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INTERNATIONAL ADVANCED LEVEL

CHEMISTRY

TEACHER PRACTICAL GUIDE

Pearson Edexcel International Advanced Subsidiary in Chemistry (XCH11)
Pearson Edexcel International Advanced Level in Chemistry (YCH11)
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Pearson

Contents

Introduction

This guide is designed to:

1. support you and your students through all elements of practical work in the new International AS and A level specification. Although it will address assessment arrangements, its focus is to ensure good quality practical work is at the heart of teaching and learning in the subject,
2. explain how the new requirements for practical skills can be developed throughout the course using both core practicals and other specification content.

The over-arching aim of the specification is to help learners progress from being International GCSE/GCSE students towards becoming ready for the next stage of their development, whether that be university or the workplace. To some extent this can be through developing skills such as non-routine problem solving and ICT literacy but also by personal skills in communication, adaptability and self-management.

In terms of practical work, the aim is for students to become capable of thinking independently. Part of this is developing confidence in their own competence to challenge accepted practice and ask 'How do I know that?', whilst thinking about the science behind the observations. This may be exhibited by e.g. working towards thinking independently in planning and evaluating for themselves the outcome of practical work.

Over the course of the new International A level, students will develop a range of skills in practical work. This will vary from the acquisition of specific practical techniques in a range of experiments (the use of a burette or assembling apparatus for carrying out a distillation), through to the development of some investigative techniques requiring independent thinking (planning and carrying out an experiment to determine the effect of a change in concentration of a reactant in an unfamiliar reaction).

At one level, practical work undertaken by students can be simple, perhaps focusing on observational aspects of the subject (such as Core Practical 8: Analysis of some inorganic and organic unknowns), whereas other practical experiences may be truly experimental (such as Core Practical 9a: Following the rate of the iodine-propanone reaction by a titrimetric technique).

Many experimental activities will involve the collection of quantitative data; and this provides opportunities for the development of mathematical skills, which are also required as part of the specification (see Appendix 6 of the specification).

There is a students' guide designed to be used alongside this teacher resource, which is written to explain the new requirements to your students in a straightforward way and to provide exercises to allow them to develop their skills. You will find the suggested answers to these exercises are included in this teachers' guide.

Practical assessment – the whole picture

At first glance, the specification contains a number of references to practical work – both in terms of delivery and assessment – so it might be helpful, at the start of this Guide, to see what these are, and how they relate to each other.

This is particularly important because, as a teacher of the subject, you will naturally want to focus most closely on what is best for your students in terms of skills development and exam preparation.

The most obvious place to start is an assessment overview. Units 3 and 6 remain primarily focussed on the assessment of practical skills and each cover 10% of the total international A Level specification.

Unit 3 exam will assess candidate's knowledge and understanding of experimental procedures and techniques developed in Units 1 and 2.

Unit 6 will also draw on these skills but also assess candidate's knowledge and understanding of experimental procedures and techniques developed in Units 4 and 5.

These exam papers constitute the formal assessment of practical work. The assessment of the core practicals by teachers is of an informal nature and does not contribute to the A Level grade for the students but nonetheless the feedback to students will be invaluable in their preparation for examinations.

How do the core practicals cover the techniques and apparatus?

As you will see from the list of core practicals on page 11, some of the techniques and apparatus will be covered several times; whereas others may only be addressed once. This is useful because it gives you an idea of which core practicals are especially important for students to complete.

How flexible are the core practicals?

The core practicals represent our suggestion for ensuring that you cover the procedures and techniques without having to worry. Of course, they aren't the only way – so you are free to develop your own practical program.

More commonly, you may find that one or two of the core practicals do not suit you, possibly due to resources, or because you have a preferred practical activity in a particular topic area. In this case, feel free to swap our core practical for one of your own.

However, we would very much recommend that you did all the practicals that we specify – and, where time permits, more on top! Any practical work that your students attempt – not just the core practicals – will definitely help them with their preparation for their examinations.

Preparing students for questions assessing practical understanding

In addition to the new Sample Assessment Materials (SAMs), there are many suitable examples in past papers that could be used for students to prepare for this type of questions. Past papers are available on the Edexcel website at both AS (Unit 3) and A level (Unit 6) standard.

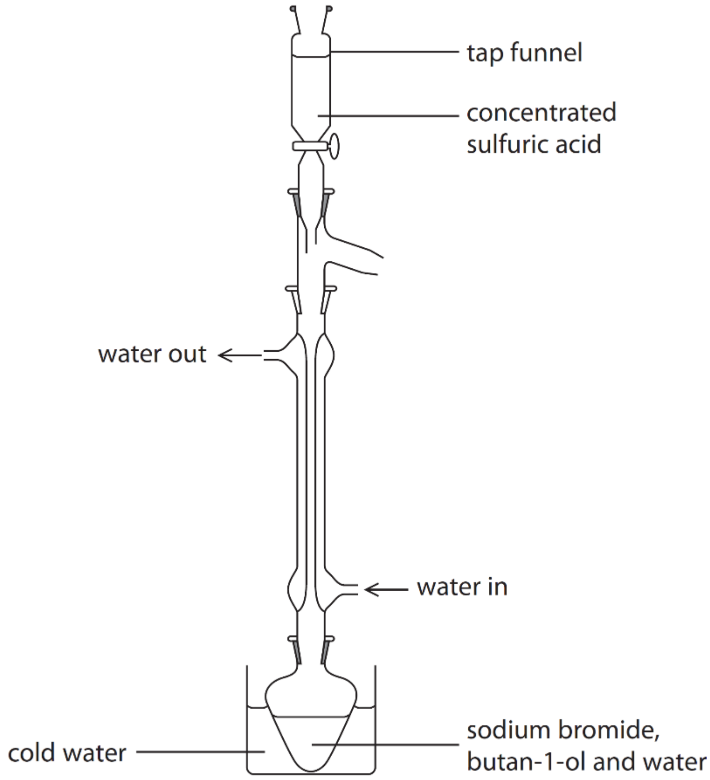
Sample questions

Here is a typical example form an International A level paper (WCHO3/01 January 2015).

4 One method of preparing 1-bromobutane from butan-1-ol is given below.

Procedure

Step 1 10 g of sodium bromide, 10 cm^3 of water and 7.5 cm^3 of butan-1-ol are placed in a flask. The flask is partially immersed in a large beaker of cold water. A condenser is fitted vertically in the neck of the flask as shown in the diagram.



Step 2 10 cm^3 of concentrated sulfuric acid is dripped slowly from the tap funnel into the reaction mixture. The flask is shaken gently.

Step 3 The tap funnel is removed from the top of the condenser and the flask is taken out of the cold water bath. The flask is then heated gently for about 45 minutes.

Step 4 The apparatus is then rearranged for distillation. The 1-bromobutane and water are distilled into a small beaker where they form two layers.

Step 5 The 1-bromobutane layer is separated from the water.

Step 6 The 1-bromobutane layer is washed with concentrated hydrochloric acid to remove unreacted butan-1-ol.

Step 7 The 1-bromobutane is then washed with dilute sodium carbonate solution.

You will need the following data to answer the questions.

Butan-1-ol, $CH_3CH_2CH_2CH_2OH$ $M_r = 74$

1-bromobutane, $CH_3CH_2CH_2CH_2Br$ $M_r = 137$

Liquid	Density / g cm^{-3}
butan-1-ol	0.81
water	1.0
concentrated hydrochloric acid	1.2
1-bromobutane	1.3

(a) The use of the beaker of cold water in **Step 1**, and the slow addition of concentrated sulfuric acid in **Step 2**, both prevent a reaction which gives unwanted **inorganic** products.

Identify **one** of these unwanted products. State the type of reaction occurring when these products form.

(2)

Product ....

Type of reaction ....

....

(b) (i) Explain why the condenser is set up so that the water flows from bottom to top, as shown in the diagram.

(1)

....

....

....

(ii) Without the reflux condenser, the procedure in **Step 2** would become more hazardous. Explain why.

(1)

....

....

(c) To achieve the best possible yield of 1-bromobutane, the purification stages should involve the minimum number of transfers of the organic product from one piece of apparatus to another.

(i) How could the water layer be removed from the small beaker in **Step 5** without transferring the organic product?

(1)

....

(ii) Name the apparatus you would use to carry out the washing of the crude 1-bromobutane in **Step 6**.

Describe how you would obtain the organic layer from this mixture.

(2)

....

....

....

....

(d) What is the purpose of **Step 7**?

(1)

....

....

(e) After **Step 7**, the crude 1-bromobutane is washed with pure water and separated again. Two further steps are needed to obtain a pure sample of 1-bromobutane.

State what these steps are. Detailed experimental procedures are not required, but you should name any reagents which are needed.

(3)

Step 8 ....

....

....

....

Step 9 ....

....

(f) (i) Calculate the mass of butan-1-ol used in **Step 1**.

(1)

(ii) In this experiment, a student obtained 7.5 g of 1-bromobutane.

Calculate the percentage yield of 1-bromobutane. Assume that each mole of butan-1-ol can produce a maximum of one mole of 1-bromobutane.

Give your answer to **two** significant figures.

(3)

(Total for Question 4 = 15 marks)

Commentary

This question could be set in either Unit 2 or Unit 3 at AS level, testing the specification statement 10C, 10.17(ii): bromination of alcohols.

Part (a) deals with possible side reactions; a useful teaching point since students often gain the impression, particularly in Inorganic Chemistry, that only one set of products is possible for a given set of reactants.

Parts (b) to (e) focus on the practical aspects of the preparation, asking questions on why and how various steps are carried out. Experience indicates that most students are adept at describing how to carry out preparations, but far fewer are capable of explaining why particular procedures are used.

Part (f)(i) is a simple mass calculation that is not level 2 mathematics. Part (f)(ii) is a slightly more sophisticated calculation of percentage yield, with at least one mark at level 2 for the appropriate use of significant figures.

Core Practicals

The Core Practicals are an integral part of your course. They are not there to get you to demonstrate some text book 'fact' or recall some simple information. They are there to help you develop the whole range of practical and mathematical skills which are essential to chemists and which will be tested in the written assessments.

List of Core Practicals

1. Measuring the molar volume of a gas
2. To determine the enthalpy change of a reaction using Hess's Law
3. Finding the concentration of a solution of hydrochloric acid
4. Preparation of a standard solution from a solid acid and use it to find the concentration of a solution of sodium hydroxide
5. Investigation of the rates of hydrolysis of halogenoalkanes
6. Chlorination of 2-methylpropan-2-ol with concentrated hydrochloric acid
7. The oxidation of propan-1-ol to produce propanal and propanoic acid
8. Analysis of some inorganic and organic unknowns
9. Following the rate of the iodine-propanone reaction by a titrimetric method and investigating a 'clock reaction' (Harcourt-Esson, iodine clock)
10. Finding the activation energy of a reaction
11. Finding the K_a value for a weak acid
12. Investigating some electrochemical cells
13. Carry out redox titrations with both: (i) iron(II) ions and potassium manganate(VII) (ii) sodium thiosulfate and iodine
14. The preparation of a transition metal complex
15. Analysis of some inorganic and organic unknowns
16. The preparation of aspirin

Using Core Practicals to Teach Skills

The most important principle, which will be reiterated throughout this guide, is that the assessments stress the idea of developing skills. Whilst students are expected to have some knowledge of the techniques and procedures they encounter throughout the course, recalling small details is the simplest part of what is required.

Developing skills implies that there is significant progression in terms of independent thinking and understanding of the underlying science behind what they are undertaking.

We have selected core practicals to be included in our specification that are accessible to all students and provide opportunities to develop the skills listed, and not because they are 'perfect' examples of experimentation or can be used to demonstrate a textbook 'fact'.

In addition to the core practicals, schemes of work need to include at least some of the experiments given in the introduction to each specification topic and in the topic itself.
For example in Topic 17: Transition metals.

Section	Practical work	In the laboratory
19	Reduction of vanadium(V) to vanadium(II) to show the colours of the oxidation states	As a teacher demonstration: Add Zn to an acidified solution of NH_4VO_3 to show the changes in colour as vanadium is reduced through its successive oxidation states
22	Addition of NaOH(aq) and NH_3(aq) to transition metal ions	On a test tube scale: add dilute NaOH(aq) and dilute NH_3(aq), until in excess, to the TM ions listed in 24.
24	Reactions of copper(II) ions The formation of: $[Cu(NH_3)_4(H_2O)_2]^{2+}$ $[CuCl_4]^{2-}$	On a test tube scale: the addition of NH_3(aq) to Cu^{2+}(aq) the addition of conc. HCl to Cu^{2+}(aq)
24	Ligand substitution: The formation of $[CoCl_4]^{2-}$ from $[Co(H_2O)_6]^{2+}$	On a test tube scale: add conc. HCl to aqueous Co^{2+}(aq)
32	Autocatalysis	As a teacher demonstration: show that the rate of reaction increases as Mn^{2+} is formed in the reaction between MnO4- and $C_2O_4^{2-}$ ions
Core Practical 14	The preparation of a transition metal complex.	Students may work in pairs and must make a record of the preparation: Preparation of $[Cu(NH_3)_4]SO_4.H_2O$ or $Fe(COO)_2.2H_2O$

Other topics in the specification may be incorporated into a scheme of work in this way. Many of the practicals are quite simple and need only take part of a lesson.
Students may always work in pairs but should keep separate records of their work.

Progression

Students beginning a two-year International A Level Chemistry course will have some experience of practical laboratory work from their International GCSE/GCSE studies.

They will have developed some practical skills and be able to recognise and safely use simple laboratory equipment. They should be familiar with common laboratory substances such as dilute acids and alkalis and be capable of using these in practical work, paying due regard to the safety of themselves and of others.

Students beginning the course should appreciate that there is a scientific method involved in Chemistry. They will have carried out enough practical work to understand that Chemistry has developed as a science on the basis of observations and measurements made in the laboratory.

In their International A Level Chemistry course, students will build on the laboratory skills developed at GCSE. They will use more complex equipment and handle a greater range of substances. It is likely that they will spend more time carrying out practical work than at International GCSE/GCSE so will need to keep detailed records of their experiments.

Planning practical lessons

It is a common belief that simply including practical sessions within the course is a 'good thing' which helps to motivate students. However, this is not always the case - as students will often say themselves! Like any other lesson, students are only engaged when they see clear aims and objectives which are relevant to their course. These aims must be relevant, achievable and explicit.

As teachers, we know what is likely to happen during any one practical session. We also know that the time available for each practical is limited so it is vital to consider what our main objectives for the lesson are going to be in terms of the practical skills requirement.

This has two main advantages:

- It avoids student frustration and end of practical negativity ('It never works!', 'That was a waste of time.')
- It enables teachers to track practical skills development. Practicals with clear skills aims can be easily recorded for each group, thereby providing confirmation that all the skills are being covered within the scheme of work. This is especially important where more than one teacher is assigned to one teaching group.

Remember that, as well as demonstrating skills, practical work will also be the vehicle for assessment in written question papers; and may frequently be associated with the assessment of mathematical skills. This needs to be reflected in teaching time during the A level course and careful planning of the use of core practicals can play a vital part in developing the practical and mathematical skills required for success at A level, rather than simply concentrating on theoretical content.

Developing maths skills through core practicals

Full details of the mathematical skills requirements can be found in **Appendix 6** of the specification along with exemplification of each assessment objective.

- All of the skills are to be examined at level 2.
- There will be **at least 20% of the marks awarded** at Advanced Level and for Advanced Subsidiary Level for mathematical skills.
- Mathematical skills will be expected in papers for units 1, 2, 4 and 5.

What is meant by level 2 mathematics?

Level 2 mathematics is of the standard of higher tier GCSE/International GCSE. The definition of level 2 can be affected by the context in which it is applied. For instance, where there is a great deal of structure or 'scaffolding' for a question then the demand is lower and may not be level 2.

Simply providing a formula and a list of data which is carefully defined would involve only simple substitution and be unlikely to meet level 2. However, selecting which data to apply and which formula to use would.

It is also useful to understand what is not regarded as mathematical skills. Questions often provide data from which students are expected to make conclusions by applying their chemical knowledge and understanding. Even though data may be involved this would not be regarded as mathematical skill. Similarly, simply defining mathematical terms or describing them would not be a mathematical skill.

The new requirements for mathematics in all science specifications have obvious implications for planning schemes of work. It is common for chemistry teaching groups to contain students with varied mathematical ability and careful thought will be needed to meet their needs.

We cannot assume that even students who perform well at higher tier GCSE/International GCSE mathematics will retain their knowledge and competence at the end of two years without further practice and development.

Past **examiners reports** show that there are several areas in which many students are not totally confident. These include;

- calculating percentage increase/decrease
- selection, application and accurate presentation of graphical formats
- manipulating formulae.

There are also areas, such as calculations using the logarithms and exponentials, that are likely to be new to students regardless of their mathematical background.

The implication of this is clear. There needs to be specific learning opportunities integrated into the course which provide reinforcement and training in these mathematical areas, if students are to develop confident mastery; enabling them to perform to the best of their ability in the terminal examinations.

Practical activities offer an excellent framework for this, as they provide all the required elements within an applied setting which has been shown to be much more effective than a purely theoretical approach. Some illustration is given in the summary of core practicals.

Graphs

The following are some useful points for students to consider when drawing graphs:

- Put the dependent variable, the quantity being measured (e.g. temperature), on the *y*-axis, and the pre-determined quantity (e.g. volume of solution) on the *x*-axis.
- Choose the scales so that the results are spread out as far apart as the size of the grid allows. However this should not be at the expense of using a sensible scale, e.g. using 1 cm on the axis to represent 3 or 4 units might spread the readings better than using 1 cm to represent 5 units, but the scale would be hard to read.
- In exams, the plots should cover **at least half** of the grid supplied for the graph.
- Always label Axes should always with the quantity being measured and the units. These should be separated with a forward slash mark, e.g. time / s.
- Data points should be marked with a cross (×), but care should be taken that data points can be seen against the grid.
- The origin (0, 0) does not need to be included on either scale if it is not relevant, for example if temperature readings between 21.0°C and 39.0°C are to be plotted there is no need to begin the *y* axis at 0. Rather it could be scaled from 20.0°C to 40.0°C.
- The line of best fit should be a continuous straight line, drawn with the aid of a ruler, or smooth curve (most likely drawn freehand, unless a 'flexicurve' is available). Since the readings are all subject to experimental error the line drawn may not necessarily pass through every point. There is no definitive way of determining where a line of best fit should be drawn. A good rule of thumb is to make sure that there are as many points on one side of the line as the other. Often the line should pass through, or very close to, the majority of plotted points. Never join Points by a series of short, straight lines.

Significant figures

Students need to understand that the final quantitative result of an experiment should only be recorded an **appropriate** number of significant figures.

As a simple guide, the number of significant figures in the final calculated value should be the same as the number of significant figures in the least accurate measurement.

Example
In an experiment to find the enthalpy of neutralisation of the reaction between an acid and a base the concentration of the acid is 1.05 mol dm^{-3}, the temperature rise 8.5°C and the volume of solution 48.55 cm^{3}. From these results the enthalpy change is calculated as $\Delta H = -52.345$ kJ mol^{-1}.

Since the temperature was only measured to two significant figures the enthalpy change should be recorded as $\Delta H = -52$ kJ mol^{-1}.

Accuracy and errors in practical work

Students need to understand the limitations of the equipment that they use in their practical work and be able to express errors and uncertainties quantitatively. It is essential that they are clear about the different terms associated with accuracy and uncertainty.

- Any piece of equipment used in a quantitative practical has a **measurement uncertainty**, however carefully it is used.
- The manufacturer of the equipment supplies the measurement uncertainty.
- The **percentage uncertainty** for a reading from the equipment may be calculated.
- A burette with an uncertainty of ±0.05 cm^3 for each reading is used to measure a volume of 24.50 cm^3. For this reading,:

  $$\text{percentage uncertainty} = \frac{2 \times 0.05}{24.50} \times 100 = 0.41\%$$

  (In this case, the burette is used twice – to measure and initial and a final reading – so the uncertainty is doubled)
- Errors due to the apparatus are **systematic errors**.
- Uncertainties may be combined to find the **total uncertainty** in a final value calculated from experimental results.

  The results of a titration are used to calculate the molar mass of a compound as 126.0 g mol^{-1}.

  The percentage uncertainties in the balance, pipette and burette readings used in the calculation are 0.12%, 0.25% and 0.55%.

  Total percentage uncertainty = 0.12 + 0.24 + 0.55 = 0.91%.

  $$\text{Total uncertainty in molar mass} = 0.91 \times \frac{136}{100} = 1.15$$

  Molar mass = 126.0 ± 1.15 g mol^{-1}.

1 Errors due to careless use of the apparatus are **random errors**. These may be reduced by improving a technique and by repeating the experiment several times, e.g. repeating a titration until concordant titres are recorded.

2 The **overall error** in a value calculated from the results of an experiment may be calculated. This error will include both systematic and random errors.

An enthalpy change for a reaction is calculated from the results of an experiment and is compared with a data book value.

By experiment $\Delta H = -200$ kJ mol^{-1}; data book $\Delta H = -220$ kJ mol^{-1}

The overall error in the experimental value is:

$$\frac{(220 - 200)}{220} \times 100\% = 9.1\%$$

Opportunities for development of maths skills

This grid shows how the core practicals can be used to develop mathematical skills within Chemistry; and also makes some links to the skills that may be tested indirectly (i.e. on written question papers).

Practical activity	Mathematical skills
1: Measuring the molar volume of a gas	0.0, 1.1, 3.1, 3.2
2: To determine the enthalpy change of a reaction using Hess's Law	0.0, 0.1, 1.1, 2.2, 2.3, 2.4
3: Finding the concentration of a solution of hydrochloric acid	0.0, 0.1, 0.2, 1.1, 1.2 1.3, 2.2, 2.4
4: Preparation of a standard solution from a solid acid	0.0, 0.1, 0.2, 1.1, 1.2 1.3, 2.2, 2.4
5: Investigation of the rates of hydrolysis of halogenoalkanes	
6: Chlorination of 2-methylpropan-2-ol with concentrated hydrochloric acid	
7: The oxidation of propan-1-ol	
8: Analysis of some inorganic and organic unknowns	
9a and 9b: Following the rate of the iodine-propanone reaction by a titrimetric method and investigating a 'clock reaction' (Harcourt-Esson, iodine clock)	0.0, 0.1, 1.1, 3.1, 3.2, 3.5
10. Finding the activation energy of a reaction	0.0, 0.1, 0.2, 1.1, 2.2, 2.3, 2.4, 2.5,
11. Finding the *K*a value for a weak acid	0.0, 0.1, 0.2, 1.1, 2.2, 2.3, 2.4
12. Investigating some electrochemical cells	
13. Redox titrations	0.0, 0.1, 0.2, 1.1, 1.2 1.3, 2.2, 2.4
14. The preparation of a transition metal complex	
15. Analysis of some inorganic and organic unknowns	0.4, 1.11, 2.3, 2.4, 3.1, 3.2, 3.3
16. The preparation of aspirin	0.0, 0.2, 1.1

Teaching approaches to core practicals

The 16 core practicals should be used to allow students to develop their practical techniques and skills, and also to use their mathematical skills.

It is expected that the course will include other practicals that also allow these techniques and skills to be introduced and practised but it is essential that the core practicals are given priority and emphasis.

For example in Core Practical 4 students are to make up a standard solution of a solid acid and use it to find the concentration of a solution of sodium hydroxide. They may then go on to complete Core Practical 2 *Finding the concentration of a solution of hydrochloric acid*.

There are experiments that may be included in the course before these two core practicals are set in order to give students the opportunity to practice the appropriate techniques. Students could make up a standard solution of sodium carbonate then use this, in a titration, to find the concentration of dilute sulphuric acid.

Most of the core practicals should be able to be carried out in one laboratory session. Some, however, will need a following session in order to be completed. For example in Core Practical 14 *The preparation of a transition metal complex*, crystals will have to be left overnight to dry out so that the yield may be accurately weighed.

Choosing the Core Practicals

We have given you as teacher the choice of experiment but, in most cases, we expect teachers will carry out the practicals for which worksheets and instructions are provided. However, you are free to use other practical activities in place of the ones selected.

For example there are a number of experiments suitable for Core Practical 2: *Determination of an enthalpy change using Hess's Law*. The example given in the table is known to be reliable, giving acceptable results. However teachers may have other experiments that they prefer to use. This is perfectly acceptable and is to be encouraged.

Commentary on the Core Practicals

1. Measuring of the molar volume of a gas
This experiment provides opportunities for students to consider procedural errors and measurement uncertainties, and to identify changes that could be made to improve the accuracy of the results obtained. Procedural errors include placing the bung into the tube after the reaction has started and collecting the gas over water. Both lead to a loss of gas collected. There are three measurements taken: 1. the volume of ethanoic acid using a 50 cm3 measuring cylinder 2. the mass of calcium carbonate using a two decimal place balance 3. the volume of gas using a 100 cm3 measuring cylinder The percentage measurement uncertainty for each can be calculated and then a comparison can be made to decide which, if any, would produce the most significant effect when a change is made. For example, using a two decimal place balance, the measurement uncertainty for each weighing is ± 0.005 g, giving an overall measurement uncertainty of ± 0.01 g, since two weighings are made. With a mass of 0.05 g this produces a percentage measurement uncertainty of 20%. This can be reduced to 2% by the use of a three decimal place balance. The percentage uncertainty for each volume measurement is considerable less than 20%. Even with the smallest mass of 0.05 g, the maximum volume of gas that can be collected is 20 cm3. The measurement uncertainty of a 100 cm3 measuring cylinder is ± 0.5 cm3. This gives a percentage measurement uncertainty of 2.5%. Hopefully students will recognise that the uncertainty involved in measurement of the volume of ethanoic acid is irrelevant, since this reagent is in excess. Students could be asked beforehand to predict which instrument will produce the most precise measurement. Many are likely to choose the balance. These calculations emphasise the principle that one does not always need to use precise instruments to obtain an acceptable level of precision
2. Determination of the enthalpy change of a reaction using Hess's Law
Students can calculate the measurement uncertainties involved with the measurement of mass, volume and temperature in the experiment. **Questions**: • Why is it not necessary to plot cooling curves is this experiment? [Compare with the determination of the enthalpy change of reaction between zinc and aqueous copper(II) ions, where it is necessary to plot a cooling curve.] • What is the link between Hess's law and the First Law of Thermodynamics? As a follow up to this practical, students could be asked to choose a reaction in which enthalpy change cannot be measured directly and explain why this is so. They could then be asked to state how the enthalpy change could be determined by making use of Hess's Law.

3. Finding the concentration of a solution of hydrochloric acid
This is a straightforward titration using the standardised sodium hydroxide from Core Practical 4. Students could be asked to calculate the overall measurement uncertainty in their calculated concentration of hydrochloric acid.
4. Preparation of a standard solution from a solid acid and use it to find the concentration of a solution of sodium hydroxide
Before this experiment, students could research why primary standard solutions cannot be made directly using some solids. In the case of sodium hydroxide it is because it is difficult to obtain it pure. It is hygroscopic and reacts with carbon dioxide when exposed to the air, so a certain amount of both water and sodium carbonate is always present. If other cases, such as sodium carbonate-10-water, the salt is efflorescent. Students could then go on to research suitable solids for making primary standard solutions that can be used to standardise aqueous acids. The next task would be to research suitable solids to use as primary standards for standardising aqueous sodium hydroxide. If sulfamic acid is not available, then it can be substituted by another suitable solid acid, such as butanedioic acid (succinic acid), ethanedioic acid (oxalic acid) or even benzoic acid, but owing to its insolubility in water, solutions of benzoic acid need to be made up in 95% ethanol. Students could calculate the overall uncertainty in their calculated concentration of the sodium hydroxide. If all students have standardised the same solution of sodium hydroxide, from a bulk sample, then an average value for the class could be calculated and a discussion of the reliability of this value, compared to that obtained by an individual student, could then take place.
5. Investigation of the rates of hydrolysis of some halogenoalkanes
This practical could be used to confirm theory already covered in lesson time, or as an investigation into the hydrolysis of halogenoalkanes. The students will need to be introduced to the classification of halogenoalkanes as 1°, 2° and 3° and also to the fact that halogenoalkanes can undergo nucleophilic substitution reactions owing to the polarity of the carbon-halogen bond. They could be asked to make predictions based on both relative polarity and relative strength of the carbon-halogen bonds, and then compare their predictions with the results obtained.
6. Chlorination of 2-methypropan-2-ol with concentrated hydrochloric acid
In preparation for this practical, students could be asked, in groups, to research the various methods of halogenating alcohols. Different groups could then give a short presentation of chlorination, bromination and iodination. Questions: ● A mixture of a chloride and conc. H2SO4 can be used to chlorinate an alcohol. Similarly, a mixture of a bromide and conc. H2SO4 can be used for bromination. However, a mixture of an iodide and conc. H2SO4 cannot be used to iodinate an alcohol. Why? ● Why is zinc chloride required when chlorinating a primary or secondary alcohol with HCl? ● When halogenating an alcohol with a phosphorus halide, pyridine (a weak base) is often added to the mixture. Suggest a reason for this.

7. The oxidation of propan-1-ol to produce propanal and propanoic acid

In preparation for this practical, students could be asked to research how to oxidise propan-1-ol separately into propanal and propanoic acid, including how to identify the final product.

Volunteers could then give a short presentation on each preparation, explaining the need for the varying conditions. The presentation could include, with reasons, any safety measures that need to be taken.

There are many questions that can be asked about the practical procedure for the preparation of propanoic acid. For example, the need to use anti-bumping granules and why the water enters the bottom of the condenser and exists the top. Why does the mixture need to be refluxed?

The students could then extend their research into the oxidation of secondary and tertiary alcohols.

8. Analysis of some inorganic and organic unknowns

In preparation for this practical, students could produce tabulated summaries of the tests for cations and anions (inorganic compounds), and for the functional groups they have so far studied (organic compounds).

They could then use these summaries as an aid to identify the compounds in the practical.

Following the practical, the students could be given a series of results, both positive and negative, of tests carried out on unknowns and asked to make deductions about each compound. There are a number of such questions available in past WCH03 papers from the 2013 specification.

Information from mass spectra and infrared spectra can also be supplied to supplement to data collected from the wet tests.

The following two resources may be useful to students:

- A reaction to form silver chloride
- Royal Society of Chemistry: Chemical Misconceptions

http://www.rsc.org/learn-chemistry/resource/res00001096/precipitation

This resource is intended for students aged 14–16 but parts would be useful at AS Level. The first two pages of the student worksheet use simple models to explain the processes going on when a precipitate forms.

The final page, the production of lead iodide, is a good way of testing whether students are able to apply these ideas.

Testing for Negative Ions
Royal Society of Chemistry: Learn Chemistry
http://www.rsc.org/learn-chemistry/resource/res00000758/testing-for-negative-ions
(This includes a test for nitrate ions which is not on the specification!)

Simple test-tube reactions allow students to discover the expected observations when testing for anions. To fulfil the requirements of the specification, students will have to be taught to test for carbonates first and only proceed to test for halide or sulphate ions when carbonate has been eliminated.

9a. Following the rate of the iodine-propanone reaction by a titrimetric method

The reaction between propanone and iodine in aqueous solution may be acid catalysed:
I2(aq) + CH3COCH3(aq) + H+(aq) * CH3COCH2I(aq) + 2H+(aq) + I-(aq)

The influence of the iodine on the reaction rate may be studied if the concentrations of propanone and acid protons effectively remain constant during the reaction. This is achieved by using a large excess of both acid and propanone in the original mixture.

A single reaction mixture is made up and samples are withdrawn at regular time intervals, quenched and titrated against standard sodium thiosulfate. The titres are then plotted against the time of quenching of the sample. The results will show that the reaction is zero order with respect to iodine.

Students can be told that the reaction is first order with respect to both propanone and hydrogen ions. They can then research the mechanism (which involves keto-enol tautomerism – not on the specification). This mechanism is one of the few examples that students at this level will meet where the first reaction is not the rate determining step.

9b. Investigating a 'clock reaction' (Harcourt-Esson, iodine clock)

The two most common 'iodine clock' reactions are:

1. the reaction between acidified hydrogen peroxide and sodium thiosulfate (the Harcourt-Esson reaction), and
2. the reaction between peroxydisulfate ions and iodide ions.

Either reaction can be investigated using the 'initial rate' method. In the case of the later, two series of experiments can be performed to determine separately the order with respect to peroxydisulfate ion and iodide ion.

The first set of reactions is carried out with different volumes of peroxydisulfate solution, keeping the volume of iodide solution and the total volume of solution constant.

In the second set of reactions, the volume of iodide solution is the variable.
Fixed volumes of starch solution and sodium thiosulfate solution are added to each reaction mixture and the time taken, t, for the blue-black colour to form is recorded. Suitable graphs can then be plotted using 1/t as a measure of initial rate of reaction.

If left to their own devices, students are likely to plot 1/t against volume of peroxydisulfate / iodide solution. They will be rewarded on this occasion since the reaction is first order with respect to each reactant.

The more mathematically able students may be able to recognise that a plot of log(1/t) against log(volume of reactant) will give a straight line whose gradient is equal to the order of reaction with respect to that reactant.

Questions:
- Is 1/t a reasonable approximation to the initial rate of reaction? Explain your answer.
- Why does the iodine formed in the reaction not immediately react with the starch to form the blue-black complex?
- Why is it important to keep the amount of starch and sodium thiosulfate constant in each reaction?
- Why is it important to keep the total volume of reaction mixture constant in each reaction?

10. Finding the activation energy of a reaction

In this activity students will calculate the activation energy of a reaction. There are many places where they may slip up. The main ones are:

1. The graph to be drawn needs to be ln t against 1/T, where T is in K. Check that they are using the 'ln' button on their calculator correctly.
2. They need to convert T from °C into K, and then work out 1/T.
3. A graph has to be plotted. Some may find the axes tricky if the numbers are very small or very large.
5. A line of best fit is then required.
6. The gradient of the line of best fit needs to be determined. The gradient is NOT the activation energy, it is = Ea/R, so the value of the gradient needs to be multiplied by R (the gas constant).
7. If they are asked to determine A, students will need to extrapolate the graph back to the y-axis, and read off the y-intercept. However, this value is NOT the value of A, it is the value of ln A.

11. Finding the Ka value for a weak acid

In this experiment an aqueous solution of a weak acid is 'half-neutralised' by an aqueous solution of sodium hydroxide.

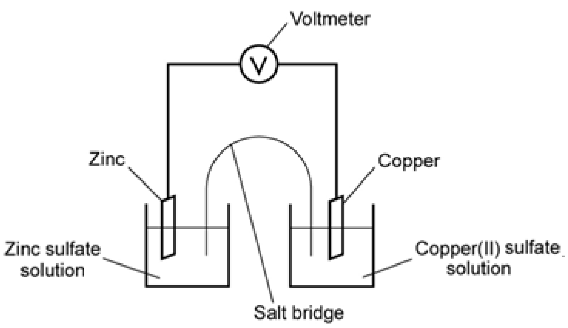
The pH of the resulting solution is then measured. The pH of this solution is equal numerically to the pKa of the weak acid.

The more mathematically able students could be encouraged to derive the Henderson-Hasselbalch equation linking pH and pKa and hence prove the above statement.

Questions:
- Why is it not necessary to known the concentration of either the acid or the sodium hydroxide used in the experiment?
- What assumption has been made regarding the hydrogen ion concentration, and hence the pH, of the final solution? Is this assumption justified?
- Why is not strictly correct to refer to end–point in a titration of a weak acid and a strong base, such as sodium hydroxide, as the 'neutralisation-point'.

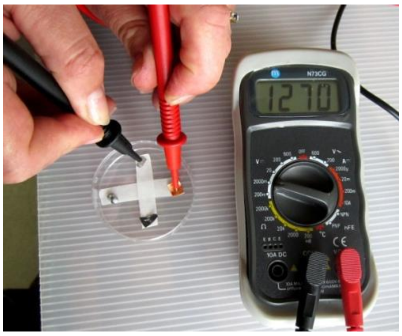
12. Investigating some electrochemical cells

This practical can be performed in the usual manner with small beakers and strips of filter paper soaked in aqueous potassium nitrate. A typical set up is as shown in the diagram:



Digital voltmeters are ideal since they have a high resistance.

A more convenient way of performing these experiments is to use the microscale apparatus shown below:



Further investigations could be based around the change in concentration of the cation. The classic experiment is a 1.0 mol dm^{-3} solution of copper(II) ions and varying concentrations of silver ions (typically 0.01, 0.0033, 0.001, 0.00033, and 0.0001 mol dm^{-3}).

The more able mathematicians can then compare the results with those obtained from the Nernst equation. A plot of E_{cell} (vertical axis) against $\ln[Ag^{+}(aq)]$ should give a straight line of gradient 0.026.

13a and 13b. Carry out redox titrations with both

i. iron(II) ions and potassium manganate(VII)
Potassium manganate(VII) is a powerful oxidising agent and is used for the quantitative estimation of many reducing agents, especially compounds of iron(II) and for ethanedioic acid and its salts.

The titrations are carried out in acidic solution. With aqueous potassium manganate(VII) in the burette, as the titration proceeds manganese(II) ions accumulate but, at the dilution used, give a colourless solution. As soon as the potassium manganate(VII) is in excess, the solution becomes pink and it therefore acts as its own indicator.

Solid potassium manganate(VII) is not suitable as a primary standard, so an aqueous solution is prepared and this is then standardised. Hydrated iron(II) ammonium sulfate, (NH4)2SO4.FeSO4.6H2O, is used to make the standard solution for this procedure. Solid iron(II) ammonium sulfate is dissolved in dilute sulfuric acid that has been previously boiled and allowed to cool.

Once standardised, the potassium manganate(VII) solution can be used in quantitative estimations. A typical experiment would be to find 'x' in the formula FeSO4.xH2O. Another is to find the percentage of ethanedioic acid and sodium ethanedioate in a mixture of the two. The later requires a standard solution of sodium hydroxide to quantitatively estimate the acid.

Questions:
- Why is solid potassium manganate(VII) not suitable as a primary standard?
- Why is the sulfuric acid boiled, and then allowed to cool, before the iron(II) ammonium sulfate is dissolved in it?
- Why is hydrated iron(II) sulfate not suitable as a primary standard?
- Why must the titration be carried out in acidic, and not alkaline, conditions?
- Why, in the titration with ethanedioic acid, does the aliquot need to be heated before the titration is performed?

ii. sodium thiosulfate and iodine
If time permits, it is desirable to carry out at least one iodine-thiosulfate titration, using starch as the indicator. For example, a standard solution of sodium thiosulfate could be used to estimate the percentage of copper(II) ion in a sample of hydrated copper(II) sulfate crystals.

14. The preparation of a transition metal complex
Two reliable preparations are: 1. [Cu(NH3)4SO4.H2O from aqueous ammonia and aqueous copper(II) sulfate. 2. Fe(COO)2.2H2O from acidified iron(II) ammonium sulfate and ethanedioic acid. In either case, the maximum possible mass of the complex can be calculated and then used to calculate the percentage yield.

15. Analysis of some inorganic and organic unknowns
The same suggestions apply here as to Core Practical 8. This time it would be sensible to use, initially at least, just transition metal compounds for the inorganic tests, and possibly limit the organic tests to carbonyls and carboxylic acids. Mass spectra, infrared spectra and NMR spectra should also be included.

16. The preparation of aspirin

Aspirin is prepared by the acylation of 2-hydroxybenzoic acid using ethanoic anhydride as the acylating agent.

The reaction can be represented as follows.

$HOOCC_6H_4OH$	+	$(CH_3CO)_2O$	→	$HOOCC_6H_4OCOCH_3$	+	CH_3COOH
2-hydroxybenzoic acid		ethanoic anhydride		aspirin		ethanoic acid

The preparation involves purification of the crude product by recrystallisation from ethanol, in which the temperature must be carefully controlled since the boiling point of ethanol is quite low at 78 °C.

To analyse the effectiveness of this method

- Calculate the theoretical yield of aspirin that should be formed from the mass of 2-hydroxybenzoic acid used.
- Calculate the percentage yield of aspirin from your experiment and comment on the reasons for the losses that have occurred during the preparation and the purification of the solid.
- Calculate the atom economy for the preparation of aspirin by this method.
- Consider the reasons why the alternative method of preparation, which uses ethanoyl chloride rather than ethanoic anhydride, is not favoured by industry even though this alternative method has a higher atom economy.
- This preparation can also be done on a microscale. The following is a link to the Royal Society of Chemistry website:

http://www.rsc.org/learn-chemistry/resource/res00000556/the-microscale-synthesis-of-aspirin?cmpid=CMP00000628

Research and referencing

Students need to understand how scientific advances are communicated and reviewed.

The process of peer-review, citations and attention to detail needed to present scientific papers should be understood, as should the role of scientific journals, conferences and the international nature of research.

Evidence revealed by research might 'support the idea that' but is not described as 'proving'. Objective scientific language is cautious and often conditional and this needs to be reflected in students' own practical recording.

Independent objective research needs to be developed to incorporate this approach. The internet is a wonderful resource but is often approached in a scientifically naive manner even by the most technologically aware students. Valid scientific information can be extracted easily but it is rarely found in the first few pages of a Google search or on an anonymous question and answer site. In contrast Wikipedia is often carefully referenced or flagged where further corroboration of details are required so simplistic views that it is weak because anyone can edit it need to be investigated further.

Independent thinking and evaluation

It is highly desirable that students are challenged to think more critically about a practical procedure from the start of the A level course. It is vital that they realise that what has served them well at International GCSE/GCSE needs to be developed to achieve the same success later.

As an example of this, consider the core practicals dealing with initial rates in an iodine clock reaction. In these investigations we use 1/t as a measure of the initial rate of reaction. However, 1/t is not an exact measure of the initial rate since as time increases the rate of reaction decreases, (the reaction is first order with respect to each reactant species). Hence, 1/t is a measure of the **average** of the rate of reaction over the time period. The longer the time period the greater the deviation of this average from the true initial rate. A more reliable value of initial rate can be obtained from a plot of a concentration-time graph. A tangent to the curve is drawn at t = 0 and its gradient is then determined.

An objective discussion and reflection on the reliability and validity of their findings provides crucial evidence of a student's independent thinking and practical competence. This will vary according to the core practical as some activities, such as identification of inorganic and organic unknowns, are limited in this respect. Others, such as the 'Investigating some electrochemical cells' offer greater potential.

Progression in evaluating practical investigations is characterised by;

- a move from descriptive comment and a subjective approach to evidence-based analysis
- cautious conditional language
- an accurate appreciation of exactly what the data shows (and what it does not)
- a clear understanding of the chemical principles underlying the methodology applied

When looking for evidence in evaluating, the obvious place to start is the data. There is little point in speculating about difficulties or mistakes if there is no evidence. Students need to appreciate that in a perfect investigation where all variables are accurately controlled then any repeats should be identical. When this is not the case then the size of these differences can provide useful information. This naturally leads to such measures as means, medians, standard deviation and standard error. Other differences may be more marked and be regarded as anomalies or outliers. All of this will provide good evidence for any judgements on the reliability of the findings. Only then is it possible to begin thinking about what might be causing these differences.

In chemistry uncertainties of measurements of instruments of volume, mass and temperature are often the most important consideration in investigations where most variables are tightly controlled.

In summary the practical assessment is not an isolated section of the specification and its assessment. A small part is simple direct learning of basic techniques and procedures as well as physical skill in handling apparatus. A far greater part will be testing students' ability to apply their skills to a wide variety of questions. Whilst papers 3 and 6 have a specific focus on indirect assessment of practical skills many, such as mathematical skills or interpreting data will also be highly relevant to papers 1, 2, 4 and 5. Using a wider perspective, the development of such skills is a key element in preparing students for higher education.

Chemical analysis

Inorganic analysis

- Topics 8 and 17 include sections on reactions that may be used to test for the presence of cations and anions
- Most of these reactions may be carried out on a test tube scale
- Students may be set these tests as part of an examination question on the analysis of inorganic unknowns
- A useful source of these exercises are the WCH03 and WCH06 exam papers in the 2013 Pearson Edexcel International Advanced Level Chemistry Specification.
- The tables that follow provide a reference for the observations made in the tests.

Aqueous, dilute solutions of transition metal compounds

Colour	Transition metal ions
blue	copper(II), $[Cu(H_2O)_6]^{2+}$
green	iron(II), $[Fe(H_2O)_6]^{2+}$; chromium(III), $[Cr(H_2O)_6]^{3+}$; nickel(II), $[Ni(H_2O)_6]^{2+}$
brown / yellow	iron(III), $[Fe(H_2O)_6]^{3+}$
red / pink	cobalt(II), $[Co(H_2O)_6]^{2+}$
yellow	chromate(VI), $CrO_4{}^{2-}$
orange	dichromate(VI), $Cr_2O_7{}^{2-}$
purple	manganate(VII), $MnO_4{}^{-}$
pale pink	manganese(II), $[Mn(H_2O)_6]^{2+}$

Flame tests

Flame tests may be carried out by using a clean nichrome wire to mix a sample of a solid compound with a few drops of concentrated hydrochloric acid. The wire is then held at the edge of a hot Bunsen flame and the colour observed.

Flame colour	Metal cation
yellow	sodium
lilac	potassium
yellow-red	calcium
pale green	barium
red*	lithium, strontium
red-violet	rubidium

*Further tests are needed to identify these metal cations.

Addition of sodium hydroxide solution

When dilute, aqueous sodium hydroxide is added to an aqueous solution of a metal ion a precipitate of the insoluble hydroxide may be formed e.g. $Cr(OH)_3$.

Some precipitates will dissolve in excess sodium hydroxide to give a solution containing a complex ion e.g. $[Cr(OH)_6]^{3-}$.

When carrying out these tests students should be told to add aqueous sodium hydroxide, drop-by-drop, until there is no further change.

Metal ion solution	Observation on adding aqueous NaOH	Observation on adding excess aqueous NaOH
chromium(III), $[Cr(H_2O)_6]^{3+}$	green precipitate	precipitate dissolves to give a dark green solution
manganese(II), $[Mn(H_2O)_6]^{2+}$	pale brown precipitate, turning darker brown on exposure to air	precipitate is insoluble
iron(II), $[Fe(H_2O)_6]^{2+}$	green precipitate, turning brown on exposure to air	precipitate is insoluble
iron(III), $[Fe(H_2O)_6]^{3+}$	red-brown precipitate	precipitate is insoluble
cobalt(II), $[Co(H_2O)_6]^{2+}$	blue precipitate, turning pink on standing	precipitate is insoluble
nickel(II), $[Ni(H_2O)_6]^{2+}$	green precipitate	precipitate is insoluble
copper(II), $[Cu(H_2O)_6]^{2+}$	blue precipitate	precipitate is insoluble
zinc(II), $[Zn(H_2O)_6]^{2+}$	white precipitate	precipitate dissolves to give a colourless solution
Group 2 cations: $Mg^{2+}(aq)$, $Ca^{2+}(aq)$, $Ba^{2+}(aq)$	white precipitate	precipitate is insoluble
Group 1 cations: $Na^+(aq)$, $K^+(aq)$	no precipitate	—

Addition of ammonia solution

When dilute, aqueous ammonia is added to an aqueous solution of a transition metal ion a precipitate of the insoluble hydroxide is formed. e.g. $Cu(OH)_2$.

Some precipitates will dissolve in excess ammonia to give a solution containing a complex ion e.g. $[Cu(NH_3)_4(H_2O)_2]^{2+}$

When carrying out these tests students should be told to add aqueous ammonia, drop-by-drop, until there is no further change.

Metal ion solution	Observation on adding aqueous NH_3	Observation on adding excess aqueous NH_3
chromium(III), $[Cr(H_2O)_6]^{3+}$	green precipitate	precipitate dissolves to a give a green solution
manganese(II), $[Mn(H_2O)_6]^{2+}$	off-white precipitate, turning brown on exposure to air	precipitate is insoluble
iron(II), $[Fe(H_2O)_6]^{2+}$	green precipitate turning brown on exposure to air	precipitate is insoluble
iron(III), $[Fe(H_2O)_6]^{3+}$	red-brown precipitate	precipitate is insoluble
cobalt(II), $[Co(H_2O)_6]^{2+}$	blue precipitate	precipitate dissolves to a brown solution
nickel(II), $[Ni(H_2O)_6]^{2+}$	green precipitate	precipitate dissolves to give a blue solution
copper(II), $[Cu(H_2O)_6]^{2+}$	blue precipitate	precipitate dissolves to a deep blue solution
zinc(II), $[Zn(H_2O)_6]^{2+}$	white precipitate	precipitate dissolves to give a colourless solution

Silver nitrate solution

Aqueous silver nitrate is used to test for the presence of halide ions in solution.

Anions such as carbonate that would form precipitates with silver nitrate are removed by adding dilute nitric acid before the silver nitrate.

The identity of the halide may be confirmed by adding aqueous ammonia to the silver halide.

Anion	Precipitate		Addition of aqueous ammonia	
	Colour	Formula	Dilute	Concentrated
chloride, Cl^-	White	$AgCl$	soluble	—
bromide, Br^-	Cream	$AgBr$	insoluble	soluble
iodide, I^-	pale yellow	AgI	insoluble	insoluble

Barium chloride solution

Aqueous barium chloride forms precipitates with a number of anions but is usually used as the test for the sulfate(VI), SO_4^{2-} anion.

When dilute hydrochloric acid is added to the anion solution before the addition of aqueous barium chloride then only the sulfate(VI) anion will form a precipitate.

Anion	Precipitate		Addition of dilute hydrochloric acid
	Colour	Formula	
sulfate(VI), SO_4^{2-}	white	$BaSO_4$	precipitate is insoluble
sulfate(IV), SO_3^{2-}	white	$BaSO_3$	precipitate dissolves
carbonate, CO_3^{2-}	white	$BaCO_3$	precipitate dissolves

Concentrated sulfuric acid

When a few drops of concentrated sulfuric acid are added to a solid halide the observed products may be used to identify the halide ion.

The test must be carried out on a small scale and in a fume cupboard.

The gaseous products in brackets will not be observed since they are colourless.

No attempt should ever be made to smell the products of these reactions.

Solid halide	Observations with concentrated sulfuric acid	Observed reaction products
chloride, Cl^-	steamy fumes	HCl
bromide, Br^-	steamy fumes, brown vapour	HBr, Br_2, (SO_2)
iodide, I^-	steamy fumes, purple vapour, black solid, yellow solid	HI, I_2, S, (H_2S)

Displacement of halide ions

When aqueous chlorine is added to a solution of a bromide or an iodide then bromine or iodine is displaced.

When aqueous bromine is added to a solution of an iodide then iodine is displaced.

The formation of aqueous solutions of bromine or iodine may be used as a test for the bromide and iodide ions.

If an organic solvent such as hexane is added to the reaction mixture the bromine or iodine dissolves in the organic layer.

Halide solution	**Observations on addition of aqueous halogen**		
	Chlorine $Cl_2(aq)$	**Bromine $Br_2(aq)$**	**Iodine $I_2(aq)$**
$Cl^-(aq)$	No reaction Pale yellow-green or colourless solution	No reaction Pale red-brown solution	No reaction Brown solution
$Br^-(aq)$	Red-brown solution Yellow-orange organic layer	No reaction. Pale red-brown solution	No reaction Brown solution
$I^-(aq)$	Brown solution Black solid Purple organic layer	Brown solution. Black solid Purple organic layer	No reaction Brown solution

Heat

Gases or vapours may be given off on heating a solid inorganic compound.

Gas or vapour given off on heating	**Possible compound**
carbon dioxide	Group 2 carbonates OR lithium carbonate
oxygen	Group 1 nitrates (except lithium nitrate)
oxygen and nitrogen dioxide	Group 2 nitrates OR lithium nitrate

Organic analysis

- Topics 10, 15, 18, 19 and 20 include sections on reactions that may be used to test for the presence of organic functional groups
- Most of these reactions may be carried out on a test tube scale
- Students may be set these tests as part of an exercise to analyse organic unknowns
- The exercise may include spectroscopic information from Topics 10 and 15
- A useful source of these exercises are the WCH03 and WCH06 exam papers in the 2013 Pearson Edexcel International Advanced Level Chemistry Specification.
- The tables that follow provide a reference for the observations made in the tests.

Ignition

Igniting an organic compound on a crucible lid in a fume cupboard may provide evidence for the identity of the compound.

Observations	Possible identity
burns with smoky flame	arene, unsaturated aliphatic e.g. cyclohexene
burns with a clean flame	saturated low molar mass compound e.g. ethanol
no residue	most lower molar mass compounds

Chemical tests

Test	Observations	Inference
shake with bromine water	yellow solution is decolourised	alkene
	if white precipitate is also formed	phenol
warm with aqueous, acidified potassium dichromate(VI)	orange to green solution	primary or secondary alcohol, aldehyde
warm with ethanol and aqueous silver nitrate	white precipitate cream precipitate yellow precipitate	chloroalkane bromoalkane iodoalkane
phosphorus(V) chloride	steamy fumes that turn damp blue litmus paper red	OH group in alcohols and carboxylic acids
2, 4-dinitrophenylhydrazine solution	yellow or orange precipitate	C=O group in aldehydes and ketones
heat with Fehling's solution or Benedict's solution	red precipitate	aldehyde
warm with Tollens' reagent (ammoniacal silver nitrate)	silver mirror	aldehyde
iodine in alkaline solution	pale yellow precipitate	methyl ketone or ethanal methyl secondary alcohol or ethanol
warm with ethanol and a few drops of concentrated sulfuric acid and pour reaction mixture into aqueous sodium carbonate	ester smell e.g. glue-like	carboxylic acid

Answers to Student Guide Questions

Page 9

1. (a) barium / Ba^{2+} (1)

(b) chloride / Cl^- (1)

(c) (i) white precipitate (1)

(ii) $Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$

All formulae correct (1). Balancing and state symbols (1).

(d) hydrogen chloride / HCl (1)

(Taken from Edexcel WCH03 January 2014)

2. (a) smoky /sooty flame (1)

(b) (i) contains phenol group (1)

(ii) aldehyde or ketone (both for mark) / carbonyl group (1)

(iii) ketone (1)

(Taken from Edexcel 6CH08 Summer 2013)

Page 10

3. Titres / cm^3 24.00 22.30 22.80 22.40

Titres 2 and 4, which have a mean of 22.35 cm^3

Page 11

4. $+63\ kJ\ mol^{-1}$ $0.1035\ mol\ dm^{-3}$ $5.8 \times 10^{-4}\ mol\ dm^{-3}$ $86\ g\ mol^{-1}$

Page 13

5. pipette = 0.24%, burette = 0.43%, thermometer = 3.2%

6. ± 0.14

Page 14

7. Titres / cm^3 27.00 26.50 26.60 26.65

Titres 2, 3 and 4, which have a mean of 26.60 cm^3

8. (a) $10^{-2.88} = 1.32 \times 10^{-3}\ mol\ dm^{-3}$

(b) $1.45 \times 10^{-5}\ mol\ dm^{-3}$

(c) 4.8

Page 15

9. $\Delta H_3 = 2\Delta H_1 - \Delta H_2 = 2(+28.5) - (-27.6) = 84.6\ kJ\ mol^{-1}$

10. ln 1/time −4.41 −3.95 −3.58 −3.30 −2.89

11. $k = 2.90 \times 10^{-5} / (0.150 \times 0.100^2) = 1.93 \times 10^{-2}\ mol^{-2}\ dm^6\ s^{-1}$

Page 16

12. The final answer will depend on the graph, but 29.0°C and 23.0 cm^3 are expected.

Page 16

13. 8.3×10^{-4} mol dm^{-3} s^{-1}

Page 18

14. (a) (i) To generate HBr (1)

(ii) Two from lower yield / Br_2 formed /less HBr formed / alkene formed (2)

(b) Dilution of sulfuric acid is exothermic (1)

(c) Round-bottomed/pear-shaped flask and heat/mantle/water bath (1)

Still-head with stoppered thermometer opposite opening to condenser (1)

Condenser with water flowing up (1)

Collection of product from a delivery tube into a flask/beaker or straight from the condenser into flask/beaker. (1)

(d) Removes/neutralizes acid/HBr (1)
Allow neutralizes sulfuric acid.

(e) Drying agent / to dry 1-bromobutane / absorbs any water present / to remove water (1)

(f) (Re-)distillation (over a narrow range either side of its boiling temperature/at its boiling temperature).

Allow fractional distillation.

(Taken from Edexcel 6CH07 January 2013)

Answers to Core Practical questions

1. (a) $CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$

(b) amount of $CaCO_3$ = 0.40 / 100.1 = 0.004 mol
(to 1 sig. fig. which is acceptable for this type of calculation)

amount of HCl = (50 / 1000) x 1.0 = 0.05 mol

0.004 mol of $CaCO_3$ react with 0.008 mol of HCl, so the HCl is in excess.

(c) Measuring cylinder. As this volume provides an excess of HCl, there is no need for the measurement to be accurate.

(d) 0.004 mol of calcium carbonate would produce 0.004 mol of carbon dioxide. This would occupy 0.004 x 24 000 cm^3 = 96 cm^3. As most gas syringes take a maximum of 100 cm^3, this seems like a sensible mass to use.

(e) A smaller volume of gas will be collected, so Greg will equate 0.40 g of gas with a smaller volume, and his value for the molar mass will be larger than expected.

2. (a)

$$CaCO_3(s) \xrightarrow{\Delta_r H} CaO(s) + CO_2(s)$$

ΔH_1 $+2HCl_{aq}$ $+2HCl_{aq}$ ΔH_2

$$CaCl_2(aq) + CO_2(g) + H_2O(l)$$

(b) Polystyrene is a good insulator, so prevents heat energy transfer with the surroundings (in this case, transfer of heat energy to the surroundings).

(c) (i) Q = m . c. ΔT = 25 g x 4.2 J g^{-1} °C^{-1} x 5.6 °C = 588 J

(ii) Molar mass of $CaCO_3$ = 100 g mol^{-1}, so amount of $CaCO_3$ used = (1.00 / 100.1)
= 0.0100 mol

Enthalpy change for reaction = − 588 / 0.0100 = 58 800 J mol^{-1} = − 58.8 kJ mol^{-1}

(iii) using 2 dp balance to weigh 1.00g: ± 0.01 / 1.00 x 100 = ± 1%
(0.01 as balance will have been used twice, each time ± 0.005 g)

volume of acid: pipette used, so ± 0.24 % (standard for pipette)

temperature: each reading ± 0.05 °C – and initial and final reading taken so,

± (0.10 / 5.6) x 100 = ± 1.79 %

total uncertainty = ± (1 % + 0.24 % + 1.79 %) = ± 3.03 %

3. (a) The pipette is rinsed with the solution that it will be used to measure i.e. the diluted hydrochloric acid. It should not be rinsed with distilled water, as some water will remain in the pipette and dilute the first sample that the pipette is used to measure.

(b) (i) Universal indicator does not have a sharp colour change at the end-point: it shows a gradual change of colour over a range of pH values.

(ii) This titration involves a strong acid and a strong base, so most indicators are suitable e.g. phenolphthalein (colourless to pink), litmus (red to blue)* or methyl orange (red to orange).

* Litmus is rarely used since it is difficult to distinguish purple from blue at the end-point.

(c) The titre values are: 24.60 cm^3, 24.25 cm^3 and 24.35 cm^3.

The first value is not concordant with the other two, so is omitted from the calculation of the mean titre. So, mean titre = (24.25 + 24.35) / 2 = 24.30 cm^3.

Note that the mean titre value is given to 2 decimal places.

(d) Amount of diluted HCl = amount of NaOH = 24.30 / 1000 x 0.350
= 8.505 x 10^{-3} mol in 25.0 cm^3 of diluted HCl

So, the 250 cm^3 volumetric flask contains $8.505 \times 10^{-3} \times 10 = 8.505 \times 10^{-2}$ moles of HCl.

This was present in 10.0 cm^3 of the original HCl sample, so the concentration of the original HCl = $8.505 \times 10^{-2} \times 1000 / 10 = 8.51$ mol dm^{-3} (to 3 sig. figs)

4. (a) Distilled water is added to the volumetric flask. The neck of the flask is marked with a ring – distilled water should be added until the **bottom** of the meniscus of the solution in the flask is resting on this ring. Kathryn needs to check this by having her eye level in line with the ring on the flask, to avoid parallax errors.

(b) She should put a stopper in the flask and invert the flask several times to ensure that the solution is mixed.

(c) Molar mass of $NaHSO_4$ = 120 g mol^{-1}, so amount of $NaHSO_4$ = 0.900 / 120 = 0.00749… mol

volume of final solution = 250 cm^3, so concentration = 0.00749… x (1000 / 250) = 0.030 mol dm^{-3}

(A final answer to 2 or 3 sig. figs is appropriate for the data)

(d) The solution lost is from the original, undiluted solution, not the final solution. So, the error is approximately 0.5 cm^3 in 25 cm^3. Therefore, the % error is (0.5 / 25) x 100 = 2%

5. (a) The three halogenoalkanes will have different densities, so the same volume of each will have a different mass, and therefore a different number of moles of each. This reduces the validity of the experiment, as the number of moles of halogenoalkane should be the same in each experiment.

(b) 1-chlorobutane = white ppt; 1-bromobutane = cream ppt; 1-iodobutane = yellow ppt

(c) There are several problems with recording of the data. Firstly, a mixture of units is used (minutes and seconds); secondly, the recording is not to the same degree of accuracy each time; thirdly, human reaction time means that the times cannot be recorded to an accuracy of more than ±0.5 s.

(d) This would not make a valid experiment, as two factors are being changed at the same time: the nature of the halogen atom in the halogenoalkane, and the structure of the haloalkane as primary, secondary or tertiary.

6. (a) Concentrated HCl, so wear safety glasses, or work in a fume cupboard or wear disposable nitrile gloves.

(b) The diagram should show two layers: the lower layer would be 2-chlorobutane, and the upper layer (the aqueous layer) would be a mixture of unreacted butan-2-ol and HCl.

(c) The liquid may start to come out of the separating funnel, but will soon stop running out, as the pressure in the sealed flask reduces below atmospheric pressure.

(d) Hazel should distil the organic layer, collecting the liquid that distils over at the correct boiling point for 2-chlorobutane. She would then shake the distillate with a drying agent, such as anhydrous calcium chloride, to remove any water from the distillate. The pure 2-chlorobutane can then be decanted off.

(e) 10 cm^3 of butan-2-ol (density 0.808 gcm^{-3}), so mass butan-2-ol = 10 x 0.808 = 8.08 g

Amount of butanol = 8.08 g / 74 g mol^{-1} = 0.109… mol

Maximum yield of 2-chlorobutane = 0.109 mol x 92.5 g mol^{-1} = 10.1 g

So, percentage yield = (2.64 / 10.1) x 100 = 26.1 % (to 3 sig figs)

(A final answer to 2 or 3 sig. figs is appropriate for the data)

7. (a) The reaction is very exothermic, and a violent reaction can occur.

(b) Propanal can be further oxidised to propanoic acid, so it is important to remove propanal from the reaction mixture as soon as it forms.

(c) The vessel used to collect the propanal should be kept cool.

(d) (i) A variety of tests would work here: 2,4-DNPH could be used to show that it contained a carbonyl group; Fehling's or Tollens' could be used to show that it contained an aldehyde group.

(ii) A melting point test is not appropriate on a liquid with a low boiling point! But the boiling point could be measured – an impure substance would have a higher than expected boiling point. Or, chromatography could be used (in this case, GLC).

8. (a) Flame test: a clean nichrome wire is dipped into concentrated hydrochloric acid and then used to introduce a small quantity of each solid, separately, into the side of a non-luminous, non-roaring Bunsen flame. A yellow flame shows the presence of sodium ions; and a lilac flame shows the presence of potassium ions.

Halide ion test: this is used on the two salts that gave a yellow flame test. A small amount of each solid is dissolved in deionised water. The solution is acidified with dilute nitric acid and a few drops of silver nitrate solution are added. A white precipitate shows the presence of chloride ions; a yellow precipitate shows the presence of iodide ions.

$$Ag^+(aq) + Hal^-(aq) \rightarrow AgHal(s)$$

Carbonate / sulfate test: this is used on the two salts that gave a lilac flame test. A small amount of each solid is dissolved in distilled water. The solution is acidified with dilute hydrochloric acid. One of the solutions will produce effervescence, indicating the presence of carbonate ions:

$$2H^+(aq) + CO_3^{2-}(aq) \rightarrow CO_2(g) + H_2O(l)$$

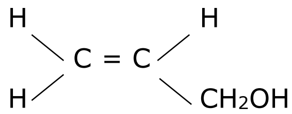
To the other solution, add a few drops of barium chloride solution and a white precipitate forms, showing the presence of sulfate ions:

$$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$$

(b) This removes any ions that might interfere with the test by also making a precipitate with silver ions (such as hydroxide ions, carbonate ions)

(c) Bromine decolourised: liquid contains C=C. Reaction with PCl_5: liquid contains −OH

Several structural formulae are possible, although it is unlikely to find the −OH group attached directly to one of the carbons on the double bond, so the best structure is:



9. (a) Each measurement needs to be made to the nearest 0.1 cm^3, so a pipette or a burette would be needed.

(b) Purple to colourless

(c) So that the final volume in each experiment would be the same (otherwise, there is an effect on the concentration e.g. going from reaction A to reaction B, the same number of moles of glucose solution would be in a different total volume, and therefore would produce a different concentration).

(d) Colorimetry could be used to measure the gradual change in colour throughout the reaction.

(e) Potassium manganate(VII): compare A and B, when concentration halves, the rate halves, so first order

Sulfuric acid: compare C and D, when concentration halves, the rate halves, so first order

Glucose: compare A and C, when concentration halves, the rate is unaffected (the effect is so small as to be discounted), so zero order.

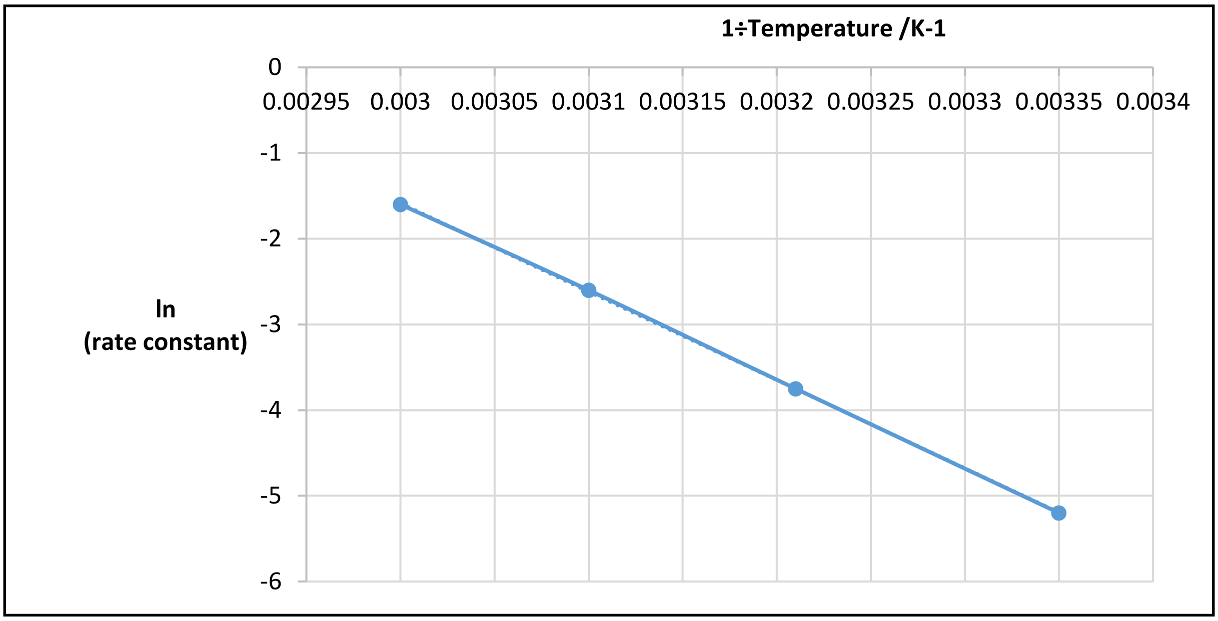
rate = k [manganate(VII)][glucose]

10. (a) The reaction is complete when Henna can no longer see the black spot through the contents of the tube.

(b) Note that the temperature in °C needs to be converted into kelvin first.

Experiment	Temperature / °C	1 ÷ Temperature / K^{-1}	ln (rate constant)
A	60.0	3.00×10^{-3}	−1.60
B	49.5	3.10×10^{-3}	−2.60
C	38.5	3.21×10^{-3}	−3.75
D	25.5	3.35×10^{-3}	−5.20

(c)



(d) Taking the first and last points: gradient = $(-5.20 - (-1.60)) / (3.35 - 3.00 \times 10^{-3})$

$= -3.60 / 0.35 \times 10^{-3} = -10\,285....$

Activation energy = − grad x R = 10 285... x 8.31 = 85 4774.. J mol^{-1} = 85 kJ mol^{-1}

(A final answer to 2 or 3 sig. figs is appropriate for the data)

11. (a) Bosun should plot the titration curve as the propanoic acid is reacted, and find the minimum volume required to exactly react with the propanoic acid. The pH of the mixture, at the point where half this volume of sodium hydroxide solution has been added to the propanoic acid, is the pK_a value for propanoic acid. K_a can then be found in the usual way.

(b) Bosun adds the sodium hydroxide solution in relatively large volumes. This makes little difference at the start of the experiment, but means that it would be difficult to find the end point with any accuracy. The method should be altered to add smaller volumes, especially near the end-point.

(c) If the pH meter is stored in acid / alkali, the solution remaining on the electrode will introduce an error when it is used. Even if the electrode is kept in water, this added water will alter the concentration of the propanoic acid, especially as the experiment uses a small volume of acid.

(d) $K_a = [H^+]^2 / [HA]$, so $[HA] = [H^+]^2 / K_a$

$[H^+] = 10^{-2.58} = 2.63 \times 10^{-3}$ mol dm^{-3}

$[HA] = (2.63 \times 10^{-3})^2 / 1.349 \times 10^{-5} = 0.51$ mol dm^{-3} (to 2 sig. figs)

(A final answer to 2 or 3 sig. figs is appropriate for the data)

12. (a) It completes the electrical circuit and also allows the flow of ions into each of the two half-cells. This prevents polarisation of the electrodes, which would be caused by the electron transfer at each.

(b) All common potassium salts and nitrate salts are soluble, so there will be no interaction between these ions and any in the half-cells to form precipitates.

(c) The voltmeter has been connected the wrong way round in the circuit i.e. against the natural direction of the flow of electrons.

(d) The value is lower than the expected value, which is 1.10 V (using the copper half-cell as +0.34 V and the zinc half-cell as −0.76 V gives +0.34 − (−0.76) = 1.10 V). This means that the solution used is not at standard concentration (1 mol dm^{-3}).

As $Cu^{2+} + 2e \rightleftharpoons Cu$ = +0.34 V, the lower contribution from this half-cell implies that the solution is less concentrated that the standard concentration (1 mol dm^{-3}).

13. (a) Potassium manganate(VII) solution is deeply coloured, even in dilute solutions, so reading the marking on the burette can be difficult (some burettes have white graduations for this purpose).

(b) The reaction is very slow to begin with, although it speeds up as it is catalysed by the Mn^{2+} ions formed as the reaction takes place.

(c) The reaction acts as its own indicator. During the titration, as potassium manganate(VII) reacts with the ethanedioic acid, the products formed are colourless, so the purple colour of the manganate(VII) solution is discharged. At the end point, when there is no ethanedioic acid left, and the manganate(VII) colour is not discharged, so there is a permanent pink colour.

(d) $2MnO_4^- + 5(COOH)_2 + 6H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

Amount of manganate(VII) used = (0.00200 x 10.5) / 1000 = 2.10 x 10^{-5} mol

Amount ethanedioic acid in 25.0 cm^3 = 5/2 x 2.10 x 10^{-5} = 5.25 x 10^{-5} mol

Amount ethanedioic acid in 250 cm^3 = 5.25 x 10^{-4} mol

Mass of ethanedioic acid = 5.25 x 10^{-4} mol x 90 g mol^{-1} = 0.047 g (to 2 sig. figs)

(A final answer to 2 or 3 sig. figs is appropriate for the data)

14. (a) +3

(b) The reaction is highly exothermic. If the water is added to the acid, the water (which is less dense than the sulfuric acid) can settle on top, and the heat energy released can be sufficient to boil the water. This leads to spitting of acid droplets out of the mixture.

(c) This reaction must be exothermic, as the passage warns that the mixture may need cooling to ensure that the temperature does not rise about 60 °C.

(d) Molar mass of potassium dichromate = 2(39.1) + 2(52.0) + 7(16.0) = 294.2 g mol^{-1}

Molar mass of chrome alum = 998.6 g mol^{-1}

Amount of chrome alum produced = amount of dichromate = 15 g / 294.2 g mol^{-1}

So, max mass of chrome alum = (15 g / 294.2 g mol^{-1}) x 998.6 g mol^{-1} = 50.9 g

15. (a) **A** and **B** both contain a carbonyl group (2,4-DNPH test). **B** must be an aldehyde, as it can be oxidised by acidified potassium dichromate(VI), whereas **A** must be a ketone. **A** must be propanone and **B** is propanal.

(b) A capillary is sealed at one end. The open end is pushed, into a sample of powdered solid **A**, the tube is inverted, and then tapped to make the solid fall into the closed end. The capillary tube is attached to a thermometer, and this is placed into a tube containing an oil (which must still be a liquid at the melting point of solid **A**). The oil is gently heated, and stirred with the thermometer. The melting temperature is taken as a range from the temperature when the crystals first begin to melt, until the temperature when they are fully liquid. Alternatively the use of a melting point apparatus may be described.

(c) (i) The solution could contain Cr^{3+}, Fe^{2+} or Ni^{2+} ions.

(ii) Red-brown precipitate is $Fe(OH)_3$, so Fe^{3+} ions are present in **D**.

Deep blue solution contains $[Cu(NH_3)_4(H_2O)_2]^{2+}$, so Cu^{2+} ions are present in **D**.

The original solution was green because it was a mixture of yellow $[Fe(H_2O)_6]^{3+}$ ions and blue $[Cu(H_2O)_6]^{2+}$ ions.

16. (a) Amount of aspirin = amount of salicylic acid = 2.00 g / 138 g mol^{-1} = 0.01449… mol

Amount of aspirin = 0.01449… mol x 180 g mol^{-1} = 2.61 g

(b) Percentage yield = 1.45 g / 2.61 g x 100 % = 55.6 %

(c) Side reactions / incomplete reaction of salicylic acid will reduce yield, as will handling losses.

(d) (i) This ensures that the solution of aspirin in the solvent is as concentrated as it can be, so that it will crystallise readily on cooling.

(ii) As the aspirin is in very high concentrations in the solution – and effectively saturates the solution – it crystallises out first. As the solvent is now holding less solute, impurities stay in the solution and do not precipitate out.

(iii) The melting point would agree with that in a data book, and would be over a narrow range. TLC could be carried out, showing a single spot on the plate.