

14

Experimental techniques and chemical analysis

FOCUS POINTS

- ★ What apparatus is most commonly used in experimental techniques?
- ★ What is qualitative chemical analysis?
- ★ How can we identify elements and ions?

This chapter shows how to safely and accurately use different experimental techniques, apparatus and materials. This will aid your ability to plan experiments and investigations.

You will also learn that there are specific criteria for purity of substances. Finally, you will learn about the branch of chemistry that deals with the identification of elements, ions or grouping of elements as ions present in a sample, which is called qualitative chemical analysis, or qualitative analysis for short. The various tests you will study would allow you to carry out a wide range of qualitative analysis.

14.1 Apparatus used for measurement in chemistry

Scientists find out about the nature of materials by carrying out experiments in a laboratory. Many of these experiments require apparatus, such as that you have used in your study of chemistry to date. A knowledge and understanding of the use of this scientific apparatus is required for successful experimentation and investigations. In an experiment, you will first have to decide on the measurements to be made and then collect together the apparatus and materials required. The quantities you will need to measure most often in a chemistry laboratory will be temperature, mass, volume of liquids as well as gases, and time.

There will be advantages and disadvantages of each method or piece of apparatus, so when you choose which to use, you will have to use your knowledge of the apparatus to decide if it will be suitable. You will have to decide:

- » What apparatus should you use to measure each of these? For example, it sounds silly but you would not use a thermometer to measure the timing of a particular reaction rate!
- » Which piece of apparatus is most suitable for the task in hand? For example, you would not use a large gas syringe to measure out 10 cm³ of liquid: you would use a small measuring cylinder because

it gives you a more accurate measurement of volume and it is easier to use.

- » How do you use the piece of apparatus correctly? Before you use a piece of apparatus, be sure you know how to use it properly. You need to be safe in your working habits and also to ensure you use good techniques, for example, measuring volume of solutions carefully, so that you make and record measurements accurately. If necessary, ask your teacher before you use a particular piece of apparatus if it is unfamiliar to you.

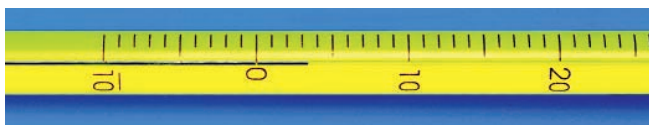
Measurement of time



▲ **Figure 14.1** This stopwatch can be used to measure the time passed in a chemical reaction. The reading is 6.3 s

Experiments involving rates of reaction will require the use of an accurate **stopwatch** – one that measures to a hundredth of a second. The units of time are hours (h), minutes (min) and seconds (s).

Measurement of temperature

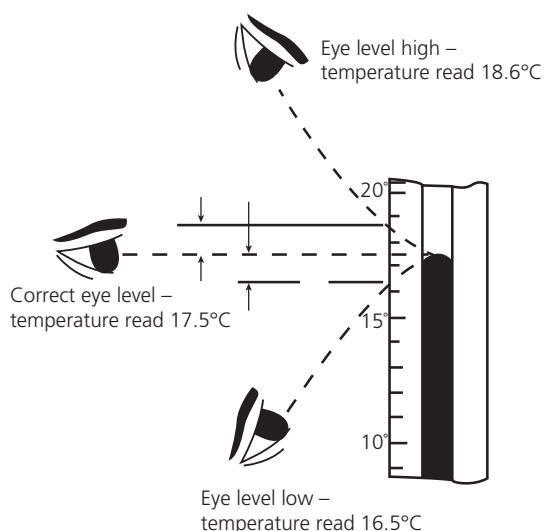


▲ **Figure 14.2** A thermometer can be used to measure temperature. The reading is 3°C

The most commonly used thermometers in a laboratory are alcohol-in-glass. Mercury in-glass thermometers can be used but should be handled with great care. The mercury inside them is poisonous and should not be handled if a thermometer breaks. The units of temperature are those of the Celsius scale. This scale is based on the temperature at which water freezes and boils, that is:

- » the freezing point of water is 0°C
- » the boiling point of water is 100°C.

The usual thermometer used is that shown in Figure 14.2, which measures accurately between -10° and 110°C at 1°C intervals. It may also be possible to read the thermometer to the nearest 0.5°C if the reading is between two of the scale marks. When reading the thermometer, always ensure that your eye is at the same level as the liquid meniscus in the thermometer to ensure there are no parallax effects (Figure 14.3). The meniscus is the way that the liquid curves at the edges of the capillary in which the liquid is held in the thermometer.



▲ **Figure 14.3** Eye should be level with the liquid meniscus

Measurement of mass



▲ **Figure 14.4** An electronic balance can be used to measure the mass of reagents. The reading is 155.12 g

There are many different electronic balances which can be used. The precision of an electronic balance is the size of the smallest mass that can be measured on the scale setting you are using, usually 0.01 g. The units for measuring mass are grams (g) and kilograms (kg): 1 kg = 1000 g.

When using an electronic balance, you should wait until the reading is steady before taking it.

Measurement of volume of liquids



▲ **Figure 14.5** The apparatus shown in the photograph (left to right: volumetric pipette, measuring cylinder and burette) are generally used in different experiments to measure volume accurately

Different experiments involving liquids will require one or other or all the various measuring apparatus available for measuring volume. Figure 14.5 shows three of the most commonly used measuring apparatus.

Acid-base titration

A burette and a pipette are needed in a titration. This technique is often used to test the strength or purity of an acid or an alkali. A titration involves the slow, careful addition of one solution, such as an acid, of a known concentration from a burette to a known volume of another solution, such as an alkali, of unknown concentration held in a flask. The solution in the burette is known as a **titrant**. This addition of acid continues until the reaction reaches neutralisation, which is indicated by a colour change in an indicator. For a further discussion of indicators, see Chapter 8 (p. 117). The known volume in the flask would require the use of a pipette. For example, in an acid-alkali titration, the acid would be the titrant. For a further discussion of titrations, see Chapter 8, p. 128.

A measuring cylinder (sometimes called a mixing or graduated cylinder) is a common piece of laboratory equipment used to measure the volume of a liquid. It has a narrow cylindrical shape. Each marked line on the graduated cylinder shows the amount of liquid that has been measured.

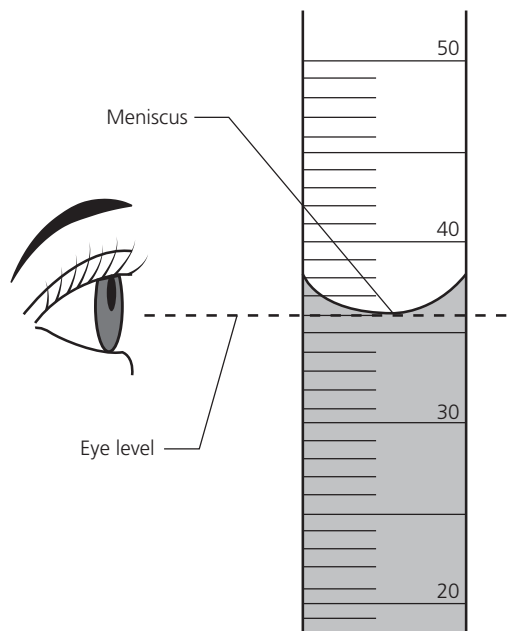
The volume of a liquid is a measure of the amount of space that it takes up. The units of volume are litres (l), cubic decimetres (dm^3) and cubic centimetres (cm^3).

$$1 \text{ litre} = 1 \text{ dm}^3 = 1000 \text{ cm}^3$$

However, some of the manufacturers of apparatus used for measuring volume use millilitres (ml). This is not a problem, however, since $1 \text{ cm}^3 = 1 \text{ ml}$.

When reading the volume using one of these pieces of apparatus, it is important to ensure that the apparatus is vertical and that your eye is level with the meniscus of the liquid being measured, as shown in Figure 14.6. The precision of the measurement will vary depending on the apparatus used. For example, if the smallest scale division on a burette is 0.1 cm^3 , the precision of any volume measured with the burette will be 0.1 cm^3 . It may

also be possible to read the burette to the nearest 0.05 cm^3 if the bottom of the meniscus is between divisions.



▲ **Figure 14.6** The volume level in this measuring cylinder should be read on the dotted line. The reading here is 36 cm^3

Measurement of volume of gases

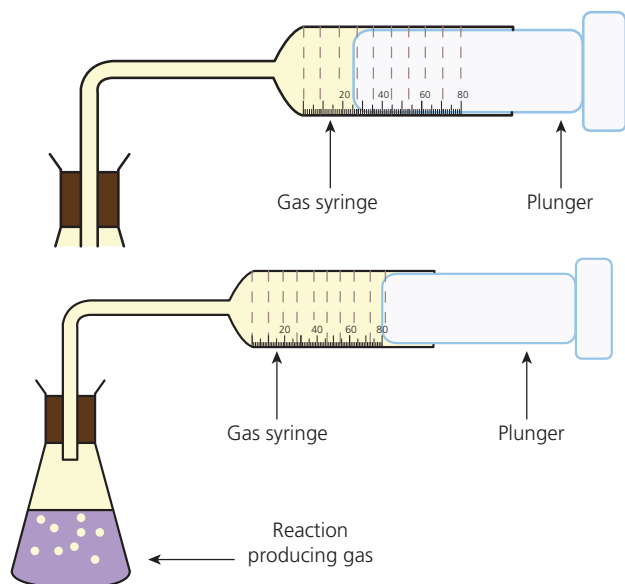
The volume of a gas can be measured with a gas syringe. This is used to measure the amount of gas collected in experiments. They have a maximum volume of 100 cm^3 .



▲ **Figure 14.7** A gas syringe

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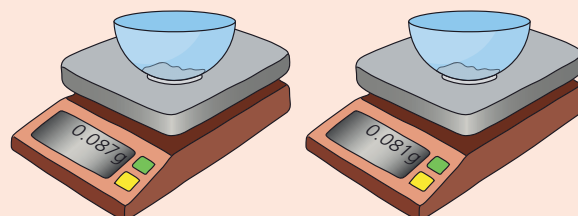
For example, in certain reactions (see p. 106) the reaction rate can be followed by collecting the volume of gas generated with time. The gas syringe is connected by a tube to the flask that is giving off the gas as shown in Figure 14.8.



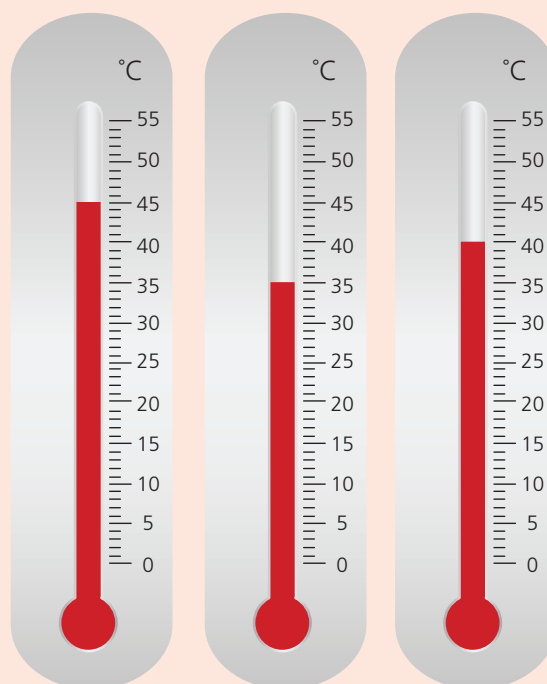
▲ **Figure 14.8** As gas is collected during the reaction, the plunger is forced out and the volume can be read from the scale on the side

The gas syringe has a scale along its length that allows the volume of the gas collected to be measured. As the volume of gas collected increases, the plunger moves further out of the syringe. The volume of collected gas can be measured in a given time, as it increases, and a graph of volume against time can be plotted. This can then be used to show the rate of the reaction (see Chapter 7).

b What are the readings on the two balances?



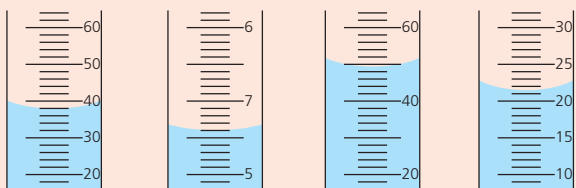
c What are the readings on these three thermometers?



d Why must mercury thermometers be handled with care?

Test yourself

- 1 a i** Copy and complete the following about the units of volume measurement.
 $1 \text{ dm}^3 = \text{_____ cm}^3$
- ii** What are the readings on the following measuring cylinders?



14.2 Separating mixtures

Many mixtures contain useful substances mixed with unwanted material. To obtain these useful substances, chemists often have to separate them from the impurities. Chemists have developed many different methods of separation, particularly for separating compounds from complex mixtures. Which separation method they use depends on what is in the mixture and the properties of the substances present. It also depends on whether the substances to be separated are solids, liquids or gases.

Separating solid/liquid mixtures

If a solid substance is added to a liquid, it may dissolve to form a **solution**. In this case, the solid is said to be soluble and is called the **solute**. The liquid it has dissolved in is called the **solvent**. An example of this type of process is when sugar is added to tea or coffee. What other examples can you think of where this type of process takes place?

Sometimes the solid does not dissolve in the liquid. This solid is said to be insoluble. For example, tea leaves do not dissolve in boiling water when tea is made from them, although the soluble materials from which tea is made are seen to dissolve from them.

Key definitions

A **solvent** is a substance that dissolves a solute.

A **solute** is a substance that is dissolved in a solvent.

A **solution** is a liquid mixture composed of two or more substances.

Filtration

When you pour a cup of tea through a tea strainer, you are carrying out a filtering process. **Filtration** is a common separation technique used in chemistry laboratories throughout the world. It is used when a solid needs to be separated from a liquid. For example, sand can be separated from a mixture with water by filtering through filter paper as shown in Figure 14.9.

The filter paper contains holes that, although too small to be seen, are large enough to allow the molecules of water through but not the sand particles. It acts like a sieve. The sand gets trapped in the filter paper and the water passes through it. The sand is called the **residue** and the water is called the **filtrate**.



▲ **Figure 14.9** It is important when filtering not to overfill the filter paper

Key definitions

Residue is a substance that remains after evaporation, distillation, filtration or any similar process.

Filtrate is a liquid or solution that has passed through a filter.

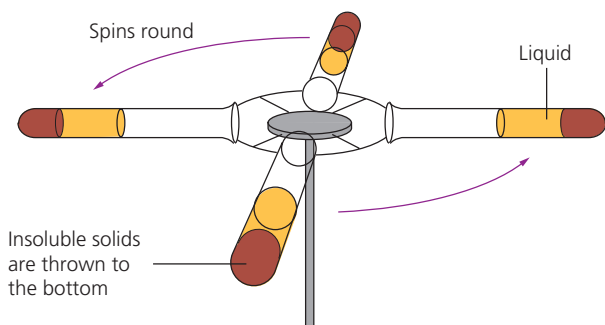
➔ Going further

Decanting

Vegetables do not dissolve in water. When you have boiled some vegetables, it is easy to separate them from the water by pouring it off. This process is called **decanting**. This technique is used quite often to separate an insoluble solid, which has settled at the bottom of a flask, from a liquid.

Centrifuging

Another way to separate a solid from a liquid is to use a centrifuge. This technique is sometimes used instead of filtration. It is usually used when the solid particles are so small that they spread out (disperse) throughout the liquid and remain in suspension. They do not settle to the bottom of the container, as heavier particles would do, under the force of gravity. The technique of centrifuging or centrifugation involves the suspension being spun round very fast in a centrifuge so that the solid gets flung to the bottom of the tube (Figure 14.10).

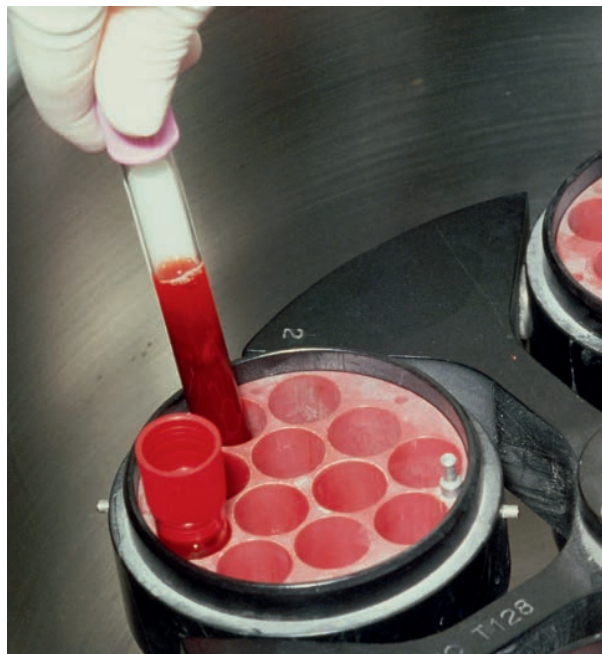


a The sample is spun round very fast and the solid is flung to the bottom of the tube



b An open centrifuge

▲ **Figure 14.10**



▲ **Figure 14.11** Whole blood (top) is separated by centrifuging into blood cells and plasma (bottom)

The pure liquid can be decanted after the solid has been forced to the bottom of the tube. This method of separation is used extensively to separate blood cells from blood plasma (Figure 14.11). In this case, the solid particles (the blood cells) are flung to the bottom of the tube, allowing the liquid plasma to be decanted.

Evaporation

If a solid has dissolved in a liquid, it cannot be separated by filtering or centrifuging. Instead, the solution can be heated so that the liquid **evaporates** completely and leaves the solid behind. For example, the simplest way to obtain salt from its solution is by slow evaporation, as shown in Figure 14.12.



▲ **Figure 14.12** Apparatus used to slowly evaporate a solvent

You may need to choose a suitable solvent to separate a mixture. For example, if one of the substances in a mixture is soluble in water, you could use water to dissolve the soluble substance, filter the mixture to separate the insoluble substance, and then evaporate the water to retrieve the soluble substance.

Crystallisation

In many parts of the world, salt is obtained from sea water on a vast scale. This is done by using the heat of the Sun to evaporate the water to leave a saturated solution of salt known as brine. A **saturated solution** is defined as one that contains the maximum concentration of a solute dissolved in the solvent. It is important to be aware that solubility of a substance varies with temperature, so the maximum concentration will vary at different temperatures. More of the solute will dissolve at a higher temperature. When the solution is saturated, the salt begins to **crystallise** and it is removed using large scoops (Figure 14.13).

Key definition

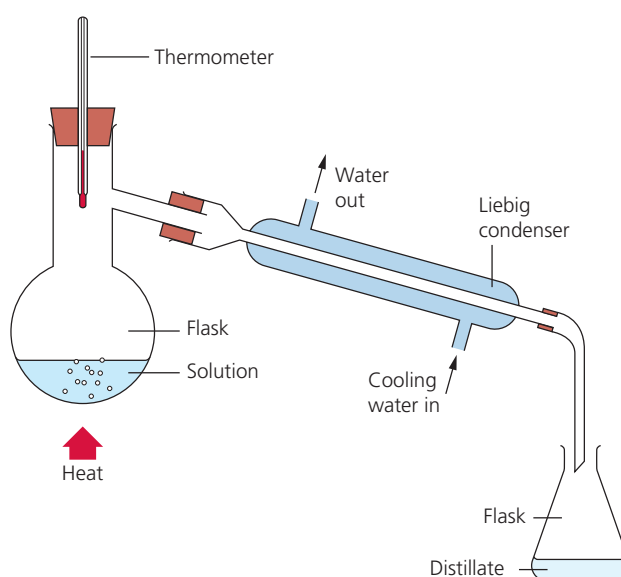
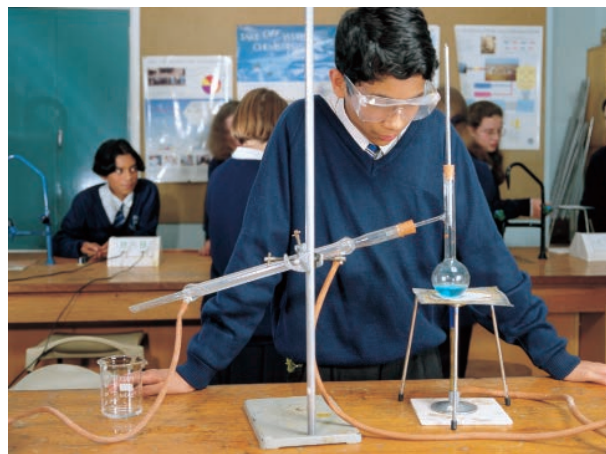
A **saturated solution** is a solution containing the maximum concentration of a solute dissolved in the solvent at a specified temperature.



▲ **Figure 14.13** Salt is obtained in north-eastern Brazil by evaporation of sea water

Simple distillation

If we want to obtain the solvent from a solution, then the process of **distillation** can be carried out. The apparatus used in this process is shown in Figure 14.14.



▲ **Figure 14.14** Water can be obtained from salt water by distillation

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Water can be obtained from salt water using this method. The solution is heated in the flask until it boils. The steam rises into the Liebig condenser, where it condenses back into water. The salt is left behind in the flask.

➔ Going further

In hot and arid countries, such as Saudi Arabia, this sort of technique is used on a much larger scale to obtain pure water for drinking (Figure 14.15). This process is carried out in a desalination plant.



▲ **Figure 14.15** This desalination plant produces large quantities of drinking water in Saudi Arabia

Separating liquid/liquid mixtures

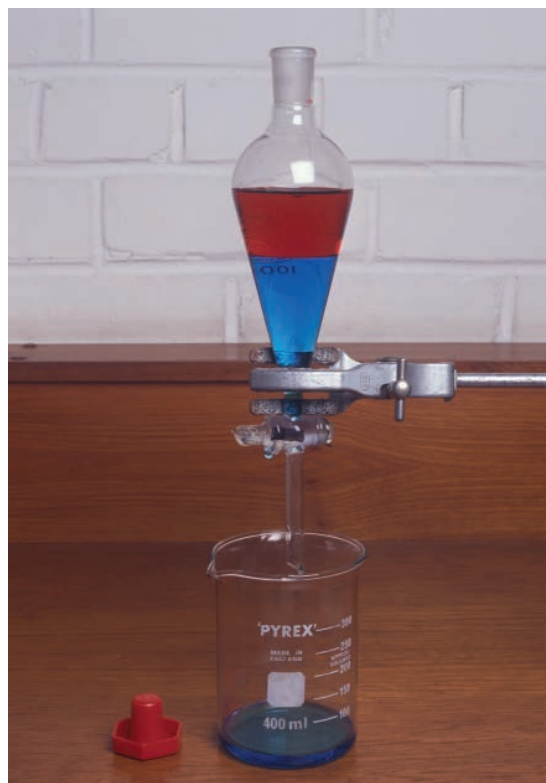
In recent years, there have been several oil tanker accidents that have led to environmental disasters, just like the one shown in Figure 14.16. These have resulted in millions of litres of oil being washed into the sea. Oil and water do not mix easily. They are said to be **immiscible**. When cleaning up disasters of this type, a range of chemicals can be added to the oil to make it more soluble. This results in the oil and water mixing with each other. They are now said to be **miscible**. The following techniques can be used to separate mixtures of liquids.



▲ **Figure 14.16** Millions of litres of oil can be spilled from oil tankers and cleaning up is a slow and costly process

Immiscible liquids

If two liquids are immiscible, they can be separated using a separating funnel. The mixture is poured into the funnel and the layers allowed to separate. The lower layer can then be run off by opening the tap as shown in Figure 14.17.



▲ **Figure 14.17** The blue liquid is more dense than the red liquid and so sinks to the bottom of the separating funnel. When the tap is opened, the blue liquid can be run off.

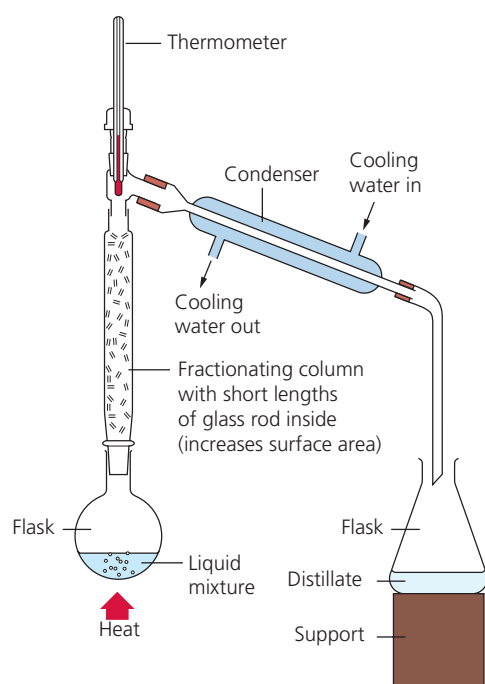
Miscible liquids

If miscible liquids are to be separated, then this can be done by fractional distillation. The apparatus used for this process is shown in Figure 14.18 and could be used to separate a mixture of ethanol and water.

Fractional distillation relies upon the liquids having different boiling points. When an ethanol and water mixture is heated, the vapours of ethanol and water boil off at different temperatures and can be condensed and collected separately.

Ethanol boils at 78°C whereas water boils at 100°C . When the mixture is heated, the vapour produced is mainly ethanol with some steam. Because water has the higher boiling point of the two, it condenses out from the mixture with ethanol. This

is what takes place in the fractionating column. The water condenses and drips back into the flask while the ethanol vapour moves up the column and into the condenser, where it condenses into liquid ethanol and is collected in the receiving flask as the **distillate**. When all the ethanol has distilled over, the temperature reading on the thermometer rises steadily to 100°C , showing that the steam is now entering the condenser. At this point the receiver can be changed and the condensing water can now be collected.



▲ **Figure 14.18** Typical fractional distillation apparatus

Fractional distillation is used to separate miscible liquids such as those in petroleum (crude oil) (see p. 89, Figure 6.4a and b), and the technique can also separate individual gases, such as nitrogen, from the mixture we call air (Figure 14.19).



▲ **Figure 14.19** Gases from the air are extracted in this fractional distillation plant

➔ Going further

Separating solid/solid mixtures

You saw in Chapter 2 (see p. 19, Figure 2.9) that it was possible to separate iron from sulfur using a magnet. In that case, we were using one of the physical properties of iron, that is, the fact that it is magnetic. In a similar way, it is possible to separate scrap iron from other metals by using a large electromagnet like the one shown in Figure 14.20.

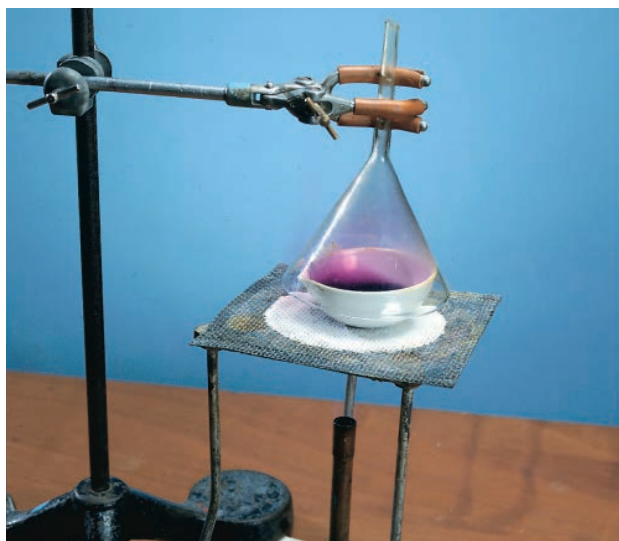
► **Figure 14.20** Magnetic separation of iron-containing materials



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It is essential that, when separating solid/solid mixtures, you pay particular attention to the individual physical properties of the components. If, for example, you wish to separate two solids, one of which sublimes, then this property should dictate the method you employ.

In the case of an iodine/common salt mixture, the iodine sublimes but salt does not. Iodine can be separated by heating the mixture in a fume cupboard as shown in Figure 14.21. The iodine sublimes and re-forms on the cool inverted funnel.



▲ **Figure 14.21** Apparatus used to separate an iodine/salt mixture. The iodine sublimes on heating

Solvent extraction

Sugar can be obtained from crushed sugar cane by adding water. The water dissolves the sugar from the sugar cane (Figure 14.22). This is an example of solvent extraction. In a similar way, some of the green substances can be removed from ground-up grass using ethanol. The substances are extracted from a mixture by using a solvent which dissolves only those substances required.



▲ **Figure 14.22** Cutting sugar cane, from which sugar can be extracted by using a suitable solvent

Test yourself

- 2 Which experimental technique(s)/processes would you use to obtain:
- a the silver bromide precipitate produced from the reaction of silver nitrate solution with sodium bromide solution?
 - b pure water from an aqueous solution of iron(II) sulfate?
 - c common salt from a mixture of the solids salt and sand?
 - d nitrogen gas (boiling point -195.8°C) from liquid air?
 - e calcium chloride solid from calcium chloride solution?

Chromatography

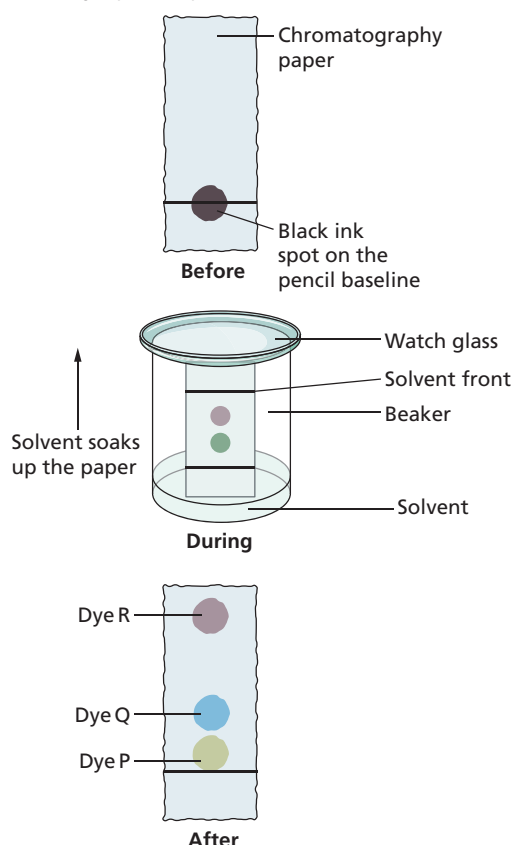
Sometimes we need to separate two or more solids that are soluble; for example, mixtures of soluble coloured

substances such as inks and dyes. A technique called **chromatography** is widely used to separate these materials so that they can be identified.

There are several types of chromatography; however, they all follow the same basic principles. The simplest kind is paper chromatography. To separate the different coloured dyes in a sample of black ink, a pencil line is drawn about 1 cm from the bottom of a piece of chromatography paper. This is called the baseline. Do not use a pen to draw the baseline as the dyes in its ink will interfere with the results. A spot of the ink is put onto the pencil line on the chromatography paper. This paper is then put into a suitable container, such as a beaker, containing a suitable solvent, ensuring that the solvent level does not reach the pencil line as shown in Figure 14.23. A lid is placed over the container to reduce evaporation of the solvent.



a Chromatographic separation of black ink



b The black ink separates into three dyes: P, Q and R

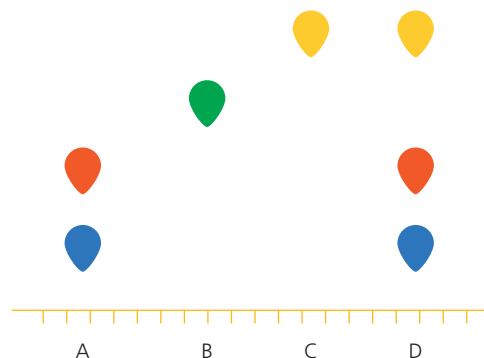
▲ Figure 14.23

As the solvent moves up the paper, the dyes are carried with it and begin to separate. They separate because the substances have different solubilities in the solvent and are absorbed to different degrees by the chromatography paper. As a result, they are separated gradually as the solvent moves up the paper. The chromatogram in Figure 14.23b shows how the ink contains three dyes, P, Q and R.

Chromatography is used extensively in medical research and forensic science laboratories to separate a variety of mixtures.

? Worked example

Several different dyes were spotted on chromatography paper and set up for chromatography. The chromatogram produced is shown below.



Which of the dyes are pure substances and which are mixtures?

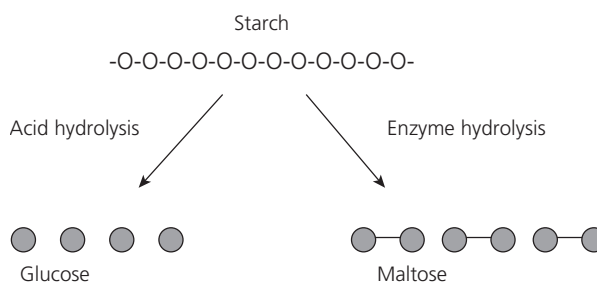
The single spots show they are a single dye. So dyes B and C are single dyes.

The dyes that have produced multiple spots are mixtures. So dyes A and D are mixtures of dyes.

➔ Going further

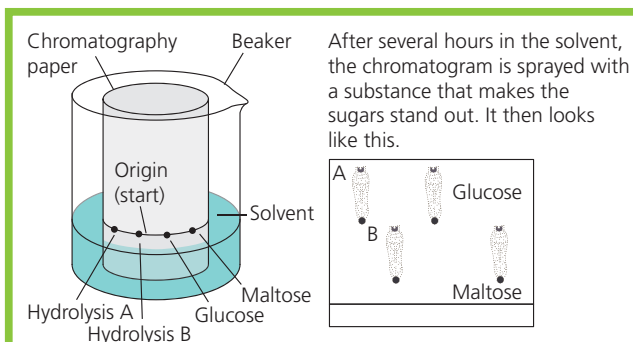
Paper chromatography of the products of starch hydrolysis

This technique can be used to identify the products of a reaction. For example, starch can be broken up using enzymes in water solution or water in dilute acid. This type of reaction is called hydrolysis. However, the different methods produce different products, as shown in Figure 14.24.



▲ **Figure 14.24** Summary of starch hydrolysis

The products of these reactions can be identified using chromatography. The chromatography paper is 'spotted' with the samples as well as pure glucose and maltose. When this is developed after some hours in the solvent, the spots may be identified (Figure 14.25).



▲ **Figure 14.25** The products of hydrolysis may be identified by chromatography

? Worked example

Which substance has been produced by which hydrolysis in Figure 14.25?

Acid hydrolysis – sample A on the chromatogram. That spot has risen to the same height as the substance glucose.

Enzyme hydrolysis – sample B on the chromatogram. That spot has risen to the same height as the substance maltose.

Numerical measurements (retardation factors) known as **R_f values** can be obtained from chromatograms. An R_f value is defined as the ratio of the distance travelled by the solute (for example, P, Q or R) to the distance travelled by the solvent from the pencil line.

$$R_f = \frac{\text{distance travelled by solute}}{\text{distance travelled by solvent}}$$

The substances to be separated do not have to be coloured. Colourless substances can be made visible by spraying the chromatogram with a **locating agent**. The locating agent will react with the colourless substances to form a coloured product. In other situations, the position of the substances on the chromatogram may be located using ultraviolet light.

? Worked example

The following values were obtained for the chromatogram produced for the amino acid glycine.

Distance travelled by glycine = 2.5 cm

Distance travelled by the solvent = 10.0 cm

$$R_f = \frac{\text{distance travelled by solute}}{\text{distance travelled by solvent}}$$

$$R_f = \frac{2.5}{10.0} = 0.25$$

R_f values are catalogued in data books for a vast array of substances. So R_f values of unknown substances can be compared with values inside a data book to identify the unknown substance.

Criteria for purity

In chemistry, a pure substance is one which contains a single element or compound not mixed with any other substance. The measure of whether a substance is pure is known as purity. This is different to the everyday meaning. For example, in the food industry, if bottles or cartons which are labelled as 'pure' apple juice are sold in shops then the label means that the contents are just apple juice with no other substances being added. However, the apple juice is not pure in the chemical sense because it contains different substances mixed together. The substances added here may include a preservative as well as other substances which are harmless to the person buying it.

However, in the pharmaceuticals industry, purity is essential because there could be significant dangers to health if any other substances were found in medicines. Drugs (pharmaceuticals) are manufactured to a very high degree of purity (Figure 14.26). To ensure that the highest possible purity is obtained, the drugs are dissolved in a suitable solvent and subjected to fractional crystallisation.

Purity can have an important effect on the chemical properties of a substance. Pure substances will form predictable products in chemical reactions. This reliability comes from the fact that there are no impurities present that could start other reactions. Reactions due to impurities may interfere with the reaction that is being studied by a chemist, so chemists often use substances which have a high degree of purity when conducting chemical research.

Throughout the pharmaceutical, food and chemical industries, it is essential that the substances used are pure. The purity of a substance can be assessed by:

- » its melting point – if it is a pure solid, it will have a sharp melting point. If an impurity is present then melting takes place over a range of temperatures to show it is a mixture of two or more substances.
- » its boiling point – if it is a pure liquid, the temperature will remain steady at its boiling point. If the substance is impure then the mixture will boil over a temperature range.
- » chromatography – if it is a pure substance, it will produce only one well-defined spot on a chromatogram. If impurities are present then several spots will be seen, as on the chromatogram in Figure 14.23, p. 231.



▲ **Figure 14.26** These pharmaceuticals must have been through a lot of testing for purity before they can be sold in a pharmacy

Test yourself

- 3 The following values were obtained for the chromatogram produced for the amino acid proline.

Distance travelled by proline = 6.0 cm

Distance travelled by the solvent = 10.0 cm

Calculate the R_f value for proline.

14.3 Qualitative analysis

The branch of chemistry that deals with the identification of elements or grouping of elements present in a sample is called qualitative chemical analysis, or qualitative analysis for short. It does not deal with quantities.

The techniques employed in qualitative analysis vary in their complexity, depending on the nature of the sample under investigation. In some cases, it is only necessary to confirm the presence of certain elements or groups for which specific chemical tests, or 'spot' tests, applicable directly to the sample, may be available. More often, the sample is a complex mixture, and a systematic analysis must be made in order that all the component parts may be identified. Often, the first simple stages of qualitative analysis require no apparatus at all. Things like colour and smell can be observed without any need for apparatus.

The following summary collects together information from throughout the book which would allow you to carry out qualitative analysis. The material which is of extreme importance as far as your revision is concerned is that given in the following tables.

- » Table 14.2 Characteristic flame colours of some metal ions
- » Table 14.3 Effect of adding sodium hydroxide solution to solutions containing various metal ions
- » Table 14.4 Effect of adding aqueous ammonia to solutions containing various metal ions
- » Table 14.5 A variety of tests for aqueous anions
- » Table 14.6 Tests for gases

Appearance or smell

A preliminary examination of the substance will give you a start. The appearance or smell of a substance can often indicate what it might contain (Table 14.1).

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▼ **Table 14.1** Deductions that can be made from a substance's appearance or smell

Observation on substance	Indication
Black powder	Carbon, or contains O^{2-} ions (as in CuO), or S^{2-} ions (as in CuS)
Pale green crystals	Contains Fe^{2+} ions (as in iron(II) salts)
Dark green crystals	Contains Ni^{2+} ions (as in nickel(II) salts)
Blue or blue-green crystals	Contains Cu^{2+} ions (as in copper(II) salts)
Yellow-brown crystals	Contains Fe^{3+} ions (as in iron(III) salts)
Smell of ammonia	Contains NH_4^+ ions (as in ammonium salts)

Flame colours

If a wooden splint, which has been soaked in an aqueous metal ion solution, is held in a colourless Bunsen flame, the flame colour can become coloured (Figure 14.27). Certain metal ions may be detected in their compounds by observing their flame colours (Table 14.2).

▼ **Table 14.2** Characteristic flame colours of some metal ions

	Metal	Flame colour
Group I (1+ ion)	Lithium	Red
	Sodium	Yellow
	Potassium	Lilac
Group II (2+ ion)	Calcium	Orange-red
	Barium	Light green
Others	Copper (as Cu^{2+})	Blue-green

A flame colour is obtained as a result of the electrons in the particular ions being excited when they absorb energy from the flame which is then emitted as visible light. The different electronic configurations of the different ions, therefore, give rise to the different colours.

Tests for aqueous cations

Effect of adding dilute sodium hydroxide solution

Aqueous sodium hydroxide can be used to identify salts of Al^{3+} , Ca^{2+} , Cr^{3+} , Cu^{2+} , Fe^{2+} , Fe^{3+} and Zn^{2+} when present in aqueous solutions. All metal cations form insoluble hydroxides when sodium hydroxide solution is added to them. The colour of the precipitate and its behaviour in excess sodium hydroxide solution will help identify the metal present (Table 14.3).

▼ **Table 14.3** Effect of adding sodium hydroxide solution to solutions containing various metal ions

Added dropwise	To excess	Likely cation
White precipitate	Precipitate is soluble in excess, giving a colourless solution	Al^{3+} , Zn^{2+}
White precipitate	Precipitate is insoluble in excess	Ca^{2+}
Green precipitate	Precipitate is insoluble in excess	Cr^{3+}
Light blue precipitate	Precipitate is insoluble in excess	Cu^{2+}
Green precipitate	Precipitate is insoluble in excess, turns brown near the surface on standing	Fe^{2+}
Red-brown precipitate	Precipitate is insoluble in excess	Fe^{3+}

▲ **Figure 14.27** The green colour is characteristic of copper

In the case of ammonium salts containing the ammonium ion, NH_4^+ , ammonia gas is produced on warming. The ammonium cation does not form an insoluble hydroxide. However, it forms ammonia and water upon heating.



▲ **Figure 14.28** Addition of sodium hydroxide will show the difference between Fe^{2+} and Fe^{3+} in aqueous solution

Effect of adding dilute ammonia solution

Ammonia gas dissolved in water is usually known as aqueous ammonia. The solution is only weakly alkaline, which results in a relatively low concentration of hydroxide ions. Aqueous ammonia can be used to identify salts of Al^{3+} , Ca^{2+} , Cr^{3+} , Cu^{2+} , Fe^{2+} , Fe^{3+} and Zn^{2+} ions. The colour of the precipitate or solution formed identifies the metal present (Table 14.4).

▼ **Table 14.4** Effect of adding aqueous ammonia to solutions containing various metal ions

Added dropwise	To excess	Cation present
White precipitate	Precipitate is insoluble in excess	Al^{3+}
No precipitate or very slight white precipitate	No change	Ca^{2+}
Green precipitate	Precipitate is insoluble in excess	Cr^{3+}
Light blue precipitate	Precipitate is soluble in excess, giving a dark blue solution	Cu^{2+}
Green precipitate	Precipitate is insoluble in excess, turns brown near the surface on standing	Fe^{2+}
Red-brown precipitate	Precipitate is insoluble in excess	Fe^{3+}
White precipitate	Precipitate is soluble in excess, giving a colourless solution	Zn^{2+}



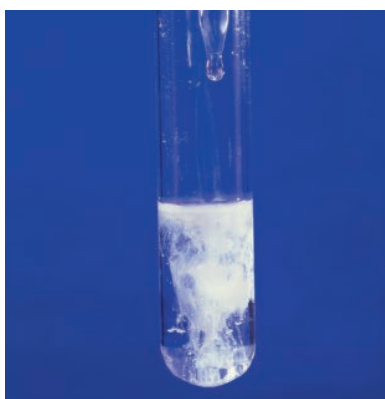
▲ **Figure 14.29** When aqueous ammonia is added to a solution containing Cu^{2+} ions, the solution forms a gelatinous light blue precipitate. As more is added to excess, the precipitate dissolves, forming a dark blue clear solution

Tests for aqueous anions

Table 14.5 shows a variety of tests for aqueous anions.

▼ **Table 14.5** Tests for aqueous anions

Anion	Test	Test result
Carbonate (CO_3^{2-})	Add dilute acid	Effervescence is seen as carbon dioxide produced
Chloride (Cl^-) [in solution]	Acidify with dilute nitric acid, then add aqueous silver nitrate	A white precipitate is produced
Bromide (Br^-) [in solution]	Acidify with dilute nitric acid, then add aqueous silver nitrate	A cream precipitate is produced
Iodide (I^-) [in solution]	Acidify with dilute nitric acid, then add aqueous silver nitrate	A yellow precipitate is produced
Nitrate (NO_3^-) [in solution]	Add aqueous sodium hydroxide, then aluminium foil; warm carefully	Ammonia gas is produced
Sulfate (SO_4^{2-}) [in solution]	Acidify, then add aqueous barium nitrate or barium chloride	A white precipitate is produced
Sulfite (SO_3^{2-})	Add a small volume of acidified aqueous potassium manganate(VII)	The acidified aqueous potassium manganate(VII) changes colour from purple to colourless



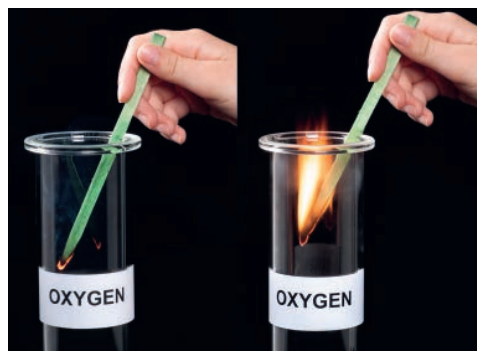
▲ **Figure 14.30** The test for sulfate ions

Tests for gases

Table 14.6 shows the common gases which may be produced during qualitative analysis and tests which can be used to identify them. These tests are used in conjunction with the tests shown above.

▼ **Table 14.6** Tests for gases

Gas	Colour (odour)	Effect of moist indicator paper	Test
Hydrogen (H_2)	Colourless (odourless)	No effect – neutral	'Pops' in the presence of a lighted splint
Oxygen (O_2)	Colourless (odourless)	No effect – neutral	Relights a glowing splint
Carbon dioxide (CO_2)	Colourless (odourless)	Pink – weakly acidic	Turns limewater a cloudy white
Ammonia (NH_3)	Colourless (very pungent smell)	Blue – alkaline	Turns damp red litmus blue
Sulfur dioxide (SO_2)	Colourless (very choking smell)	Red – acidic	Turns acidified potassium dichromate(VI) from orange to green Turns acidified potassium manganate(VII) from purple to colourless
Chlorine (Cl_2)	Yellow-green (very choking smell)	Bleaches moist indicator paper after it initially turns pale pink	Bleaches damp litmus paper
Water (H_2O)	Colourless (odourless)	No effect – neutral	Turns blue cobalt chloride paper pink Turns anhydrous copper(II) sulfate from white to blue



▲ **Figure 14.31** Testing for oxygen gas



Practical skills

Testing a compound

For safe experiments/demonstrations which are related to this chapter, please refer to the Cambridge IGCSE Chemistry Practical Skills Workbook, which is also part of this series.

Safety

- Eye protection must be worn.

A student was given small quantities of a compound labelled A. After putting on their eye protection, the student carried out a variety of tests to help them identify compound A. Table 14.7

shows the tests the student did on compound A and the observations made.

Complete the table below by giving the possible conclusions to tests (a), (b), (c) and (d). Suggest both the test and observation that lead to the conclusion in test (d).

- 1 What is the name and formula for compound A?
- 2 It was suggested by the teacher that the final test (d) should be carried out in a fume cupboard. Explain why this procedure should be carried out in a fume cupboard.

▼ **Table 14.7** Tests on compound A and observations made

Test	Observation	Conclusion
(a) Compound A was dissolved in water and the solution produced divided into three parts for the tests (b), (c) and (d)	When dissolved in water, a colourless solution was produced.	
(b) (i) To the first sample, a small amount of aqueous sodium hydroxide was added. (ii) An excess of aqueous sodium hydroxide was added to the mixture from (i).	(i) White precipitate was produced. (ii) The white precipitate dissolved in excess sodium hydroxide.	
(c) (i) To the second part, a small amount of aqueous ammonia was added. (ii) An excess of aqueous ammonia was then added to the mixture from (i).	(i) A white precipitate was formed. (ii) The white precipitate was insoluble in excess.	
(d) Aqueous sodium hydroxide solution was added to some of the solution of A followed by some aluminium foil. The mixture was then warmed carefully.	A gas was produced which turned moist litmus paper blue.	

Test yourself

- 4 What will be the colour of the precipitate formed with silver nitrate for the following?
a chloride, Cl^- **c** iodide, I^-
b bromide, Br^-
 Choose from yellow, white, green, cream, blue.
- 5 Match the gas shown in the table to the correct test for that gas and the positive result.

Gas	Test result
1 Oxygen	a Damp indicator paper turns white (bleached)
2 Carbon dioxide	b Burning splint produces a 'pop' sound when plunged into a test tube of gas
3 Hydrogen	c Damp litmus paper turns blue
4 Ammonia	d Limewater turns milky white
5 Chlorine	e Relights a glowing splint

Revision checklist

After studying Chapter 14 you should be able to:

- ✓ Name appropriate apparatus for the measurement of time, temperature, mass and volume.
 - ✓ Suggest advantages and disadvantages of experimental methods and apparatus.
 - ✓ Know the terms 'solvent', 'solute', 'solution', 'saturated solution', 'residue' and 'filtrate'.
 - ✓ Describe an acid–base titration and know how to identify the end-point of a titration.
 - ✓ Describe how paper chromatography is used to separate mixtures of soluble substances.
- ✓ Describe the use of locating agents when separating mixtures.
- ✓ Interpret simple chromatograms.
- ✓ State and use the equation:
$$R_f = \frac{\text{distance travelled by solute}}{\text{distance travelled by solvent}}$$
- ✓ Describe and explain methods of separation and purification.
 - ✓ Suggest suitable separation and purification techniques.
 - ✓ Identify substances and assess their purity using melting point and boiling point information.
 - ✓ Describe tests using aqueous sodium hydroxide and aqueous ammonia to identify aqueous cations mentioned in this chapter in Tables 14.3 and 14.4.
 - ✓ Describe the use of a flame test to identify the cations mentioned in this chapter in Table 14.2.
 - ✓ Describe tests to identify the anions mentioned in this chapter in Table 14.5.
 - ✓ Describe tests to identify the gases mentioned in this chapter in Table 14.6.

Exam-style questions

- 1 a Which of the following:
cm³, kilograms, hours, degrees Celsius, dm³,
minutes, seconds, grams
are units of:
- i time [1]
 - ii temperature [1]
 - iii volume [1]
 - iv mass? [1]
- b In experiments, you will require the use of accurate measuring instruments. State the precision you would expect from the following measuring instruments:
- i a stopwatch [1]
 - ii a thermometer [1]
 - iii an electronic balance [1]
 - iv a burette. [1]
- 2 State the method which is most suitable for separating the following:
- a oxygen from liquid air [1]
 - b red blood cells from plasma [1]
 - c petrol and kerosene from crude oil [1]
 - d coffee grains from coffee solution [1]
 - e pieces of steel from engine oil [1]
 - f amino acids from fruit juice solution [1]
 - g ethanol and water. [1]
- 3 Three solid dyes are to be separated. One is yellow, one is red and one blue. The following facts are known about the dyes.
- a The blue and yellow dyes are soluble in cold water.
 - b The red dye is insoluble.
 - c When chalk is added to the solution of blue and yellow dyes, the mixture stirred and then filtered, the filtrate is blue and the residue is yellow.
 - d When the residue is mixed with alcohol and the mixture stirred and filtered, the filtrate is yellow and the residue is white.
- Describe an experiment to obtain solid samples of each of the dyes from a mixture of all three. [10]

- 4 Forensic scientists recovered several samples from a crime scene. Chromatography was carried out on these samples. The scientists placed the samples onto chromatography paper. The chromatogram produced was sprayed with a *locating agent*. The R_f values were then measured for each of the samples taken.

- a What do you understand by the term:
- i locating agent? [2]
 - ii R_f value? [2]
- b The samples were thought to be amino acids. Use your research skills to find out the normal locating agent used for amino acids. [1]
- c The table below shows R_f values for several amino acids thought to be at the crime scene and for three samples.

Amino acid	R_f value
Alanine	0.65
Glycine	0.25
Threonine	0.57
Proline	0.60
Lysine	0.16
Methionine	0.50
Sample 1	0.64
Sample 2	0.18
Sample 3	0.24

- i Identify the samples taken from the crime scene using the data from the table. [3]
 - ii Explain how you made your choices in part i. [1]
- 5 For each of the following pairs of substances, describe a chemical test you would carry out to distinguish between them.
- a potassium sulfate and potassium sulfite [3]
 - b ammonium chloride and aluminium chloride [3]
 - c zinc nitrate and calcium nitrate [3]
 - d sodium chloride and sodium iodide [3]
 - e iron(II) sulfate and copper(II) sulfate [3]
- 6 Sodium carbonate hydrate contains water of crystallisation. When it is heated strongly, it gives off the water of crystallisation, which can be collected.
- a The substance left behind is anhydrous sodium carbonate. Describe a chemical test to show that this substance contains sodium (cation) and carbonate (anion). [2]
 - b Describe two chemical tests to show that the colourless liquid produced and collected is indeed water. [4]
 - c Describe another test to show that the colourless liquid given off in this experiment is pure water. [1]