, tripod or ring, gauze, and asbestos squares

Stands and clamps

Thick-walled pressure tubing

Vacuum pump

Broken porcelain

Distilled water

PROCEDURE

Set up the apparatus as shown in figure 16.1 paying particular attention to the following points:

1. Pieces of broken porcelain should be added to promote smooth boiling.

2. Glass to glass connections should be of thick-walled pressure tubing and the bungs must fit tightly.

3. Use of the Winchester bottle as an air reservoir prevents large fluctuations in pressure and allows the pressure to be changed by small amounts. The Winchester bottle should be screened by asbestos squares so that its temperature remains as constant as possible.

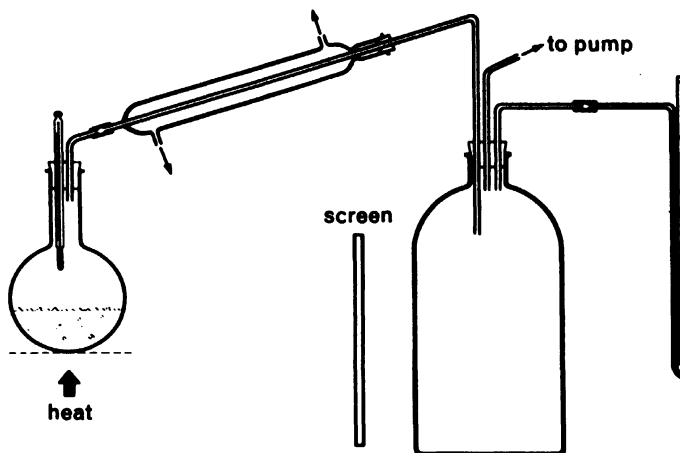


Figure 16.1

Boil the water in the flask and record its boiling point at the known atmospheric pressure, P . Attach the pump and reduce the pressure in the apparatus until the manometer indicates a pressure difference of about 2-3 cm. Boil the water gently in the flask and record this temperature and both mercury levels of the manometer. Continue reducing the pressure in stages of about 2-3 cm and recording the boiling temperatures until sufficient results have been obtained to plot a graph of pressure against boiling temperature.

ALTERNATIVE METHOD

This experiment should be done by the teacher.

APPARATUS

Flask, round-bottomed, 500 cm³, fitted with a two-hole bung, short delivery tube and rubber tubing

Bunsen clip

Thermometer, -10° to $+110^{\circ}\text{C}$

Stand and clamp or ring

Bunsen burner, tripod, gauze, and asbestos square

Cloth

Distilled water

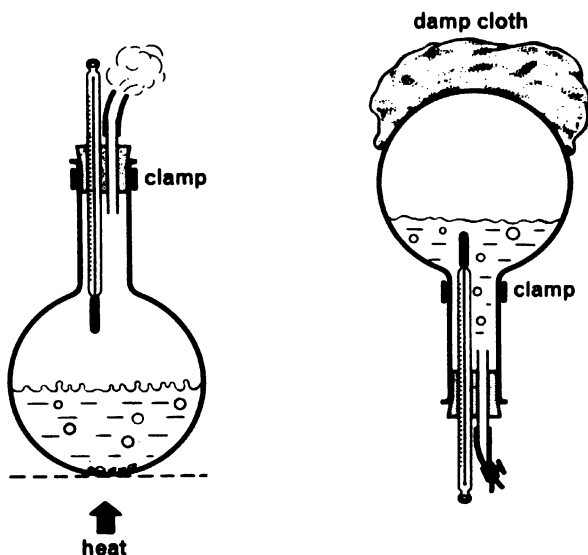


Figure 16.2

PROCEDURE

Fill the flask to about one-third with distilled water and arrange the position of the thermometer so that the thermometer bulb will be in the water when the flask is inverted later on. Boil the water and maintain its boiling for about half a minute to ensure that the steam has swept out all the air from the flask. Remove the Bunsen burner and immediately close the rubber tubing by the Bunsen clip. Invert the flask and apply a cloth soaked in water which will cool and condense some of the water vapour in the flask. This results in a lowering of the pressure inside the flask and the water begins to boil vigorously once more. Record this boiling temperature.

By further applications of the damp cloth the boiling may be maintained until the temperature has been lowered to 50°C or thereabouts.

A disadvantage of this method is that no direct measurement of the internal pressure is available. Reference can be made to tables of saturated water-vapour pressures in the *Book of Data* for pressures corresponding to the observed boiling temperatures.

Experiment E16.3

To compare the amounts of heat required to warm and to boil liquids

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 15.2)

Conical flask, 100 cm³, wide-necked, or beaker, 100 cm³

Thermometer, -10° to +110°C

Bunsen burner and asbestos square

Stand and clamp

Supply of asbestos squares to protect apparatus from draughts

Stop clock, or watch with seconds hand

Graph paper

Trichloroethane or tetrachloroethane

PROCEDURE

This experiment may be done by pupils using water, and repeated by the teacher using, say, trichloroethane or tetrachloroethane.

Place a steady Bunsen burner flame (about 5 cm high) below a conical flask or beaker containing a known weight of the liquid (a volume of about 50 cm³ is suitable). Support the apparatus in a stand and clamp, and protect it from draughts by using asbestos squares. Place a thermometer in the liquid and measure the rate of rise of temperature in degrees C per minute. This is conveniently done by recording the temperature every quarter minute and plotting a graph of temperature against time. The gradient of the best straight line that can be drawn through the points obtained (omitting the later ones) is the required rate.

If this rate is then multiplied by the weight of the liquid in the flask and by its specific heat, the number of calories given out by the flame per minute will be obtained. (This calculation neglects the heat absorbed by the flask, which is small in comparison with that absorbed by the contents.) *Without altering the flame in any way*, allow the liquid to boil for a known time, say, ten minutes for the water or five for the more volatile liquids. Then remove the burner, allow the flask and contents to cool, and determine the weight of the residual liquid.

Using shielding and clamping the flask without a gauze gives results of about 85 per cent of the accepted result with water and 95 per cent with tetrachloroethane.

Experiment E16.4

To find out what happens when benzene is frozen

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 15.1c)

Test-tube, 100×16 mm

Beaker, 100 cm^3

Thermometer, -10° to $+110^\circ\text{C}$

Stop clock or watch with seconds hand

Crushed ice

Benzene

PROCEDURE

Put about 5 cm^3 of benzene in the test-tube, place the test-tube in a beaker half-filled with water, and record the temperature. Then fill the beaker up with ice.

Use the thermometer to stir the benzene and record its temperature at regular intervals as the benzene is cooled. Soon the benzene will solidify but leave the thermometer in place and continue the temperature observations for a further five minutes.

Plot a graph of temperature against time. Definite breaks in the curve should be noticeable and the pupils should suggest what they may represent.

If there is time, repeat the experiment, this time making no attempt to stir the benzene. When the results are plotted, does the second graph differ in any respects from the first? Suggest reasons for any differences.

Experiment E16.5

To measure the heat of combustion of a liquid

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 23.1a)

Metal can of capacity about 300 to 500 cm^3 . (Any sort of tin can will serve but it is an advantage to have the same size and variety for each working group)

Spirit lamp (improvised, see diagram)

Stand and clamp with wide jaws

Measuring cylinder, 500 cm^3

Thermometer, -10° to $+110^\circ\text{C}$

Draught shield. Two asbestos squares normally used to protect the bench tops are quite suitable
Teat pipette

Access to:

Balance

Methanol

Ethanol

Propan-1-ol

Butan-1-ol

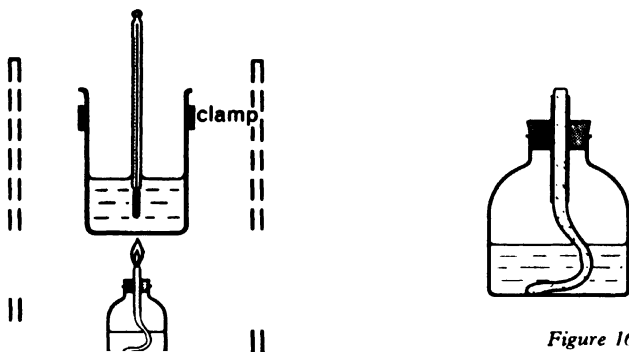


Figure 16.3

PROCEDURE

Place 250–300 g of water in the can. This weight can be measured by taking 250–300 cm³ from a measuring cylinder, but it is the *weight* that matters. Fill the lamp with one of the alcohols, and weigh it. Clamp the can, insert the thermometer, and read the temperature of the water. Place the lamp under the can, put the draught shield round it and light the lamp. Stir the water at frequent intervals until the temperature has risen by about 30°C. With 250 cm³ of water this takes about seven minutes. Extinguish the lamp and take the temperature of the water after a thorough stirring. Reweigh the lamp.

If W grams of alcohol have been burned then $\frac{W}{M}$ g-mol have been burned where M is the mass of 1 g-mol. If the temperature rises by $t^\circ\text{C}$ and 250 cm³ of water are heated, then $\frac{250}{1000}t$ kcal of energy have gone into the water as heat. The energy lost as heat when 1 g-mol of alcohol is burned is therefore $\frac{250}{1000}t \times \frac{M}{W}$ kcal. This is the heat of combustion in kcal per g-mol.

Note the water equivalent of the calorimeter and then fill it to within about 1 cm of the top with a known volume of water. Clamp the calorimeter securely at a suitable working height. Weigh the crucible containing about half a gram of the carbon and support the crucible on the asbestos platform which should be lowered a few centimetres below the calorimeter.

Apply moderate suction by connecting the filter pump to the spiral; stir the calorimeter contents and record the temperature when this becomes constant. Connect the oxygen supply to the asbestos platform and carefully regulate the gas so that only a gentle stream of oxygen is obtained. (About 3 p.s.i. on the outlet pressure gauge should be enough.) Ignite the carbon lump with a small Bunsen flame and when the carbon is just glowing raise the asbestos platform to fit securely below the base of the calorimeter. The carbon will now glow brightly in the air enriched with oxygen. The oxygen pressure needs careful adjustment so that the carbon burns quietly with no spluttering and consequent loss of weight. Should the carbon splutter, the oxygen supply must be reduced until quieter combustion is obtained. The water is stirred throughout the combustion; the combustion should last about five minutes for the weight of carbon indicated.

When combustion has ceased, turn off the oxygen supply and the filter pump; stir until the maximum temperature is observed and record this temperature.

When cool, remove the crucible and re-weigh it to find the amount of carbon consumed.

From the results of the experiment, the heat of combustion of this form of carbon can be calculated in kilocalories per gram-atom.

b. For a series of alcohols

The apparatus may also be used to compare the heats of combustion of a series of alcohols. A special 'spirit' burner is used and the loss in weight during combustion of a few cm³ of an alcohol is measured.

An adequate air supply should be maintained by the filter pump, which may be supplemented, if necessary, by leaving a small space between the asbestos platform and the calorimeter. *Do not use an oxygen supply in this experiment.*

A temperature rise of 5°C is required and the close-fitting ground-glass cap is placed over the hot spirit burner to prevent evaporation of alcohol as it cools down before the final weighing is made.

Experiment E16.7

To measure the heat evolved in some reactions

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 23.3)

Polythene bottle, about 70 cm³, fitted with rubber stopper and thermometer, -10° to $+110^{\circ}\text{C}$, or -5° to $+50^{\circ}\text{C}$

Measuring cylinder, 25 cm³

0.5M silver nitrate solution, 25 cm³

Either 0.5M sodium chloride solution, 25 cm³

or 0.5M potassium chloride solution, 25 cm³

or 0.5M ammonium chloride solution, 25 cm³

0.2M copper(II) sulphate solution, 25 cm³

Either zinc filings, about 0.5 g

or iron filings, about 0.5 g

PROCEDURE

The method of using the Polythene bottle depends to some extent on the type of thermometer used. The short type is most robust but the thread is then inside the bottle when this is upright and readings are impossible. In this case the bulb should be just inside the rubber stopper and readings taken with the bottle *inverted*.

For the precipitation reaction measure 25 cm³ 0.5M silver nitrate solution into the Polythene bottle. Wash the measuring cylinder with distilled water and put 25 cm³ chloride solution into it. Take the temperature ($t^{\circ}_{\text{initial}}$) of the chloride solution.

Wash the thermometer bulb and insert the rubber stopper and thermometer into the bottle. If the solutions and apparatus are prepared well in advance and left on the bench, they will all be at $t^{\circ}_{\text{initial}}$. Remove the stopper, add the chloride solution, replace the stopper, shake the bottle, and record the highest temperature reached (t°_{final}).

Because the heat capacity of the solution *and* the Polythene bottle is very nearly 1 kcal l⁻¹ deg⁻¹, the heat which must be *lost* for 1 litre to return to room temperature is $1 \times (t^{\circ}_{\text{final}} - t^{\circ}_{\text{initial}})$ kcal. The concentrations we have used are such that 1 litre of solution would have had $\frac{1}{4}$ g-equation of change taking place in it. It follows that the energy change for the precipitation is

$$\Delta H = -4 (t^{\circ}_{\text{final}} - t^{\circ}_{\text{initial}}) \text{ kcal g-equation}^{-1}$$

The measurement for the replacement reaction is made similarly, but 0.2M solutions are used. Put 25 cm³ 0.2M copper(II) sulphate solution

in the bottle. Insert stopper and thermometer, shake, and read the temperature ($t^{\circ}_{\text{initial}}$). Add 0.5 g metal filings (about a two-fold excess), shake, and record the highest temperature reached (t°_{final}). Arguing along the same lines as above, but bearing in mind that 1 litre of solution would only contain 0.2 g-equation of change

$$\Delta H = -5 (t^{\circ}_{\text{final}} - t^{\circ}_{\text{initial}}) \text{ kcal g-equation}^{-1}$$

Experiment E16.8

To measure ΔH and ΔG for a copper-silver cell

a. To measure ΔH for reaction



APPARATUS

Each pupil or pair of pupils will need:

Polythene bottle, capacity about 50 cm³, fitted with stopper carrying thermometer, 0° to 50° $\times$ 0.1°C

Measuring cylinder, 25 cm³

0.2M silver nitrate solution

Copper powder, about 0.5 g, prepared by reducing copper(II) oxide by heating in town gas

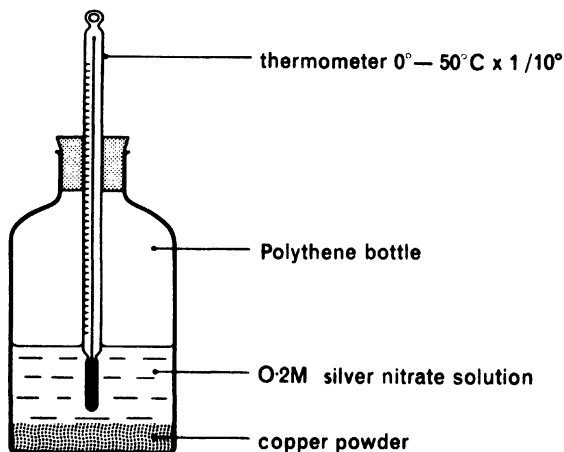


Figure 16.5

Put 25 cm³ of silver nitrate solution in the Polythene bottle, insert the cork and thermometer and read the temperature of the solution, when this is steady. Remove the stopper and add about 0.5 g copper powder (this is about a fourfold excess). Replace the stopper and shake the

bottle. Record the highest temperature reached. ΔH is calculated from:

$$\Delta H = 10 \Delta t^\circ \text{ kcal g-equation}^{-1}$$

where Δt° is the rise in temperature.

If the thermal capacity of the bottle is to be taken into account this is found as follows:

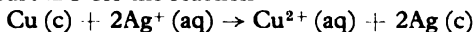
Insert the stopper and thermometer into the empty bottle and read the temperature (t°_1). Warm some distilled water in a beaker to about 45°C . Pour 25 cm^3 of this into a measuring cylinder and take the temperature (t°_2) with the thermometer from the bottle. Pour the water quickly into the bottle, insert the cork plus thermometer, and shake the bottle so that the warm water contacts the whole of the interior. Read the temperature when it is steady (t°_3).

Then the thermal capacity of bottle, c , = $\frac{25(t^\circ_2 - t^\circ_3)}{(t^\circ_3 - t^\circ_1)} \text{ cal } ^\circ\text{C}^{-1}$

ΔH is now calculated from:

$$\Delta H = \frac{10(25 + c)\Delta t^\circ}{25} \text{ kcal g-equation}^{-1}$$

b. To measure ΔG for the reaction



APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Length of silver wire, 10 cm

Strip of copper foil

Saturated potassium nitrate solution, 50 cm^3

Filter paper

Two beakers, 100 cm^3

Evaporating basin

Thick glass rod

Thin glass rod

0.2M silver nitrate solution

0.2M copper(II) nitrate solution

Also available for use by the class:

High resistance voltmeter, with leads and crocodile clips

The procedure is the same as for experiment E17.3. Silver nitrate solution is placed in the inner compartment of the cell and silver wire, held in the split cork, dips into this. Copper(II) nitrate solution is used in the outer compartment with the strip of copper foil wedged between specimen tube and cork.

Measure the e.m.f. of this cell. If the value is E volts, ΔG can now be calculated from:

$$\Delta G = -23nE \text{ kcal g-equation}^{-1}$$

where n is the number of moles of electrons transferred in the equation as written, i.e. $n = 2$ in this case.

Experiment E16.9

To measure the heat evolved when powdered metals react with an acid

APPARATUS

Each pupil or pair of pupils will need:

Test-tubes, 125×16 mm

Test-tube rack, modified for thermal insulation, by enclosing each side, between top and bottom, with slabs of expanded polystyrene (e.g. 1 foot square $\frac{3}{8}$ in thick standard ceiling tile). Cut this using a sharp razor blade, with sawing action, into 3 in. wide strips – one each side of rack, and smaller sections in the spaces between adjacent tube-holes. Secure with pins, for ease of dismantling. Do *not* use polystyrene cement or similar adhesives.

Metals in powder form, as uniform as possible.

Thermometer, -10° to $+110^\circ\text{C}$

Burette or pipette to measure 5 cm^3

2.5M hydrochloric acid

PROCEDURE

Measure 5 cm^3 portions of 2.5M hydrochloric acid into test-tubes in the rack and note temperature.

Add one or two measures of powdered metal (excess); stir at intervals and note the highest temperature reached; record this temperature and inspect the tube contents to assess the vigour of reaction. Leave the tube in the rack and proceed to the next test.

Typical Results (using 2.5M hydrochloric acid)

Magnesium	maximum rise 68.5°C . Very vigorous reaction.
Aluminium	rise during 15 minutes 5°C , then after 25–35 minutes' delay, sudden rise to maximum 78° and violent action.
Zinc	9°C rise, moderately brisk action.
Iron (60 mesh, grease-free filings)	5°C , slow to start then fairly brisk.
Tin (very fine powder)	1.2°C , action not very evident.

Lead (medium powder)	0.5°C, very little action evident.
Copper (fine powder – dark)	8.5°C, marked change to pink powder!
Copper (medium turnings)	no rise and no change seen.

Note: The results with aluminium, after the period of delay, are quite spectacular. After noting the results with copper powder and the cessation of action when it goes pink, a student might suggest that oxide has dissolved in the acid and hence heat of neutralization is involved.

Experiment E16.10

To measure the rise in temperature when one metal displaces another from its salt

APPARATUS

Each pupil or pair of pupils will need:

Test-tubes and test-tube rack as described for experiment E16.8 *or* Polythene test-tubes, 10 cm³ capacity

1M copper(II) sulphate solution

Pipette, 10 cm³

Thermometer, –10° to +110°C

Metals in powdered form: zinc, iron (reduced), aluminium, tin, magnesium, lead

PROCEDURE

10 cm³ of 1M copper(II) sulphate solution are run by means of a pipette into a Polythene tube. The temperature is taken and recorded.

One measure of powdered metal is added.

The solution and powdered metal are stirred by means of the thermometer and the highest temperature recorded.

There is no need to weigh the powdered metals, as long as excess over the equivalent weight of copper present in the 10 cm³ of solution has been added.

Typical Results

Rise in temperature

Zinc replacing copper from copper sulphate	49.0°
Magnesium replacing copper from copper sulphate	39.0°
Iron replacing copper from copper sulphate	32.5°
Tin replacing copper from copper sulphate	22.0°
Aluminium replacing copper from copper sulphate	nil
Lead replacing copper from copper sulphate	nil

Chapter 17

Cells

Introduction

Investigations of voltaic cells are closely linked with electron transfer processes and energy. Experiments on these subjects are grouped in Chapters 9 and 16 respectively.

Thus cells can be used in the building of an electron-transfer or redox series, and an experiment to investigate some redox reactions using cells is included in Chapter 9 (experiment E9.5). An experiment in which the energy terms ΔH and ΔG for a cell are measured is described in Chapter 16 (experiment E16.8).

Experiments in this chapter are mostly concerned with the methods of construction and properties of cells. Apart from experiment E17.1 they are most suitable for use towards the end of the pupils' first five-year course.

References

The subject of voltaic cells is discussed in the *Handbook for Teachers*, Chapter 8, and the way the theme is developed in the Sample Scheme is also described there.

The possibility of obtaining electricity from chemical reactions is first mentioned in the Sample Scheme, Part 1, *Basic Course*, in Stage 1 B, Topic 6. It is again introduced in Stage II, Topic 23: 'Chemicals and energy', and developed in detail in the *Course of Options*, Option 5, 'Chemical changes and the production of electrical energy'.

List of experiments

- E17.1 Can electricity be obtained from chemical reactions?
- E17.2 The conversion of chemical energy into useful work
- E17.3 To find the polarities of the terminals and measure the e.m.f. of some simple voltaic cells

- E17.4 To investigate the effect of the concentration of the electrolyte solutions on the e.m.f. of a voltaic cell
- E17.5 To measure the e.m.f. of a voltaic cell using the counter-voltage method
- E17.6 To measure the standard electrode potential of copper
- E17.7 How to predict the course of possible metal-ion reactions and check them experimentally
- E17.8 To measure the e.m.f. of a copper-iodine cell
- E17.9 To measure the e.m.f. of a copper-iron cell
- E17.10 Testing predictions made about an iodine-iron cell
- E17.11 To investigate the structure of a 'dry' cell
- E17.12 Experiments with a rechargeable cell
- E17.13 A very simple hydrogen-oxygen fuel cell

Experiment E17.1

Can electricity be obtained from chemical reactions?

Experiment a.

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment B6.5)

Support for electrode foils

1.25 volt 0.25 amp bulb and holder

Two lengths of connecting wire, fitted with crocodile clips

Sand paper

Beaker, 100 cm³

M or 0.5M sulphuric acid

Copper foil, 5 × 3 cm approximately

Magnesium ribbon, 15–20 cm approximately

PROCEDURE

Clean the copper foil and magnesium ribbon thoroughly using sand paper. Form the ribbon into a suitable shape round a glass rod or ruler and fasten the magnesium and copper in the electrode support. Place the magnesium and copper in the beaker containing approximately half

molar or molar sulphuric acid. A vigorous reaction occurs and the bulb glows steadily as long as some magnesium remains. The distance apart of the electrodes may need adjustment to obtain the maximum illumination.

After the pupils have done this experiment discuss the importance of batteries and lead to the points: that different batteries (*a*) use different metals and (*b*) produce different voltages. A lead accumulator and a nickel-iron cell or carbon-zinc torch battery may be quoted as examples. Which metals make the best batteries? It is possible to measure the potential difference set up between two different metals very simply by means of the following experiment.

Experiment b.

A projection microammeter, range 250-0-250 μA and having low internal resistance, may be used in series with a suitable resistor to give direct e.m.f. values. The instrument may be mounted in a film strip-slide projector or placed on an overhead projector and the experimental results projected to the class. With a projection microammeter of internal resistance of about 30-50 Ω a series resistor of 10,000 Ω is needed for a deflection of 200 $\mu\text{A} \approx 2$ volts, or 5,000 Ω for a deflection of 200 $\mu\text{A} \approx 1$ volt, neglecting the resistance of the instrument.

Should a projection microammeter not be available a large demonstration instrument could be substituted. However, if instruments of the type of the model 8 Universal Avometer or Multiminor Mk IV are available the pupils could record their own results.

APPARATUS

The teacher will need:

Projection microammeter and leads fitted with crocodile clips

Projector

Beaker, 250 cm^3

Metal foils or strips: copper, zinc, iron, magnesium, silver, nickel, lead, gold

PROCEDURE

Copper or iron may be selected as the reference metal. In turn other metal strips are placed in the beaker containing tap water, to which a little sodium chloride or potassium nitrate may be added if necessary, and the projection microammeter with its resistor connected in circuit. The e.m.f. of the pairs of electrodes should be projected in a vertical plane on the board and the needle position marked for each metal. A list of metals will thus be drawn up on the board in order of the e.m.f. that they produce when coupled to the copper or iron electrode.

Experiment E17.2**The conversion of chemical energy into useful work**

This experiment should be done by the teacher.

APPARATUS

Four Daniell cells

Electric motor with pulley attached to operate from 4 volts or less

Cord and small weight

PROCEDURE

The object of this experiment is to show the pupils that chemical energy can be converted in a motor into useful work. The most effective way to do this experiment is to set up the four cells in series with the electric motor, without the electrolyte in the cells. Place the motor at the edge of the bench. Attach the cord to the electric motor shaft with the weight on the floor at one end so that when the motor starts, the cord winds round the shaft and the weight is lifted off the ground. Now add the electrolyte to the cells and show that the weight is raised.

Experiment E17.3

To find the polarities of the terminals and measure the e.m.f. of some simple voltaic cells

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see diagram below)

Two beakers, 100 cm³ (for transporting solutions)

Evaporating basin

Filter paper

Thick glass rod

Thin glass rod

The following will also need to be available:

Saturated potassium nitrate solution, 50 cm³

0.1M solutions of copper(II) sulphate

zinc sulphate

copper(II) nitrate

zinc nitrate

lead nitrate

silver nitrate

aluminium sulphate

iron(II) sulphate

tin(II) chloride
magnesium sulphate
nickel sulphate
mercury(II) chloride

Copper, zinc, iron, lead, silver, tin, aluminium, magnesium, nickel, in the form of foil, wire (fairly thick) or ribbon.

High-resistance voltmeters (one can be made to serve for thirty-two pupils but two are preferable) with leads and crocodile clips. Avometers are excellent.

Shears (or stout scissors) for cutting foil and wire

Iron wool or emery cloth

Distilled water

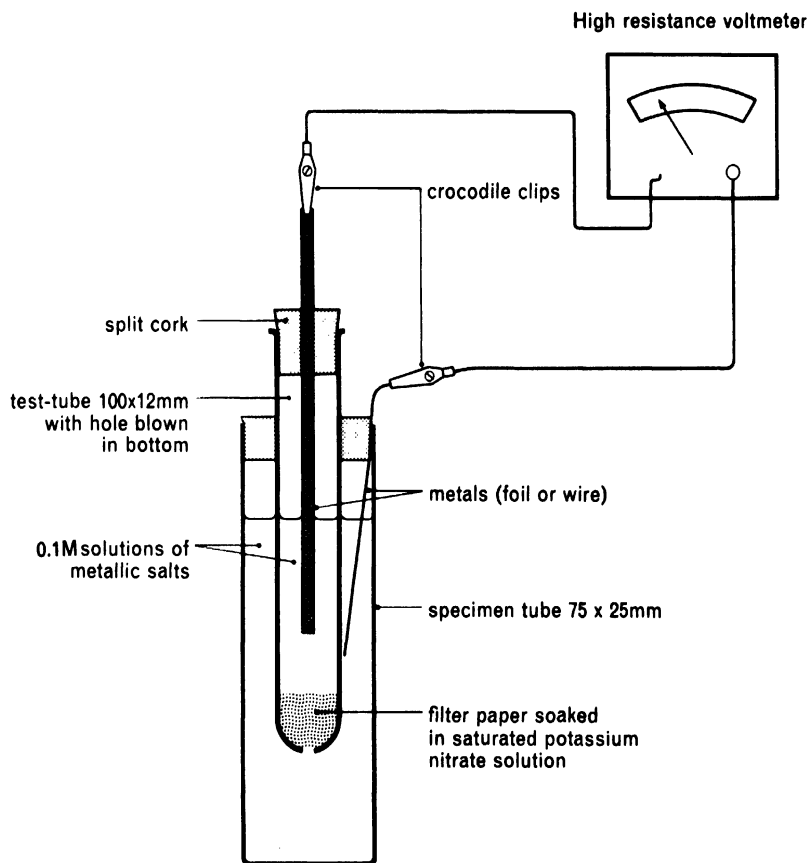


Figure 17.1

PROCEDURE

The pupils tear a filter paper into small pieces and immerse them in saturated potassium nitrate solution. Some of the pulp thus obtained is squeezed with the fingers, placed in the inner vessel of the cell (removed for this purpose) and compacted into a layer 1 cm thick with a stout glass rod. This layer should be packed fairly tightly.

The specimen tube is now filled to about two-thirds with a suitable solution of a metallic salt, a piece of metal foil or wire (cleaned with iron wool or emery cloth – aluminium should be amalgamated by dipping in mercury(II) chloride for a few minutes and washing with distilled water immediately before use) is added and the cork, with inner tube, inserted so that the metal is held between cork and outer tube. The second metallic salt solution is added to the inner vessel until the levels in both vessels are the same. Finally the second electrode is wedged in the inner vessel by means of a split cork. In order to obtain consistent results, it is necessary to keep the area of electrodes immersed constant in the case of each metal.

Without wasting time (because diffusion occurs across the salt bridge), pupils now take the assembled cell to the high resistance voltmeter, find the correct terminal connections, using the crocodile clip leads attached to the instrument, and measure the e.m.f. of the cell. Each pair of pupils can easily set up and measure the e.m.f. of three cells in a double period. *A freshly prepared filter paper partition must be used for each experiment, the used one being pushed out with a thin glass rod.* When allotting cell combinations make sure that a number of these have common electrodes, so that polarities of the terminals and e.m.f. of other cells can be predicted. Avoid using solutions which will react to give precipitates, e.g. avoid copper(II) sulphate and lead nitrate in the same cell.

The results obtained by the whole class can be collected together on the blackboard, with the cell diagrams. From the list discuss with the pupils the positive and negative terminals they would expect to find in cells not included in the list, and their e.m.f. The class now sets up some of these cells and checks its predictions.

Experiment E17.4

To investigate the effect of the concentration of the electrolyte solutions on the e.m.f. of a voltaic cell

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Two beakers, 100 cm³

Evaporating basin

Filter paper

Thick glass rod

Thin glass rod

Measuring cylinder, 25 cm³

M zinc sulphate solution

M copper(II) sulphate solution

Zinc foil

Copper foil

Saturated potassium nitrate solution, 50 cm³

The following will also need to be available:

High-resistance voltmeter (0-3 volt)	} each with leads and crocodile clips
High-resistance millivoltmeter (0-50 millivolts)	

PROCEDURE

Pupils will fit up the cell:



using molar solutions for each electrode, and measure the e.m.f. They then repeat this keeping the molar zinc sulphate solution for the negative electrode and using 0.1M, 0.01M, 0.001M, and 0.0001M solutions of copper(II) sulphate (obtained by successive dilution in the measuring cylinder). The small but definite change in e.m.f. can be discussed and reasons for this sought. The fact that the e.m.f. changes with the concentration suggests that a cell is possible using the same electrode materials but different concentrations of electrolyte.

The pupils can now fit up concentration cells with different molarity copper(II) sulphate solutions, measuring the e.m.f. on a millivoltmeter.

Experiment E17.5

To measure the e.m.f. of a voltaic cell using the counter-voltage method

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Galvanometer, centre-zero, 5-0-5 milliamp

Voltmeter, 0-3 volt (need not be high resistance)

Rheostat

2 volt accumulator (or 2-3 volts from low voltage d.c. supply)

Wire leads for connections

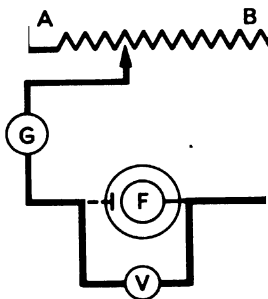


Figure 17.2

PROCEDURE

The circuit is shown in the diagram; the cell to be measured is assembled and connected as shown. The sliding contact is moved from A to B until the current, as shown by the galvanometer, is zero. The voltmeter reading at this point is the maximum e.m.f. of the cell.

Experiment E17.6

To measure the standard electrode potential of copper

APPARATUS

(To save expense on platinum, this experiment can be done by the teacher or by groups of pupils.) Each group will need:

Hydrogen electrode (see diagram below)

Apparatus for constructing rest of cell (see experiment E17.3)

Two beakers, 100 cm³

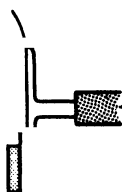
Evaporating basin

Filter paper

Thick glass rod

Thin glass rod

High resistance voltmeter, with leads and crocodile clips



platinum wire

Hydrogen gas

Cork with groove in side
(to allow escape of gas)

0.1M HCl

Platinum deposit
(electrolysis of Pt Cl_4 /solution)filter paper soaked in
saturated potassium
chloride solution
(renewed for each experiment)*Figure 17.3*

Hydrogen is best obtained from a cylinder. If gas generated from zinc and dilute acid is used, impurities liable to poison the platinum catalyst must be removed by bubbling it through some dilute permanganate solution containing a little dilute sulphuric acid.

PROCEDURE

The procedure is the same as for experiment E17.3. The hydrogen electrode forms the inner compartment of the cell, with 0.1M copper(II) sulphate solution and copper foil in the outer compartment.

Experiment E17.7

How to predict the course of possible metal-ion reactions and check them experimentally

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Two beakers, 100 cm³

Evaporating basin

Filter paper

Thick glass rod

Thin glass rod

Test-tubes, 100 × 16 mm, in rack

Saturated potassium nitrate solution, 50 cm³

The following will also need to be available:

Iron wool or emery cloth

High-resistance voltmeter, with leads and crocodile clips
A range of metals and 0.1M salt solutions

PROCEDURE

The reactions can either be allocated by the teacher or pupils can choose which they will investigate.

As an example, suppose a pair of pupils is to investigate the possibility of a reaction involving iron, tin, and their salts. They first write the cell diagram, using the table in the *Book of Data* to calculate the e.m.f. and find its sign. From this they predict the most likely course that the reaction will take. These predictions they report to the teacher.

The pair then check their conclusions by:

- Fitting up the cell, finding the polarities of the terminals, and measuring the e.m.f.
- Putting a piece of tin into a solution of an iron(II) salt in a test-tube and a piece of iron into a tin(II) solution in another test-tube.

The tubes are examined after standing for five to ten minutes.

If time presses, (a) could be omitted.

Experiment E17.8

To measure the e.m.f. of a copper-iodine cell

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Copper foil

Platinum electrode (two types are shown in the diagram below)

Saturated potassium chloride solution, 50 cm³

Filter paper

Measuring cylinder, 25 cm³

Beakers, 100 cm³

Evaporating basin

Thick glass rod

Thin glass rod

0.2M potassium iodide solution

0.2M iodine solution (in 0.2M KI solution)

0.1M copper(II) sulphate solution

Also available for use by the class:

High-resistance voltmeter, with leads and crocodile clips

*Figure 17.4***PROCEDURE**

The iodine/iodide solution is made by mixing 5 cm³ 0.2M potassium iodide solution with 5 cm³ 0.2M iodine solution. The mixture is placed in the inner compartment of the cell (after the 'salt bridge' has been inserted). The platinum electrode dips into this solution, being held in place by a grooved split cork. The outer compartment is filled with 0.1M copper(II) sulphate solution, into which dips a strip of copper foil.

The polarities of the terminals and the e.m.f. of the cell are determined as in previous experiments.

From the values obtained the standard electrode potential for the $I_2(aq) \mid 2I^-(aq) \mid Pt$ electrode is calculated.

Experiment E17.9

To measure the e.m.f. of a copper-iron cell

APPARATUS

As for experiment E17.8 but with iodide and iodine solutions replaced by 0.2M iron(II) sulphate and iron(III) sulphate solutions. $Fe_2(SO_4)_3 \cdot 9H_2O$ or iron(III) alum solution, which is 0.2M with respect to $Fe^{3+}(aq)$ ions, must be used for this experiment.

PROCEDURE

The procedure is the same as for experiment E17.8, but the inner compartment holds a mixture of equal volumes of 0.2M Fe^{2+} (aq) and 0.2M Fe^{3+} (aq) solutions.

Experiment E17.10

Testing predictions made about an iodine-iron cell

APPARATUS

Each pupil or pair of pupils will need:

Apparatus for constructing cell (see experiment E17.3)

Two platinum electrodes

Filter paper

Thick glass rod

Thin glass rod

Measuring cylinder, 25 cm³

Two beakers, 100 cm³

Evaporating basin

Three test-tubes, 100 × 16 mm

Teat pipette

Starch solution

0.2M potassium iodide solution

0.2M iodine solution (in 0.2M KI solution)

0.2M Fe^{2+} solution } see experiment E17.9

0.2M Fe^{3+} solution }

Saturated potassium chloride solution, 50 cm³

Also available for use by the class:

High-resistance voltmeter, with leads and crocodile clips

PROCEDURE

The pupils, having made their own predictions about this cell combination, are now allowed to check these experimentally.

To check the polarities of the terminals and the e.m.f. they assemble the cell in the usual way, and use the voltmeter without loss of time because diffusion soon affects the reading. The iodine/iodide solution should be put in the inner compartment, to save unnecessary expense on chemicals. The platinum electrode for the outer compartment is inserted through a vertical groove cut in the outside of the cork.

The reaction which provides the energy for the cell is checked by test-tube experiments. About 2 cm³ of Fe^{3+} (aq) solution are put into a test-tube; potassium iodide solution is added, one drop at a time, from a teat pipette. Reaction is indicated by a deepening of colour and the presence of iodine confirmed by adding a drop of the reaction mixture

to starch solution in a separate test-tube. A check that the reverse reaction does not take place to any appreciable extent may be made by adding one drop of iodine solution to 2 cm³ of Fe²⁺ (aq) solution.

Experiment E17.11

To investigate the structure of a 'dry' cell

APPARATUS

Each pupil or pair of pupils will need:

Run down 'dry' cell

Filtration apparatus

Beaker, 100 cm³

Evaporating basin

Two test-tubes, 100 × 16 mm, in rack

Glass rod

Bunsen burner, tripod, gauze, and asbestos square

Penknife or screwdriver

Sodium hydroxide solution

Silver nitrate solution

PROCEDURE

Remove the pitch seal from the top of the cell, scrape out the mixture in the zinc case into the beaker, and take out and clean the carbon rod. After examining the contents of the beaker, add 40 cm³ of water to them, stir the mixture and heat to boiling. Filter the mixture whilst hot and evaporate the filtrate to about a quarter of its bulk. White crystals will separate which can be identified as ammonium chloride by suitable tests with sodium hydroxide solution and silver nitrate solution.

Some possible reactions taking place in the 'dry' cell

(The experimental details given here are based mainly on information given in Andrew, H. G., *School Science Review* (1960), 41 (144) 203).

APPARATUS

Each pupil or pair of pupils will need:

Two connecting leads, each fitted with crocodile clips

Teat pipette, with 1 cm³ graduation

Two crystallizing dishes

Working 'dry' cell

Piece of natural pyrolusite

Carbon rod

Strip of zinc foil

M ammonium chloride solution

M sodium chloride solution

Phenolphthalein solution

Also available for use by the class:

High-resistance voltmeter, with leads and crocodile clips

PROCEDURE

1. Using the voltmeter the pupils identify the positive and negative terminals of the 'dry' cell, and measure the e.m.f. The crocodile clips attached to the voltmeter may be held in contact with the two ends of the cell for this purpose.

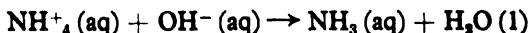
2. M ammonium chloride solution is poured into a crystallizing dish to a depth of about 2 cm. A piece of pyrolusite and a strip of zinc foil are fixed into crocodile clips attached to leads. The bare ends of the leads are then held in the crocodile clips attached to the voltmeter and the pyrolusite and zinc dipped into the solution. Care must be taken that only the pyrolusite is immersed and not the crocodile clip holding it. The e.m.f. of the system is measured.

3. The same procedure is used as in (2) substituting a short carbon rod for the pyrolusite. The ammonium chloride solution is retained for use later.

4. M sodium chloride solution is poured into another crystallizing dish to a depth of about 2 cm and 1 cm³ phenolphthalein solution added. Pyrolusite and zinc foil are fixed into crocodile clips attached to leads as before. The bare ends of the leads are twisted together to complete the external circuit of the cell. The pyrolusite and zinc are now dipped into the liquid and held there for several minutes. Any changes in appearance of the solution around the electrodes are noted.

5. 1 cm³ of phenolphthalein solution is added to the ammonium chloride solution reserved from (3) and the procedure in (4) repeated with this solution.

In discussion, pupils should be asked to account for differences observed in (4) and (5). This will lead to an appreciation of the function of the ammonium chloride in the 'dry' cell as a source of ammonium ions, which combine with hydroxyl ions formed at the positive electrode:



Concentration of hydroxyl ions round the positive electrode would prevent the cell from exerting its maximum e.m.f.

Experiment E17.12**Experiments with a rechargeable cell****APPARATUS**

Each pupil or pair of pupils will need:

Pair of lead plates (about 8×2 cm) fixed to wooden framework (see diagram)

Two connecting leads, fitted with crocodile clips

Low voltage d.c. supply (2-4 volts)

Beaker, 100 cm^3

2-volt bulb in holder

Test-tube, 100×12 mm, with hole in bottom (as for experiment E17.3, with split cork to fit)

Strip of copper foil

Filter paper

Thick glass rod

Thin glass rod

Evaporating basin

4M sulphuric acid

0.1M copper(II) sulphate solution

Saturated potassium nitrate solution

Also available for use by the class:

High-resistance voltmeter with leads and crocodile clips

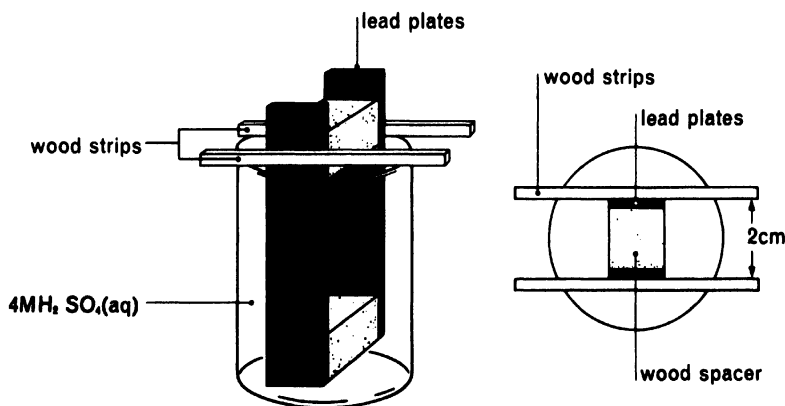


Figure 17.5

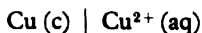
PROCEDURE

The pupils assemble the cell shown in the diagram, connect the lead

plates to the low-voltage d.c. supply (or a suitable battery), and pass a current at 3-4 volts through the sulphuric acid for five minutes. The low-voltage supply is then disconnected and the lead plates examined. The teacher discusses with the class the changes which have occurred, especially the formation of a dark brown solid at the anode.

The cell is taken to the communal voltmeter, the polarities of the plates determined and the e.m.f. measured. Using leads and crocodile clips, the cell is connected to the 2-volt lamp and allowed to remain until the lamp ceases to emit light. The e.m.f. of the cell is re-determined.

The cell is now recharged for 5 minutes, during which time a



electrode is prepared as before, from a 100×12 mm test-tube, with a hole in the bottom, a plug of filter paper soaked in saturated potassium nitrate solution, 0.1M copper(II) sulphate solution and a strip of copper foil. After the low-voltage supply has been disconnected the copper electrode is placed in the beaker, as shown in diagram below.

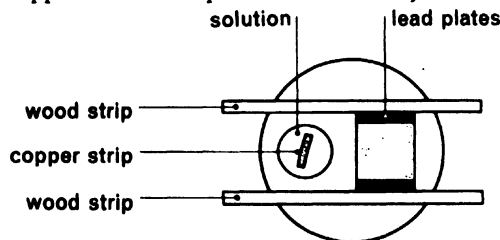
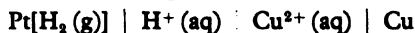


Figure 17.6

The e.m.f. between this electrode and each of the lead plates is measured in turn. The pupils then calculate the E value for each electrode against the hydrogen electrode, using the value for the cell



obtained earlier (experiment E17.6). From the E° table in the *Book of Data* they find the electrodes which correspond most closely to the values calculated and, from these, work out the over-all equation for the cell reaction.

Experiment E17.13

A very simple hydrogen-oxygen fuel cell

APPARATUS

Each pupil or pair of pupils will need:

Page from *Laboratory Investigations* (experiment 23.5)

Electrolysis cell, with carbon electrodes

Two test-tubes, 75×10 mm

D.C. supply (4–6 volts)

Two leads with crocodile clips on one end

1M sodium hydroxide solution

Access to a high-resistance voltmeter fitted with leads and crocodile clips.



d.c.

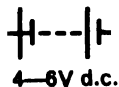


Figure 17.7

PROCEDURE

The pupils electrolyse the alkaline solution until both tubes are filled with gas. They then disconnect the d.c. supply and put a high-resistance voltmeter across the carbon electrodes, finding which is the positive terminal of the cell and measuring the voltage. The gases absorbed on the carbon electrodes provide the reactants for the cell reaction. Some carbon dioxide is bound to be produced during electrolysis but the alkali removes this.

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Blundell's School

Blyth Grammar School

Bootham School, York

Bourneville Boys' Technical School, Birmingham

Braintree County High School

Carolan Grammar School, Belfast, 7

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