



IB Chemistry IA Handbook

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(For use with the IB Diploma Programme)

(Fourth edition)

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This criterion assesses the extent to which the student establishes the scientific context for the work, states a clear and focused Research Question and uses concepts and techniques appropriate to Diploma level. Where appropriate, this criterion also assesses awareness of safety, environmental, and ethical considerations. The following table is an extract from the IB Chemistry Guide and is the basis that will be used by your teacher and the moderator for the assessment of your work.

MARK	DESCRIPTOR
0	The student's report does not reach a standard described by the descriptors below.
1-2	<p>The topic of the investigation is identified and a Research Question of some relevance is stated but it is not focussed.</p> <p>The background information provided for the investigation is superficial or of limited relevance and does not aid the understanding of the context of the investigation.</p> <p>The methodology of the investigation is only appropriate to address the Research Question to a very limited extent since it takes into consideration few of the significant factors that may influence the relevance, reliability and sufficiency of the collected data.</p> <p>The report shows evidence of limited awareness of the significant safety, ethical or environmental issues that are relevant to the methodology of the investigation *</p>
3-4	<p>The topic of the investigation is identified and a relevant but not fully focused Research Question is described.</p> <p>The background information provided for the investigation is mainly appropriate and relevant and aids the understanding of the context of the investigation.</p> <p>The methodology of the investigation is mainly appropriate to address the Research Question but has limitations since it takes into consideration only some of the significant factors that may influence the relevance, reliability and sufficiency of the collected data.</p> <p>The report shows evidence of some awareness of the significant safety, ethical or environmental issues that are relevant to the methodology of the investigation*.</p>
5-6	<p>The topic of the investigation is identified and a relevant and fully focused Research Question is clearly described.</p> <p>The background information provided for the investigation is entirely appropriate and relevant and enhances the understanding of the context of the investigation.</p> <p>The methodology of the investigation is highly appropriate to address the Research Question because it takes into consideration all, or nearly all, of the significant factors that may influence the relevance, reliability and sufficiency of the collected data.</p> <p>The report shows evidence of full awareness of the significant safety, ethical or environmental issues that are relevant to the methodology of the investigation.*</p>

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Guiding questions:

- *To what extent does the choice of investigation allow for concepts and techniques appropriate to Diploma level to be employed?*
- *To what extent has the student stated a clear and focused Research Question?*

2.1 The Research Question

2.1.1 Choosing a topic

Introduction

The Individual Investigation seeks to develop the IB candidate's investigative skills and to provide opportunities for self-motivation, independent learning and the planning and designing of appropriate experiments. It also provides the candidate with an opportunity to write in a scientific manner which reveals the significance of the findings of the Individual Investigation by analysing and interpreting the results in a critical and scientific manner and demonstrating knowledge and understanding of the chemical basis of the Individual Investigation.

The Individual Investigation is assessed internally through the five Group 4 Assessment Criteria and moderated externally by the IBO (if sampled). The length of the Individual Investigation work is 10 hours and this includes the planning stage and the experimental work. After completion of the Individual Investigation, the Report for the assessment is likely to take a further 5-10 hours.

It is likely that consultation with your Chemistry Teacher will ensure an early focus and clear direction as to the suitability of the topic chosen for your Individual Investigation. While you will be involved in initial reading and research, it is important that this aspect of the Investigation does not take too much time of your time.

You should consult a wide variety of sources in selecting topics for investigation. The sources that might be consulted could include: Chemistry text books and practical books, journals and periodicals, e.g., *New Scientist*, *Chemistry Review* (published by Philip Allan) (especially its project pages section), *The Australian Journal of Education in Chemistry*, *Journal of Chemical Education*, *Education in Chemistry* and *School Science Review* published by the ASE (print and on-line) and Internet web sites.

Focused investigations completed in the time available are likely to be the most successful. Well-controlled investigations will score higher marks than investigations with too many input variables from which valid conclusions cannot be drawn. A good investigation will generate a variety of raw data that can be manipulated via calculations and presented by graphs.

Time constraints, chemical laboratory facilities, availability of chemical equipment/apparatus, chemicals, costs and safety are all factors that need to be considered when you choose investigation topics.

While your chemistry teacher should encourage you to be creative and original, the Individual Investigation does not require it to be a piece of original research but should be new to you as an IB Chemistry candidate.

Examples of investigation topics

Let us consider chemical kinetics as topic to select a reaction as an Individual Investigation. You may have carried out practicals involving studying the reaction between calcium carbonate and hydrochloric acid. You may have investigated the effect of surface area of the calcium carbonate, or the temperature or concentration of the acid. This practical is unlikely to score high marks as an Individual Investigation.

A more suitable reaction for a kinetic study might be a more complex reaction investigated by a method not specified in the IB Chemistry syllabus. For example, the *Harcourt Essen* reaction. First find the order of reaction with respect to hydrogen peroxide, iodide and acid using a 'clock' reaction.

Therefore you could find the rate equation, rate constant and suggest a possible mechanism. Then find the order of reaction with respect to catalyst, or look at the effect of catalyst on order of reaction of hydrogen peroxide or find the activation energy with and without catalyst.

Another 'clock' reaction is the reaction between iodide and persulfate ions. First find the order with respect to iodide and persulfate using a 'clock' reaction. Therefore find the rate equation, rate constant and suggest a possible mechanism. Then find the activation enthalpy, or look at the effect of ionic strength or explore the effect of d-block ion catalysts.

A suitable Individual Investigation may take you beyond the IB Chemistry syllabus and build on your strengths in mathematics, for example, investigating the *Freundlich* adsorption isotherm from surface chemistry which is a curve relating the concentration of a solute on the surface of an adsorbent to the concentration of the solute in the liquid with which it is in contact.

You could shake different concentrations of ethanoic acid with activated charcoal, filter and find the concentration of acid remaining in solution by titration with sodium hydroxide solution. Use the data to calculate the *Freundlich* constants and extend to other acids such as methanoic, propanoic and ethanedioic acids.

The IB Learner Profile encourages diploma students to be ‘risk takers’ and a suitable Individual Investigation may involve growing crystals via precipitation in a sodium silicate gel. This is a diffusion technique and it is often found that crystals grow in banded arrangements known as Liesegang Rings (*Figure 201*). The formation of these rings has not been fully explained and a number of variables can be investigated, for example, pH and the passage of an electric current (AC or DC.). *Figure 201* is taken from <http://en.wikipedia.org/wiki/Liesegang_rings>



Figure 201 Liesegang Rings

2.1.2 Characteristics of a good Research Question

A Research Question is a focused and challenging question addressing a chemical problem or controversy with the aim of answering it by a conclusion that is based on the analysis and interpretation of chemical data.

The Research Question is a critical component of the investigation. When the aims of your study are clearly defined, this helps determine all other aspects of your investigation. The Research Question dictates the whole process; it drives your investigation design including how data are to be collected and processed and what evidence is required. It guides your analysis and interpretation of the processed data and the chemical arguments associated with them.

Hence, the Research Question can certainly make or ‘break’ your Individual Investigation. If your Research Question is weak, it would be too difficult to compensate for it in the other aspects of the study; and the whole investigative process is unlikely to be successful. Remember, a question well asked can lead to a question well answered.

Therefore, a good Research Question is at the heart of a great investigation. At the IB Diploma level, it is expected that the Research Question has a narrow focus and yet, it allows for an extensive exploration of the topic appropriate to Diploma level, given the available time, resources and ethical constraints.

A Research Question is deemed appropriate to IB Diploma level when it has the following characteristics:

- The Research Question encourages a complex answer. It is not answerable by a simple ‘yes or no.’ The answer to it should not be immediately obvious. It should have multiple possible answers and it is capable of generating multiple insights and possible surprises. The Research Question that aims to verify a known principle or physical or chemical law is unacceptable, as the answer to it is already known.
- The Research Question is amenable to the formulation of a testable hypothesis. It is grounded on a theoretical chemical framework and has the potential to lead to a meaningful investigation design and methodology.
- The answer to the Research Question transcends the raw data. It is expected that the analysis and interpretation implemented to answer the Research Question go beyond the raw data. It involves variables that can be determined from the raw data and includes cause and effect relationships. It allows for a reasonable amount of data processing that shall include some calculations and graphing of processed chemical data (not just raw data) and a meaningful interpretation of the graphs (including gradients and intercepts) and tables.
- The Research Question takes ethical and safety issues into consideration. You need to carry out a risk assessment but you need to strike a balance. You must be aware of any toxic reagents you are using and suggest suitable safe practices. But there is no need to list putting on gloves, lab coat and safety glasses or goggles at every stage of the investigation.
- Equally important, the Research Question must be communicated effectively, that is, the Research Question is well written and well phrased. It is both clear and concise. It identifies both the independent and dependent variables, and where appropriate, the controlled variables and possibly the method.

It uses simple language and includes scientific terms only when they add meaning to the statement. It is such a disservice to your study if you have an interesting investigation but your Research Question was stated inadequately.

For example, you may be investigating the growth processes, for example, tube formation, (and a variable that may control it) that occurs during the formation of a ‘crystal garden.’ Chemical gardens are obtained from the precipitation reaction on adding crystals of soluble metal salts to an aqueous solution of sodium silicate or introducing sodium silicate into a solution of a transition metal salt.

However, almost a hundred years later, and several centuries on from the first observations of so-called metallic trees by the early chemists such as *Glauber*, crystal gardens remain incompletely understood.

Hence to write your Research Question, ‘Investigating a Crystal garden’ is insufficient and does not reveal the complexity of the investigation.

2.1.3 Examples of unsuitable and suitable Research Questions

Shown below are some examples of how to formulate and write the Research Question.

Example 1 (Unsuitable)

How does temperature affect the solubility of potassium chloride?

This is a simplistic Research Question and probably not suitable for an Individual Investigation, though it may be suitable as a simple practice exercise. It is unfocussed because the solvent is not specified. It may be helpful to include the units for solubility in the Research Question. This Research Question does not offer the possibility of employment of concepts and techniques appropriate to Diploma level. Little data processing may be generated from the practical work.

Example 2 (Unsuitable)

To investigate rusting of iron

This Research Question is unfocussed. Are you trying to find out the chemical nature of rust? Are you trying to find out which substances in the air are involved in rust formation? Here is a more focused Research Question:

What is the effect of the concentration of dissolved sodium chloride solution on the rate of rusting of iron nails as measured by their increase in mass?

Example 3 (Suitable)

What is the relationship between the concentration of nitric acid (at constant temperature) and the rate of reaction between hydrogen ions (hydrochloric acid) and thiosulfate ions (sodium thiosulfate) as measured by the time taken for a sulfur precipitate to obscure a cross under the flask?

This is a very focused Research Question that identifies the independent and dependent variables as well as the method and controlled variables.

Example 4 (Suitable)

What is the relationship between the temperature and the rate of reaction between magnesium and excess hydrochloric acid as measured by the volume of gas collected per second with a gas syringe?

This is a very focused Research Question that identifies the independent and dependent variables as well as the method and units of rate.

Exercise

Write suitable focused Research Questions for examples 1 and 2.

2.2 The Investigation In Context

Guiding questions:

- *To what extent has the student established the scientific context for the work through a discussion of its significance?*
- *How well has the student justified their choice of Research Question and approach to the investigation?*

2.2.1 Justification for the investigation

The Research Question can be put into context by:

- **stating** why this is an important question to be answered and/or why you found this to be a particularly interesting question. A question is worth investigating if it is unresolved or unanswered by existing chemical literature. Your methodology may also be an innovative process to find supporting data or falsify an existing hypothesis.
- **discussing** the background material that is particularly relevant to the Research Question and by summarising the current understanding of the problem you are investigating.
- **addressing** how your investigation will help to fill in the holes in our current chemical knowledge of the topic.

Scientific hypotheses are testable hypotheses. This means that you need to be able to perform an experiment and take measurements and formulate hypotheses to establish how two variables are related.

- A good hypothesis states an expected relationship between the dependent and independent variables and clearly explains or justifies the relationship between variables.
- A good hypothesis reflects the theory or literature on which they are based. A good hypothesis has a substantive link to existing chemical literature and chemical theory.
- The Exploration criterion does not explicitly require a hypothesis but formulating one can help you give direction to your investigation and form an important part of the background of your planning and report. Formulating a hypothesis requires you to state clearly what you intend to change and measure. This is crucial as it can lead you to a reasonable experimental design.
- The ultimate value of the hypothesis is that its explanation, if linked convincingly to existing theory and literature, can give you the opportunity to demonstrate higher level order thinking skills necessary to access the assessment criteria at a higher achievement level.

2.2.2 Writing the background from the literature

A review of the chemical literature related to your Individual Investigation has the following functions:

- To justify your choice of Research Question, theoretical or conceptual framework, and method.
- To establish the importance of the chemical topic.
- To provide background information needed to understand the study.
- To show your IA Assessor you are familiar with significant and/or up-to-date research relevant to the topic.
- To establish your study as one link in a chain of research that is developing knowledge in this field of chemistry.

The review will provides a historical overview of the theory and the research literature, with a special emphasis on the literature specific to the Individual Investigation topic. It serves as well to support the argument/proposition behind your thesis, using evidence drawn from authorities or experts in your research field.

For example you may have chosen to study an oscillating reaction such as the *Belousov* and *Zhabotinskii* (BZ) reaction for your Individual Investigation. A literature search will reveal that the study of non-linear reactions began in 1968. At a conference in Prague that year, the work of *Belousov* and *Zhabotinskii* on oscillating chemical reactions, done in Moscow in the fifties and early sixties, became widely known to Western scientists. The field of oscillating reactions received a further boost in 1972, when *Noyes* at the University of Oregon suggested a chemical mechanism for the

Belousov-Zhabotinskii reaction. In the mid-seventies, a French research group pioneered the use of the continuous-flow stirred tank reactor for the study of chemical oscillations and chaos. One importance of oscillating reactions is their possible connection to pattern formation in embryos.

However, it is important that you do not include any advanced material that is not directly relevant to the Individual Investigation or you do not understand and explain clearly.

2.2.3 Formulating the hypothesis

In chemistry, a hypothesis is a prediction and associated explanation of the type of chemical behaviour or result expected during a chemical investigation. Hypotheses enable the design of investigations so that predictions based upon a hypothesis may be tested and either tentatively ‘supported’ or ‘disproved’. Many IB Chemistry investigations will start with a hypothesis or a pair of competing hypotheses. They should be supported by a detailed justification using relevant chemical concepts (especially those at the ionic, atomic or molecular level).

The IB Learner Profile encourages IB students to be ‘risk takers’ and this can be interpreted as encouraging you to investigate physical relationships not included in the IB Programme, for example, to establish whether there is a relationship between the strength of an acid (as measured by its acid dissociation constant, K_a) and its enthalpy change of neutralization.

Examples of hypotheses

Introduction

Hypotheses may be qualitative, for example, an increase in temperature will increase the rate of reaction between zinc granules and dilute aqueous hydrochloric acid. This behaviour is accounted for by the increase in collision rate between zinc atoms (on the surface of the zinc) and hydrogen ions (in aqueous solution) and, more importantly, the increase in the fraction of collisions between these species that have combined kinetic energies greater than, or equal to, the activation energy.

Where possible, the hypothesis should be quantitative, and describe the relationship between a dependent and independent variable. For example, every rise of ten degrees Celsius will double the rate of reaction between zinc and dilute aqueous hydrochloric acid.

Arrhenius temperature dependence

This is known as Arrhenius temperature dependence and a detailed quantitative ‘proof’ of this behaviour using the principles of kinetic theory is shown below.

We need to find the value of the activation energy, E_a

that would give a doubling of reaction rate between 0 °C and 10 °C, i.e., for $\frac{k_2}{k_1} = 2$

where k_1 and k_2 are the rate constants at absolute temperatures T_1 and T_2 .

Then with this value of E_a we shall see what the effect is on $\frac{k_2}{k_1}$ at two different 10 °C ranges.

For the calculation of E_a , take $\frac{k_2}{k_1} = 2$, $T_1 = 273$ K, $T_2 = 283$ K and $R = 8.314$ J K⁻¹ mol⁻¹:

Arrhenius equation:

$$\ln\left(\frac{k_2}{k_1}\right) = \left(\frac{E_a}{R}\right)\left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

Therefore:

$$\frac{\ln\left(\frac{k_2}{k_1}\right)}{\left(\frac{1}{T_1} - \frac{1}{T_2}\right)} = \frac{E_a}{R} \Leftrightarrow E_a = \frac{\ln\left(\frac{k_2}{k_1}\right)}{\left(\frac{1}{T_1} - \frac{1}{T_2}\right)} \times R$$

Substituting the values given, we have:

$$E_a = \frac{0.693}{\left(\frac{1}{273} - \frac{1}{283}\right)} \times 8.314 = 44500 \text{ J mol}^{-1}$$

$$= 44.5 \text{ kJ mol}^{-1}.$$

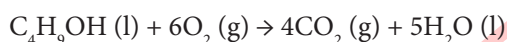
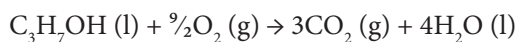
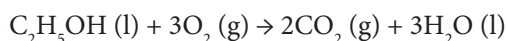
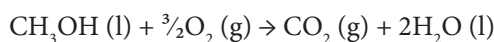
It is assumed in this calculation that $\frac{k_2}{k_1}$ does not vary with temperature or the value of the activation energy.

Enthalpies of combustion of alcohols

A linear relationship is predicted between the change of the enthalpy of combustion of monohydric alcohols and the number of carbon atoms. It is predicted that there will be a similar difference from one monohydric alcohol to the next as the homologous series is ascended.

These predictions arise because each methylene group, $-\text{CH}_2-$, is responsible for a fixed increment in the enthalpy of combustion. In addition, each bond makes a characteristic contribution to the enthalpy of the alcohol.

When an alcohol undergoes complete combustion the bonds between the atoms within the alcohol molecule are broken and carbon, hydrogen and oxygen atoms are released. New bonds form between carbon and oxygen atoms to form carbon dioxide and between hydrogen and oxygen atoms to form water (see Figure 202). Each alcohol has one more carbon-carbon bond and two more carbon-hydrogen bonds than the previous alcohol. As the alcohol molecules increase in molar mass, more energy is required to break the bonds, but even larger amounts of energy are released as these atoms form carbon dioxide and water. Upon combustion, each alcohol molecule forms one more carbon dioxide molecule and one more water molecule than the previous alcohol.



NAME OF ALCOHOL	C-C BONDS BROKEN	C-H BONDS BROKEN	C-O BONDS BROKEN	O-H BONDS BROKEN	O=O BONDS BROKEN	C=O BONDS FORMED	O-H BONDS FORMED
METHANOL	0	3	1	1	1.5	2	4
ETHANOL	1	5	1	1	3	4	6
PROPAN-1-OL	2	7	1	1	4.5	6	8
BUTAN-1-OL	3	9	1	1	6	8	10

Figure 202 Summary of bonds broken and formed during combustion of monohydric alcohols

There is a constant difference between the monohydric alcohols as the homologous series is ascended:

$$1 \text{ extra C—C bond is broken} = 1 \times 348 = 348 \text{ kJ extra energy needed}$$

$$2 \text{ extra C—H bonds are broken} = 2 \times 412 = 824 \text{ kJ extra energy needed}$$

$$1.5 \text{ extra O=O bonds are broken} = 1.5 \times 496 = 744 \text{ kJ extra energy needed}$$

$$\text{Hence, total extra energy needed} = 348 + 824 + 744 = 1916 \text{ kJ mol}^{-1}$$

$$2 \text{ extra C=O bonds are formed} = 2 \times 805 = 1610 \text{ kJ extra energy produced}$$

$$2 \text{ extra O—H bonds are formed} = 2 \times 463 = 926 \text{ kJ extra energy produced}$$

$$\text{So, total extra energy produced} = 1610 + 926 = 2536 \text{ kJ mol}^{-1}$$

The extra energy released is greater than the extra energy needed and thus, as the homologous series is ascended, the enthalpies of combustion become more exothermic. More importantly, because the change in the structures of the alcohols is fixed, the difference in the enthalpy of combustion from one homologue to the next will also be fixed and will be: $\Delta H = (1916 - 2536) \text{ kJ mol}^{-1} = -620 \text{ kJ mol}^{-1}$.

This means that one mole of ethanol will release 620 kJ more energy than one mole of methanol when combusted; one mole of propan-1-ol will release 620 kJ more energy than one mole of ethanol when combusted, and so on. The position of the alcohol functional group (-OH) will have little effect since it is the number and type of bonds that are being broken and made that dictate the size of the enthalpy change of combustion.

Exercise

Carry out a similar exercise with the first four members of the alkanes homologous series. Refer to the IB chemistry data booklet for the relevant data.

Predictions

Predictions are based upon your hypothesis. They are descriptions of what you expect to observe and why.

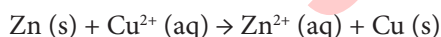
For example, if you predict first kinetics for a reactant in a particular chemical reaction then you would predict its initial rate of reaction will double if the concentration of the reagent is doubled.

This behaviour can be accounted for by the doubling in the number of collisions involving that reactant in the rate determining step, that is, the elementary step that exerts a dominant effect on the overall rate.

It is important to realise that reaction mechanisms are simply theories, and cannot be proven beyond doubt. Research Chemists are led to accept a theory of the mechanism of a particular reaction because it provides the most satisfactory or easiest approach to understanding the observed kinetic behaviour of that reaction. However, new results may later be obtained which are not consistent with this theory, which must then be rejected or modified.

Consider the replacement reaction between zinc powder and aqueous copper(II) sulfate solution. Since zinc is more reactive than copper (it is higher up in the reactivity or activity series), it is predicted to be able to replace copper from a solution of copper(II) sulfate. (This could also be predicted from a consideration of standard electrode potential data: zinc is a more powerful reducing agent than copper).

If the mass of zinc powder is increased, then the mass of copper replaced should also increase. You must provide a chemical explanation for this qualitative prediction.



An appropriate ionic equation should be provided which should be explained in words, for example, 'one atom of zinc displaces one atom of copper'. Therefore if the mass of zinc increases and the number of atoms of zinc increases, this will cause the number of copper ions replaced and therefore the mass of copper produced to also increase.

A quantitative prediction and explanation should then be given, for example, if the number of atoms of zinc doubles then the number of atoms of copper replaced will also double (assuming that the copper(II) sulfate solution is present in excess). This means that the mass of zinc used is directly proportional to the mass of copper displaced, but the masses will not be in a 1:1 ratio by mass, since the two metals have different relative atomic masses. Suitable calculations involving the mole concept must be included to support the statement.

2.3 The Investigative Process

2.3.1 Selecting, manipulating and controlling variables

Guiding questions:

- *To what extent has the student devised a methodology that shows awareness of the factors that may influence the collection of data relevant to the Research Question?*

Variables

Variables are factors that can be measured and/or controlled. Independent variables are those that are manipulated, and the result of this manipulation leads to the measurement of the dependent variable. A controlled variable is one that should be held constant so as not to obscure or hide the effects of the independent variable on the dependent variable. The variables need to be explicitly identified by you as the dependent (measured), independent (manipulated) and controlled variables (constants). Relevant variables are those that can reasonably be expected to affect the outcome. For example, consider the Research Question, 'How does changing the acid catalyst concentration affect the rate of the esterification reaction between propan-1-ol and propanoic acid?'

You must clearly state that the independent variable is the concentration of concentrated sulfuric acid (the catalyst and dehydrating agent) and the dependent variable is the concentration of propanoic acid after a fixed time interval. The relevant controlled variables are temperature of the reaction mixture, the initial concentrations in the reaction mixture of propan-1-ol and propanoic acid and any solvent, so that one concentration can be varied independent of the others.

Control of variables refers to the manipulation of the independent variable and your attempt to maintain the controlled variables at a constant value. Your method should include explicit reference to how the control of variables is achieved. If the control of variables is not practically possible, such as atmospheric pressure, some effort should be made to monitor the variable(s).

A standard measurement technique, for example, a redox titration or a flame calorimeter, may be used as part of a wider investigation but it should not be the focus of that investigation. You will be assessed on your individual design of the wider investigation. If a standard measurement technique is used you should reference it.

For example, while planning an investigation to study the factors that influence the rate of oxidation of vitamin C in fresh fruit juices, you may have adapted a method, for example, an acid-base titration or redox titration, for vitamin C determination from a literature source. A standard reference would then be expected as a footnote in your investigation 'write-up'.

A variable is a factor present in an experiment that can be changed and controlled to see what effect it has on the results of the experiment. Each chemical test should change only one variable at a time, in other words, each test should be a fair test. If more than one variable is changed during a test, it will not be possible to conclude which variable was responsible for the change in results.

For example, in the reaction between zinc and dilute aqueous hydrochloric acid the following continuous variables (variables that can be expressed as a decimal number) affect the rate of reaction: temperature of the aqueous hydrochloric acid, concentration of the aqueous hydrochloric acid (assuming the zinc is present in a large excess) and the surface area of the zinc (assuming the acid is present in a large excess). A change in the surface area of the zinc has a much greater effect on the rate than either changing the amount of zinc or acid.

Note that the rate of reaction is not affected by a change in the mass or amount of zinc (in grams or mol) used in the tests. The acid and the metal are also variables and are known as categorical variables (variables that can only be described by words). The initial rate will also be independent of the volume of hydrochloric acid (assuming the zinc is covered), though the way the rate changes with time will be affected.

Consider a simple practice investigation into the replacement reaction between zinc and aqueous copper(II) sulfate. For example, a possible Research Question could be to find the relationship between the mass of zinc and mass of copper produced in the replacement reaction between zinc and copper(II) sulfate.

You need to carefully identify all the variables in the investigation, for example: the mass of zinc powder (independent variable), the mass of copper produced (the dependent variable) and the controlled variables (to make the investigation a fair test): concentration and volume of aqueous copper(II) sulfate solution, temperature, time taken for reaction and the time left to dry the copper powder before weighing.

Consider another simple practice investigation into the effect of particle size on the rate of reaction between calcium carbonate and dilute aqueous acids (in a thermostatted water bath).

A possible Research Question could be to determine the effect of particle size on the rate of reaction between calcium carbonate and dilute hydrochloric acid.

The independent variable is the size of the particles (powder, small grains and large chips); the dependent variable is the time take for the calcium carbonate to react completely (that is the time effervescence occurs); the controlled variables will be temperature, type of acid used, volume of acid and concentration of the acid. It is assumed that the heat released during the reaction will be absorbed by the water bath and will not affect the rate of reaction. It is also assumed that the salt produced when the carbonate reacts with acid has virtually infinite solubility in water.

The identification and control of variables is an important part of IB Chemistry coursework. It may be helpful (where relevant) to have a separate section in your 'write-up' under the heading 'Exploration' that explicitly identifies and classifies the variables inherent in your investigation. The control of variables is the essential difference between scientific experimentation and trial-and-error.

Figure 203 (below) summarises the variables that affect selected various chemical and physical processes or reactions. These processes and reactions would make very suitable practice investigations where the Exploration criterion is assessed.

Chemical/Physical process or reaction	Major variables
Conductivity of an aqueous electrolyte	Strength of electrolyte, nature of electrolyte, temperature and concentration
Hydrolysis of a vegetable oil	Concentration of lipase, concentration of oil, type of oil, presence of detergent (concentration and type), pH, temperature
Solubility of a sparingly soluble ionic compound	Temperature, presence of common ions
Development of oxidative rancidity in an oil	Temperature, light, oxygen concentration, type of oil
Simple electrochemical cell	Nature of electrodes, temperature, immersion area of electrodes, distance between electrodes, concentration of electrolyte
Electroplating in aqueous solution	Current density (current per unit area of the cathode), temperature, presence of other ions and time
Electrolysis of aqueous sodium chloride	Concentration, temperature, distance between the electrodes, immersion depth of electrodes
Hydrolysis of halogenoalkanes	Nature of halogen atom, nature of carbon backbone, nature of solvent, temperature and pH
Thiosulfate ions and hydrogen ions reaction rate in aqueous solution	Concentrations of reactants, temperature, presence of a catalyst e.g. iron(III) ions
Formation of an ester from an alcohol and a carboxylic acid (equilibrium constant)	Use of excess reagent, removal of ester (via distillation), length of reflux time

Figure 203 Variables affecting common chemical processes or reactions

Exercise

Magnesium blocks are used to provide protect to iron objects, such as ships and piers.

- Design an experiment to investigate the effect of corrosion by sea water on magnesium.
- Identify the independent, dependent and controlled variables.

2.3.2 Establishing the rationale for the method**Guiding questions:**

- *To what extent does the methodology allow for the collection of sufficient relevant data that could enable a reasoned conclusion to be drawn?*
- *To what extent has the student shown how their method has been developed and modified?*
- *When appropriate, to what extent does the investigation indicate an awareness of safety, environmental, and ethical considerations?*
- *To what extent does the student engage with the investigation and make it their own?*

Planning a scientific investigation is one of the most demanding and difficult skills to learn. In order for training in planning skills to be effective you must have confidence in your practical abilities. It is not sufficient that you have simply learnt to follow instructions; you must be able to apply the experience you have got from earlier exercises in order to see the consequences of a given choice on the outcome of your plan. It is therefore vital that you understand the rationale for using particular approaches, pieces of equipment, recording and analysing techniques, rather than simply performing a given exercise in a particular prescribed way.

An appreciation of precision and reliability is essential when choices of measuring equipment or apparatus are made, and when experimental procedures are suggested. An understanding of random errors and possible systematic, associated with individual pieces of apparatus is fundamental to the successful choice of apparatus for a given task. A similar argument applies to the identification of variables that need to be controlled, and the proposing of suitable measures to control them.

The advantages and limitations of one type of measuring device, control measure or practical approach compared to other possibilities must be understood if the appropriate equipment, approach and quantities are to be used. The proposed experimental procedure should be workable. It should, given that the apparatus is assembled appropriately, allow data to be collected without great difficulty.

There should be a description, including labelled diagrams in cross section, of how the experiment should be performed and how the key variables are to be controlled. However, drawings perhaps should be perhaps be limited to complex set-ups, non-standard equipment or standard equipment being in an unusual manner.

Equipment or apparatus, of a level of precision appropriate for the measurements to be made, and quantities (and concentrations) of chemicals (IUPAC names and formulas) to be used should be specified.

The use of control experiments should be considered, if appropriate, especially if enzymes are present. Also, details of how the raw data are to be recorded, manipulated, analysed and evaluated should be given. Your method should not be a 'recipe' or simple list. It should justify the techniques and apparatus selected. It is suggested that it is in two sections: the planning and development of the method and the method actually used.

You may build your own apparatus for use in your Individual Investigation, for example a simple polarimeter (to study optical activity in sugars) or an Evans balance (to measure paramagnetism in transition metals salts).

You may also be using a standard method such as dual indicator approach together with a data-logger to record pH. You may also be using column chromatography with a vacuum pump to carry out 'flash chromatography'. It is very important to document and justify any modifications to standard methods. For example, you may have had to dilute a solution to get better titration results. There may be a need for controls. For example you may have made up a solution of known concentration to check the accuracy of one or more techniques.

Method

Your method should clearly allow another IB Chemistry student to replicate your Individual Investigation and results (within experimental error). *Figure 204* shows two lists of the same apparatus and materials used for a simple practice investigation that is assessed against the Exploration criterion.

Student A	Student B
Apparatus	Apparatus
Beakers	2 × 250 cm ³ glass beakers
Balance	Electronic balance (±0.0001 g)
Volumetric flask	50.00 cm ³ volumetric flask (TC) (±0.05 cm ³)
Burette	50.0 cm ³ burette (TD) (±0.1 cm ³)
Pipette	50.0 cm ³ pipette (TD) (± 0.05 cm ³)
Thermometer	Mercury thermometer (-10 to + 110 °C, ± 0.2 °C)
Conical flasks	3 × 100 cm ³ glass conical flasks
Clock	Manually operated electronic stop watch (± 1 s)
Spatula	Nickel spatula
Data logger	pH data logger sensor (manufacturer: Vernier Logger Pro 3.1: ± 0.02 pH units).
Chemicals	Chemicals
Methanoic acid	2.00 mol dm ⁻³ methanoic acid, HCOOH(aq)
Propanoic acid	2.00 mol dm ⁻³ propanoic acid, C ₃ H ₇ COOH(aq)
Sodium carbonate	10.00 g of sodium carbonate-ten-water, Na ₂ CO ₃ ·10H ₂ O(s)
Potassium hydroxide	0.0915 mol dm ⁻³ potassium hydroxide, KOH(aq)

Figure 204 Comparing equipment lists

Student A's list is imprecise and hence the method is not reproducible. Any deviations from the original method given or suggested by you should be documented and justified.

Most importantly you must carefully make sure that you have described a fair test. For example consider again the replacement reaction between zinc powder and aqueous copper(II) sulfate solution.

Here is what a student might think or write during a simple practice investigation into metal ion replacement that is going to be assessed against the Exploration criterion.

'Every time I change the mass of zinc powder I will use the same volume and concentration of aqueous copper (II) sulfate (present in excess – I will provide a calculation to prove this). I will perform the experiment at the same temperature in the same clean, dry glass apparatus and leave the mixture to react for the same period of time in the same air-conditioned laboratory. I will leave the copper to dry for the same length of time in a thermostatted oven (at the same temperature) before weighing (on the same electronic balance).'

Finally your method must include a description of sufficient raw data that you intend to collect. This means that you need to suggest how many times you should change variables and the values that you will use.

For example, consider changing the mass of zinc powder added during an investigation of the replacement reaction between zinc powder and aqueous copper(II) sulfate solution.

'I will use approximately 0.5 g of zinc powder the first time and record the precise mass and then the following approximate values 1.0 g, 1.5 g, 2.0 g, 2.5 g and 3.0 g and record the precise masses so that six tests are performed'.

Always make sure that you suggest at least five different and equally spaced values (unless it is not appropriate).

What will you measure?

For example, 'I will be measuring the mass of the dry copper powder produced after each experiment'. What will I control?'

For example, the volume of aqueous copper(II) sulfate solution used will be 50.0 cm³ (heated to a temperature of 50.0 degrees Celsius) and its concentration will be 1.50 mol dm⁻³ to ensure that it is present in excess (see appendix for calculation). I will leave each hot reaction mixture for 10 minutes to react (in an open 100 cm³ glass beaker). I will stir for two minutes after the reaction begins. I will also allow each sample of copper powder to dry for twenty four hours in an oven at 80 °C for 4 hours before weighing'.

How will you ensure accuracy?

'Each experiment will be repeated three times and then averaged to obtain accurate and reliable results for each mass of zinc used'.

Organic chemistry preparations

A reporting style, with quantities in brackets after each chemical reactant or solvent, is often favoured for an organic investigation. For example:

'Sodium metal (2.65 g; 0.12 mol) was added, a little at a time with caution, to ethanol (75.00 cm³) in a round-bottomed flask fitted with a reflux condenser.

The solution became warm and a gas (assumed to be hydrogen) was evolved as the sodium reacted. After 15 minutes...'

Where applicable describe the chemical reaction by writing a balanced or stoichiometric equation at the beginning (Figure 205). Displayed structural formulas are normally more helpful than molecular formulas. Include the relative molecular masses or molar masses of the reactants and products, the mass of the limiting reagent and hence the theoretical yield.

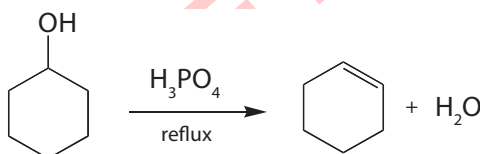


Figure 205 Skeletal formula showing the dehydration of cyclohexanol to form cyclohexene

It may also be helpful to explain and justify the use of particular organic practical techniques. For example, you may reflux ethanol with acidified potassium dichromate(VI) to form ethanoic acid. The refluxing is to ensure that any ethanal (an intermediate oxidation product) is cooled, condensed (by the cold glass wall of the reflux condenser and returned to the hot reaction mixture for further oxidation to ethanoic acid.

When you have prepared and purified an organic product, give the experimental yield in grams and then as a percentage of the theoretical yield.

Measure and record physical data required such as the melting point (range) if the product is a solid or its boiling point (range) if a liquid, together, for the purposes of comparison, with values from the chemical literature—most likely a data booklet or a text book appendix.

For example:

... produced X as a colourless liquid (6.6 g; 30%), boiling point 68-72 °C at one atmosphere pressure;

(literature boiling point 70 °C at one atmosphere pressure. (*Dictionary of Organic Compounds* (3rd edition), Singapore University Press, 1997.)

On-line resources for practical work

The Royal Society of Chemistry has information on a range of standard chemical techniques including associated health and safety guidance (Figure 206).

The URL is <http://www.nuffieldfoundation.org/practical-chemistry/standard-techniques> used with permission (see below).

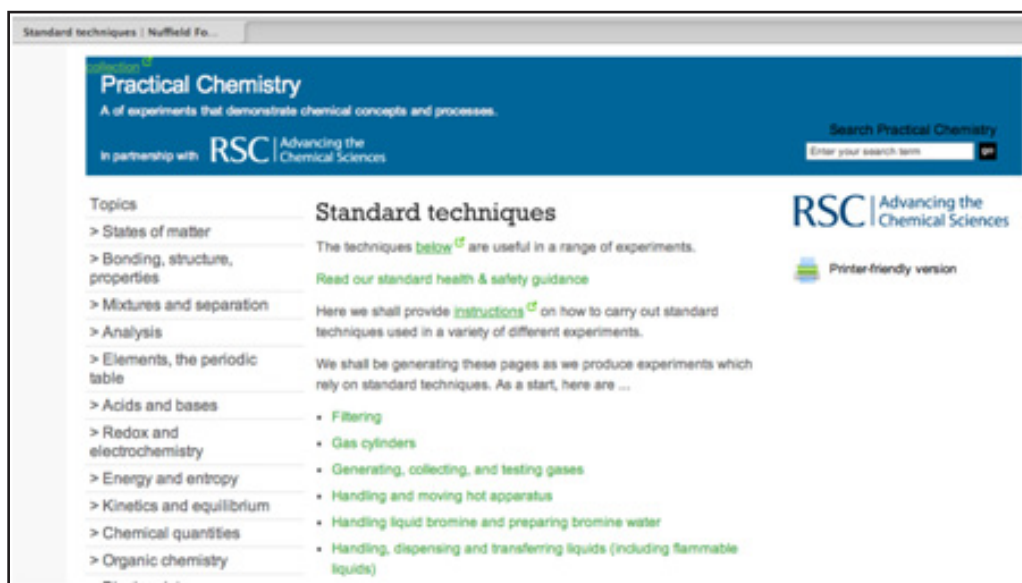


Figure 206 Website for The Royal Society of Chemistry

The Royal Society of Chemistry has another web site that shows various experimental techniques including organic techniques, such as refluxing and distillation.

The URL is <http://www.rsc.org/Education/Teachers/Resources/practical/> (see Figure 207)

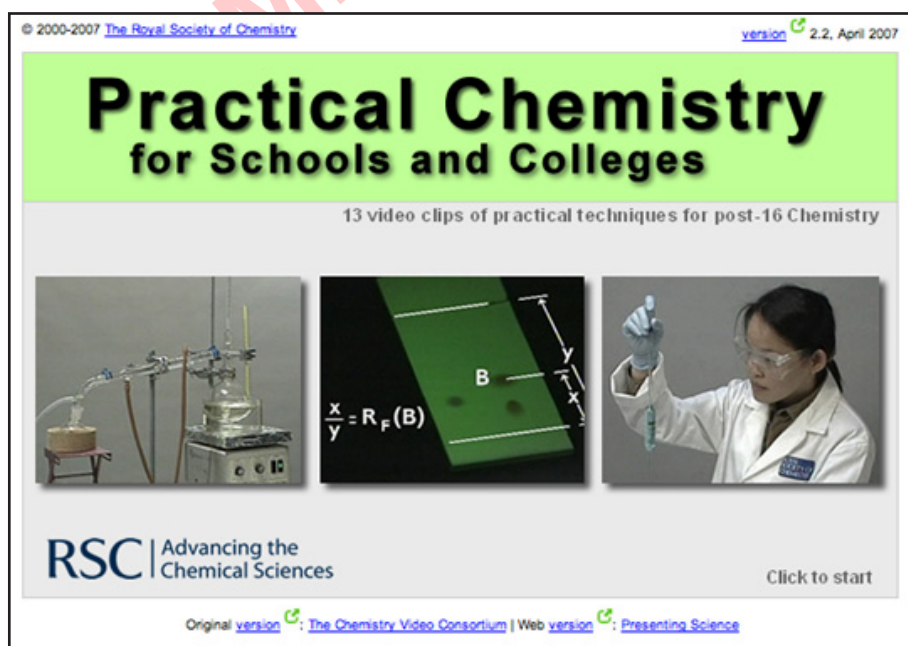


Figure 207 Information about Practical techniques

Drawing Chemical Apparatus for a Design Investigation

General guidelines for drawing good diagrams of Chemical apparatus for the planned method of a practice investigation being assessed against the Exploration criterion.

- Draw diagrams in cross section, not as three-dimensional pictures.
- Start at the top of your lab book page and work downwards, so that you are less likely to run out of space.
- Make drawings sufficiently large so the detail can be seen.
- Do not draw Bunsen burners, the bench, gas taps or clamps.
- A water or an oil bath should be shown if used.
- Label important points on the diagram or unusual items.
- If you name the glassware, then use the correct names.

Simple Distillation

Note the following points:

- The bulb of the thermometer is located opposite the entrance to the condenser since you want to record the temperature of the vapour.
- The delivery bend is vented so that when the apparatus is heated the glass joints are not pushed apart by the expanding gas.
- Never draw closed apparatus.

Note the internal structure of the Liebig condenser: the water jacket is separate and sealed from the tube that runs down the middle.

Heating Under Reflux

Note the following points:

- The water enters at the bottom of the Liebig condenser and leaves from the top.
- There must not be a stopper inserted into the top of the condenser – the apparatus must not be sealed.
- No thermometer should be placed into the top of the condenser

A description and diagrams of refluxing and distillation (simple and fractional) may be found elsewhere. *Figure 208* is a cross sectional diagram showing the standard laboratory preparation of hydrogen gas. It is provided as an example of a well drawn and labelled diagram.

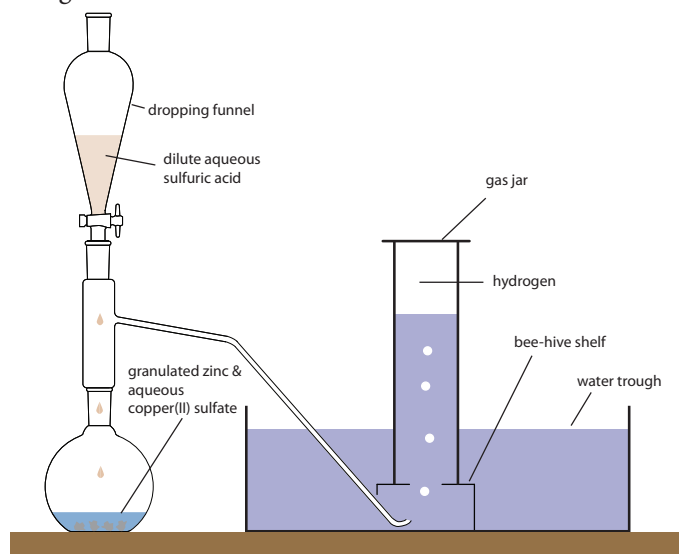


Figure 208 Collection of hydrogen gas

Experimental procedures for an Exploration Investigation

A number of issues have to be considered in a successful experimental design for an IB Chemistry Individual Investigation. Listed below are some questions that should be asked during the design of any experimental procedure involving a chemical reaction. You should get into the habit of asking appropriately critical questions of every practical procedure that you have experienced during your IB Chemistry practical programme.

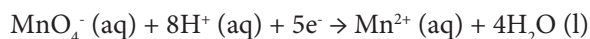
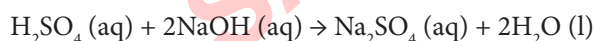
- In quantitative experiments, have the quantities (masses and/or volumes) been calculated correctly and are they are of a reasonable magnitude?
- In a preparation, is the scale on which it is planned appropriate for the aim?
The use of small quantities during organic or medicinal chemistry experiments can often result in little or no yield, simply due to mechanical losses. However, the use of large reacting quantities may raise waste and safety considerations.
- Have the quantities of each reagent been calculated correctly?
Thus, if a particular reagent has to be present in excess, for example, during a kinetics or replacement investigation, the calculation will need to be checked.
- Are the concentrations of the solution used in an investigation, for example a titration, reasonable?
Use of very concentrated solutions may give rise to inaccuracy, since one drop of the reagent solution contains a relatively large amount of dissolved solute in terms of the likely error or uncertainty in the measurement of the solution volume.

Conversely, the use of very dilute solutions may give rise to relatively large titre volumes, which means the burette has to be refilled. It reduces the percentage error, but it increases costs and also gives less flexibility for a titre being higher than expected.

If a titration is planned then you should aim to use concentrations of approximately 0.1 mol dm^{-3} and have an end point between 15 and 40 cm^3 . This will ensure that the last drop at the end point contains only a relatively small amount of reagent.

It is usually best to use the same concentration for the two reacting solutions. However, there are exceptions to this:

- For the reaction between aqueous sulfuric acid (dibasic) with aqueous sodium hydroxide it is best to use 0.05 mol dm^{-3} sulfuric acid with 0.1 mol dm^{-3} sodium hydroxide in order to keep the volumes identical.
- For redox titrations, the 'electron concentration' should be approximately 0.10 mol dm^{-3} . Potassium manganate(VII) is therefore normally used at a concentration of 0.02 mol dm^{-3} .



In experiments involving the measurement of heat changes, the situation is different since you need to produce a conveniently measurable temperature change. In order to produce a significant temperature rise then more concentrated solutions are required, of the order of 1 or 2 mol dm^{-3} .

A relatively large volume of solution is preferable since its surface area to volume ratio for heat loss is minimised. It is also easy to stir without the risk of spillage.

- If limits have been set on the amount of material or substance to be weighed out, have these limits been adhered to?

It is necessary to know the number of times the test is likely to be replicated. For example, if a standard solution needs to be prepared for a titration, then 250 cm^3 is more than sufficient and will allow up to nine sets of results using 25 cm^3 aliquots.

Safety

All practical work should be carried out in accordance with the health and safety legislation of the country in which it is done. You should not attempt any activities that conflict with this legislation and all your planned investigations must be approved by your chemistry teacher. Some safety information is provided in the front of the book.

Hands-on practical work can be carried out safely in schools but to ensure that it is safe, you must identify the hazards and reduce any associated risks to insignificant levels by adopting suitable control measures.

Risk assessment involves answering two basic questions:

How likely is it that something will go wrong?

For example, if you are pushing a delivery tube through a tight bung it is possible that the tubing will break and cut you.

How serious would it be if it did go wrong?

For example, the consequences of a spark from an experiment landing in an open bottle of magnesium powder are likely to be serious, and include spraying burning magnesium all over the laboratory, perhaps burning many students and setting the chemical laboratory ceiling on fire.

Once you have the answers to these questions, it is possible to plan your Individual Investigation to minimise the risk of an accident occurring and, if it does, to minimise its possible severity. In the second example, this could include bringing only the amount of magnesium powder required for the activity into the laboratory and making sure other students are aware of the magnesium powder.

Eye protection is the main control measure for preventing injury and glasses or goggles should be worn at all times in the laboratory. If you expect a problem, a range of control measures may be adopted, the following being the most common.



Use:

- a less hazardous (substitute) chemical.
- as small a quantity as possible.
- as low a concentration as possible.
- a fume cupboard and safety screens.

Shown below are the stages in carrying out a Risk Assessment for an IB Chemistry Investigation

1. Write down the chemicals and procedures you will be using (chemicals used or made, quantities, concentrations (if solutions), techniques and non-chemical hazards).
2. Use reference sources to identify any hazardous chemicals you are planning to use or make. Warning symbols will be printed on reagent bottles and in supplier's catalogues.
3. Record the type of hazards involved and the way you might be exposed to the hazard. There are standard reference sources with this information, such as the 'Hazcards' published in the United Kingdom by CLEAPSS. In North America MSDS (material safety data sheets) sheets should be read (*Figure 209*). Ensure you consult reliable sources such as chemical manufacturers, chemical societies and safety bodies.

Section 2: Composition and Information on Ingredients		
Composition:		
Name	CAS #	% by Weight
Sodium hydroxide	1310-73-2	100
Toxicological Data on Ingredients: Sodium hydroxide LD50: Not available. LC50: Not available.		
Section 3: Hazards Identification		
Potential Acute Health Effects: Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, of inhalation. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by burning, sneezing and coughing. Severe over-exposure can produce lung damage, choking, unconsciousness or death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.		
Potential Chronic Health Effects: CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Mutagenic for mammalian somatic cells. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to mucous membranes, upper respiratory tract, skin, eyes. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated exposure of the eyes to a low level of dust can produce eye irritation. Repeated skin exposure can produce local skin destruction, or dermatitis. Repeated inhalation of dust can produce varying degree of respiratory irritation or lung damage.		

Figure 209 MSDS card for sodium hydroxide

- Decide what protective or control measures to take so that you can carry out your practical work healthily and in safety.
- Find out how to dispose safely of any hazardous residues from your practical work and record this in your report with your reference.
- Check your plans with your Chemistry Teacher before starting any practical work.

An example of a Chemistry Investigation Risk Assessment form is shown in the Figure below.

TITLE OF THE IB CHEMISTRY INDIVIDUAL INVESTIGATION			
Outline of the procedures			
Hazardous substances being used or made	Nature of the hazards (e.g. highly flammable, toxic)	Quantities and concentrations being used or made	Safety measures (precautions)
Any non-chemical hazards and precautions to be taken			Signed (student) Signed (Teacher)
Disposal of residues			Date:

Handling Chemicals

You should regard all the chemicals you handle in the chemical laboratory to be hazardous. The most commonly used 'dangerous' chemicals are acids ($>2 \text{ mol dm}^{-3}$), halogens in solution and alkalis ($>2 \text{ mol dm}^{-3}$). All of these are corrosive and will damage skin and eyes. If you have an accident or spill acid, halogen or alkali then wash the chemical off with lots of running water and immediately report the accident to your Chemistry Teacher.

Another group of 'dangerous chemicals' are oxidising agents, such as potassium dichromate(VI), $\text{K}_2\text{Cr}_2\text{O}_7$, and potassium manganate(VII), KMnO_4 . These substances, when in solid form, may produce large amounts of heat as they react with other substances. They can create a fire risk. Your Chemistry Teacher will warn if there is any potential hazard with chemicals made available for your Individual Investigation or for chemicals that you have requested for a planned investigation.

You must make careful note of any safety instructions your Chemistry Teacher gives you. Look for the safety symbols shown in *Figure 210* which indicate you need to wear gloves and goggles. You must also read any practical instructions, noting any warnings given. You must also look at the labels on the reagent bottles. There are international symbols for chemical hazards and these are shown in the front of this publication.

Make sure you get information about chemicals before you start to use them. If you are using solutions it is much easier to use the correct solution if you already have a mental image of Benedict's solution as the 'blue solution', iodine solution as the 'brown solution'. Where possible use colours to distinguish between chemicals. If the chemicals are colourless or the same colour then make sure you carefully label or mark them carefully.



Figure 210 Some chemical hazard symbols

Sample Risk Assessment

A detailed method needs to be submitted to your IB Chemistry teacher before you can begin your Individual Investigation.

List of reactants and products

Chromium(III) chloride-6-water (solid)	Irritant; harmful by ingestion
Potassium hydroxide (solid)	Corrosive
Chromium(III) hydroxide	Minimal hazards
20 volume hydrogen peroxide solution	Irritant
Potassium chromate(VI) (solid)	Toxic
Glacial ethanoic acid	Corrosive, flammable
Potassium dichromate(VI) (solid)	Toxic (by inhalation)

Risk Assessment

Safety glasses must be worn at all times as potassium hydroxide is corrosive to eyes. Potassium hydroxide and glacial ethanoic acid are corrosive and hence disposable gloves and a buttoned up lab coat should be worn at all times. Inhalation of glacial ethanoic acid may cause lung and tooth damage. Any spillages should be reported immediately to your IB Chemistry Teacher and extensive dilution with water performed. Potassium chromate(VI) and potassium dichromate(VI) are toxic, and chromium(III) chloride and 20 volume hydrogen peroxide are irritants. Skin contact should be avoided. As hydrogen peroxide can be react explosively with glacial ethanoic acid, the school technician should be instructed to dilute and destroy any excess by boiling to decomposition. Potassium dichromate(VI) should be converted to chromium(III) ions by reaction with sodium metabisulfite, $\text{Na}_2\text{S}_2\text{O}_5$.

2.3.3 Establishing the rationale for the data processing

Your plan should include data tables that can be used to record relevant quantitative data. Your plan should also indicate what qualitative data should be recorded, for example, colour changes.

Your plan should also clearly outline how the raw data will be processed. For example, mass differences may be calculated, times (s) may be converted to ‘rates’ (s⁻¹) and pH values or rate constants may be transformed using a logarithmic function. These involve the conversion of dependent variables into processed variables.

The chemical and mathematical rationale behind the data processing must be clearly outlined in detail in your plan or report for the Individual Investigation.

It is likely that one or more graphs displaying processed data will be generated during your Individual Investigation. The graphs that will be drawn should be outlined, perhaps including sketches with blank axes.

For example if you are planning to generate an Arrhenius plot (of $\ln k$ versus reciprocal of absolute temperature) the mathematical transformation of the rate constants and times must be described, explained and justified, both chemically and mathematically.

You may be carrying out investigation that involves an acid-base titration where pH is followed by a pH meter. When a weak acid is titrated with a strong base then at the half-neutralisation point $\text{p}K_{\text{a}} = \text{pH}$ (the pH when half the volume of base required to exactly neutralise the acid has been added to the acid). This needs to be justified mathematically and chemically and any assumptions from acid-base theory outlined.

Any statistics, including simple averaging, must be outlined and justified.

Sample Exploration hypotheses

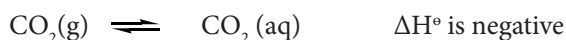
Below is an example of a series of chemical hypotheses based on a series of related acid-base equilibria. It may make an excellent series of practice investigations for assessing the Exploration criterion.

A variety of Research Questions may be generated from this system. For example, to find the relationship between pH and partial pressure of carbon dioxide above distilled water in a sealed container.

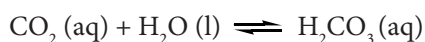
Background information

Carbonated drinks contain carbon dioxide dissolved under pressure. When carbon dioxide dissolves in and reacts with water, several chemical equilibria are established.

Carbon dioxide molecules in the gas phase in the space above the surface of the drink, known as the head space, are in equilibrium with hydrated molecules in the aqueous phase:



A proportion of the carbon dioxide also reacts with the water to form carbonic acid:



Carbonic acid is a weak acid and dissociates to give hydrogencarbonate and carbonate ions:

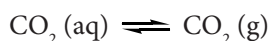
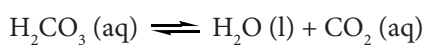
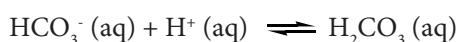
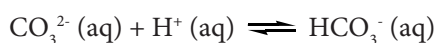


Carbonated drinks are manufactured by dissolving carbon dioxide gas under pressure in cold water.

Henry’s law is approximately obeyed, namely, that doubling the pressure of carbon dioxide will double the amount of gas dissolved in the water.

(This law is only, however, obeyed exactly by an ideal gas that does not chemically react with water).

The loss of carbon dioxide involves a reversal of the equilibria described above:



Soda water is acidic due to the presence of hydrogen ions, $\text{H}^+(\text{aq})$. The pH of the solution is related to the concentration of hydrogen ions, $\text{H}^+(\text{aq})$, present and hence to the concentration of dissolved carbon dioxide, $\text{CO}_2(\text{aq})$.

Hypotheses

Effect of temperature on the equilibrium of the system

If a carbonated drink is chilled and its temperature is decreased, then the forward reaction for equilibrium 1 is favoured resulting in more carbon dioxide dissolving in the water and a smaller rate of carbon dioxide bubble production and consequently a decrease in pH.

The forward reaction is favoured due to the system obeying Le Chatelier's Principle, namely, that any change imposed on the system will cause the system to respond by opposing the change. Le Chatelier's Principle is a consequence of a reaction minimising its Gibbs free energy change, ΔG .

If the temperature of the system is lowered then the system responds by increasing the temperature and thus favours the forward reaction of equilibrium 1 which is exothermic, namely, that heat is released into the surroundings. When the temperature is raised the rates of both the forward and reverse reactions are increased, but the rate of the forward reaction is increased by a higher factor.

If a carbonated drink is warmed and its temperature is increased, then the backward reaction for equilibrium 1 is favoured which is endothermic, namely, that heat is absorbed from the surroundings. This results in less carbon dioxide dissolving in the water and greater bubble production and consequently an increase in pH. When the temperature is lowered the rates of both the forward and reverse reactions are increased, but the rate of the backward reaction is increased by a higher factor.

Effect of adding sodium hydrogen carbonate

If a solution of sodium hydrogencarbonate is added to a carbonated drink then there will be an increase in the concentration of hydrogen carbonate ions. Le Chatelier's Principle predicts that equilibrium 3 will shift from right to left to remove it, favouring the backward reaction, which results in equilibrium 2 and then equilibrium 1 also shifting from right to left, resulting in increased formation of carbon dioxide and consequently decrease in pH.

If solid sodium hydrogencarbonate is added to the carbonated drink the chemical effect described above will take place, but the rate at which bubbles are produced is increased due to the introduction of nuclei, that is, sites for the formation of gas bubbles on the solid surface.

Effect of adding hydrochloric acid

Hydrochloric acid will supply hydrogen ions that will shift equilibria three, four and then two and one from right to left resulting in an increased rate of production of carbon dioxide bubbles.

Effect of stirring

When dissolved carbon dioxide escapes from solution and enters the gas phase, this can only occur at the interface between the solution and the atmosphere, that is, at the surface of the solution. Stirring may increase the diffusion of molecules towards the surface and vigorous stirring may also increase the surface area of the interface between the surface of the liquid and the atmosphere by creating bubbles of gas in the liquid. Hence, stirring may increase the rate at which carbon dioxide is lost but on its own would not affect the position of the equilibrium.

Sample Exploration Plan

Below is an incomplete outline plan describing how to address the Exploration criterion when designing a simple kinetics investigation. This practical is probably only suitable as a practice for the Individual Investigation. The author's comments are in italics.

Aim

Investigating the reaction between magnesium ribbon and hydrochloric acid.

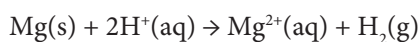
Research Question

The aim is to identify the relationship between the concentration of hydrochloric acid and the initial rate of reaction (at constant temperature) as measured by the reciprocal of the time taken for the reaction to go to completion

(Many other variables could be investigated, for example, temperature of the hydrochloric acid, solvent used for the acid, stirring and the surface area of magnesium, metal used, acid used).

Introduction and background

It is assumed that the reaction between magnesium and dilute aqueous hydrochloric acid can be described by the following ionic equation:



It is assumed that magnesium atoms do not directly react with the spectator chloride anions. The reaction is a redox reaction with the magnesium changing its oxidation number from 0 to +2 and the hydrogen from +1 to 0.

It is also assumed that the reaction is sufficiently dilute to ensure that the redox properties of hydrochloric acid are not observed.

It is also assumed that the magnesium is pure and no impurities are present causing release of gas.

Hypothesis

It is predicted that as the concentration of hydrochloric acid is increased the time taken for the reaction to go to completion will decrease. This means that the 'rate' (reciprocal of time) will increase. This can be explained by the increase in collision rate between hydrogen ions and magnesium atoms on the surface of the magnesium. A higher concentration of acid means a higher number of hydrogen ions per unit volume and hence a corresponding increase in the collision rate and hence reaction rate.

(Some simple diagrams illustrating collision theory may be a useful inclusion).

Risk Assessment

Safety glasses and disposable gloves are to be worn at all times. No naked flames will be allowed due to the release of hydrogen. Residual hydrochloric acid will be neutralised with sodium hydrogen carbonate before being flushed down the sink with copious amounts of water. Inhalation of acid spray is to be avoided by maximising the distance from the reacting chemicals. The laboratory must be well ventilated.



Classification of variables

Dependent variable: Time for reaction to go to completion

Processed variable: 'Rate' (reciprocal of time)

Independent variable: Concentration of hydrochloric acid

Controlled variables: Mass and purity of magnesium
Surface area of magnesium
Temperature of hydrochloric acid
Stirring (none)

It is important to control the temperature of the hydrochloric acid and the surface area of magnesium since both affect the rate of reaction. The reaction will be carried out in an air-conditioned laboratory.

(Stirring can be controlled and quantified by means of a magnetic stirrer; a thermostatted water bath is required for proper control of temperature).

Materials and apparatus

Magnesium ribbon (99% purity)

(Approximate dimensions: width 3mm, length 22 mm and thickness 0.25mm)

Fine sand paper

Metal rule (± 1 mm)

2.00 mol dm⁻³ aqueous hydrochloric acid

25 cm³ pipette 0.06 cm³ (Class B)

50 cm³ burette ± 0.1 cm³ (Class B)

250 cm³ glass conical flask

Electronic stopwatch (± 0.005 s)

Discussion about possible methods and methodologies

A simple and easily reproducible method is to use a fixed length of cleaned magnesium ribbon and time how long it takes for all the metal to react with the acid and form soluble magnesium chloride. The reciprocal of time can be taken as a measure of the rate. This is essentially a variant of the initial rate method.

Note that for this method to be valid, a large excess of acid must be used. Provided that a large excess of hydrochloric acid is used, by the time the magnesium strip has reacted the concentration of hydrochloric acid is still virtually the same as it was at the start, so the rate of reaction throughout the dissolving process only decreases slowly. Therefore, since rate is inversely proportional to time, a measure of the rate can be obtained from the reciprocal of time. It is assumed that the magnesium ribbon will have constant dimensions.

To determine how much of the chemicals I need for the apparatus I plan to use, I need to refer to the stoichiometric equation for the reaction: $\text{Mg (s)} + 2\text{HCl (aq)} \rightarrow \text{MgCl}_2 \text{ (aq)} + \text{H}_2 \text{ (g)}$

Hence one mole of magnesium reacts with two moles of hydrochloric acid to give one mole of hydrogen and one mole of magnesium chloride.

“To determine the amount of hydrochloric acid I need to know its volume and concentration. To estimate the length of magnesium ribbon I will need to weigh it (after cleaning it thoroughly) on an electronic balance. I will divide the mass by the molar mass of magnesium to obtain the amount in moles. I will then compare the amounts (in mol) of hydrochloric acid and of magnesium. I plan to use ten times as many moles of the acid relative to the magnesium to ensure that the acid is present in excess and the magnesium is the limiting reagent.”

(A clearly explained calculation of the amount (moles) of magnesium from the determined dimensions of the strip must be given.)

An alternative method is to collect and measure the volume of hydrogen produced over measured time intervals. This would allow me to plot the volume of product formed against time to and to determine the rate at selected times by finding the gradient of the graph at these times.

This could be done by collecting the gas in a sealed gas syringe in the apparatus shown below in *Figure 212*. However, this approach may not give such reliable results as the method chosen since it may be difficult to make the apparatus air tight, the gas syringe may stick.

(It is perfectly acceptable and indeed you are encouraged to explore alternative methods and justify why they were not employed).

A clear and reproducible method must also be given. Each experiment at one concentration of hydrochloric acid must be repeated before the acid concentration (independent variable) is changed.

(Blank results tables also need to be given and an outline of the data processing, which in this investigation would include averaging and calculating reciprocals. A sketch of the expected graphical relationship between the independent and dependent variable may also be helpful.)

Exercise 1

Refer to the setup in *Figure 213*. The electrodes are clean copper sheets. Try to generate a focused Research Question and identify and classify relevant variables.

Exercise 2

Suppose you are carrying out a simple investigation to verify Boyle's law: the relationship between the pressure and volume of a gas at constant temperature. Generate a table classifying the variables and stating a method for their measurement/control and a reason for the control of the controlled variables.

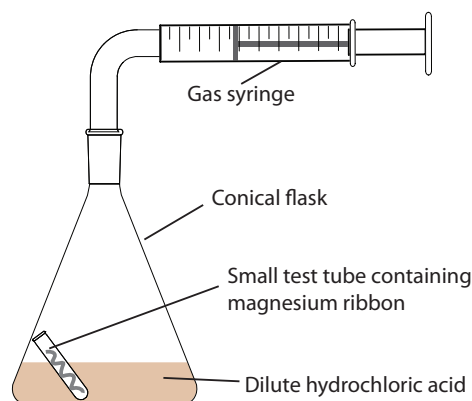


Figure 212 Using a syringe to collect gas

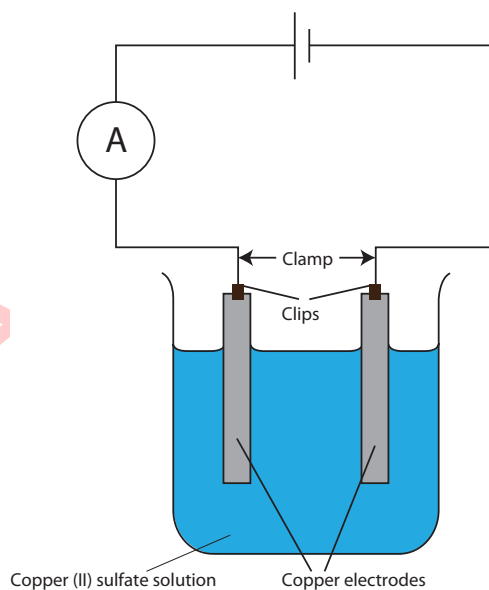


Figure 213 Electroplating apparatus

2.4 Use Of Apparatus And Instruments

It is important that you are able to effectively and safely handle chemicals and laboratory equipment, set up chemical apparatus properly, and safely and competently perform out a range of successful practical procedures. Your risk assessment and safety considerations during these your Individual Investigation and practice investigations will be assessed. An ability to be able to adapt to changing circumstances during an investigation is also critical.

You will also be assessed on your attention to environmental issues involved in your investigation. This could be demonstrated by wastage of materials or resources, for example, distilled water, and improper disposal of chemicals and waste during your practical. Safety issues should always be clearly addressed and chemical spillages should always be dealt with in the appropriate manner. Chemical apparatus and chemicals must also be stored away safely and effectively.

The correct use of common laboratory apparatus and the development of good techniques will allow you to score highly in the Exploration and the Analysis criteria. They will also help reduce random errors and prevent systematic errors.

Volumetric Glassware

Pipette

Always rinse the pipette with a small quantity of the solution to be measured before beginning the titration. Discard this rinse solution. Check that the tip of the pipette is not damaged before use. Ensure the tip of the pipette is placed well below the surface of the solution before using a pipette filler to draw the solution above the scratch mark. Wipe the outside of the pipette after filling it with solution.

Allow the excess solution to run out into a beaker until the meniscus is on the scratch mark. Check that no air bubbles are present, especially at the tip. Remove the pipette filler and allow the solution to drain naturally for thirty seconds before touching the tip to the inside of the flask for about three seconds. This allows the correct retention of the last drop of the solution by the pipette: do not attempt to remove it by blowing. Check that the pipette drains cleanly without leaving drops clinging to the side. If it does not, it will need a thorough cleaning with detergent.

Burette

Rinse this with the solution you are going to put in it. Make sure you rinse through the tap and jet. Discard the rinse solution. Fill the burette to above the 0.00 cm³ mark. Run out excess solution into a beaker to fill the tap and jet, check no air is trapped in the jet. Loss of bubbles will add to the measured volume even though no corresponding solution is delivered to the flask. There is no need to adjust the volume of solution to exactly 0.00 cm³ (but it may reduce the risk of arithmetical errors). Remember to remove the filter funnel used to help fill the burette.

Aqueous solutions of alkalis are generally not placed into burettes since they gradually absorb carbon dioxide from the atmosphere forming deposits of solid carbonates. The presence of alkali in a burette may therefore lead to the jet being blocked and can also lead to deposits of solid carbonates in the socket of the burette tap. This makes the tap difficult to clean and, additionally, the alkali could slightly dissolve the glass causing the ground glass of the stopcock and socket to fuse together.

Readings should always be taken with the eye level with the bottom of the meniscus. It also helps to use a 'burette reader' (see Figure 214): a piece of card behind the burette, the upper half being white and the lower half black (which is positioned just below the meniscus). A magnifying glass can also be used.

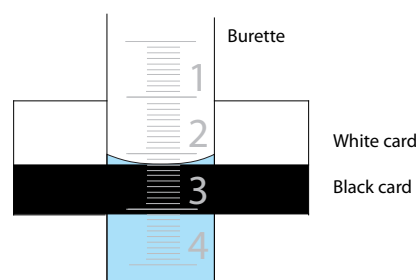


Figure 214 A burette reader

The Volumetric Flask

This should be rinsed with distilled water before use. If you are dissolving an accurately weighed solid sample you must ensure that all the sample is transferred to the flask. One way to do this is to transfer the solid from the weighing bottle to a clean beaker. Wash the weighing bottle at least three times with distilled water, adding the washings to the beaker. Now add enough water to dissolve the solid and warm the solution if necessary. Cool and transfer the solution to the volumetric flask using a small funnel. Wash the beaker and funnel several times with distilled water adding the washings to the volumetric flask. Now make up to the mark with distilled water. Add the water in drops when close to the mark. Stopper the flask and invert several times so the resulting air bubbles ensure thorough mixing of the solution.

Graduated glassware should never be heated, otherwise the glass expands and the scale or mark loses some accuracy. Ideally, the flask should be filled at 20 °C, or the temperature that is recommended by the manufacturer.

Weighing can be done by difference, for example,

Mass of weighing bottle and anhydrous sodium carbonate = 11.045 ± 0.001 g

Mass of weighing bottle and residual solid = 10.155 ± 0.001 g

Therefore, mass of anhydrous sodium carbonate used = 11.045 g – 10.155 g = 0.890 ± 0.002 g

Titration Flask

A conical/Erlenmeyer flask should be used for titrations as the narrow neck reduces the chance of losing any solution through splashing. This should be thoroughly rinsed with distilled water before use and between titrations. During a titration, rinse down splashes on the sides of the flask, with a little distilled water from a wash bottle.

During the actual titration, the conical/Erlenmeyer flask should be placed on a white ceramic tile and it should be held close to the burette to minimise losses by splashing. The contents of the flask should be continuously swirled to avoid any local build up of reagent added from the burette.

If a burette with a tap is being used, then the tap may be held in the left hand (if you are right handed) and the flask in the right hand. As the end-point is approached, transient or temporary colour changes occur in the flask that hint at the colour of the indicator at the end point.

The titration should be continued with the slow addition of the chemical from the burette one drop at a time until the end-point is reached, then:

1. The solution changes colour, if a two-colour indicator is used for example, methyl orange or bromothymol blue; or
2. The solution is decolourised or coloured if a one-colour indicator is used, for example phenolphthalein. The intensity of phenolphthalein is determined not only by the pH of the solution but also by the total amount of indicator (provided the pH is $pK_a \pm 1$).

If you are unsure whether the colour has changed, record the reading and add one or more drops of the reagent from the burette to see if there is a noticeable difference. The colour of the indicator before and at the end point should be recorded. The colour at the end point should be maintained for at least thirty seconds. A trial titration is often useful so that the end point can be approached rapidly and carefully in subsequent titrations.

A number of these titrations (see Table) should be performed until you obtain consistent results (i.e. ones which agree to within 0.1 cm^3 , since the titration volumes are taken from the difference of two readings each: hence add absolute uncertainties. The conical flask should be washed out thoroughly after each titration.

For example see *Figure 215*.

Titration Number	Titration Readings		
	TRIAL	1	2
Second Burette Reading / cm^3	(24.20)	48.10 ± 0.05	23.90 ± 0.05
First Burette Reading / cm^3	(0.00)	24.20 ± 0.05	0.00 ± 0.05
Titre Volume / cm^3	(24.20)	23.90 ± 0.10	23.90 ± 0.10

Figure 215 Sample titration data

The two accurate titrations are both $23.90 \pm 0.10 \text{ cm}^3$.

If, however, the spread of values that you are averaging is greater than 0.1 cm^3 then the systematic errors of your titration are greater than the random errors inherent in the experiment. This error could be estimated to be half the spread of the averaged values. Use whatever value is greater, but you do not need to add the errors.

Balances

The Use and Misuse of Balances

Weighing is one of the most fundamental procedures performed in the laboratory and can create all manner of problems during quantitative practical work if not carried out properly. The following list describes several weighing techniques and procedures that should be avoided and how they can lead to errors.

Weighing hot samples

This will generate an upward convection of warm air that will cause an upward force to be applied to the balance pan. The result will be an apparent mass that is less than the actual mass. In the case of a weighing, the final result will appear lower than the actual value.

Weighing materials that lose water rapidly or are extremely hygroscopic on an open balance pan or in an open vessel.

The loss or gain of water during weighing will give false low or high masses. Always weigh wet or hygroscopic materials in a closed container such as a covered weighing bottle.

Weighing objects that are too large for the balance pan or weighing off-centre.

This can cause instability and buoyancy effects resulting in variable and unpredictable results.

Weighing volatile liquids in an open vessel.

The weight will continually decrease due to loss of sample by evaporation. As a result the mass recorded will be greater than the true mass because of further evaporation between the time the weight is recorded and when the sample is transferred and diluted with a suitable solvent.

Sample is spilled on a balance pan.

If this is not noticed, then the amount of sample actually being used in the analysis will be less than the recorded mass. The recorded mass will be low. Always make sure the balance pan is clean before and after using a balance.

Forgetting to weigh the stopper

Consider taring (calibrating the balance to zero) a stoppered volumetric flask on a balance pan, removing the flask, transferring a liquid sample into the flask, and then reweighing the flask plus the sample without the stopper. This is easily recognised by an unexpectedly low or negative sample mass.

Open balance doors

Drafts and air currents will cause weighing uncertainties and inaccuracies in analytical results.

Excessive vibration

If a balance is not on a surface that is vibration free, then accurate balance readings will be impossible to record.

Weighing on a balance that is out of calibration, out of level or not properly damped

Weighing performed on a balance that is past its calibration due date cannot be considered reliable. Accuracy may also be affected when a balance is not level. Improper damping affects balance sensitivity because a balance that is underdamped will fluctuate to the point where a steady reading is difficult or impossible to obtain. Overdamping, on the other hand, will inhibit a balance from responding quickly enough to changes in weight as a sample is applied to the balance pan. The results may then lack accuracy.

Spectrophotometers and Colorimeters

These instruments are most sensitive if the light falling on the solution under investigation corresponds to the range of wavelengths actually absorbed by the substance. The difference between the solvent and the solution is then greater, and the absorbance reading higher. Errors due to wavelength drift and the finite bandwidth of wavelengths selected by the monochromator are minimised because the spectrum varies least with wavelength at the absorbance maximum.

It is therefore important to select the appropriate range of wavelengths. In colorimeters this is achieved by placing a colour filter in the path of the machine's light beam. The colour of the filter should be complementary to the colour of the solution under investigation. The colour wheel in *Figure 216* will help in choosing a suitable filter.

The scale of a spectrophotometer is shown in *Figure 217*. Note that the absorbance is a logarithmic scale and % transmittance is a linear scale. The absorbance reading shown is 0.234, but the last figure, 4, is an estimate.

The transmittance is 58.3%. Note that because the transmittance scale is smaller than the absorbance scale, at this point, there is more uncertainty in the last digit of transmittance.

The transmittance may be quoted as $58.3 \pm 0.2\%$.

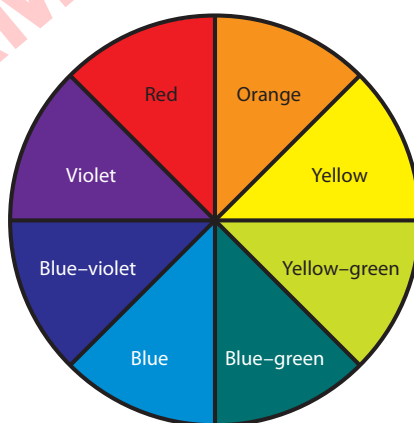


Figure 216 Colour wheel

For example, an orange filter should be chosen if an aqueous solution of copper(II) sulfate, which is blue in colour, is under investigation. If the solution is not strongly coloured a 'trial and error' process can be carried out. Try each filter to find out which one gives the maximum absorbance.

Since an instrument of this type may 'drift' (leading to random errors), it is important (time permitting) to re-check the zero reading with your 'blank' (usually distilled water) before each measurement is made.

Since the temperature of the cell compartment is generally several degrees higher than the surrounding room temperature, it is important to remove the cell containing the reaction mixture immediately after a measurement has been taken.

All vessels should be covered to protect them from dust, which scatters light and therefore makes it look like the absorbance of the sample has increased. Cuvettes should be handled by the sides that the light will not be passing through (usually opaque or ridged) to avoid placing fingerprints on the optical faces, which must be kept clean to avoid surface contamination, which also leads to scattering.

It is also important to place a cuvette in the machine as reproducibly as possible. A slight misplacement of the cuvette in its holder, or turning a flat cuvette round by one hundred and eighty degrees, or rotation of a circular cuvette, all lead to random errors in absorbance measurements.

These instruments are probably most accurate when measuring absorbances of around 0.5 to 1.0. The scale may tend to become slightly non-linear with absorbance in the region 1.5 to 2.0, when the intensity of the transmitted light is very low, because under these conditions the response of the photo-detectors may no longer be linear and stray light becomes significant. See *Figure 217*.

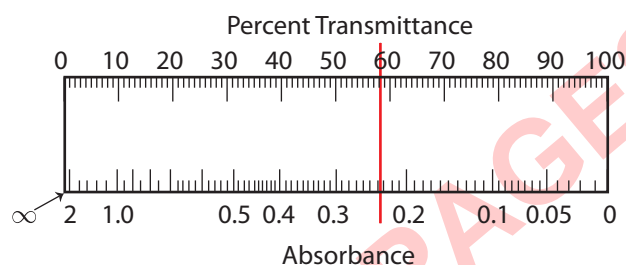


Figure 217 Scale of a spectrophotometer

Conversely, if too much light leaves the sample (low absorbance) because of its low concentration of solute, it is difficult for the machine to distinguish the transmittance of the sample from that of the reference.

pH Meter (glass electrode type)

Take care to clean the electrode thoroughly by rinsing it with distilled water from a wash bottle every time you change solutions. Do not leave the electrode un-immersed for longer than you need, or let the electrode dry out: salt deposits will form interfering with the electrode response.

Standardise the meter (see *Figure 215*) (as in the instructions or demonstrated by your IB Chemistry teacher) at a particular pH and temperature using the buffer(s) provided. The buffer solutions provided must be made up with care since the accuracy of your pH measurements will depend on the accuracy of the buffer solutions used in calibrating the probe.

The buffer provided should reflect the nature of solutions whose pH values you are going to measure. For example, if you are investigating acidic solutions then an acidic buffer solution (e.g., pH 4.0) should be provided. The measurements would be a little less accurate should a neutral (pH 7.0) or alkaline buffer solution (e.g., pH 9.0) be used. Check to ensure that the buffer solutions have not 'expired'.

Figure 218 shows a pH METER (manufactured by Hanna Instruments) (Photograph by Robert Balcer, formerly of Overseas Family School, Singapore)

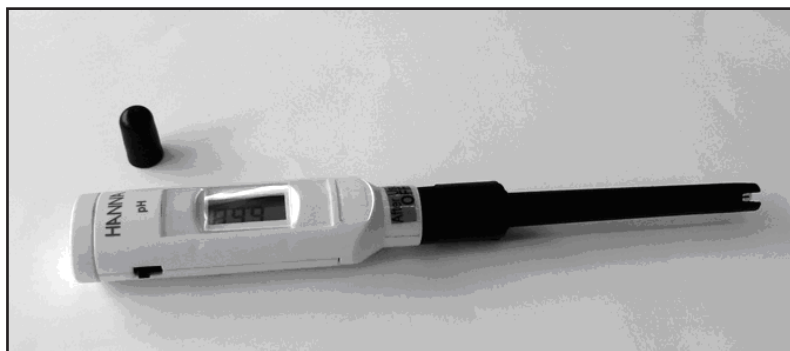


Figure 218 shows a pH meter

However, calibrating with two buffers allows the 'slope' of the pH meter to be set so that it reads correctly at all pH values. Attention to the following points will enable you to obtain the highest accuracy:

Do not touch the electrode or move about near it whilst a reading is taken. If you are using a magnetic stirrer then switch it off when you take the readings.

The electrode needs time to reach equilibrium in solution, so do not rush to record the measurement, but wait patiently until the meter reading becomes steady.

If the meter is not steady, try to establish whether, over a period of a minute or so, it is:

- approaching a steady reading exponentially.
- oscillating regularly about a mean value.
- drifting continuously in one direction.

If one of these patterns can be identified then you should be able to decide the best way to record accurate readings and an appropriate uncertainty for your particular meter in a consistent way. This might be to wait one minute before taking the reading, to average the high and low points of the oscillation, or to take the reading immediately.

pH meters actually measure $-\log_{10}$ [activity of hydrogen ions]. At high concentrations, ions interact with each other, so that their effective concentration is reduced. Consequently, pH meters tend to record higher values than you would expect for concentrated solutions.

It may be possible for you to connect your pH meter and probe to a personal computer to record a series of pH measurements over a period of time

Extracting and Studying Enzymes

In some of the Topics you are likely to perform some experiments involving plant or animal enzymes. The main features of such experiments are the difficulties associated with delicate and sensitive biological materials.

The enzymes may be supplied in a purified form or you may have to extract them from fresh plant materials. A common approach involves grinding up the materials in a pestle and mortar, with a little fine sand to break down the cellulose cell walls. For particularly tough plant tissues a food blender or food processor may be used for short periods of time.

Enzymes are very sensitive to changes in pH, the polarity of the solvent, the presence of dissolved salts and temperature. The crude (impure) extracts of enzyme are therefore placed into a buffer solution of specified pH. To minimise enzyme degradation the crude enzyme extract is kept cool at all times, from extraction, through filtration to final use.

Enzyme activities will often decrease as the enzyme solution stands, even if stored in a refrigerator, hence experiments involving enzymes must be performed relatively quickly on the same day.

Handling Gases

During your IB Chemistry Practical Programme you may have to collect gases for testing, or to measure the volumes released during kinetics or stoichiometry investigations.

Collecting Gases

The method used will depend on whether the gas is soluble in water. If it is soluble the gas will have to be collected directly in a gas jar (see *Figure 219*). If the gas is insoluble in water it can be collected over water in a test tube or measuring cylinder (see *Figure 220*).

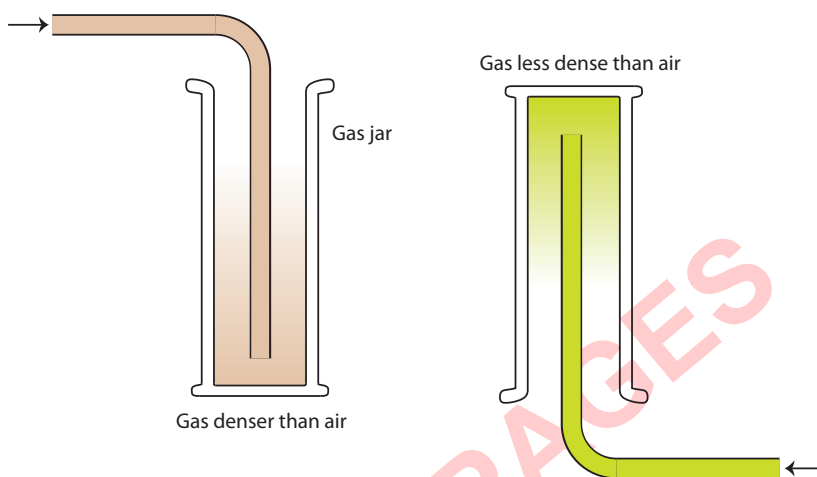


Figure 219 Collecting gases by displacement of air

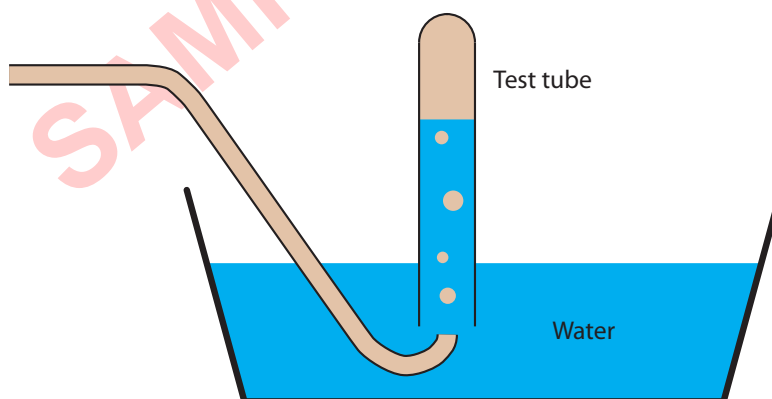


Figure 220 Collecting insoluble gases over water

Measuring Volumes of Gases

The gas can be collected over water in an inverted burette or in a gas syringe (see *Figures 221 (a) and (b)*). Before you commence any experimental work ensure that the system is airtight and check that the rubber or plastic tubing fits tightly. Do not clamp the syringe too tightly since this will prevent the gas from pushing the inner section freely.

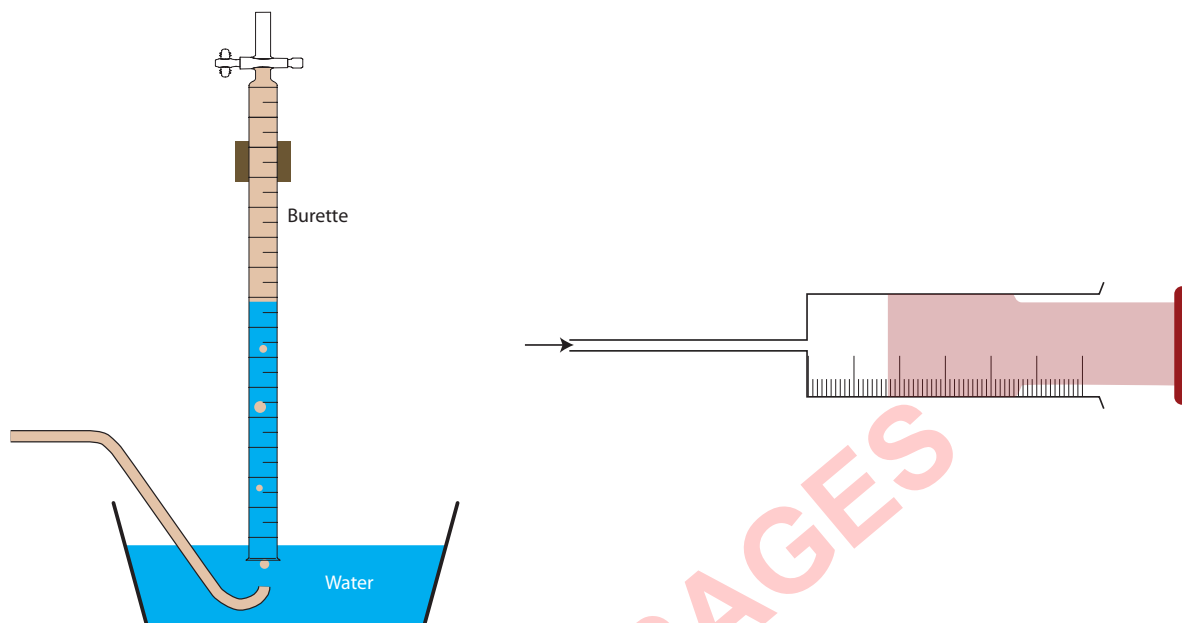


Figure 221 (a) and (b) Techniques for collecting and measuring volumes of gases

Washing and drying gases

A gas washing bottle can be used. For example, if a gas is contaminated by acid it can be 'washed' or 'scrubbed' by bubbling it through aqueous sodium hydroxide which neutralises the acid. The commonest way of drying gases is to bubble through them concentrated sulfuric acid which removes the water vapour from the gas (*Figure 222*). You must consult your IB Chemistry teacher or instructor before using concentrated sulfuric acid.

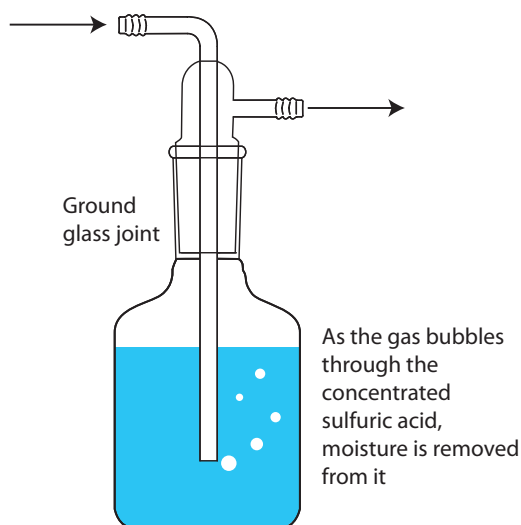


Figure 222 Drying gases

Constructing Simple Electrochemical Cells

An electrochemical or galvanic cell is set up so that the two redox reactions (described by half equations) are separated in space. Each electrode is immersed in aqueous solution in a small beaker. The two solutions of metal ions are linked by a salt bridge which allows the movement of ions in both directions and the electrodes are connected via wires forming an external circuit, around which electrons flow.

To determine the maximum potential difference or voltage that can be generated between the electrodes, a high-resistance voltmeter is connected to the external circuit. A high-resistance voltmeter is required so that very little current is drawn from the circuit.

Several precautions are needed to ensure accurate values of cell potentials are obtained:

- The surface of the electrodes must be thoroughly cleaned with carborundum (silicon carbide) or ‘wet-and-dry’ paper to ensure any unreactive metal oxide layers are removed. The electrodes should then be washed with distilled water and dried. To ensure they are grease-free they should be rubbed with cotton soaked in propanone (acetone) before a final rinsing. The cleaned electrodes should now only be handled by tweezers at the edges. This cleaning process is particularly important if relatively reactive metals like magnesium are used. Magnesium will not be able to reach the thermodynamic equilibrium potentials because of the competing hydrogen evolution reaction that takes place from the reduction of water.
- A good electrical contact is needed between the electrodes and the external circuit. Clean rust-free crocodile clips must be used to minimise the resistance introduced into the circuit. The circuit should then be completed with a salt bridge—this normally takes the form of a piece of filter paper dipped in saturated aqueous potassium nitrate solution. Potassium chloride can also be used, unless silver ions are present in one of the half cells: when insoluble silver chloride would be precipitated. Salt bridges should be left in the solutions for the minimum time in order to reduce contamination. A high-resistance voltmeter should then be connected between the electrodes and the potential difference or voltage measured and recorded. If the voltmeter is a digital voltmeter and gives a negative reading, or if with an analogue meter the needle moves below zero, then the voltmeter needs to be connected the other way round to get a positive reading.

Measuring Molar Masses of Gases

The simplest method for determining the molar mass of a gas is via direct weighing: a flask is weighed empty, that is, full of air; the flask is then weighed full of gas and then finally the flask is weighed full of water. An appropriate sized measuring cylinder is then used to determine the volume of water, that is, the volume of the flask.

It is not a good approach to measure the volume of the flask at the beginning of the experiment since the drying of the flask will be a time consuming process. If the flask is not dried properly and is damp, then the gas used may dissolve in the remaining water droplets, thus leading to an error in the measurement of the volume of gas contained in the flask.

Another error inherent in this approach is that some water remains in the flask when its volume is determined. This is unavoidable, but introduces a systematic error into the experiment.

Some gases, especially hydrogen chloride, sulfur dioxide and ammonia are very soluble in water. The molar masses of these gases can be determined using an experimental approach similar to the one just described. A flask is weighed empty (that is, full of air), then the gas is passed into it for a short period of time before the tubes are then sealed and the flask and contents reweighed.

One of the tubes leading into the flask is opened under water and owing to the solubility of the gas, the pressure inside the flask is reduced and air pressure pushes water into it, but once all the gas has dissolved, no more water enters the flask. The amount of water that did enter is measured by pouring it into a measuring cylinder of an appropriate size. Finally, the volume of the flask is found by filling it completely with water.

It is common for this type of experiment to overestimate the molar mass of the gas under investigation. This occurs, in part, because not all the water is transferred from the flask to the measuring cylinder – a so-called mechanical loss. This underestimates the volume of the gas, thus leading to a higher calculated molar mass.

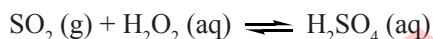
It is important to dry the flask and tubing thoroughly before they are used in the experiment, otherwise the gas will dissolve and react with the moisture. This will increase the mass of gas in the apparatus, but it will add negligible volume. Therefore the molar mass of the gas will be overestimated. Another source of error is the non-ideal behaviour of these gases.

Measuring Equilibrium Constants

To measure a chemical equilibrium constant, K_c the following issues need to be taken into account:

- The reactants and products of the reaction must have actually reached chemical equilibrium. You can test if this has happened by removing small samples out of the reaction mixture at different times and analysing them. When identical concentrations are obtained for successive analyses, then you can assume that equilibrium has been achieved.
- It may also be possible to analyse the system without physically removing samples. For example, depending on the reaction under study, a colorimeter may be used.
- Equilibrium constants should only be measured for systems where the concentrations of the chemical species are relatively low. At high concentrations interactions occur between the particles and substances in a mixture may behave as if their concentration were less than its real value due to molecular or ionic interactions. Hence, you are not justified in calculating experimental equilibrium constants to more than two significant figures.
- The temperature at which the measurements are performed must be known and kept constant. Equilibrium constants vary with temperature and are quoted for specific temperatures. This is usually achieved by performing the reactions in a water bath whose temperature is kept constant using a thermostat.
- The concentrations for gases must be found. The most common approach to measure concentration of water soluble gases is by performing an acid-base or redox titration. The reaction mixture should be quenched in cold water to slow the reaction down. The solutions of the gases are then analysed by a direct titration or by a back titration. Datalogging probes can also be employed, for example, a dissolved oxygen probe.

For example, the quantity of sulfur dioxide can be determined by reacting it with hydrogen peroxide and titrating the sulfuric acid formed with standardised alkali.



Ammonia gas can be reacted with an excess of dilute hydrochloric acid. The resulting solution can be made alkaline, heated and the ammonia liberated is dissolved in a large excess of hydrochloric acid. The acid is then back titrated with standardised sodium hydroxide.

Common Organic Chemistry Practical Techniques

Glassware with interchangeable ground glass joints (refluxing and distillation)

The joints should be greased only lightly: excessive grease is difficult to remove and leaves the glass looking dirty and may contaminate the product. A suitable building point for your apparatus is normally the reaction flask. Try to use the minimum number of clamp stands and bosses to hold your apparatus in place. A Liebig condenser is best secured by a clamp to the same retort stand as that holding the flask. This will require the use of a variable boss which can be set at an appropriate angle. Such an arrangement will avoid the condenser separating slightly from the rest of the refluxing or distillation apparatus. You will also find it easier to move your apparatus around if only one retort stand is used.

Refluxing

Many organic reactions are relatively slow and their reaction mixtures need to be kept hot for significant lengths of time. In addition, many of the organic liquids used as either reactants or solvents are volatile, flammable and toxic. Some organic reactions may also produce volatile liquids or gases.

All of these problems can be overcome using a technique called refluxing (see *Figure 223*). It involves fitting a Liebig condenser to the top of the reaction flask. Cold water is then passed through the outer jacket of the condenser. When the contents of the flask are heated, the organic liquid will boil and the vapour will rise up inside the condenser. There it meets the cold walls of the condenser where it is cooled below its boiling point and condenses back to a liquid. This then drips down the walls of the condenser and is returned by gravity to the reaction mixture in the flask.

To ensure smooth boiling anti bumping granules—in the form of very fine pure sand—are placed into the flask prior to refluxing. Often the heating is carried out in a water bath or a steam bath, rather than heating directly with a Bunsen burner. This is done if any of the organic products or reactants are especially volatile and flammable, or if the reaction mixture must not be allowed to rise above 100 °C.

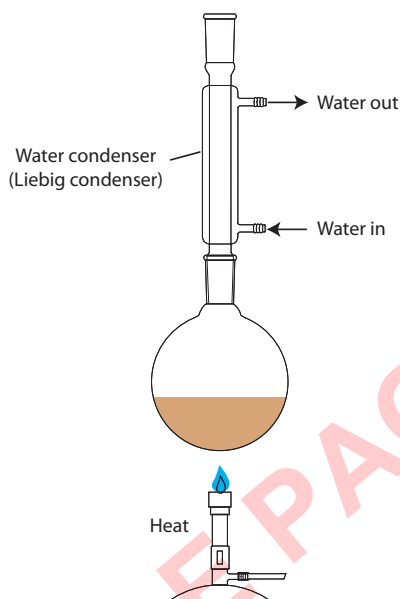


Figure 223 Heating under reflux

Recrystallisation

Recrystallisation is a valuable technique for purifying the impure solid product obtained from a reaction, typically an organic reaction. The substance to be purified is dissolved in a solvent that dissolves more solute when it is hot than when it is cold. A suitable solvent can be found by a process of 'trial-and-error' or by consulting an appropriate organic text. For many organic solids ethanol is used.

The aim is to use just enough solvent to dissolve the solid when the mixture is hot. This will enable the maximum amount to crystallise on cooling (the use of ice may be necessary). Some solids with low melting points may form an oil. If this occurs, you will need to add more solvent until the oil has been dissolved. Check that the solution does not contain any insoluble solid impurities, for examples particles of carbon (frequently formed during organic reactions by a so-called 'charring process'), if it does it will need filtering through a pre-heated funnel. This filtration can be carried out using one of two methods: a filter funnel, conical flask and 'fluted' filter paper or a Büchner flask and funnel. The fluted filter paper increases the surface area for filtration so that filtration is rapid. If it is not rapid then the solvent will cool down and the product may crystallise out in the filter paper.

Leave the hot solution to cool slowly and undisturbed. Occasionally supercooling occurs and no crystals form; the solution is said to be supersaturated. If this occurs shaking the flask may be all that is required to start rapid crystallisation. Alternatively, scratching the side with a glass rod (to release tiny crystals of glass) or, if available, a small seed crystal of the pure solid, may initiate crystallisation.

When the pure product has crystallised out, leaving soluble impurities in solution, filter off the crystals using a clean, dry Büchner funnel and flask. Wash the product with a minimum of cold solvent to remove any solution adhering to it, and then dry by drawing air through the crystals to evaporate most of the solvent and finally in a dessicator.

Determination of boiling point

There are two methods which can be used to measure the boiling point of a liquid. The first involves simple distillation and a relatively large volume of the liquid (see Figure 224). A flask is half-filled with the organic liquid and a few anti-bumping granules to ensure the liquid does not 'bump' and boil explosively. The apparatus for simple distillation is then assembled and the bulb of the thermometer placed at the entrance to the condenser. The boiling point of the liquid is the steady temperature shown as the liquid distils over.

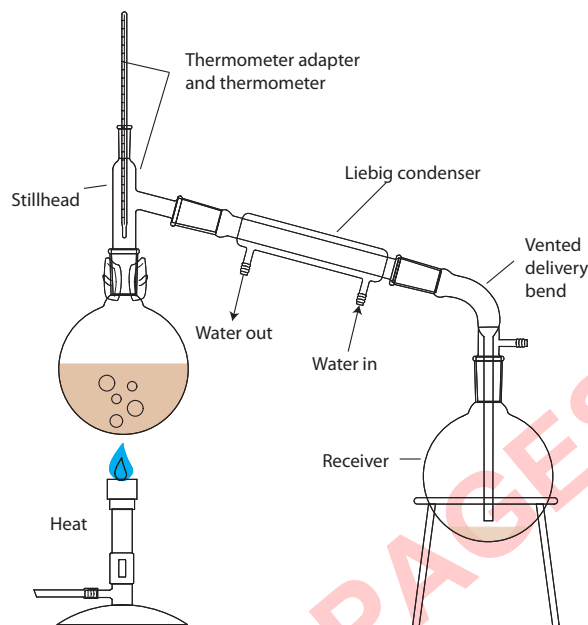


Figure 224 Simple distillation to determine the boiling point of an organic liquid

The second method of determining boiling points is known as the *Siwoloboff* method (Figure 225) and is applicable to both pure liquids and mixtures of liquids (they would be separated by distillation). It also can be used with relatively small volumes of liquid.

The technique is based upon the principle that the boiling point of a liquid is temperature at which the vapour pressure of the liquid is equal to atmospheric pressure. A small volume of the organic liquid is placed into a small ignition tube into which is placed a capillary tube, sealed at one end, open end down. The tube is attached to a thermometer and placed in a beaker filled with a high boiling point oil or water. The oil or water are heated very slowly and stirring is carried out to ensure the heat is evenly distributed.

Heating is stopped when a rapid and continuous stream of bubbles emerges from the capillary tube. The temperature is noted when the bubbling has stopped and the organic liquid appears about to be sucked up into the capillary.

This is the boiling point of the liquid. This is because at the boiling point the pressure of the vapour of the liquid in the capillary tube is just overcome by the atmospheric pressure (one atmosphere), and the liquid is pushed back into the capillary tube.

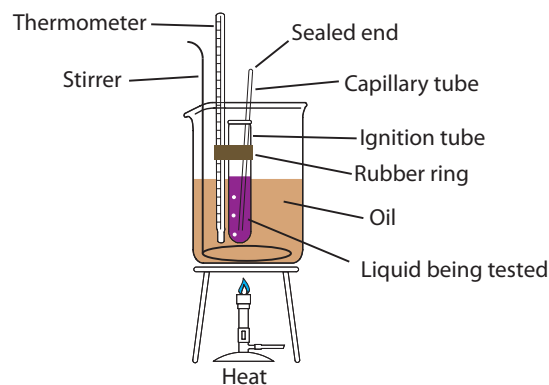


Figure 225 The Siwoloboff method to determine the boiling point of an organic liquid

Büchner or Suction Filtration

To set up the funnel for filtration, a filter paper with a similar diameter to the flat bottom of the funnel is needed. If necessary, but less preferably, a large filter paper can be cut to size. The filter paper is then moistened with a few drops of the solvent used in the solution to be filtered. The flask is connected to a pump and the funnel placed in the rubber cone. It may be necessary to press down on the funnel to help it seal with the rubber. It also helps to clamp the Büchner flask. If the filter paper tears under the pressure developed by the pump, then place two filter papers on top of each other. The mixture to be filtered is then directed on to the centre of the filter paper by slowly pouring it down a glass rod, whilst suction is applied by the pump. Before doing this swirl the mixture so that crystals are carried into the funnel. It may be necessary to scrape out the remaining crystals into the Büchner funnel using a glass rod. Alternatively, some of the filtrate may be returned to the flask containing the crystals to help wash them all out.

Determination of a melting point

The melting point of an organic solid will give some indication of the purity of the substance. Pure solids have 'sharp' melting points and melt over a narrow range of temperatures. The melting point of a solid can also help determine the identity of an organic substance, either directly, or indirectly, through the melting point of a derivative

The sample for the melting point determination should be dry and crystalline. It should then be ground to a fine powder using a clean, dry pestle and mortar. Some of the sample should be introduced into the melting point tube. You need sufficient sample to view clearly. Using the ridged edge of a coin may help to vibrate the melting point tube and allow the solid to fall to the bottom of the tube.

The sample is observed closely and the temperature over which melting starts and ends noted. Repeat the melting point determination with a fresh sample and approach the melting point with a very slow increase in temperature. If the substance is recrystallised again, it is likely to have a slightly higher, sharper melting point. This is because impurities have the effect of lowering the melting point (by reducing the intermolecular forces within the lattice) and making it less sharp, that is, broader.

The simplest form of melting point apparatus are boiling or Thiele tubes filled with a high boiling point oil (see Figure 226). The capillary tube with its sample is attached to a suitable thermometer. The oil is then heated and the crystals carefully observed until melting begins. The temperatures over which melting occurs is recorded.

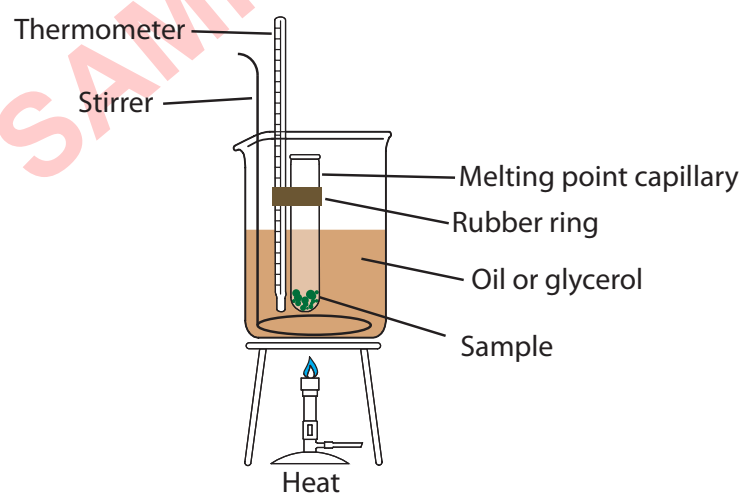


Figure 226 Simple melting point apparatus

Melting point determinations are best performed in an electrical melting point apparatus. The melting point tube and thermometer are placed into holes located in a large block of high resistance metal which is heated electrically. The crystals are observed through a magnifying glass and the temperature of the block is raised until the crystals begin to melt.

Drying liquids

A liquid organic product may require drying before a final separation by distillation. A small quantity of the drying agent (typically anhydrous calcium chloride or anhydrous magnesium, sodium or calcium sulfates) is added to the solution in a suitable stoppered flask. The mixture is swirled and allowed to stand before filtering, preferably using a small plug of wool to remove the excess drying agent.

Use of a separating funnel

Preparing a pure organic liquid involves several stages. After refluxing and distillation, the product will be present in a mixture which may contain water, unreacted reagents and a variety of side products. Further purification of organic liquids often involves the use of an immiscible solvent in which the product is more soluble than the impurities and this can be separated off using a separating funnel.

Ensure that that the ground glass joints are lightly greased and the tap of the separating funnel is securely closed. The mixture of solvents is placed in the separating funnel—ensure the tap is not leaking. The funnel is stoppered and the stopper held firmly in place with a forefinger and the funnel inverted (turned upside down). Any pressure build-up may be released through the tap while the funnel is inverted. The separating funnel is now placed in a clamp stand and the contents allowed to settle, a process which may take several minutes. Remove the stopper and drain the lower layer (the one with the lower density) into a clean flask. Close the tap so that the meniscus between the two layers is caught in the tap. If the upper layer is required this is poured out of the top of the funnel. This avoids contamination with the last trace of lower liquid in the tap.

Distillation

Simple distillation is frequently used during organic synthesis to separate the desired organic product from a reaction mixture that may contain less volatile liquids, or occasionally organic solids or involatile inorganic solids (for example sodium bromide used to prepare hydrogen bromide *in situ*).

The technique, which employs the apparatus previously shown in *Figure 226*, is based on differences in boiling points and is most efficient when there is a relatively large difference in boiling point between the components of the reaction mixture.

The thermometer bulb is placed in the side arm adaptor so the bulb is located at the entrance to the condenser. It records the temperature of the vapour as it 'distills over' into the condenser, this is the boiling point of the distillate.

The heating of the reaction flask is adjusted so that the distillate is being collected at a steady slow rate. The temperature will remain constant while a particular product is distilling over. Liquids with higher boiling points and involatile solids remain in the flask. To promote smooth boiling some anti-bumping granules should be placed in the flask with the liquid.

The process of separating of two or more volatile liquids is improved if fractional distillation (see *Figure 227*) is employed. In this technique a long column filled with inert glass beads is placed between the flask and the side arm adaptor.

Before you perform a distillation you need to have an idea of the fractions that you are likely to obtain, for example, solvent, pure product, starting reagent etc. and you should, if possible, know their boiling points. You should have several labelled flasks ready to receive these fractions. You will notice that the temperature remains steady while one product is distilling over.

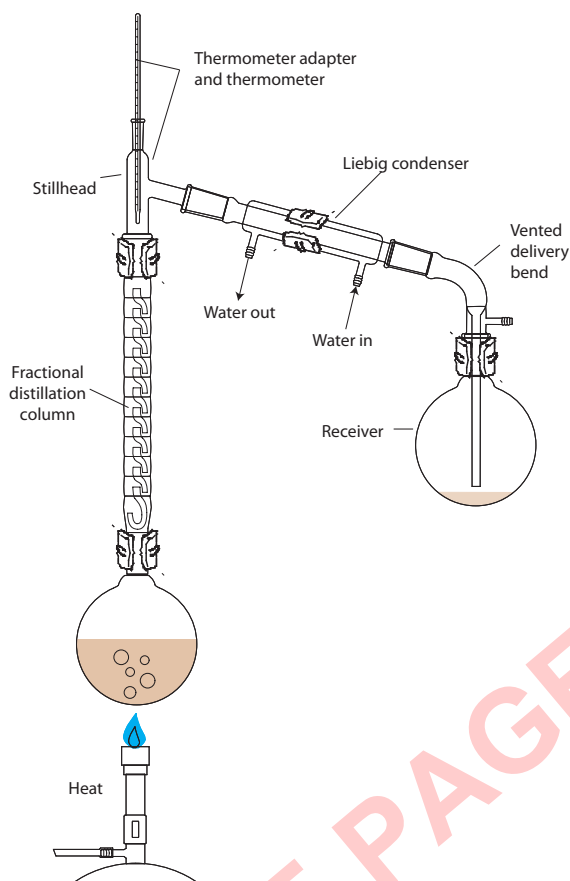


Figure 227 Apparatus for fractional distillation

Calorimetry

Calorimetry is the name given to the experimental technique used to determine the heat energy absorbed or released during a chemical reaction.

Calorimetry experiments can be performed using a polystyrene cup and a thermometer. This simple apparatus can be used to investigate enthalpy changes for a wide variety of reactions involving aqueous solutions, for example, enthalpies of replacement and neutralisation.

The heat energy released when a fuel burns, for example, an alcohol, can also be determined using a similar procedure. A 'spirit' burner containing a liquid fuel is placed under the copper can (which acts a calorimeter) containing a known volume of water. Figure 228 shows a simple apparatus for measuring the enthalpy change of combustion of a liquid fuel.

The initial temperature, the mass of the water and the initial mass of the spirit burner are measured before the start. The wick is then lit and the heat produced as the fuel burns is used to heat the water in the copper can. After a suitable period of time, the flame is extinguished, the lamp reweighed and the final temperature of the water recorded. However, this simple apparatus suffers from significant heat losses.

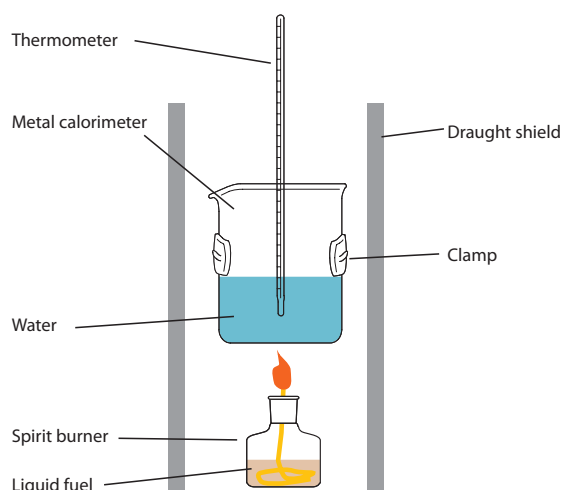


Figure 228 A simple calorimeter

The apparatus in *Figure 229* is designed to overcome some of the problems associated with heat losses and will therefore allow more accurate values to be obtained for enthalpies of combustion. The spirit burner is surrounded by water jacket, which helps to prevent draughts and ensures a more efficient transfer of heat from the flame to the water. The warm gases produced during combustion, namely, steam and carbon dioxide, are drawn through the apparatus using a water pump, and heat energy is transferred to the water via a copper coil.

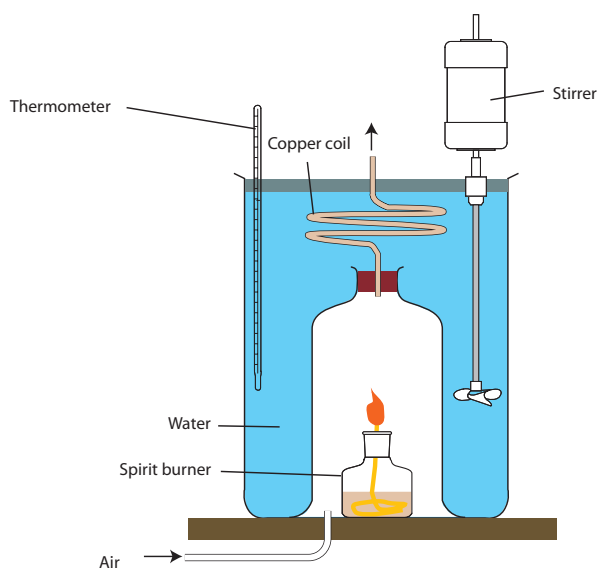


Figure 229 A (flame) combustion calorimeter

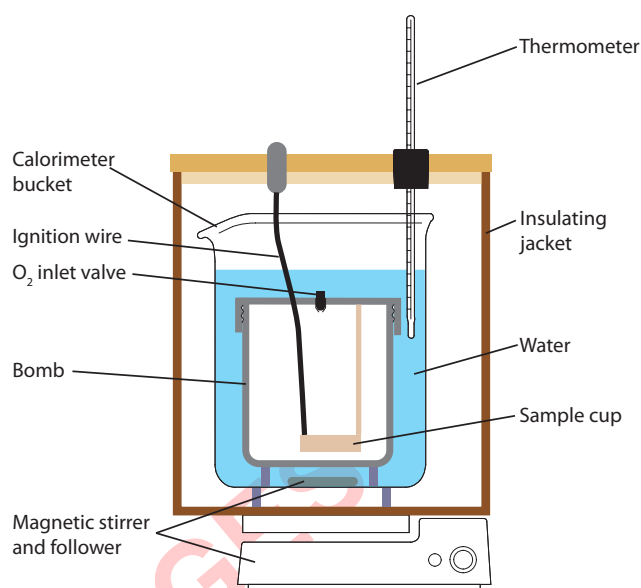


Figure 230 The bomb calorimeter

However, some heat energy is used to warm the glass and copper from which the calorimeter is constructed. The apparatus is therefore normally 'calibrated', by burning a fuel of known enthalpy of combustion over the same temperature range as used for the unknown fuel.

One drawback with this calorimeter is that some of the fuel may undergo incomplete combustion, resulting in an error. The most accurate measurements of enthalpies of combustion are obtained by the use of a bomb calorimeter (see *Figure 230*).

A weighed sample of the substance to be burnt is placed in a steel 'bomb' capable of withstanding large increases in pressure. The bomb is filled with oxygen, sealed and surrounded by water. After the initial temperature of the water has been recorded, the contents of the bomb are ignited electrically. The heat produced during the combustion reaction is transferred to the water surrounding the bomb and the final temperature recorded. Calibration is required, using a known amount of energy (usually found from the electrical energy required to give a similar temperature rise) to determine the heat capacity of the calorimeter. This gives the internal energy change (that is the heat change at constant volume) rather than the enthalpy change (which refers to constant pressure), though one can be calculated from the other.

Experiments involving Electricity

In investigations involving electrolysis or electrochemical cells you will need to connect circuits using a circuit diagram or you may have to construct your own circuits if the investigation involves the Exploration criterion.

Connecting an Ammeter and a Voltmeter

An ammeter is connected in series with the device, whilst the voltmeter is connected in parallel. The terminals of the ammeter and the voltmeter have a polarity. They need to be connected to the right poles of the power source. The red terminal is positive while the black terminal is negative. To work properly, the positive terminal of the ammeter or of the voltmeter must be connected to the positive side of the power source. Refer to *Figure 231*.

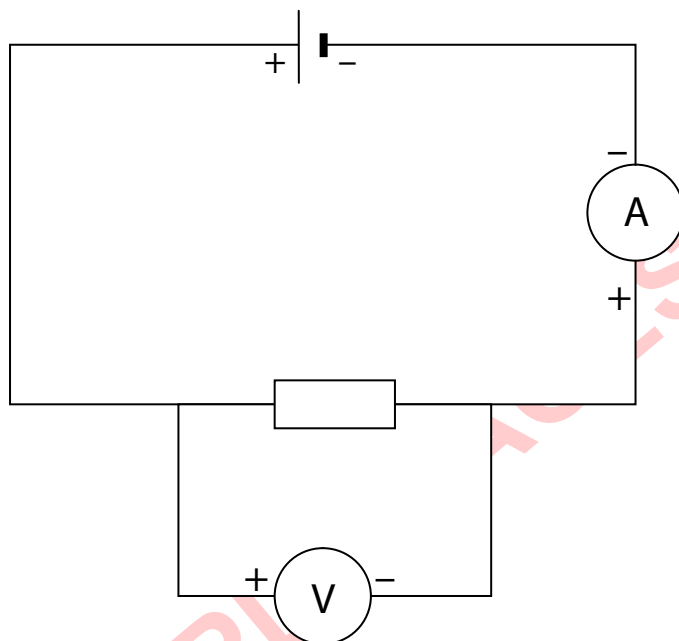


Figure 231 Circuit diagram showing the correct connection of a voltmeter and an ammeter

Listed in Figure 232 is a summary of what you need to do to score well in the Exploration criterion.

Assessment criteria	Evidence required	What you must do
Identifies a topic, a relevant and fully focused Research Question is clearly described. Relevant and appropriate background information is provided to increase the understanding of the context of the investigation	A topic, a relevant and fully focused Research Question are described	Identify the topic and state the Research Question, for example: The investigation will determine how the 'independent variable' affects the 'dependent variable'. The following will be kept constant: controlled variable 1, controlled variable 2,... and the method for measuring the dependent variable.
	Relevant and appropriate chemical background is included	Give relevant background information including a summary of the chemical literature and a testable hypothesis and perhaps reference to a relevant chemical model or chemical theory and identifies the main chemical processes involved and the likely causes at the molecular, atomic or ionic level. Makes quantitative predictions in words and in the form of graph. (<i>Where appropriate</i>)
	State the relevant variables explicitly	Classifies and tabulates key variables. <ul style="list-style-type: none"> • Independent variable (one only) • Dependent variable (one only) • Processed variables (one or more) • Controlled variables (typically more than one) • Identify variables over which little control can be exerted
Designs a methodology that allows relevant, reliable and sufficient data to be collected. Shows full awareness of safety, ethical and environmental issues	Appropriate choice of chemicals, materials and apparatus	List of all chemicals (physical states, purity, volumes and concentrations) (where appropriate) and apparatus and instrumentation (state manufacturer (where appropriate)) with specifications including precision/random uncertainty. A labelled and cross-sectional diagram of any set-up of apparatus with a justification and explanation for the methodology.
	Effective control and manipulation of variables	A clear description of the method which <ul style="list-style-type: none"> • describes how the independent variable is to be varied and measured accurately • describes how the dependent variable is measured • includes a logical sequence of steps to be taken and their rationale • includes details of any modification or adaptations of standard methods and justification for their use • includes a clear account of how and why the controlled variables are kept constant. • includes a statement of how the plan will produce relevant, reliable and sufficient results. • includes a statement of how the plan will produce accurate and precise results. • draws up blank results tables and graph axes and describes how the raw data will be processed • includes where appropriate, an explanation of the chemical and physical principles behind your plan. • includes control experiments should be described (if relevant) • includes a detailed statement of how the plan ensures an ethical and safe investigation (risk assessment) that minimises the impact on the environment. • describes safety precautions taken to keep risks to a minimum • describes how chemicals are to be disposed of and stored
	Appropriate number and range of readings to be taken	State you will take measurements for at least five values of the independent variable. Also consider what the reading at zero will be. State the range of values for the independent variable i.e. the lowest and highest values and the size of the increment. State the number of repetitions for each value of the independent variable.

Figure 232 Summary of the Exploration criterion