

*Leaving Certificate Chemistry*  
*Student Laboratory Notebook*

**Suggested Answers**

**by Declan Kennedy**  
**and**  
**Don O'Shea**

**Experiment 3.2. To carry out flame tests with salts of lithium, sodium, potassium, barium, strontium and copper.**

**Questions on this experiment**

**1. Why do these metal salts give off a characteristic colour?**

Since each element has its own electron configuration this means that each element has different electron transitions. Therefore each metal has a characteristic colour. (Alternatively, one could say that each element has a unique atomic spectrum due to the fact that each element has its own arrangement of energy levels.

**2. How could you determine what metal is contained in an unknown substance?**

Hold the salt in the blue flame of the Bunsen burner and observe the colour given off. Compare this colour with the standard colours given off by different metals.

**3. How would you prevent cross contamination?**

(1) Use a new disposable wooden splint for each salt tested.

(2) If using platinum wire, dip the platinum wire in concentrated hydrochloric acid after each salt has been tested. Alternatively use a separate platinum wire for each salt sample.

**4. Why is a platinum wire or a damp wooden splint used to hold the salt?**

Neither the platinum wire nor the damp wooden splint gives off a colour that would interfere with the characteristic colour emitted by the salt.

**5. Give one practical application of flame tests.**

Flame tests are used in chemistry to identify the presence of certain metals.

**Experiment 5.1. (a) To obtain a pure sample of benzoic acid from an impure sample by the technique of recrystallisation and (b) to measure the melting point of benzoic acid.**

**Questions on this experiment**

- 1. In this experiment it is advisable to use the minimum amount of solvent possible. What is the reason for this and how is it achieved?**

It is important to use the minimum of solvent (hot water) so as to maximise the yield, i.e. so that the benzoic acid does not remain in solution on cooling. This is achieved by adding the water in small portions until the benzoic acid has just dissolved (see Step 3 of procedure).

- 2. Is benzoic acid an ionic or covalent compound? Give a reason for your answer.**

It is a covalent compound. The fact that it is only sparingly soluble in water is evidence for this fact. Since the melting point of benzoic acid is reasonably low ( $122\text{ }^{\circ}\text{C}$ ), this indicates that it must be a molecular crystal rather than an ionic crystal. If it were an ionic compound, the melting point would be very high (greater than  $700\text{ }^{\circ}\text{C}$ ).

- 3. Describe how the speed of the filtering is increased.**

The speed of filtration is increased by attaching the Buchner flask to a suction pump. This helps the liquid to pass more quickly through the filter paper.

- 4. If little or no crystals appeared, what steps could be undertaken?**

A number of steps can be taken:

- The conical flask containing the filtrate could be cooled by surrounding it with ice.
- Scratching the inside of the flask with a glass rod helps to speed up the formation of crystals.
- If both of the above steps fail to produce crystals, this suggests that too much solvent has been used. Therefore, allow the water to evaporate either by boiling it off or by placing it in an evaporating basin overnight.

- 5. Describe the appearance of the pure benzoic acid.**

Pure benzoic acid is a white crystalline solid.

- 6. Outline how you would know the sample was pure.**

The pure sample should melt at a sharp temperature (exactly  $122\text{ }^{\circ}\text{C}$ ). If the sample were impure it would melt over a wide range of temperatures. In addition an impure sample melts at a lower temperature than that given for pure benzoic acid.

- 7. Give two differences that would be observed when measuring the melting points of the pure and impure samples.**

The two differences are:

- (1) The pure sample melts at a sharp temperature. The impure sample melts over a wide range of temperatures.
- (2) The impure sample melts at a lower temperature than the pure sample.

**Experiment 6.1. To test for the presence of chloride, carbonate, nitrate, sulfate, phosphate, sulfite and hydrogencarbonate anions in aqueous solutions.**

**Questions on this experiment**

- 1. What term is used to describe this type of analysis? Define the term *anion*.**

This type of analysis is known as qualitative analysis. An anion is a negatively charged ion.

- 2. Why is deionised water used in all of the tests?**

Since water is a good solvent, tap water contains many dissolved ions. These ions could interfere with the tests. Deionised water is used because it contains no dissolved ions.

- 3. What is a precipitate? In the test for the chloride ion, how can the presence of silver chloride be confirmed?**

A precipitate is a material that settles out of solution.

In the test for the chloride ion, the presence of silver chloride is confirmed by adding dilute ammonia solution to the silver chloride. Since silver chloride is soluble in dilute ammonia, the cloudiness disappears.

- 4. What is the difference between a sulfate ion and a sulfite ion? How can these two ions be distinguished from each other in the laboratory?**

The sulfate ion has the formula  $\text{SO}_4^{2-}$  and the formula for the sulfite ion is  $\text{SO}_3^{2-}$ .

The two ions are distinguished by adding a few drops of barium chloride solution to a solution of each ion. A white precipitate is observed in each case. Add dilute hydrochloric acid to the precipitate. The cloudiness disappears in the case of the sulfite ion but remains in the case of the sulfate ion.

- 5. Why is it best to make up a fresh solution of limewater before using it to confirm the presence of carbon dioxide?**

It is best to make up a fresh solution since limewater reacts with carbon dioxide in the air, and this makes the solution cloudy.

- 6. In testing for the presence of the nitrate ion, why must extreme care be taken when using concentrated sulfuric acid?**

Concentrated sulfuric acid is very corrosive and causes burns to the skin.

**Experiment 10.1. To measure the relative molecular mass of a volatile liquid.**

**Questions on this experiment**

**1. Define the term *relative molecular mass*?**

The relative molecular mass of a compound is the mass of one molecule of that compound compared to one twelfth of the mass of the carbon-12 isotope.

**2. In this experiment, a volatile liquid is used. What does the term *volatile* mean?**

A volatile liquid is one with a low boiling point (evaporates easily).

**3. Apart from the liquid you used in this experiment, give one other example of a suitable liquid.**

Examples of suitable liquids are propanone, cyclohexane, hexane and ethanaol.

**4. What does the term *s.t.p.* represent?**

Standard temperature and pressure. Standard temperature is 273 K and standard pressure is  $1 \times 10^5$  pascals.

**5. Why must a very small hole be punctured in the aluminium foil?**

The small hole is necessary to allow the excess vapour to escape from the flask.

**6. What units are normally used to measure atmospheric pressure?**

Pascals (newtons per metre squared) or symbols Pa ( $\text{N m}^{-2}$ ).

**7. What must be done to ensure that all of the volatile liquid has vaporised fully?**

The flask should be left in the boiling water for several minutes.

**8. Indicate one source of error in this experiment.**

Sources of error:

- Forgetting to ensure that the inside of the flask is clean and dry.
- Measuring the volume of the conical flask inaccurately.
- Not leaving the flask in the boiling water for a sufficient length of time.
- Forgetting to dry the outside of the flask when re-weighing.
- Not using a balance accurate enough to 2 decimal places.
- Forgetting to measure the temperature of the boiling water (it may not be exactly 373 K).
- Lack of accuracy in the measuring instruments, e.g. the graduated cylinder and the gas syringe can only read to  $0.5 \text{ cm}^3$ .
- There may be bubbles of air in the hypodermic syringe.
- Parallax errors may be involved in taking readings.

**9. What does *R* represent in the equation  $pV = nRT$ ?**

*R* is the universal gas constant. (The value of *R* is  $8.31 \text{ J mol}^{-1} \text{ K}^{-1}$ .)

**10. Why does the liquid have to be volatile?**

The liquid must be volatile so that when the conical flask is placed in the boiling water the liquid will be completely changed into a gas and fill the conical flask.

### Experiment 13.1. To prepare a standard solution of sodium carbonate.

#### Questions on this experiment

**1. Describe the appearance of sodium carbonate.**

Sodium carbonate is a white solid.

**2. Why must the mass of the sodium carbonate dissolved in the water be known precisely?**

The mass must be known precisely so that the concentration of the sodium carbonate solution can be calculated accurately.

**3. Why is sodium carbonate suitable as a primary standard? Distinguish between a primary standard and a standard solution.**

Sodium carbonate is suitable as a primary standard because it can be obtained in a pure and stable state and it dissolves easily in water.

A primary standard is a substance which can be obtained in a stable, pure and soluble solid form so that it can be weighed out and dissolved in water to give a solution of accurately known concentration.

A standard solution is a solution of precisely known concentration.

**4. Outline the correct procedures for transferring the sodium carbonate to the volumetric flask.**

The main points are:

- Accurately measure the known mass of sodium carbonate using a clock glass.
- Correctly transfer the sodium carbonate to the beaker containing water.
- Wash down traces of sodium carbonate from the clock glass (note filter paper is not suitable).
- Stir the solution to ensure that all solid has dissolved completely.
- Use the glass rod and funnel to transfer solution to the volumetric flask.
- Wash all traces of solution from the beaker, rod and funnel into the volumetric flask.
- With the volumetric flask on a level surface, remove the funnel, add deionised water drop by drop, and bring the bottom of the meniscus to the graduation mark on the volumetric flask.
- Stopper the flask and invert it about 20 times.

**5. Why is it necessary to *slowly* add the solid sodium carbonate to the water in the beaker?**

Adding the sodium carbonate slowly to the water helps prevent lumps being formed in the solution.

**6. State two benefits of a volumetric flask for making up the solution.**

The two benefits are:

- (1) The volume of the solution in the volumetric flask is accurately known.
- (2) The design of the volumetric flask allows it to be shaken easily to ensure a homogenous mixture, i.e. to ensure that the solution is uniform throughout.

**Experiment 13.2. To use the standard solution of sodium carbonate solution prepared in experiment 13.1 to standardise a given hydrochloric acid solution.**

**Questions on this experiment**

**1. Why is it not recommended procedure to pipette directly from the volumetric flask?**

This is not recommended because any impurities on the pipette would contaminate the entire solution in the volumetric flask.

**2. Why is it important not to add too much indicator?**

Indicators are either weak acids or weak bases and adding too much indicator could affect the accuracy of the result. Also, adding too much indicator could give a very intense colour that may make it difficult to see the colour change at the end point.

**3. Why do we have to standardise HCl solution?**

The HCl solution must be standardised because HCl itself is not a primary standard, i.e. it is not possible to take an accurately known amount of HCl and dissolve it in a fixed volume of water to give a solution of known concentration.

**4. What is the purpose of carrying out the titration more than once?**

Carrying out the titration more than once gives a more accurate titration figure since the average of two readings agreeing to within 0.1 cm<sup>3</sup> is used in the calculations.

**5. Describe the appearance of the isolated sodium chloride.**

Sodium chloride is a white crystalline solid.

**6. Why is the result of the first titration usually ignored?**

It is ignored because it is likely to be inaccurate, since the purpose of the first titration is to tell you approximately where the end point lies.

**7. Name the gas given off during the titration.**

Carbon dioxide. ( $\text{Na}_2\text{CO}_3 + 2\text{HCl} \rightarrow 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2$ )

**Experiment 13.3. To make up an approximate 0.1 M solution of sodium hydroxide, to standardise it (i.e. obtain the exact concentration) with a standard hydrochloric acid solution and hence to prepare a sample of sodium chloride.**

### Questions on this experiment

1. **Why is it important not to allow solid sodium hydroxide to come in contact with your skin?**

Solid sodium hydroxide is corrosive and causes severe burns.

2. **How is it possible to standardise the NaOH in this experiment when the HCl is not a primary standard?**

Even though the HCl is not a primary standard, the concentration of the solution is known from the previous experiment, i.e. the HCl solution is a standard solution.

3. **What is the purpose of the white tile under the burette during the titration?**

The white tile enables us to detect colour changes more easily.

4. **Why is the volumetric flask inverted several times?**

To ensure that the solution is homogeneous, i.e. that the solution is the same concentration throughout.

5. **Outline the correct precautions for using a pipette.**

The main points:

- Rinse out the pipette with water to remove any impurities.
- Rinse out the pipette with the liquid it will contain so as to remove any drops of water.
- Fill the pipette using a pipette filler.
- Avoid bubbles of air entering the pipette when it is being filled.
- Ensure that the bottom of the meniscus is level with the graduation mark.
- Touch the tip of the pipette against the side of the flask when the discharge is complete.
- Do not blow out the liquid remaining in the tip of the pipette into the conical flask.

6. **Draw a diagram to illustrate the shape of the meniscus of the HCl in the burette.**

See *Chemistry Live!*, page 157, Fig 13.15.

7. **If the two accurate titrations do not agree to within 0.1 cm<sup>3</sup>, what must be done?**

If they do not agree, further titrations should be carried out until agreement to within 0.1 cm<sup>3</sup> is obtained.

8. **Why is the indicator left out when performing a titration to isolate a sample of the sodium chloride?**

The indicator would contaminate the sample.



**Experiment 13.4. To determine the concentration of ethanoic acid (acetic acid) in vinegar.**

**Questions on this experiment**

**1. Why is it important to use colourless vinegar rather than brown vinegar in this experiment?**

Because the brown colour of the vinegar would mask the colour change at the end point.

**2. Give the name and formula of the acid commonly found in vinegar.**

Ethanoic acid (acetic acid),  $\text{CH}_3\text{COOH}$ .

**3. Why is the vinegar diluted?**

It is diluted to ensure that the end point is within the limits of the burette, i.e. within  $50 \text{ cm}^3$ .

If undiluted vinegar were used a very large quantity of sodium hydroxide solution would be required to neutralise it, i.e. a very high end point would be obtained. This would be wasteful of chemicals and give rise to increased costs.

**4. What colour change is observed in this experiment?**

Using phenolphthalein indicator, the colour change is from pink to colourless.

(If the acid is in the conical flask and the base in the burette, the colour change is from colourless to pink).

**5. What precautions should you take when setting up the burette for titration?**

The main precautions:

- Clamp the burette vertically using the retort stand.
- Rinse the burette including the space below the tap with deionised water.
- Rinse the burette with the solution it is going to contain.
- Using a funnel, fill the burette with the solution.
- Open the tap to ensure that the space below the tap is filled.
- Remove the funnel from the burette.
- Adjust the level of the liquid so that the bottom of the meniscus is on the zero graduation mark.
- Check that there are no air bubbles in the nozzle.

**6. Outline the correct procedure for diluting the vinegar.**

The main points:

- Pipette two  $25 \text{ cm}^3$  samples of vinegar into a clean  $250 \text{ cm}^3$  volumetric flask.
- Add deionised water.
- Adjust the level of the liquid so that the bottom of the meniscus is on the engraved mark.

**7. What is meant by the term %w/v? What type of answer would you expect for the %w/v of ethanoic acid in vinegar? (Check the bottle of vinegar in your home!)**

The term %w/v stands for percentage weight per volume. The %w/v of ethanoic acid in vinegar varies depending on the brand of vinegar, but it is approximately 5%.

**8. Name the salt formed in this titration.**

The salt formed is called sodium ethanoate (sodium acetate).

**9. Give two safety precautions you would take when carrying out the experiment.**

Examples of safety precautions:

- Wear safety glasses.
- Use a pipette filler.
- Wear a laboratory coat.

**Experiment 13.5. To determine the percentage of water of crystallisation in hydrated sodium carbonate (washing soda).**

**Questions on this experiment**

**1. Describe the appearance of sodium carbonate crystals.**

Sodium carbonate is a colourless crystalline solid in its hydrated state.

**2. What is water of crystallisation?**

Water of crystallisation is water that makes up part of the crystal structure of a compound.

**3. What name is commonly given to hydrated sodium carbonate?**

Washing Soda

**4. Why is it necessary to stir the solution after adding the crystals of sodium carbonate to the water?**

To ensure that the crystals are fully dissolved in the water.

**5. What is the purpose of inverting the volumetric flask about 20 times?**

To ensure that the solution is homogeneous, i.e. that the solution is the same concentration throughout.

**6. What colour change is observed during this titration?**

The colour changes from yellow (orange) to pink (for methyl orange indicator).

**7. Why is it important to use as little indicator as possible?**

Indicators are either weak acids or weak bases and adding too much indicator could affect the accuracy of the result. Also, adding too much indicator could give a very intense colour that may make it difficult to see the colour change at the end point.

### Experiment 14.1. To study some oxidation-reduction reactions.

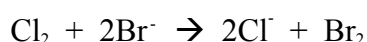
#### Questions on this experiment

**1. Why are halogens suitable to use as oxidising agents?**

Halogens are suitable as oxidising agents as they all have high electronegativity values, i.e. they have a strong attraction for electrons and they remove electrons from other substances. These substances are then oxidised.

**2. What is observed when chlorine gas is bubbled into a solution containing bromide ions? Explain this observation.**

It is observed that the colourless solution of bromide ions becomes orange. The orange colour is due to the formation of Br<sub>2</sub>. The Cl<sub>2</sub> is oxidising the Br<sup>-</sup> ion to Br<sub>2</sub>.

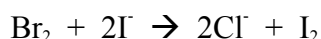


**3. Why is fluorine not used as an oxidising agent in the school laboratory?**

Fluorine is the most electronegative of all the halogens. Therefore, it is highly reactive and this reactivity makes it too dangerous to be stored in the school laboratory.

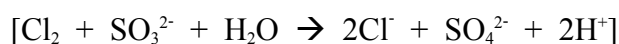
**4. What is observed when bromine water is added to a solution containing iodide ions? Explain this observation.**

When bromine water is added to a solution containing iodide ions, the colourless solution of iodide ions turns reddish/brown. This colour is due to the presence of iodine. This iodine is formed by the bromine oxidising the iodide ions to iodine.



**5. Name the main products formed when chlorine gas is added to a solution of sulfite ions.**

The products formed are : Chloride ions (Cl<sup>-</sup>), Sulfate ions (SO<sub>4</sub><sup>2-</sup>) and hydrogen ions (H<sup>+</sup>).



**6. When making up a solution of FeSO<sub>4</sub>, it is important to acidify the solution with dilute sulfuric acid. Why is this necessary?**

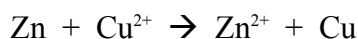
It is necessary to acidify the solution because the dilute sulfuric acid prevents the Fe<sup>2+</sup> ion being oxidised to the Fe<sup>3+</sup> ion by the oxygen in the air.

**7. What ion is formed when chlorine gas is bubbled into a solution of Fe<sup>2+</sup> ions? How would you test for the presence of the ion formed?**

The ion formed is the Fe<sup>3+</sup> ion. To test for the Fe<sup>3+</sup> ion add dilute sodium hydroxide solution. A greenish/brown precipitate is formed.

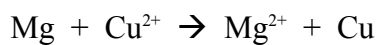
**8. What is observed when a piece of zinc metal is dipped into copper sulfate solution? Explain this observation.**

The immersed zinc becomes coated with a reddish brown substance. This substance is copper metal and has been formed by the zinc reducing the Cu<sup>2+</sup> ion to Cu metal. The equation for the reaction is



- 9. What is observed when a piece of magnesium metal is dipped into copper sulfate solution? Explain this observation.**

The immersed magnesium becomes coated with a brown substance. The brown substance is copper metal and has been formed by the magnesium reducing the  $\text{Cu}^{2+}$  ion to Cu metal. The equation for the reaction is



- 10. Give one use for the displacement of one metal by another.**

Displacement reactions of metals are used to protect metals from corrosion and also for extracting metals from solutions of their salts (see *Chemistry Live!* Workbook, Chapter 28, page 116).

**Experiment 15.1. To prepare a standard solution of ammonium iron(II) sulfate and use this to standardise a solution of  $\text{KMnO}_4$  by titration.**

**Questions on this experiment**

**1. Describe the appearance of the ammonium iron(II) sulfate crystals.**

Ammonium iron(II) sulfate is a green crystalline material.

**2. Why do you use ammonium iron(II) sulfate rather than just use iron(II) sulfate?**

Ammonium iron(II) sulfate is used for two reasons:

(1) It can be obtained in a high degree of purity.

(2) It is not affected by the air, i.e. the  $\text{Fe}^{2+}$  ions in ammonium iron(II) sulfate are not oxidised to  $\text{Fe}^{3+}$  ions by the oxygen in the air.

$\text{FeSO}_4$  is not a primary standard because (a) the crystals are oxidised slightly by the air and (b) the crystals lose their water of crystallisation when exposed to the air (efflorescence).

**3. What colour change is observed during this titration?**

The colour change at the end point is from colourless to pink.

**4. Why was it not necessary to weigh out exactly 9.8 g of ammonium iron(II) sulfate?**

In order to calculate the concentration of the ammonium iron(II) sulfate solution we need to know the exact amount that has dissolved in the dilute acid. This amount need not be exactly 9.8 g. It can be any amount that has been accurately measured. When calculating molarity all we need to know is the precise amount of salt in the solution.

**5. Describe how you would take the reading in the burette when using  $\text{KMnO}_4$ ?**

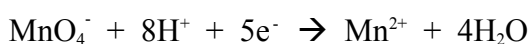
When reading the volume of  $\text{KMnO}_4$  in the burette, take the reading from the top rather than the bottom of the meniscus.

**6. Dilute sulfuric acid is added on two occasions during this experiment. Give the reason for its addition on each occasion.**

Dilute sulfuric acid is added:

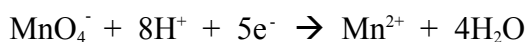
(1) when making up the solution of ammonium iron(II) sulfate. It is used to prevent the  $\text{Fe}^{2+}$  ions being oxidised to  $\text{Fe}^{3+}$  ions by oxygen in the air or oxygen dissolved in the water;

(2) to the conical flask at the beginning of the titration to supply the  $\text{H}^+$  ions in order for the following reaction to occur:



**7. When carrying out the titration another student observed that the first few drops of  $\text{KMnO}_4$  added to the conical flask were decolourised slowly, but subsequent drops were decolourised rapidly. Explain this observation.**

This observation is due to autocatalysis, i.e. the reaction is catalysed by the  $\text{Mn}^{2+}$  ions formed in the reaction:



As soon as the  $\text{Mn}^{2+}$  ions are formed the rate of the reaction increases.

- 8. When performing this experiment a student noticed a dark brown colour being formed in the conical flask. What conclusion would you draw from this observation?**

The student has probably forgotten to add the dilute sulfuric acid at the beginning of the titration. This omission causes a dark brown precipitate of  $\text{MnO}_2$  to be formed.

## Experiment 15.2. To determine the amount of iron in an iron tablet.

### Questions on this experiment

**1. Why do doctors recommend 'iron tablets' for certain patients?**

Iron tablets are often prescribed for people with anaemia, i.e. a shortage of haemoglobin in the blood.

**2. Give the name and formula of the main active ingredient in iron tablets.**

Iron(II) sulfate, FeSO<sub>4</sub>.

**3. Why do you use a number of iron tablets in this experiment?**

By using a number of tablets a solution of sufficiently high concentration is obtained so that the end point is within the limits of the burette. Also, using 5 tablets rather than 1 tablet allows greater accuracy to be obtained as it lowers the percentage error.

**4. Why is it necessary to grind up the iron tablets?**

To help them dissolve more easily.

**5. What items of laboratory equipment are commonly used to grind up the iron tablets?**

A pestle and mortar.

**6. Why is it not necessary to use an indicator in this experiment?**

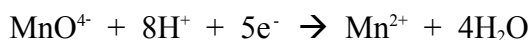
An indicator is not necessary since the potassium permanganate acts as its own indicator, i.e. as soon as all the Fe<sup>2+</sup> ions have reacted with the KMnO<sub>4</sub>, the next drop of KMnO<sub>4</sub> solution gives a permanent pink colour.

**7. Why are the tablets dissolved in dilute sulfuric acid?**

The tablets are dissolved in dilute sulfuric acid as it prevents the Fe<sup>2+</sup> ions being oxidised to Fe<sup>3+</sup> ions by oxygen in the air or oxygen dissolved in the water.

**8. Why is it necessary to add dilute sulfuric acid to the conical flask?**

It is added to the conical flask at the beginning of the titration to supply the H<sup>+</sup> ions in order for the following reaction to occur



**9. Why is it important to rinse out the burette thoroughly with water at the conclusion of this experiment?**

It is important to rinse out the burette because potassium permanganate stains glassware.



**Experiment 15.3. (i) To prepare a solution of sodium thiosulfate and (ii) to standardise it by titration against a solution of iodine.**

**Questions on this experiment**

- 1. Describe the appearance of the sodium thiosulfate crystals.**

Sodium thiosulfate is a colourless crystalline solid.

- 2. Give one advantage of using a clock glass rather than paper when weighing out the sodium thiosulfate crystals.**

The clock glass is more suitable than paper since any sodium thiosulfate remaining on the clock glass can be washed off. This is not possible with paper.

- 3. Why is it necessary to remove the funnel from the burette prior to carrying out the titration?**

Failure to remove the funnel may introduce an error into the readings as drops of liquid may fall from the funnel in the course of the titration. This will cause the titration result (titre) to be inaccurate.

- 4. Why is it advisable to use deionised water rather than tap water when making up solutions in this experiment?**

Since water is a good solvent, tap water contains many dissolved ions. These ions could interfere with the reactions taking place.

- 5. Describe how you would take the reading in the pipette when using  $\text{KMnO}_4$ ?**

The reading should be taken from the top of the meniscus.

- 6. Why is it not possible to make up a solution of  $\text{I}_2$  directly? How is this overcome?**

It is not possible to make up the solution directly because iodine vaporises slightly at room temperature. In addition iodine does not dissolve in water.

A standard solution of iodine is obtained by reacting a standard solution of acidified potassium permanganate with excess potassium iodide solution.

- 7. What colour change is observed in the conical flask when the dilute sulfuric acid and potassium iodide solution are added to the  $\text{KMnO}_4$ ?**

The colour in the conical flask changes from purple to a reddish/brown colour.

- 8. What colour change is observed at the end point in this titration?**

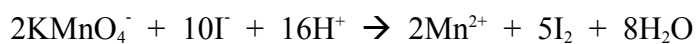
The colour changes from blue/black to colourless.

- 9. Why is it not necessary to know precisely the exact volume of KI added?**

The KI is present in excess in order to ensure that the amount of iodine formed during the titration depends **only** on the amount of potassium permanganate present. In addition, the  $\text{I}^-$  ions are necessary in order to keep in solution the  $\text{I}_2$  that is formed.

**10. Calculate the number of moles of I<sub>2</sub> liberated in the above titration.**

This calculation depends on the average titration figure obtained by the student. From the titration figure and the concentration of potassium permanganate, calculate the number of moles of KMnO<sub>4</sub> that have reacted. From the balanced equation for the reaction:



we see that 1 mole of KMnO<sub>4</sub> gives 2.5 moles I<sub>2</sub>. Hence, the number of moles of I<sub>2</sub> liberated can be easily calculated.

**Experiment 15.4. To determine the percentage (w/v) of sodium hypochlorite in household bleach.**

**Questions on this experiment**

**1. Why was it necessary to dilute the household bleach?**

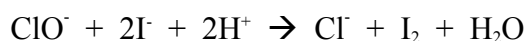
Household bleach is too concentrated to be titrated directly. If the bleach were not diluted, excessive amounts of potassium iodide and sodium thiosulfate would be needed in the experiment. This would increase the cost of performing the experiment.

**2. Why is it particularly important in this experiment to use a pipette filler when placing the household bleach in the volumetric flask?**

Since bleach is a harmful substance it should never be pipetted by mouth in case some of it is accidentally swallowed.

**3. Why was dilute sulfuric acid added to the bleach solution in the conical flask?**

The dilute sulfuric acid is necessary in order to supply the  $H^+$  ions for the following reaction to occur:



**4. Explain why hydrochloric acid should not be used when acidifying the bleach.**

Hydrochloric acid should not be used when acidifying the bleach as chlorine gas could be produced [ $ClO^- + 2Cl^- + 2H^+ \rightarrow Cl^- + Cl_2 + H_2O$ ]

**5. What colour is observed in the conical flask after the addition of the dilute sulfuric acid and the potassium iodide solution to the bleach solution?**

A reddish/brown colour is observed in the conical flask.

**6. What is the indicator used in this experiment? What colour change is observed at the end point?**

The indicator used is starch. The colour change is from blue/black to colourless.

**7. Why is the indicator not added until the solution in the conical flask becomes pale yellow?**

The indicator is not added until the solution becomes pale yellow because if the starch is added at an early stage of the titration, the iodine present may become strongly absorbed onto the starch and this makes the titration less accurate. In addition when the pale yellow colour is observed in the conical flask, it tells us that the end point is quite near. Therefore, when the starch is added at this stage, adding the thiosulfate drop by drop from this stage onwards helps to obtain an accurate titration figure.

**Experiment 16.1. To monitor the rate of production of oxygen from hydrogen peroxide using manganese dioxide as a catalyst.**

**Questions on this experiment**

**1. Define the term *catalyst*.**

A catalyst is a substance that alters the rate of a chemical reaction but is not consumed in the reaction.

**2. Describe the appearance of the manganese dioxide before use.**

A black powdery solid.

**3. Name the products of the decomposition of hydrogen peroxide.**

Water and oxygen.

**4. The hydrogen peroxide is said to be '20 volume'. What does this mean?**

This means that a certain volume of hydrogen peroxide would give off 20 times that volume when it decomposes.

**5. What property of the gas allows collection under water?**

The fact that oxygen is only sparingly soluble in water allows it to be collected under water.

**6. Describe the appearance of the manganese dioxide at the end of the reaction. Would you expect to see a change in appearance? Explain your answer.**

Manganese dioxide is a black powdery solid.

No, because the manganese dioxide acts as a catalyst and a catalyst is usually unchanged at the end of the reaction.

**7. Give one everyday use of manganese dioxide.**

It is used in the manufacture of 'dry' batteries.

**Experiment 16.2. To study the effect of concentration on the rate of reaction between sodium thiosulfate and hydrochloric acid.**

**Questions on this experiment**

**1. What is meant by the term *concentration*?**

The concentration of a solution is the amount of solute that is dissolved in a given volume of solution.

**2. What is the purpose of placing a mark on the paper?**

The X serves as a standard marker that helps us to determine the stage at which the same amount of sulfur has been precipitated for each experiment. It helps us to make sure that the comparison between each experiment is a 'fair test', i.e. that the reaction has proceeded to the same extent in each experiment.

**3. Describe the appearance of the suspension formed.**

The suspension has a cloudy (milky) yellow colour.

**4. Name the element responsible for the cloudy precipitate. Apart from the element causing the yellow suspension, what other products are formed in this reaction?**

The element is sulfur.

The other products formed in the reaction are sodium chloride, sulfur dioxide and water.

**5. Why must the flask and graduated cylinder be rinsed out after each reading?**

This is done in order to avoid contamination of the next set of solutions from the previous experiment, i.e. to ensure that droplets of the previous solutions do not alter the concentrations of the next solutions.

**6. What happened to the rate of reaction as the concentration was decreased?**

As the concentration was decreased, the rate also decreased.

**7. Time and rate are said to be inversely proportional. Explain what this means.**

This means that as time is increased the rate is decreased, i.e. the longer the time it takes the cross to be obscured, the slower is the rate of reaction; the smaller the time taken for the cross to be obscure, the faster the rate of reaction.

**Experiment 16.3. To study the effect of temperature on the rate of reaction between sodium thiosulfate and hydrochloric acid.**

**Questions on this experiment**

**1. What is the variable in this experiment?**

The variable in this experiment is temperature.

**2. Why is a dilute solution of sodium thiosulfate used in this experiment?**

The solution of sodium thiosulfate must be dilute so that the measured times will not be too short at the higher temperatures, i.e. since the rate of reaction increases at higher temperatures, if a concentrated solution were used, the reaction would proceed so fast that it would be difficult to measure the rate.

**3. Why is the temperature taken *after* the hydrochloric acid is added?**

Some cooling may have occurred on addition of the hydrochloric acid.

**4. Why is a stop-clock used in this experiment?**

It is used to accurately measure the time taken to obscure the cross.

**5. The graph for temperature versus rate is a curve, unlike the linear graph for concentration versus rate. Why is this so?**

This is because the rate of reaction is not directly proportional to the temperature.

**6. Give two everyday examples of temperature affecting the rate of a reaction.**

Examples:

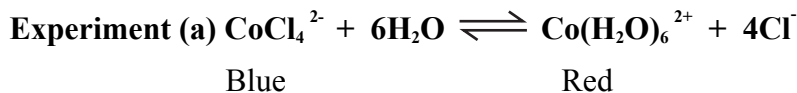
- Food being stored in a fridge. The cold temperature slows down the rate of decomposition of the food.
- Catalytic converters in a car work most efficiently when heated as the rates of the various reactions in the catalytic converter are increased at higher temperatures.

**7. Name the gas given off when sodium thiosulfate reacts with HCl in this experiment? Describe the smell of this gas.**

Sulfur dioxide is given off. It has an irritating (strong, pungent) smell.

**Experiment 17.1. To investigate Le Chatelier's Principle using three named experiments.**

**Questions on this experiment**



- 1. What colour change is observed when concentrated HCl is added to the solution? Explain your observation.**

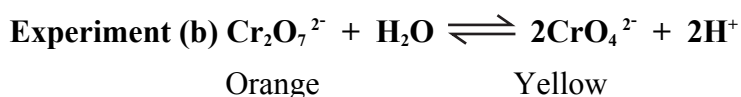
Adding the concentrated HCl causes the red colour to change to blue. The reason for this is because adding the HCl increases the concentration of  $\text{Cl}^-$  ions and, to relieve this stress, the equilibrium is shifted from right to left in accordance with Le Chatelier's Principle. Thus, the concentration of  $\text{CoCl}_4^{2-}$  (blue) is increased and hence the solution turns blue.

- 2. What colour change is observed when water is added to the solution? Explain your observation.**

Adding water to the solution in 1. above, causes the colour to change from blue to red. The reason for this is because, in accordance with Le Chatelier's Principle the equilibrium is shifted from left to right in order to absorb the stress of the increased concentration of water.

- 3. What colour change is observed when the solution is placed in a beaker of hot water? Explain your observation.**

When the solution is placed in a beaker of hot water, the colour changes from red to blue. This is explained by the fact that the forward reaction is exothermic and the backward reaction is endothermic. In keeping with Le Chatelier's Principle, in order to absorb the added heat, the equilibrium is shifted in the direction of the endothermic reaction (reverse reaction). Hence the colour of the  $\text{CoCl}_4^{2-}$  (blue) predominates.



- 4. What colour is observed when sodium dichromate is dissolved in water?**

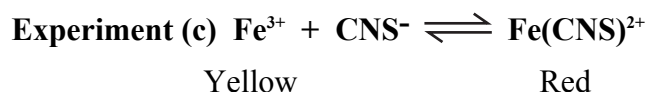
Orange.

- 5. What colour change is observed when sodium hydroxide solution is added to the sodium dichromate solution? Explain this observation.**

When sodium hydroxide is added, the orange colour changes to yellow. The sodium hydroxide reacts with the  $\text{H}^+$  ions on the right-hand side of the above equation and removes them (in the form of water). This gives rise to a stress and in keeping with Le Chatelier's Principle, the forward reaction predominates. This forward reaction produces more  $\text{H}^+$  ions, i.e. the colour of the solution changes from orange to yellow since more  $\text{CrO}_4^{2-}$  (yellow) ions are produced.

- 6. How would you reverse the colour change observed in 5 above?**

To reverse the colour change add dilute hydrochloric acid until the yellow colour changes to orange, i.e. the  $\text{Cr}_2\text{O}_7^{2-}$  (orange) predominates since the equilibrium is shifted to the left to absorb the excess of  $\text{H}^+$  ions.



7. Describe the colour observed when a solution of potassium thiocyanate is mixed with a solution of iron(III) chloride.

A red colour is observed. The red colour is due to the presence of the  $\text{Fe}(\text{CNS})^{2+}$  ion

8. Describe what is observed if some dilute HCl is added to the mixture in 7 above. How would you explain this observation?

If dilute HCl is added to the mixture, the red colour becomes colourless or pale yellow. The reason for this is that the equilibrium is shifted to the left-hand side of the equation in order to absorb the  $\text{Cl}^-$  ions added. This is in keeping with Le Chatelier's Principle since adding the  $\text{Cl}^-$  ions gives rise to a stress.

**Note:** If a lot of HCl is added, the pale yellow colour (due to  $\text{FeCl}_3$ ) may be difficult to observe and the solution appears colourless.



**Experiment 19.1. To determine the total hardness in a water sample.**

**Questions on this experiment**

**1. What is the function of the buffer solution?**

The buffer solution is used to keep the pH constant at around 10. This is necessary in order for the indicator to work satisfactorily.

**2. Why is the edta solution not stored in glass?**

The edta solution cannot be stored in glass as it reacts with the ions in glass.

**3. What colour change is observed during the titration? Explain the reason for the colour change?**

The colour changes from wine red to blue. In the presence of the Eriochrome Black T indicator calcium and magnesium ions have a wine red colour. As the edta solution is added from the burette, the edta forms a complex with the calcium and magnesium ions. When all the calcium and magnesium ions have reacted the indicator changes to a blue colour since there are no calcium and magnesium ions left in solution.

**4. Why is this type of titration called a *complexometric* titration?**

The titration is called a complexometric titration because edta works by wrapping itself around the calcium and magnesium ions in the water. The type of structure formed is called a complex.

**5. Distinguish between temporary hardness and permanent hardness.**

Temporary hardness is hardness that can be removed by boiling the water. Permanent hardness cannot be removed by boiling the water (it is usually removed by ion exchange).

**Experiment 19.2. To determine (a) the total suspended solids (in p.p.m.) of a sample of water by filtration, (b) the total dissolved solids (in p.p.m.) of a sample of water by evaporation and (c) the pH of a sample of water.**

### Questions on these experiments

1. **Suggest a possible cause of high levels of total suspended solids.**

Heavy rainfall results in large amounts of suspended solids (from soil, plants, etc) being washed into rivers and lakes. Water taken from these rivers and lakes would have high levels of total suspended solids.

2. **What undesirable effects could result from high levels of total suspended solids?**

High levels of suspended solids cause the water to be cloudy or brown (due to soil).

3. **How are suspended particles removed in water treatment plants?**

Suspended particles are removed in the flocculation stage, i.e. the suspended solids are made to form larger particles by adding flocculating agents such as aluminium sulfate. The larger particles sink to the bottom of the tank when the water is passed into settlement tanks.

4. **In the experiment to determine the concentration of dissolved solids, why must filtered water be used?**

Filtered water must be used in order to ensure that any suspended solids have been removed from the water. Otherwise an inaccurate reading would be obtained since the suspended solids would be weighed with the dissolved solids when the water is evaporated to dryness.

5. **Suggest a possible reason for high levels of total dissolved solids.**

Since water is such an excellent solvent, it always contains some levels of dissolved solids. High levels of total dissolved solids may be caused by the water flowing through areas that contain large amounts of substances which dissolve in water, e.g. calcium hydrogencarbonate and calcium chloride.

6. **A volume of 1200 cm<sup>3</sup> of water was found to contain 0.09 g of dissolved solids. Express the concentration of the dissolved solids in p.p.m.**

$$0.09\text{g} = 90 \text{ mg}$$

i.e. in 1200 cm<sup>3</sup> there are 90 mg of the dissolved solid.

In 1 cm<sup>3</sup> there are  $\frac{90}{1200}$  mg of dissolved solids.

Therefore, in 1000 cm<sup>3</sup> there are  $\frac{1000 \times 90}{1200}$  mg of dissolved solids

$$= 75 \text{ mg per litre dissolved solids}$$

$$= 75 \text{ p.p.m. dissolved solids.}$$

**7. Suggest a reason for a relatively high pH value in a water sample.**

There are two possible reasons for this:

- (1) The water may come from a limestone region and this can cause the water to be very alkaline.
- (2) It may be due to the fact that the water authority may have softened the water by adding a substance like sodium carbonate at the treatment stage. Since sodium carbonate is a base, this will raise the pH value.

**Experiment 19.3. To measure the amount of dissolved oxygen in a sample of water by means of a redox titration.**

**Questions on this experiment**

**1. Why must the bottles be completely filled?**

The bottles must be completely filled to ensure that no air is present in the bottles as this would raise the level of dissolved oxygen and give an incorrect result.

**2. Why is it necessary to analyse the water immediately after sampling?**

It is necessary to analysis the water immediately after sampling in order to accurately measure the amount of dissolved oxygen in the water at that time. If the water is allowed to stand, it is possible that dissolved oxygen will be consumed by bacteria in the water. Furthermore, the oxygen content may be increased due to photosynthesis by plant material.

**3. When measuring the amount of dissolved oxygen, why should the solutions used be concentrated?**

By using concentrated solutions only small volumes of these solutions need be added to the water being tested. Therefore, the amount of water lost due to displacement is minimised. Also, by using concentrated solutions we ensure that a small volume supplies an excess of reagents to ensure that **all** of the dissolved oxygen takes part in the reactions.

**4. When adding the chemical to the water, why should the tip of the graduated dropper be placed under the surface of the water in the bottle?**

The tip must be under the surface of the water to ensure that the chemicals being added do not react with the oxygen in the air. Also the dropping motion of the chemicals may introduce bubbles of air into the sample.

**5. What precaution should be taken when replacing the stopper of the bottle after the additions of the various chemicals have been made?**

Care must be taken to avoid trapping any oxygen from the air.

**6. Why is it important to shake the bottle vigorously after adding the various chemicals?**

This is done to ensure that all the oxygen dissolved in the water reacts with the reagents.

**7. Assuming that the sample of water contains oxygen, what colour is observed when the concentrated sulfuric acid is added to the water?**

A brown colour is observed. (This colour is caused by the liberated iodine.)

**8. What problems could be caused by chlorine in the water?**

If chlorine is present in the water, it would react with the iodide ions of the potassium iodide to liberate iodine. This additional iodine would give rise to an inaccuracy in our calculations.

**9. What colour change is observed in this titration?**

The starch indicator changes from blue-black to colourless

**10. In carrying out the experiment, what would you conclude if the white precipitate did not turn brown after the addition of the manganese(II) sulfate solution and the alkaline potassium iodide solution?**

The failure of the white precipitate to turn brown indicates that there is no dissolved oxygen in the water.

- 11. In measuring the B.O.D. of a sample of water, it is normal practice to keep a sample in the dark at 20 °C for five days. Why is this necessary?**

The bottle is kept in darkness to prevent photosynthesis taking place as this would increase the level of oxygen in the water. The fixed temperature of 20 °C gives a valid comparison of different water samples since the amount of oxygen dissolved in water depends on temperature. Also, the rate at which oxygen is consumed depends on temperature.

Five days is used to ensure a standard period of time to allow all the dissolved oxygen to be used up.

**Experiment 19.4. To estimate the concentration of free chlorine in swimming-pool water or bleach using (a) a comparator or (b) a colorimeter.**

**Questions on this experiment**

**1. What is meant by the term *free chlorine*?**

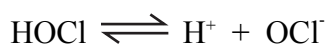
Free chlorine is chlorine that exists in water as hypochlorous acid and hypochlorite ion.

**2. What problem is caused for swimmers when the concentration of free chlorine is too high in swimming pools?**

The excess chlorine causes irritation to swimmers' eyes.

**3. Is free chlorine an acidic or basic substance? Explain your answer.**

Free chlorine is an acidic substance since the hypochlorous dissociates to form  $H^+$  ions and  $OCl^-$  ions. The  $H^+$  ions make the water acidic.



**4. What is the principle behind the working of either (a) a comparator or (b) a colorimeter?**

(a) When using a comparator, a chemical called DPD is added to the water and reacts with the chlorine in the water. A pink/coloured solution is formed. The more chlorine that is present in the water, the pinker the colour. The intensity of the pink colour is then compared against a coloured disc or strip of paper with different shades of pink. This disc/paper has already been calibrated using solutions of known concentration of chlorine. Hence by comparing the pink colour of the swimming pool water with the pink colours of known concentrations of chlorine, the concentration of chlorine in the sample of water may be estimated.

(b) In the colorimeter a narrow beam of light of a certain wavelength is passed through the solution of swimming pool water containing DPD. The light that emerges from the solution falls on a photocell. The photocell converts light energy into an electrical signal which registers on a digital display. The amount of light absorbed depends on the concentration of the coloured substance in the solution being tested. By plotting a graph of the amount of light absorbed vs the concentration of free chlorine in a number of standardised solutions, it is possible to calculate the concentration of chlorine in the sample being tested. See *Chemistry Live!*, p. 287, Fig. 19.23.

**5. What do the letters DPD stand for?**

Diethyl-phenylenediamine

**6. What is observed when a DPD tablet is added to water containing free chlorine?**

The water turns a pink colour.

**7. Why is it necessary to use DPD tablets in this experiment?**

DPD is used because it reacts with the chlorine in the water to give a coloured solution. The intensity of the colour depends on the concentration of chlorine present. Hence it is possible to estimate the concentration of chlorine in the water using either method.

**8. In a certain school, swimming pool water was not available. What alternative could be used for this experiment?**

A dilute solution of bleach could be used.

**9. In what units is the concentration of free chlorine normally measured?**

Parts per million (p.p.m.).

**10. What is the main source of nitrogen compounds in swimming pool water?**

Sweat.

## Experiment 21.1. To prepare ethene and to examine its properties.

### Questions on this experiment

1. Describe the physical appearance of aluminium oxide.

Aluminium oxide is a white powder.

2. Why is the glass wool not heated directly?

The glass wool is not heated directly as the alcohol would evaporate too quickly. By heating the aluminium oxide rather than the glass wool, the heat is transferred slowly to the glass wool causing the alcohol to evaporate at a steady rate. Also, the catalyst works better at the higher temperature.

3. The delivery tube must be removed from the water before the heating is stopped. Explain the reason for this.

If the Bunsen is turned off while the delivery tube is dipping into the water, the air inside the boiling tube contracts as it cools. This gives rise to a partial vacuum and water would be sucked in to fill this vacuum. In short, to prevent a suck back.

4. The aluminium oxide powder serves two purposes. Name them.

The aluminium oxide serves as both a catalyst and a dehydrating agent.

5. Why do we not perform any tests on the first test-tube of gas collected?

The reason for this is because the first test-tube of gas collected consists mainly of air that has been displaced from the apparatus.

6. Describe the type of flame produced when ethene is burned.

Ethene burns with a clear (not sooty) luminous flame. The colour of the flame is blue if plenty of air is present or yellow if insufficient air is present.

7. Describe what you observe when bromine water is added to ethene in a test-tube and the stoppered test-tube is shaken.

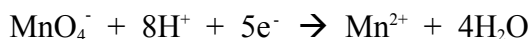
The yellow/red colour of the bromine water changes to colourless.

8. What do you observe when acidified potassium permanganate solution is added to ethene and the stoppered test-tube is shaken?

The purple colour of the potassium permanganate solution changes to colourless.

9. Why is it necessary to use *acidified* potassium permanganate solution in the above test?

If  $H^+$  ions are not present, the potassium permanganate cannot act as an oxidising agent since the  $H^+$  ions are needed for the reaction:



10. Give one use of ethene.

Ethene is used to speed up the ripening of fruit and is also the starting material from which many other substances are made. It is often referred to as industry's most important organic chemical.



## Experiment 21.2. To prepare ethyne and to examine its properties.

### Questions on this experiment

1. **Why is the water added drop-wise and not simply poured on top of the calcium carbide?**

The water is added drop-wise in order to control the rate at which ethyne is formed.

2. **Describe the physical appearance of calcium carbide.**

Calcium carbide is a solid material usually coloured either grey or brown (depending on what impurities are present).

3. **What do you observe happening inside the flask?**

The calcium carbide changes to a white powder and bubbling/effervescence is noticed inside in the flask. If you place your hand on the outside of the flask you will also notice that heat is given off in the reaction.

4. **How would you determine whether the reaction between calcium carbide and water is exothermic or endothermic?**

Measure the temperature before the water is added to the calcium carbide and after the water is added to the calcium carbide.

5. **Why is it necessary to bubble ethyne through acidified copper sulfate solution?**

It is necessary to bubble ethyne through acidified copper sulfate solution in order to remove impurities in the gas. (The impurities may be hydrogen sulfide  $\text{H}_2\text{S}$ , phosphine  $\text{PH}_3$ , and ammonia  $\text{NH}_3$ .)

6. **Describe the type of flame produced when ethyne is burned in a gas jar.**

The ethyne burns with a smoky luminous flame.

7. **What is observed when bromine water is added to ethyne?**

The yellow/red colour of the bromine water changes to colourless.

8. **Describe what you observe happening when acidified potassium permanganate is added to ethyne.**

The purple colour of the acidified potassium permanganate changes to colourless.

9. **At the end of the experiment, a white substance is left behind in the Buchner flask. What is the name of this compound?**

Calcium hydroxide.

10. **Give one use for ethyne gas.**

It is used for welding and cutting metals.

**Experiment 21.4. To determine the heat of reaction of hydrochloric acid with sodium hydroxide.**

**Questions on this experiment**

**1. Why are the liquids mixed in an insulated container?**

They are mixed in an insulated container in order to prevent heat loss to the surroundings. It is important that heat is not lost to the surroundings as we wish to ensure all the heat produced in the reaction is used to raise the temperature of the solution.

**2. Explain why it is important that both solutions should be at the same temperature before they are mixed.**

Since the calculations involve measuring the rise in temperature of the final solution, it is important that both solutions must be at the same temperature initially in order to measure the rise in temperature accurately.

**3. A teacher reminded a student that when adding the base to the acid care should be taken to avoid splashing. Explain why it is important to avoid splashing.**

It is important to avoid splashing as this splashing would give rise to loss of liquid and hence cause an inaccuracy in the calculations.

**4. What is the purpose of placing a lid on the plastic cup?**

The lid helps prevent loss of heat to the surroundings.

**5. Students were reminded by a teacher to record the maximum temperature reached on stirring the mixture of liquid continuously. Why is the *maximum* temperature recorded? Why is continuous stirring important?**

The maximum temperature is recorded in order to accurately calculate the heat liberated.

Heat liberated = mass of solution  $\times$  specific heat capacity  $\times$  temperature rise

If the maximum temperature is not recorded then the value for temperature rise will be incorrect. Therefore the calculation of heat liberated will be inaccurate.

Continuous stirring is important to ensure that the heat liberated is uniformly distributed throughout the solution, i.e. to make sure that the temperature measured does not depend on the position of the thermometer in the solution.

**6. Why is a plastic container particularly suitable for use in this experiment?**

A plastic container is ideal for use in this experiment since plastic absorbs very little heat, i.e. the specific heat capacity of plastic is negligible. Hence a more accurate result is obtained.

**7. When carrying out the calculations associated with this experiment, what assumptions are made about the density of the solutions and the density of water?**

It is assumed that the density of the solutions is the same as the density of water. This is a valid assumption as the solutions used are not highly concentrated.

**8. Comment on the sources of experimental error in this experiment.**

Possible sources of experimental error:

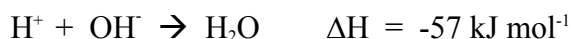
- Splashing.
- Mixing solutions which are not at the same initial temperature.
- Forgetting to wash the thermometer after measuring the temperature of the acid and before measuring the base.
- Forgetting to stir the final solution after mixing.
- Not measuring the maximum temperature rise.
- Forgetting to place a lid on the final mixture.
- The thermometer being used may only give an accurate reading to the nearest degree.

**9. Would you expect a similar result if nitric acid were used instead of HCl?**

Yes, a similar result would be obtained since nitric acid is similar to HCl in that both are strong acids, i.e. both fully dissociate in water.

**10. Why does the heat of neutralisation of a strong acid and a strong base always have a constant value?**

They have a constant value because the same reaction is taking place in all cases, i.e:



The other ions are 'spectator ions' and are not involved in the reaction.

## Experiment 22.2. To extract clove oil from cloves by steam distillation.

### Questions on this experiment

1. Write a brief note to explain how steam distillation is used to extract oils from plant materials.

When steam is passed through the plant material, it forms a gaseous emulsion with the oil in the plant material. This very hot emulsion is forced under pressure into a Liebig condenser which converts the gaseous emulsion into a liquid. The oil may then be extracted from the emulsion using an organic solvent.

2. Why is it not possible to distil the clove oil directly from the cloves?

If the cloves were heated directly, the heat would cause them to char and the oil would be destroyed. Steam is used as the temperature is kept slightly above 100 °C and at this temperature the clove oil is not destroyed.

3. Why is the product of this experiment referred to as an *emulsion*? Name one other common emulsion found in everyday life.

An emulsion is formed when droplets of oil are dispersed in a liquid like water. Many paints are sold in the form of emulsions. Mayonnaise is also an emulsion of oil in water.

4. In carrying out this experiment a long glass tube open at both ends is usually placed in the steam generator. What is the reason for this?

The long glass tube is a safety tube and is used to ensure that there is not a build up of pressure in the steam generator. As the pressure increases inside the steam generator, water is pushed up the safety tube.

5. What is the purpose of the steam trap in the experiment?

As the name suggests, the steam trap collects or traps steam that has condensed to water. If the trap were not present, the flask containing the cloves would quickly fill up with water from the condensed steam.

6. Describe how you would isolate the clove oil from the distillate.

The clove oil can be isolated using a process called **solvent extraction**. This involves adding an organic solvent to the emulsion. A suitable organic solvent is petroleum ether or dichloromethane. The emulsion and organic solvent will be added to a separating funnel and the two liquids shaken for a few minutes in the stoppered funnel. The two liquids separate into layers and the water layer is discarded. The organic solvent is allowed to evaporate leaving the oil behind in the evaporating basin.

7. What name is given to the main constituent of the oil found in cloves?

Eugenol.

8. Why are cloves usually picked before they flower?

They are picked before they flower because this is the stage at which they are richest in oil.

9. Give two uses for clove oil.

Clove oil is used for flavouring food products, in antiseptics, and in perfumes and soaps.

10. Why is it better to use whole cloves rather than powdered cloves in this experiment?

Powdered cloves have usually lost a lot of their oil as a result of being ground to powder.

### Experiment 23.1. To prepare a sample of soap.

#### Questions on this experiment

1. **This experiment is performed over two stages. Name each stage. Why is each stage necessary?**

The first stage involves **refluxing** and the second stage involves **distillation**. Refluxing is necessary in order to bring about saponification, i.e. hydrolysis of the ester (found in the oil or fat) to form sodium stearate and the alcohol glycerol. Distillation is necessary in order to remove the ethanol solvent.

2. **Describe what is observed in the first stage of this experiment.**

During the reflux stage it is observed that the walls of the flask become coated with an oily or fatty substance. This is caused by the fact that some of the oil or fat has not yet been hydrolysed. It is also observed that some of the liquid is continuously being changed to a gas, condensing to a liquid in the condenser and falling back into the flask.

3. **Why is it necessary to swirl the flask from time to time in the first stage of the experiment?**

It is necessary to swirl the flask in order to remove the coatings from the inside of the flask so that they mix with the solution in the flask and become hydrolysed. In addition, swirling the flask helps to distribute the heat uniformly among the reactants in the flask.

4. **What is the function of the anti-bumping chips?**

The anti-bumping chips help to bring about smoother boiling and prevent the flask from shaking.

5. **Why is the sodium hydroxide kept in excess in the first stage of the experiment?**

The sodium hydroxide is kept in excess in order to ensure that the hydrolysis reaction takes place completely, i.e. to maximise the amount of saponification that takes place.

6. **In the second stage of the experiment, why is it necessary to remove the ethanol by distillation?**

It is necessary to remove the ethanol by distillation in order to isolate the soap. If too much ethanol is present the soap will remain in solution.

7. **What is brine? What is its function in this experiment?**

Brine is a solution of salt (NaCl) in water. Its function in this experiment is to precipitate the soap as soap does not dissolve in brine.

8. **Describe the appearance of the soap you have isolated.**

The soap is isolated as a white solid. It may be coloured due to impurities present in the original lard or oil.

9. **Describe how the hardness of water affects soap.**

Soap does not form a lather with hard water but forms a scum instead.

10. **Why is the soap washed with salt solution at the end of the experiment?**

It is washed with salt solution as this removes any sodium hydroxide still present in the soap.

## Experiment 23.2. To prepare ethanal and examine its properties.

### Questions on this experiment

1. Describe the physical appearance of sodium dichromate.

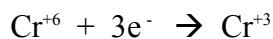
Sodium dichromate is an orange crystalline solid.

2. Why is excess alcohol rather than excess sodium dichromate used in this experiment?

Excess alcohol rather than excess sodium dichromate is used in order to prevent further oxidation of the ethanal to ethanoic acid.

3. What colour change is observed during the experiment? Explain why this colour change occurs.

The colour change observed during this experiment is from orange to green. This is caused by the fact that the sodium dichromate is reduced from Cr in an oxidation state of +6 (orange) to an oxidation state of +3 (green), i.e the following reaction occurs:



Orange                      Green

4. When diluting concentrated sulfuric acid, why is the concentrated sulfuric acid added to the water rather than water added to the sulfuric acid?

Concentrated sulfuric acid is more dense than water. Therefore when added to water it sinks into the water and is distributed throughout the water by careful mixing. However if water is added to concentrated sulfuric acid, the water floats on top of the concentrated sulfuric acid and the heat generated converts some of the mixture into a hot acid spray.

5. What impurities may be found in the ethanal prepared? Suggest how *one* of these impurities could be removed.

The impurities are mainly ethanol, ethanoic acid and water.

The water can be removed by adding a substance like anhydrous sodium sulfate, which absorbs the water.

The other impurities are removed by distilling the ethanal and collecting the fraction boiling between 20 °C and 23 °C. This gives pure ethanal.

6. Why is the collecting flask normally surrounded by ice?

The ice is necessary as ethanal has a very low boiling point of 21 °C and hence a lot of ethanal could be lost to evaporation.

7. Describe the colour change observed when acidified potassium permanganate solution is added to ethanal. Name the organic product formed.

The purple colour of the potassium permanganate changes to colourless.

The organic product formed is ethanoic acid.

- 8. Describe the colour change observed when Fehling's solution is added to ethanal. Name the organic product formed.**

The blue colour of the solution disappears and a red precipitate is formed. The organic product formed is ethanoic acid.

- 9. What is observed when ammoniacal silver nitrate is added to ethanal in a clean test-tube?**

When added to ethanal, the clear ammoniacal silver nitrate solution causes a silver mirror to be deposited on the inside of the test-tube.

- 10. What would you expect to observe when Fehling's reagent is added to some propanone in a test-tube? Explain your answer.**

No change is observed since Fehling's reagent does not oxidise ketones.

**Experiment 23.3 and 22.1. To prepare ethanoic acid and examine its properties.**

**Questions on this experiment**

1. **Describe the physical appearance of ethanol.**

Ethanol is a colourless liquid.

2. **What is meant by the term *refluxing* in chemistry?**

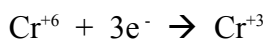
Refluxing is a laboratory technique in which a liquid is boiled in a container that is attached to a vertical condenser. Vapour from the boiling liquid condenses in the cooled vertical condenser and flows back into the flask. Thus the liquid is kept at its boiling point without loss of vapour.

3. **Give one advantage of passing the mixture of ethanol and water through the Liebig condenser.**

Adding the mixture of water and alcohol through the Liebig condenser helps to cool the mixture of alcohol and water. As a result the reaction between the alcohol and the acidified sodium dichromate solution is more controlled.

4. **What colour change do you observe taking place in the flask during the reaction? Explain your observation.**

The colour change observed in the flask during this experiment is from orange to green. This is caused by the fact that the sodium dichromate is reduced from Cr in an oxidation state of +6 (orange) to an oxidation state of +3 (green) i.e the following reaction occurs:



Orange                      Green

5. **From your observation of the reaction that takes place in the flask during reflux, is the reaction exothermic or endothermic? Give a reason for your answer.**

The reaction is endothermic since it is observed in the flask that each time the ethanol solution comes in contact with the acidified sodium dichromate solution, boiling occurs due to the heat given out.

6. **Why is it necessary to reflux the contents of the flask for about 20 minutes?**

It is necessary to reflux the contents of the flask in order to ensure that the ethanol is oxidised completely to ethanoic acid. Since this is an organic reaction it proceeds slowly.

7. **In the second part of the experiment, why is the mixture heated using a Bunsen burner rather than a water bath?**

The mixture is heated using a Bunsen burner rather than a water bath because ethanoic acid has a boiling point greater than 100 °C. (Boiling point ethanoic acid = 118 °C).

8. **Describe the appearance of the distillate formed in this experiment.**

The distillate is a clear colourless liquid.



**9. Name three possible impurities present in the final distillate.**

Possible impurities present in the final distillate are ethanal, water, ethanol and ethyl ethanoate.

**10. Describe the smell of the product formed when ethanoic acid reacts with ethanol in the presence of sulfuric acid.**

The product formed is an ester called ethyl ethanoate and has a sweetish or 'glue'-like smell.

**11. What general term is used to describe the reaction between a carboxylic acid and an alcohol in the presence of sulfuric acid?**

Esterification.

**Experiment 23.4. To separate a mixture of indicators or coloured substances using paper chromatography or thin-layer chromatography or column chromatography.**

**Questions on this experiment**

**1. What is meant by the term chromatography?**

Chromatography is a separation technique in which a mobile phase carrying a mixture moves in contact with a selectively adsorbent stationary phase.

**2. Name the scientist who invented the technique of chromatography.**

Mikhail Tswett.

**3. What is the principle on which all chromatographic separation techniques are based?**

The mobile phase flows over the stationary phase and the components of the mixture are separated on the stationary phase. The separation is caused by the fact that components which tend to be held by the stationary phase move more slowly than those which mix well with the mobile phase.

**4. In the experiment undertaken by you, what is the mobile phase and what is the stationary phase?**

| Type                      | Mobile       | Stationary             |
|---------------------------|--------------|------------------------|
| Paper Chromatography      | Ink solution | Paper                  |
| Thin Layer Chromatography | Food dyes    | Aluminium oxide plate  |
| Column Chromatography     | Food dyes    | Aluminium oxide column |

**5. Why should chromatography paper be handled as little as possible with bare hands?**

To avoid oils from the hands contaminating the results.

**6. State which colour inks may be good for separating. Explain your answer.**

Black and brown ink are good for separating because they are usually made by mixing a number of colours.

**7. In chemistry what do the letters TLC represent?**

Thin Layer Chromatography.

**8. What is the process of passing a solvent through a column called?**

Elution.

**9. Name another substance, other than inks and dyes, that could be analysed by chromatography?**

Universal indicator or chlorophyll or alcohol levels in blood.

**10. In paper chromatography why is it important that the level of solvent is below the location of the sample being analysed?**

The level of solvent must be below the location of the sample to allow the solvent to slowly rise through the paper and hence get a good separation of colours. If immersed in the water the sample would simply dissolve without separation taking place.