

# Experimental techniques and chemical analysis

Mark scheme and worked solutions for 30 questions: 15 multiple choice, 15 structured.

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**1** Core Experimental design

[1]

Answer: C

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## Worked solution

Think about what each piece of apparatus is designed to do. A volumetric pipette measures one fixed volume very accurately, but you cannot use it to add "a little at a time" and read how much has been added. A measuring cylinder can measure different volumes, but its scale is too coarse to read to  $0.05\text{ cm}^3$ . A gas syringe measures the volume of a gas. A burette has a tap so liquid can be added drop by drop, and its scale can be read to the nearest  $0.05\text{ cm}^3$ , which is why it is used in titrations.

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**2** Core Experimental design

[1]

Answer: B

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## Worked solution

"Saturated" means no more solute can dissolve: the solution holds the maximum concentration of solute. However, how much solute can dissolve changes with temperature (for most solids, more dissolves in hot water than in cold water). So a saturated solution must always be described at a stated temperature. That rules out C. Options A and D just compare the masses of solute and solvent; a solution can be saturated even when it contains far less solute than solvent.

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**3** Core Acid-base titrations

[1]

Answer: D

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## Worked solution

Start by deciding what is in the flask at the beginning: hydrochloric acid, so methyl orange is red. Alkali is then added from the burette. At the end-point the acid has just been neutralised and the indicator turns orange (one more drop of alkali would make it yellow). So the change is red to orange. If the flask had contained the alkali and the acid were added from the burette, the change would be yellow to orange instead. Blue and colourless are the colours of thymolphthalein, which is a different indicator.

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**4****Core**

Chromatography

[1]

**Answer: B**

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**Worked solution**

The baseline marks where the samples start, so that distances can be measured. Ink is itself a mixture of soluble dyes. If the line were drawn in ink, these dyes would dissolve in the solvent and travel up the paper, producing extra spots that confuse the results. Pencil does not dissolve, so the line stays put and does not interfere. The pencil has no effect on how fast the solvent rises and does not react with the substances.

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**5****Supplement**

Chromatography

[1]

**Answer: C**

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**Worked solution**

Chromatography separates the colourless substances just as it would separate coloured ones, but you cannot see where they have gone. The answer is to treat the dried chromatogram with a locating agent. This reacts with the substances to produce coloured spots, so their positions can be seen and marked. Using more sample does not make a colourless substance coloured. A solvent in which the substances are insoluble would mean nothing moves off the baseline, so there would be no separation.

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**6****Core**

Separation and purification

[1]

**Answer: D**

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**Worked solution**

Look at the information given. Both substances are liquids and they mix completely, so there is no solid to filter off or crystallise. Their boiling points are different (78 °C and 100 °C). Liquids that mix but have different boiling points are separated by fractional distillation: the liquid with the lower boiling point (ethanol) reaches the top of the fractionating column and distils over first.

Answer: A

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**Worked solution**

Flame test colours have to be learned:

- lithium,  $\text{Li}^+$ : red
- sodium,  $\text{Na}^+$ : yellow
- potassium,  $\text{K}^+$ : lilac
- calcium,  $\text{Ca}^{2+}$ : orange-red
- barium,  $\text{Ba}^{2+}$ : light green
- copper(II),  $\text{Cu}^{2+}$ : blue-green

Light green matches barium. Take care not to confuse it with copper(II), which gives a blue-green flame.

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**Mark scheme**

(a) [1]

- gas syringe (1)

**ALLOW** (upturned / inverted) measuring cylinder or burette filled with water (over water)

**REJECT** measuring cylinder alone (not inverted over water); beaker

(b) [1]

- stop-watch (1)

**ALLOW** stop-clock / timer / stopwatch

(c) [1]

- balance (1)

**ALLOW** (top-pan / electronic / digital) balance / scales

(d) [1]

- volume measured is not accurate / not precise (1)

**ALLOW** the markings / scale on a beaker are only approximate / widely spaced

**IGNORE** "it is hard to read" on its own

(e) [1]

- any one from (1):

- it gives the actual amount (volume) of gas produced / gives a measurement (with units)
- bubbles can be different sizes
- bubbles can be produced too quickly to count
- it is more accurate

**IGNORE** "it is easier" on its own

**Worked solution**

Each quantity needs its own measuring instrument: volume of gas with a gas syringe (or an upturned measuring cylinder full of water), time with a stop-watch and mass with a balance.

A beaker is designed to hold liquids, not to measure them. Its markings are only a rough guide, so the volume of acid would not be accurate. A measuring cylinder would be better.

Counting bubbles is a poor method because bubbles are not all the same size and can form too fast to count. Measuring the volume of gas in a gas syringe gives a real measurement in  $\text{cm}^3$  that can be compared between experiments.

**Mark scheme**

(a)

[3]

- any three from (1 each, max 3):
  - draw a baseline in pencil near the bottom of the paper
  - put small spots / drops of each sample (E, P, Q, R and S) on the baseline
  - stand the paper in a container of the solvent with the solvent level below the baseline
  - (put a lid on and) leave until the solvent has risen most of the way up the paper
  - remove the paper, mark the solvent front and let the paper dry

**REJECT** baseline drawn in ink**REJECT** solvent level above the baseline

(b)(i)

[2]

- P and R (1)
- the spots of P and R are at the same distances (2.1 cm and 5.4 cm) as the two spots of E / they match spots in E (1)

**REJECT** answers that include S or Q (0 for the first mark)**ALLOW** second mark for: S is not present because E has no spot at 6.7 cm, if P and R are given

(b)(ii)

[1]

- S (1)

**Worked solution**

(a) The baseline is drawn in pencil because pencil does not dissolve and move. Small spots of each sample are placed on the line. The paper is dipped into the solvent so that the solvent is below the baseline; otherwise the samples would dissolve straight into the solvent in the container instead of moving up the paper. As the solvent rises, each dye moves a different distance depending on its solubility. When the solvent is near the top, the paper is removed, the solvent front is marked and the paper is dried.

(b)(i) Because all samples ran on the same paper with the same solvent, a dye always travels the same distance. E has spots at 2.1 cm and 5.4 cm. P has a spot at 2.1 cm and R has a spot at 5.4 cm, so E contains P and R. Q (3.8 cm) does not match any spot in E. S contains a substance at 2.1 cm, but it also contains a substance at 6.7 cm, and E has no spot at 6.7 cm, so S is not in E.

(b)(ii) A pure substance gives only one spot. S gives two spots, so S is a mixture of at least two substances.

**Mark scheme**

(a)

[3]

- X: chromium(III) /  $\text{Cr}^{3+}$  (1)
- Y: copper(II) /  $\text{Cu}^{2+}$  (1)
- Z: iron(II) /  $\text{Fe}^{2+}$  (1)

**ALLOW** names or formulae of the ions**REJECT** iron(III) for Z; copper(I) for Y

(b)

[1]

- iron(III) /  $\text{Fe}^{3+}$  (1)

**REJECT** iron(II)

(c)

[2]

- add aqueous sodium hydroxide and warm (1)
- gas / ammonia given off turns damp red litmus paper blue (1)

**ALLOW** damp universal indicator paper turns blue/purple**REJECT** dry litmus paper; blue litmus turns red**Worked solution**

(a) Use the colour of each precipitate and whether it dissolves in excess.

- X forms a green precipitate that dissolves in excess sodium hydroxide but not in excess ammonia: this is chromium(III),  $\text{Cr}^{3+}$ .
- Y forms a light blue precipitate with both reagents; only with excess ammonia does it dissolve to give a dark blue solution: this is copper(II),  $\text{Cu}^{2+}$ .
- Z forms a green precipitate that is insoluble in excess of both reagents and turns brown near the surface on standing (the iron(II) hydroxide slowly changes to iron(III) hydroxide in air): this is iron(II),  $\text{Fe}^{2+}$ .

Notice that X and Z are both green; the test with excess sodium hydroxide is what tells them apart.

(b) A red-brown precipitate, insoluble in excess of both reagents, is iron(III),  $\text{Fe}^{3+}$ .

(c) Ammonium ions do not form a precipitate. Instead, warming with aqueous sodium hydroxide releases ammonia gas. Ammonia is alkaline, so it turns damp red litmus paper blue. The paper must be damp because the gas must dissolve to act as an alkali.

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**11** Core Acid-base titrations

[1]

Answer: A

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**Worked solution**

The whole point of a titration is to stop adding solution at exactly the end-point, which is shown by the indicator changing colour. Against a white tile, even a slight change in colour is easy to spot. The tile is just a background: it does not react with anything and does not heat or cool the flask.

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**12** Core Chromatography

[1]

Answer: C

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**Worked solution**

Count all the spots, including any left on the baseline. A substance stays on the baseline when it does not dissolve in the solvent, so it is not carried up the paper. Here there are three spots in total: two soluble substances that moved and one insoluble substance that stayed on the baseline. So the ink is a mixture of at least three substances. We say "at least" because two different substances can sometimes travel the same distance and appear as a single spot.

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**13** Core Separation and purification

[1]

Answer: D

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**Worked solution**

A pure substance melts sharply at one fixed temperature, here 122 °C. An impurity disturbs the regular arrangement of particles in the solid, so it starts to melt at a lower temperature and melts gradually over a range. So the impure sample melts over a range of temperatures below 122 °C. Melting point measurements are therefore a quick way to check purity.

Answer: A

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**Worked solution**

The halide test: acidify with dilute nitric acid, then add aqueous silver nitrate, and look at the colour of the precipitate.

- chloride,  $\text{Cl}^-$ : white precipitate
- bromide,  $\text{Br}^-$ : cream precipitate
- iodide,  $\text{I}^-$ : yellow precipitate

A yellow precipitate means iodide ions are present.

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**Mark scheme**

(a) [4]

- any four from (1 each, max 4):
  - use a (volumetric) pipette to put  $25.0\text{ cm}^3$  of aqueous potassium hydroxide into a conical flask
  - add a few drops of methyl orange (to the flask)
  - fill a burette with the hydrochloric acid and record the initial (start) reading
  - add the acid from the burette to the flask, swirling the flask
  - stop when the indicator (just) changes colour / turns orange
  - record the final burette reading and subtract to find the volume of acid added
  - repeat (the titration) until concordant results are obtained

**REJECT** measuring cylinder for measuring the  $25.0\text{ cm}^3$  of potassium hydroxide

**ALLOW** acid in the flask and alkali in the burette only if pipette used for  $25.0\text{ cm}^3$  and burette for the other solution

(b) [1]

- yellow (1)

**REJECT** orange; red

(c) [1]

- so that the end-point is not overshoot / too much acid is not added / to find the exact volume at the end-point (1)

**ALLOW** so the colour change is seen at exactly the right moment

**Worked solution**

(a) A volumetric pipette measures exactly  $25.0\text{ cm}^3$  of the alkali into a conical flask. A few drops of methyl orange are added. The burette is filled with hydrochloric acid and its starting reading is recorded. Acid is run in from the burette while the flask is swirled to mix the solutions. When the indicator changes colour, the tap is closed and the final reading is recorded. The volume of acid used (the titre) is the final reading minus the initial reading. The titration is repeated until two results are concordant (very close together).

(b) Methyl orange is yellow in alkali and red in acid. At the start the flask contains potassium hydroxide, an alkali, so the indicator is yellow. It turns orange at the end-point.

(c) Near the end-point, a single drop of acid can change the colour. Adding the acid drop by drop means the student can stop as soon as the colour changes and does not add more acid than is needed.

**Mark scheme**

(a)

[3]

- add water (to the mixture) and stir (to dissolve the potassium nitrate) (1)
- filter (1)
- wash the residue / zinc oxide with (distilled) water and dry it (1)

**ALLOW** dry in a warm oven / between filter papers / leave to dry

**ALLOW** warm the water to help the potassium nitrate dissolve

(b)

[3]

- heat / warm the filtrate to evaporate some of the water until crystals start to form / until the solution is saturated (1)
- leave (the solution) to cool so that crystals form (1)
- filter off (or remove) the crystals and dry them between filter papers / wash with a little cold water and dry (1)

**REJECT** heat until all the water has evaporated / heat to dryness (for the first mark)

**Worked solution**

(a) Potassium nitrate dissolves in water but zinc oxide does not. Adding water and stirring dissolves the potassium nitrate. Filtering then separates the two: the zinc oxide is left on the filter paper as the residue, and the potassium nitrate solution passes through as the filtrate. Washing the residue with water removes any potassium nitrate solution left on it, and drying it gives pure, dry zinc oxide.

(b) This is crystallisation. The filtrate is heated to evaporate some of the water until the solution is saturated (crystals start to appear, for example on a cold glass rod dipped in it). The solution is then left to cool. As it cools, less potassium nitrate can stay dissolved, so crystals form. The crystals are filtered off and dried between filter papers. The water is not boiled away completely, because that would give a powder mixed with any soluble impurities rather than pure crystals.

**Mark scheme**

(a) [1]

- potassium /  $K^+$  (1)

(b) [1]

- sulfate /  $SO_4^{2-}$  (1)

**REJECT** sulfite

(c) [2]

- calcium (1)
- bromide (1)

**ALLOW**  $CaBr_2$  (2)

**REJECT** calcium bromine

(d) [2]

- dip a clean wire (loop) into the (solid) salt (1)
- hold it in a (blue / hot, non-luminous) Bunsen burner flame and observe the colour (of the flame) (1)

**ALLOW** moistened / damp splint or wire loop; nichrome / platinum wire

**ALLOW** first mark for "clean the wire (by dipping in acid) and dip into the salt"

(e) [1]

- hydrochloric acid contains chloride ions, which would (react with silver ions to) give a white precipitate (of silver chloride) (1)

**ALLOW** chloride ions would give a false positive / interfere with the test

**Worked solution**

(a) A lilac flame shows potassium ions,  $K^+$ .

(b) Acidifying with dilute nitric acid and then adding aqueous barium nitrate is the test for sulfate ions. A white precipitate (barium sulfate) shows sulfate is present. So T is potassium sulfate.

(c) With aqueous sodium hydroxide, a white precipitate that does not dissolve in excess could be calcium ions (aluminium and zinc precipitates would dissolve in excess). With aqueous ammonia, calcium ions give no precipitate (or only a very slight one), which confirms calcium. The cream precipitate with acidified silver nitrate shows bromide ions. So V is calcium bromide,  $CaBr_2$ .

(d) A clean wire loop is dipped into the salt and then held in a hot, blue Bunsen flame. The colour of the flame is observed. The wire must be clean so that traces of other salts do not colour the flame.

(e) The test works because silver ions form a precipitate with halide ions. Hydrochloric acid contains chloride ions, so adding it would produce a white precipitate of silver chloride whatever the salt was. Nitric acid adds no halide ions, so it does not interfere.

**Mark scheme**

(a) [1]

- sugars are colourless / the spots have no colour (1)

(b) [2]

- spray the (dried) chromatogram with (or dip it in) a locating agent (1)
- the spots become coloured / visible, so their positions can be marked (1)

**IGNORE** names of specific locating agents

(c)(i) [2]

- $R_f = \text{distance travelled by substance} \div \text{distance travelled by solvent}$ , e.g.  $2.4 \div 9.6$  (1)
- 0.25 and 0.75 (1)

**ALLOW** first mark for either value correct with method shown

**REJECT** values greater than 1 (e.g.  $9.6 \div 2.4 = 4$ ) for the second mark

(c)(ii) [1]

- sucrose and fructose (1)

**ECF** from (c)(i) if the  $R_f$  values are between 0 and 1 and match two sugars in the table

(d) [1]

- the  $R_f$  value of a substance depends on the solvent / a substance has different  $R_f$  values in different solvents (1)

**ALLOW** the solubility of the sugar (and so the distance it travels) is different in different solvents

**Worked solution**

(a) The sugars do separate as the solvent moves up the paper, but because they are colourless, nothing can be seen on the dried paper.

(b) After drying, the chromatogram is sprayed with (or dipped in) a locating agent. This reacts with the sugars to make coloured spots, so their positions can be marked with a pencil and measured.

(c)(i)  $R_f = \text{distance travelled by substance} \div \text{distance travelled by solvent}$ .

- spot 1:  $2.4 \div 9.6 = 0.25$
- spot 2:  $7.2 \div 9.6 = 0.75$

Check: each value is less than 1, as it must be, because a substance cannot travel further than the solvent front. Working backwards,  $0.25 \times 9.6 = 2.4$  cm and  $0.75 \times 9.6 = 7.2$  cm, which match the measured distances.

(c)(ii) An  $R_f$  of 0.25 matches sucrose and an  $R_f$  of 0.75 matches fructose.

(d) How far a substance moves depends on how soluble it is in the solvent. In a different solvent the same sugar would have a different  $R_f$  value, so comparisons are only valid if the same solvent is used.

**Mark scheme**

(a)(i) [1]

- water (1)

(a)(ii) [1]

- copper(II) sulfate (1)

**ALLOW** CuSO<sub>4</sub>

**IGNORE** copper sulfate without (II)

(b) [1]

- saturated (solution) (1)

**REJECT** concentrated

(c)(i) [1]

- residue (1)

(c)(ii) [1]

- filtrate (1)

(d) [1]

- the maximum amount / concentration of solute that can dissolve (the solubility) changes with temperature (1)

**ALLOW** more copper(II) sulfate dissolves at higher temperature

**Worked solution**

(a) The solvent is the substance that does the dissolving: water. The solute is the substance that is dissolved: copper(II) sulfate. Together they make a solution.

(b) When no more solute will dissolve and some stays undissolved, the solution contains the maximum concentration of solute. It is a saturated solution.

(c) When a mixture is filtered, the solid that stays on the filter paper is the residue (here, the undissolved copper(II) sulfate crystals). The solution that passes through the paper is the filtrate.

(d) The maximum concentration of solute that can dissolve depends on temperature: for copper(II) sulfate, more dissolves in hot water than in cold. So "saturated" only has a definite meaning at a stated temperature.

**Mark scheme**

(a)(i) [1]

- filtration (1)

(a)(ii) [1]

- (simple) distillation (1)

**REJECT** evaporation; fractional distillation

(a)(iii) [1]

- crystallisation (1)

**ALLOW** evaporate some of the water then leave to cool / crystallise

**REJECT** evaporation to dryness; filtration

(a)(iv) [1]

- fractional distillation (1)

(b) [2]

- (sea water is heated and) the water boils / evaporates, but the salt does not / the salt stays in the flask (1)
- the water vapour / steam is cooled and condenses (in a condenser), and the liquid water is collected (1)

**ALLOW** salt has a much higher boiling point than water

**Worked solution**

(a) Choose the method from the substances involved.

- (i) Sand is an insoluble solid in a liquid, so it is separated by filtration.
- (ii) Pure water must be collected from a solution of salts, so the water is boiled off and condensed: simple distillation. Evaporation alone would lose the water to the air.
- (iii) A solid is needed from its solution, as crystals, so crystallisation is used: evaporate some water, then cool so crystals form.
- (iv) Petroleum is a mixture of many liquids with different boiling points, so it is separated by fractional distillation.

(b) In simple distillation, sea water is heated in a flask. The water boils and turns to steam, but the dissolved salts have much higher boiling points and stay behind in the flask. The steam passes into a condenser, where it is cooled and condenses back to liquid water, which is collected. This water (the distillate) contains no dissolved salts, so it is pure.

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**21** Core Experimental design

[1]

Answer: B

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**Worked solution**

The amount of acid needed to neutralise the alkali is fixed by the chemistry, not by the apparatus, so A is wrong. The problem with a measuring cylinder is precision: its scale can only be read roughly (often to the nearest 0.5 or 1 cm<sup>3</sup>), and it has no tap to add the acid a drop at a time. A burette can be read to the nearest 0.05 cm<sup>3</sup> and lets you stop exactly at the end-point.

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**22** Supplement Chromatography

[1]

Answer: C

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**Worked solution**

Rearrange the R<sub>f</sub> equation:

distance travelled by substance = R<sub>f</sub> × distance travelled by solvent = 0.60 × 7.5 = 4.5 cm.

Check by working forwards: 4.5 ÷ 7.5 = 0.60, which matches the R<sub>f</sub> given. A sensible answer must be less than 7.5 cm, because a substance cannot move further than the solvent, so 12.5 cm is impossible.

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**23** Core Separation and purification

[1]

Answer: B

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**Worked solution**

To separate two solids, find a solvent that dissolves one but not the other. Water dissolves neither, so water is useless (A leaves both solids on the filter paper, D leaves nothing in the filtrate). Ethanol dissolves Q but not R. After adding ethanol and filtering:

- the residue on the filter paper is R (C would give R, not Q)
- the filtrate is a solution of Q in ethanol.

Evaporating the ethanol from the filtrate leaves solid Q.

Answer: A

### Worked solution

A useful reagent must give a different result for each solution.

- Aqueous sodium hydroxide:  $\text{Zn}^{2+}$  gives white precipitate, soluble in excess;  $\text{Al}^{3+}$  also gives white precipitate, soluble in excess. Same result, so no use.
- Aqueous ammonia:  $\text{Zn}^{2+}$  gives white precipitate, soluble in excess;  $\text{Al}^{3+}$  gives white precipitate, insoluble in excess. Different results, so this works.
- Barium nitrate tests for the anion, and both compounds are sulfates, so both give the same white precipitate.
- Silver nitrate tests for halide ions, which neither solution contains.

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**Mark scheme**

(a) [1]

- measuring cylinder (1)

| **ALLOW** burette / (volumetric) pipette

(b) [1]

- lets the (carbon dioxide) gas escape but stops acid / liquid spray escaping (1)

| **ALLOW** stops acid splashing out (which would give an extra loss of mass)

| **REJECT** stops the gas escaping

(c) [1]

- any one from (1):

- measures the volume of gas directly
- not affected by loss of acid spray / water vapour from the flask
- the gas is collected (so it could be tested)
- gives larger readings that are easier to measure than small changes in mass

| **IGNORE** "more accurate" on its own

(d) [1]

- any one from (1):

- the gas syringe has a limited volume / may fill up before the reaction ends
- the plunger may stick
- gas may leak from the joints / connections
- (some) gas escapes before the bung is put in / the syringe is connected

| **IGNORE** "more expensive" on its own

(e) [2]

- hydrogen has a very low mass / is very light / has a very low density ( $M_r = 2$ ) (1)
- so the loss in mass is too small to measure accurately on the balance (1)

| **ALLOW** the change in mass would be too small to detect

**Worked solution**

(a)  $50 \text{ cm}^3$  of acid can be measured with a measuring cylinder, which is accurate enough for a rate experiment.

(b) The cotton wool is loose, so carbon dioxide can escape (that loss of mass is what is being measured). It stops small droplets of acid being carried out of the flask by the fizzing; if they escaped, the mass would fall by more than the mass of gas and the results would be wrong.

(c) and (d) A gas syringe measures the volume of the gas itself and collects it, so spray or evaporation from the flask does not affect the reading. However, a gas syringe can only hold a limited volume, its

plunger can stick, and gas can leak from the connections, while a balance has none of these problems.

(e) Method 1 relies on measuring the mass lost as gas escapes. Carbon dioxide ( $M_r$  44) is fairly heavy, so the mass change is easy to measure. Hydrogen ( $M_r$  2) is so light that the mass lost would be tiny, too small to measure accurately on a normal laboratory balance. Method 2, with a gas syringe, is much better for hydrogen.

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**Mark scheme**

(a) [1]

- fractional distillation (1)

**REJECT** simple distillation; distillation alone

(b) [3]

- any three from (1 each, max 3):
  - the mixture is heated (and both liquids start to evaporate)
  - the fractionating column is hotter at the bottom and cooler at the top
  - K (the liquid with the higher boiling point) condenses in the column and runs back into the flask
  - J (the liquid with the lower boiling point) reaches the top of the column (as a vapour)
  - J vapour passes into the condenser, where it is cooled and condenses (and is collected)

**ALLOW** J boils / evaporates first because it has the lower boiling point

(c) [1]

- the temperature (on the thermometer) rises above 81 °C (1)

**ALLOW** temperature rises towards 111 °C

**ALLOW** no more liquid distils over at 81 °C

(d) [2]

- measure the boiling point of the liquid; if it is pure J it boils at exactly 81 °C / at a sharp, fixed temperature (1)
- if it is impure it boils over a range of temperatures / not at exactly 81 °C (1)

**ALLOW** impure liquid boils at a higher temperature

**Worked solution**

(a) J and K are miscible liquids with different boiling points, so fractional distillation is used.

(b) The mixture is heated. The fractionating column is hot at the bottom and cooler at the top. Vapour rises up the column. K has the higher boiling point, so its vapour condenses on the cooler parts of the column and drips back into the flask. J has the lower boiling point, so its vapour reaches the top of the column, passes into the condenser, is cooled back to a liquid and is collected.

(c) While J is distilling, the thermometer at the top of the column stays at J's boiling point, 81 °C. Once all of J has distilled over, K vapour starts to reach the top of the column, so the temperature rises towards 111 °C. The rise in temperature tells the chemist that J has all been collected.

(d) A pure liquid boils at one fixed temperature. If the first liquid boils sharply at 81 °C, it is pure J. If it contains an impurity (for example some K), it will boil over a range of temperatures rather than at exactly 81 °C.

**Mark scheme**

(a)(i) [1]

- hydrogen (1)

(a)(ii) [2]

- (hold) a lighted splint (at the mouth of the tube) (1)
- (squeaky) pop (1)

**REJECT** glowing splint (0 for the first mark); "relights" for the second mark

(b) [1]

- oxygen (1)

(c) [2]

- damp litmus paper (1)
- is bleached / turns white (1)

**ALLOW** damp blue litmus turns red and then is bleached

**REJECT** dry litmus paper for the first mark

(d) [1]

- sulfur dioxide /  $\text{SO}_2$  (1)

(e) [1]

- ammonia /  $\text{NH}_3$  (1)

**Worked solution**

(a) Calcium is a reactive metal that reacts with cold water to form calcium hydroxide and hydrogen gas. The test for hydrogen is a lighted splint: hydrogen burns with a squeaky "pop".

(b) Manganese(IV) oxide is a catalyst for the decomposition of hydrogen peroxide into water and oxygen. A gas that relights a glowing splint is oxygen.

(c) Chlorine is tested with damp litmus paper, which it bleaches (turns white). The paper must be damp because chlorine needs to dissolve in water to bleach it.

(d) Sulfur dioxide turns acidified aqueous potassium manganate(VII) from purple to colourless.

(e) Ammonia is the only common gas that is alkaline, so it turns damp red litmus paper blue.

Learn the six gas tests as a set:  $\text{NH}_3$  (damp red litmus turns blue),  $\text{CO}_2$  (limewater turns milky),  $\text{Cl}_2$  (damp litmus bleached),  $\text{H}_2$  (lighted splint pops),  $\text{O}_2$  (glowing splint relights),  $\text{SO}_2$  (acidified potassium manganate(VII) purple to colourless).

**Mark scheme**

(a) [3]

- add aqueous sodium hydroxide and aluminium foil (1)
- warm (gently) (1)
- gas / ammonia given off turns damp red litmus paper blue (1)

**ALLOW** damp universal indicator paper turns blue/purple

**REJECT** "nitrogen" given off; blue litmus turns red

(b) [2]

- add dilute (hydrochloric / nitric / sulfuric) acid; effervescence / bubbles (1)
- gas / carbon dioxide turns limewater milky / cloudy (1)

**REJECT** concentrated acid

(c) [2]

- add (a small volume of) acidified aqueous potassium manganate(VII) (1)
- turns from purple to colourless (1)

**ALLOW** add dilute acid and warm, then test the gas given off (sulfur dioxide) with acidified aqueous potassium manganate(VII), which turns from purple to colourless (2)

**REJECT** potassium manganate(VII) without "acidified" for the first mark

(d) [1]

- both compounds contain sodium ions / both give a yellow flame (1)

**Worked solution**

(a) Nitrate ions are reduced to ammonia by aluminium in alkaline conditions. Add aqueous sodium hydroxide and a piece of aluminium foil, then warm gently. If nitrate is present, ammonia gas is released, which turns damp red litmus paper blue. (Check first that there are no ammonium ions, which would also give ammonia with sodium hydroxide.)

(b) Carbonates react with dilute acid to give carbon dioxide, so bubbles are seen. Bubbling the gas through limewater turns it milky, which confirms carbon dioxide.

(c) Sulfite ions are oxidised by acidified aqueous potassium manganate(VII). The purple colour of the manganate(VII) disappears, leaving a colourless solution.

(d) A flame test identifies the cation, not the anion. Both products contain sodium ions, so both give the same yellow flame. The anion tests in (a) and (c) are needed to tell them apart.

**Mark scheme**

(a) [2]

- rough 24.60, run 1 23.80, run 2 24.35, run 3 23.90 (cm<sup>3</sup>) (2)
- three of the four correct (1)

**ALLOW** 24.6, 23.8, 23.9 (ignore missing trailing zeros)

(b) [2]

- run 1 and run 3 / 23.80 and 23.90 (1)
- mean =  $(23.80 + 23.90) \div 2 = 23.85 \text{ cm}^3$  (1)

**ECF** from (a) for a correct mean of the two titres chosen if they are within 0.10 cm<sup>3</sup>

**REJECT** a mean that includes the rough titre or run 2

(c) [1]

- to find the approximate volume needed / approximate end-point (so that in the accurate runs acid can be added drop by drop near the end-point) (1)

**ALLOW** to know roughly when to slow down

(d) [1]

- blue to colourless (1)

**REJECT** colourless to blue

**Worked solution**

(a) Titre = final reading – initial reading.

- rough:  $24.60 - 0.00 = 24.60 \text{ cm}^3$
- run 1:  $24.30 - 0.50 = 23.80 \text{ cm}^3$
- run 2:  $24.55 - 0.20 = 24.35 \text{ cm}^3$
- run 3:  $24.50 - 0.60 = 23.90 \text{ cm}^3$

Check by adding back:  $0.50 + 23.80 = 24.30$ ,  $0.20 + 24.35 = 24.55$  and  $0.60 + 23.90 = 24.50$ , matching the final readings.

(b) Look for accurate titres within 0.10 cm<sup>3</sup> of each other. Runs 1 and 3 differ by 0.10 cm<sup>3</sup>, so they are concordant. Run 2 is 0.45 cm<sup>3</sup> above run 3, so it is left out, and the rough titre is never used in the mean. Mean =  $(23.80 + 23.90) \div 2 = 23.85 \text{ cm}^3$ .

(c) The rough titration is done quickly to find roughly where the end-point is. It usually overshoots, which is why it is not used in the mean. In the accurate runs the student can add acid quickly up to about 1 cm<sup>3</sup> before this volume, then drop by drop.

(d) Thymolphthalein is blue in alkali and colourless in acid. The flask starts with sodium hydroxide, so it is blue; at the end-point it turns colourless.

**Mark scheme**

(a) [1]

- the substance(s) in the dye are insoluble in water (1)

**ALLOW** the dye does not dissolve in water

(b) [2]

- ethanol (1)
- the substances are separated / gives two (separate) spots, because the substances have different solubilities in ethanol (and so travel different distances) (1)
- the second mark depends on ethanol being chosen

(c) [2]

- $R_f = \text{distance travelled by substance} \div \text{distance travelled by solvent}$ , e.g.  $1.6 \div 8.0$  (1)
- 0.20 and 0.55 (1)

**ALLOW** 0.2

**REJECT** values greater than 1 for the second mark

(d) [1]

- the substances are (all) very soluble in propanone so they travel together / are not separated (and give one spot) (1)

**ALLOW** the ethanol result shows the dye contains (at least) two substances

**ALLOW** one spot in one solvent does not prove the dye is pure

**Worked solution**

(a) A substance only moves up the paper if it dissolves in the solvent. Nothing moved in water, so the substances in the dye are insoluble in water.

(b) Ethanol is the best solvent because it separated the dye into two spots. The two substances have different solubilities in ethanol, so they travel different distances up the paper. In water nothing moved, and in propanone everything moved together with the solvent front, so neither separated the substances.

(c)  $R_f = \text{distance travelled by substance} \div \text{distance travelled by solvent}$ .

- spot 1:  $1.6 \div 8.0 = 0.20$
- spot 2:  $4.4 \div 8.0 = 0.55$

Check:  $0.20 \times 8.0 = 1.6$  cm and  $0.55 \times 8.0 = 4.4$  cm, matching the measured distances.

(d) In propanone, both substances are so soluble that they move with the solvent front and stay together as one spot. One spot in one solvent does not prove a substance is pure. The ethanol chromatogram shows that the green dye actually contains at least two substances.