Your attention is drawn to the Risk Assessment section on page 1 of this booklet, and to the hazards indicated in Appendices 1 and 2. While every effort has been made to ensure that appropriate safety indications are given, CIE accepts no responsibility for the safety of these experiments and it is the responsibility of the teacher to carry out a full risk assessment for each experiment undertaken, in accordance with local rules and regulations. Hazard data sheets should be available from your suppliers.



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#### Introduction

It is expected that this booklet will be used as a follow-on from, or in parallel to, "Teaching AS Practical Skills" and therefore a full introduction is not included. It is recommended that the introductory material in "Teaching AS Practical Skills" is read before starting a course based on this booklet.

This booklet aims to help you design a well-structured scheme of practical work in order to develop your students' practical skills and their understanding of theory, as well as preparing them for the exam. The practical skills of planning, analysis and evaluation are assessed as A2 and this booklet is designed to show how these skills can be developed and honed through hands-on practical work much more effectively than through a theoretical treatment of the skills. The skills learned through the experiments suggested in this booklet will provide a good foundation for those wishing to pursue science further as well as for those entering employment.

#### Risk assessment

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.

Hands-on practical work can be carried out safely in schools. However, to ensure that it is safe, you must identify the hazards and reduce any associated risks to insignificant levels by adopting suitable control measures. You should carry out these risk assessments for all the activities involved in running practical science classes, including storage of materials, preparatory work undertaken by the teacher and any technical support staff, and practical activities carried out in the classroom, whether demonstrations by the teacher or practical activities undertaken by the students. Such risk assessments should also be carried out in accordance with the health and safety legislation of the country in which you are working.

Risk assessment involves answering two basic questions:

- 1 How likely is it that something will go wrong? For example, students using a double-sided razor blade to cut up carrots are quite likely to cut themselves.
- 2 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, burning many students and setting the laboratory ceiling on fire (this scenario is based on a real accident).

Once you have the answers to these questions, it is possible to plan the practical activity to minimise the risk of an accident occurring and, if it does, to minimise its possible severity. In our first example, this could include cutting up the carrot before giving it to young students or providing older students with an appropriate sharp knife rather than a razor blade; in the second, it could include bringing only the amount of magnesium powder required for the activity into the laboratory.

The likelihood that something will go wrong depends on who is carrying out the activity and what sort of training and experience they have had. Obviously you would not ask 11-year-old students to heat concentrated sulphuric acid with sodium bromide or to transfer *Bacillus subtilis* cultures from one Petri dish to another, simply because their inexperience and lack of practical skills would make a serious accident all too likely. However, by the time they reach post-16, they should have acquired the skills and maturity to carry such activities out safely.

Decisions need to be made as to whether an activity should only be carried out as a teacher demonstration or whether it could be performed by students. Clearly, some experiments should normally only be done as a teacher demonstration or by older students. Well-motivated and able students may be able to carry out such an experiment at a younger age,



but any deviation from the model risk assessment needs to be discussed and a written justification must be prepared beforehand.

There are some activities that are intrinsically dangerous and, if included in the suggested procedure, should always be changed to include safer modes of practice. For example, there are **no** circumstances under which mouth pipetting is acceptable – pipette fillers of some sort should **always** be used.

Teachers tend to think of eye protection as the main control measure for preventing injury. In fact, personal protective equipment, such as goggles or safety spectacles, is meant to protect from the unexpected. If you expect a problem, more stringent controls are needed. 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 (more than one is usually needed, to protect both teacher and students).

The importance of using the lowest possible concentrations is not always appreciated, but the following examples, showing the hazard classification of a range of common solutions, should make the point.

ammonia (aqueous)	irritant if ≥ 3 mol dm <sup>-3</sup>	corrosive if ≥ 6 mol dm <sup>-3</sup>
sodium hydroxide	irritant if $\geq 0.05 \text{ mol dm}^{-3}$	corrosive if ≥ 0.5 mol dm <sup>-3</sup>
hydrochloric acid	irritant if ≥ 2 mol dm <sup>-3</sup>	corrosive if ≥ 6.5 mol dm <sup>-3</sup>
nitric acid	irritant if ≥ 0.1 mol dm <sup>-3</sup>	corrosive if ≥ 0.5 mol dm <sup>-3</sup>
sulphuric acid	irritant if ≥ 0.5 mol dm <sup>-3</sup>	corrosive if ≥ 1.5 mol dm <sup>-3</sup>
barium chloride	harmful if $\geq 0.02 \text{ mol dm}^{-3}$	toxic if $\geq 0.2 \text{ mol dm}^{-3}$ (or if solid)

Reference to the above table shows, therefore, that if sodium hydroxide is in common use, it should be more dilute than 0.5 mol dm<sup>-3</sup>. Using more concentrated solutions requires measures to be taken to reduce the potential risk.

Your risk analysis should consider the hazards associated with the materials you propose to use.

#### **Eye protection**

Clearly students will need to wear eye protection. Undoubtedly, chemical splash goggles give the best protection but students are often reluctant to wear goggles. Safety spectacles give less protection, but may be adequate if nothing classed as corrosive or toxic is in use.

Your risk assessment should not be restricted simply to the materials, procedures and equipment that will be used, but should have a wider remit that covers the time from when the students enter the room until they leave it.

Practical science can be - and should be - fun. It must also be safe. The two are not incompatible.

Further relevant information on health and safety can be obtained from the following publications:

Safeguards in the School Laboratory, 10th edition, ASE, 1996 Topics in Safety, 2nd edition, ASE, 1988 Hazards, CLEAPSS, 1998 (or 1995) Laboratory Handbook, CLEAPSS, 1997 Safety in Science Education, DfEE, HMSO, 1996 Hazardous Chemicals Manual, SSERC2, 1997



#### A2 skills

#### Links to the AS syllabus

In many different activities, the skills learned by performing simple tasks are similar, if not identical, to those required for more complex tasks. Progress is made when basic skills and principles are applied to progressively more complex tasks. Without a firm grounding in the basics, however, this progression will be hindered, or may be stopped altogether. A simple story and a PhD thesis will involve quite different vocabularies and contents, and will be targeted at quite different audiences, but both rely on the same rules of grammar if they are to be clearly understood. In a similar manner, the experiments described in the A2 section of this booklet rely, in the main, on the same basic practical skills that were introduced and practised in the AS course. While it will be necessary to develop new skills for some of the A2 exercises, firm mastery of basic AS skills is crucial if the A2 practical course is to be successful. It would be fair to say that the skills learned and developed at AS level are fundamental to the success of the A2 practical course; none are redundant.

As the A2 practical assessment is made by written examination, some students may choose to ignore or deny the relevance of A2 practical work; as a consequence, they will not accept the need to hone and perfect their practical skills. This view must be **robustly opposed**, as a student without a thorough familiarity with practical chemistry will be in a very weak position to plan, analyse or evaluate an experiment. To be successful in the examination room, when sitting Paper 5, students will have to have been successful and hardworking in the laboratory.

### Extensions to the skills practised at AS level, and new skills to learn

There will be new manipulative skills to learn at A2 level and help is provided in the fully written-up exercises in Appendix 2. Before these new skills and techniques are introduced to the students in a lesson, it is important that the teacher practises the exercise. By doing this, the teacher should gain sufficient practical experience of the exercise to be able to offer constructive advice, and to identify likely problems areas.

Some of the solvents and reagents used in these exercises carry significant safety risks; however, this should not be used as an excuse for not performing a given experiment. Providing the teacher is well practised and familiar with the experiment, there is no reason why any of the experiments in Appendix 2 should not be performed safely. It may be decided, as a result of performing a risk analysis, that the experiment would be better performed as a demonstration. Providing all the students are in a position to see clearly, a demonstration can produce effective teaching and learning. However, where it is thought appropriate to do so, the hands-on experience gained by students as they perform an experiment for themselves does significantly enhance the learning process.

The main areas for extension in the A2 course are in Planning, Analysis and Evaluation. While some exercises may offer more opportunities than others to practise these skills formally, whenever possible and appropriate it is in these areas that the focus of an exercise should be directed. Casually asked questions such as 'How would you ....' or 'What would you do if .....' or 'What does this show us about....', 'Why is this result anomalous?' or 'What would you change to make this work better/give more accurate results?' about a current experiment can generate a classroom discussion that very effectively drives a point home, as it is specific and relevant to the task in hand.

#### Teaching students to plan experiments

Planning is arguably one of the most demanding and difficult skills to learn. In order for training in planning skills to be effective, students must develop confidence in their practical abilities. It is not sufficient that they simply learn to follow instructions; they must be able to apply the experience they have gained from earlier exercises in order to visualise the consequences of a given choice on the outcome of their plan. It is therefore essential that they understand the rational for using particular approaches, pieces of equipment, recording and analysing techniques, rather than simply being trained to perform a given exercise in a particular prescribed way. An appreciation of precision and reliability is essential when choices of measuring equipment are made, and when experimental procedures are worked out. An understanding of the limits of reliability, frequently described as errors, 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. It cannot be overstressed that students will only be in a position to plan such details successfully with speed and confidence if they have followed a comprehensive course of hands-on practical work in their A2 year.

The writing of a plan divided into clearly defined stages, each of which must be addressed when producing an effective plan. However, given that practical exercises vary widely in their nature and purpose, it would be unwise to assume that there is a standard 'formula', with predetermined stages, to follow when planning an exercise.

The requirements of a quantitative exercise, the purpose of which is to propose and test a hypothesis, or to measure a trend (such as is found in the  $K_a$  plan in Exercise 6, and the enzyme catalysis plan in Exercise 13, in Appendix 2) will be very different to the requirements of a quantitative organic preparation, where the student is required to prepare a plan for the preparation of a given mass of product (as in the preparation of N-phenylethanamide plan in Exercise 25 in Appendix 2). The plan for an analytical investigation (as in the identification of carbonyl compounds in Exercise 21 in Appendix 2) will be different again.

Thus, students need to develop a flexible approach to planning, which allows them to tackle a wide range of different types of exercise. To do this they will have to be able to identify and understand the specific requirements of a particular planning exercise and devise for it a tailor-made plan. To reinforce what has been said earlier, this takes time and practice.

#### 1 Defining the problem

#### For a quantitative exercise

- Students should be able to use information provided about the aims of the investigation, or experiment, to identify both the independent and the dependent variables. Also, the other key variables must be identified and effective measures proposed to control them.
- They may be required to use their knowledge and understanding of the topic under consideration to make a quantitative prediction of the likely outcome of the experiment.
- The purpose of the plan would then be to test this hypothesis in a manner which is reliable, unambiguous and, above all, reproducible.
- Even if making a formal hypothesis is not a requirement of an exercise, students will still need to have a clear idea of what they expect the results to show if they are to analyse and evaluate their results effectively.

The data obtained in the exercise will then require processing in some way in order to allow for analysis and evaluation.



- The plan must contain details of how these processes are to be carried out.
- If the experiment is to generate quantitative data, then the recording, graphical and numerical processes involved in the data analysis must be clearly laid out.
- The steps by which the analysed data, and the experimental procedure, are to be evaluated should also be described.

#### For a qualitative exercise

Examples of this type of planning exercise would be the preparation of a required mass of product in an organic synthesis (see exercise 25) or the devising of an analysis scheme to identify an unknown compound (see exercise 21). Clearly, a different approach is required for such examples to that used to plan a quantitative exercise but students are likely to find them to be just as demanding.

Whatever the type of planning exercise, the plan should be sufficiently robust that, when performed by competent chemists, the outcomes will not vary beyond anticipated limits. Without first-hand experience of the approach and procedures to be used, it is highly likely that the plan will be flawed.

#### 2 Methods

The proposed experimental procedure should be workable. It should, given that the apparatus is assembled appropriately, allow data to be collected without undue difficulty. There should be a description, including diagrams, of how the experiment should be performed and how the key variables are to be controlled. Equipment, of a level of precision appropriate for the measurements to be made, and quantities to be used should be specified. The use of control experiments should be considered. Also, details of how the data are to be recorded, manipulated, analysed and evaluated should be given (see point 3 below).

Aspects of the planning process which students frequently find difficult are deciding on a suitable scale for the experiment and choosing suitable apparatus. It is suggested that the teacher frequently asks questions such as 'why do we use this amount of solid?' or why do we choose this volume of liquid?' when students are following an experiment from a worksheet, rather than allowing them to blindly follow a recipe. Similarly, the choice of apparatus should be questioned. By doing this, students will gain experience in these areas and so be better prepared when they have to make such decisions for themselves. In some cases, the choice of volume or apparatus will have a significant influence on the precision and reliability of an experiment. Exercise 9 requires students to consider the effect of the inherent error in a burette measurement on the overall reliability of a titration exercise as small and large volumes are measured from the burette. Such knowledge is of great value when choosing suitable volumes in a planning exercise.

It is often in the fine detail that students tend to let themselves down. Many students will be able to produce a broad overview of what is to be done but it is likely that far fewer will produce a plan sufficiently detailed for it to be successfully used by their peers.

Exercise 6 incorporates several different planning tasks. Firstly, students are required to plan how a pH curve for the titration might be obtained, and they are asked to predict the shape of this curve. Performing their planned titration exercise would then allow this hypothesis to be tested. To be effective, this part of the plan must contain sufficient detail, in terms of quantities, the number and range of measurements and the means by which the results will be used to draw the pH curve, to allow an experienced chemist to perform the task. Students must then plan how they are to deduce the p $K_a$  value for the acid using their pH curve. Finally, the plan must describe how the required buffer solution is to be prepared, and how its effectiveness as a buffer is to be tested. This latter part of the plan provides a significant test of a student's understanding of buffer theory and so is likely to reveal problem areas.

In Exercise 25, students are provided with sufficient background information regarding the chemistry involved in the organic preparation so that no previous experience of this particular preparation is needed. What students do need, however, is experience of the various



processes involved in the preparation; together with experience in mole and percentage yield calculations. Students who have sound practical skills and who are experienced in measuring volumes, filtration, recrystallisation and melting point determination will find the exercise to be straightforward. Those students who lack familiarity with such processes will struggle.

The approach to planning required in Exercise 21 is very different to the approaches employed above. In this exercise, students must identify the different types of carbonyl compounds given in the question, decide how they differ in their chemical properties, select suitable chemical tests to allow them to be distinguished from each other and then produce a logical sequence for these tests. Finally, students must be able to demonstrate their understanding of the part played in the characterisation of a compound by the preparation of derivatives, and the use of spectroscopic analytical techniques. The exercise provides a sound test of the chemistry involved but it also effectively tests communication skills.

Whichever type of planning exercise a student undertakes it is important, both for the safety of the student and also for the safety of those around, that a realistic risk assessment is made as part of the plan. By requiring students to focus on the risks in advance of the practical class, they will gain experience in this area and so will be less likely to take risks in the practical class.

#### 3 Analysis, conclusions and evaluation

Consideration should be given in the plan of how the data obtained in an experiment are to be analysed, interpreted and evaluated. Valid and reliable conclusions can be drawn only if the strategies devised to address these points are effective. This may involve the generation of a results table and the use of graphical techniques, as in Exercise 6, the determination of yield and purity in a preparative experiment such as Exercise 25 or the devising of a logical framework for deciding on the identity of an unknown sample, such as in Exercise 21. Again, the wide range of possible planning topics requires students to show flexibility, and perhaps even ingenuity, in their response to planning tasks.

## Teaching students to analyse and evaluate experiments, and to draw conclusions

#### 1 Analysing data

This skill requires students to apply their understanding of underlying theory. Even when that understanding is present, however, many students still struggle. The presentation of a clear, lucid, watertight argument does not come naturally to most people and therefore much practice in this area is recommended.

Any conclusion made on the basis of data obtained from an experiment must be fully reasoned and justified. This justification may take the form of a written argument, for example in the interpretation of the results of a series of tests designed to determine the identity of an unknown compound, as in Exercise 21. It may take the form of an extended calculation, as would be required in the determination of the Avogadro constant in Exercise 3 or it may involve graphical steps, as in rate order determinations in Exercise 15.

The steps followed as an argument is developed, must be sequential, clear and easy to follow. In the examinations, students will have to convince a stranger that their reasoning is sound. The plea 'well you know what I meant' might convince a teacher who is familiar with that student's work, but will not convince an external examiner.

To confidently analyse numerical data, students must be both proficient in handling the mathematics involved and experienced in the calculation sequence involved. Similarly, students will struggle with, for example, inorganic or organic analysis experiments if their theoretical knowledge is weak. Therefore, it follows that success in the practical class will depend to an extent on success in the theory class.



Students should be aware that the number of significant figures to which the answer is expressed shows the precision of a measured quantity. Therefore, great care should be taken with regard to the number of significant figures quoted in an answer. The general rule is to use the same number of significant figures as are found in the least precisely measured quantity.

Another skill which many students find difficult to master is that of error analysis. Students need to be familiar with two types of 'error'. The first type of 'error' is that which is inherent in the use of a particular piece of equipment/apparatus. Although we refer to this as an apparatus error, we really mean that there is a 'range of uncertainty' associated with measurements made with that piece of apparatus. This uncertainty will be present no matter how skilled the operator might be.

The second type of error is appropriately called experimental error and is a direct consequence of the level of incompetence of the operator or of flaws in the experimental procedure. If the overall experimental error, as measured against a reliable benchmark, is greater than the combined apparatus errors, students should look for flaws in technique or experimental procedure.

One of the aims of exercise 9 is to help students to focus on this point by requiring them to compare the error in burette measurements when the same burette is used to measure volumes of 100 cm<sup>3</sup> and 5 cm<sup>3</sup>. Clearly, the error, or uncertainty, in measuring the 5 cm<sup>3</sup> volume will be twenty times that when 100 cm<sup>3</sup> is measured.

#### 2 Evaluation

Arguably, this is one of the most important, and probably one of the most difficult, skills to acquire. In order for an evaluation to be effective, students must have a clear understanding of the aims, objectives and predicted outcomes of the exercise. Without such knowledge they will be in no position to judge the effectiveness of the procedures used.

The evaluation procedure may include:

- (i) the identification of anomalous values, deducing possible causes of these anomalies and suggesting appropriate means of avoiding them.
- (ii) the adequacy of the range of data obtained.
- (iii) the effectiveness of the measures taken to control variables.
- (iv) an informed judgement on the confidence with which conclusions may be drawn.

Anomalous results are those which do not fit in with the pattern formed by the other results of an experiment. If such results are to be identified, the expected pattern must be known. This pattern could be predicted as part of a hypothesis or deduced from the clear trend shown by the remaining results. However, when the anomalous value is identified, the selection of this value must be supported by evidence. Once an anomalous value has been identified, it is necessary for students to attempt to explain the origin of the anomaly and to propose strategies to deal with it. These strategies might include repeating the measurement or omitting it.

In experiments such as the  $K_a$  determination in Exercise 6, once the pH curve has been plotted, it may become clear that the experiment has not provided sufficient data in some parts of the curve. Thus, the number measurements taken and/or the range of measurements taken were not adequate. When evaluating such an experiment, it would be sensible to propose the inclusion of extra measurements to clarify and to specify where these extra measurements should be made.

When the results of a quantitative experiment are know to be clearly inaccurate, the results should be evaluated. Students must be able to deduce whether the errors in the data obtained in an experiment exceed those expected due to the apparatus used.



If the errors in the data do exceed the apparatus errors, then flaws in the procedure that have generated these excess errors must be identified. Having identified potential flaws, students must have sufficient knowledge of the process involved, that they are able to suggest changes to the procedure that will result in a more accurate or reliable outcome. If the perceived flaw lies, for example, with temperature fluctuation, it would be of little use to simply state that the temperature should be controlled. What is needed is a practical solution to this problem, such as the use of a thermostatically-controlled water bath.

If the errors in the data do not exceed those expected due to the apparatus used, then improvements to accuracy would be achieved by changing the apparatus used. If, for example, the perceived flaw lies with the precision of a mass measurement, rather than a vague reference to the use of a 'more precise balance', the precision of the replacement balance to be used should be specified, for example, 'a balance weighing to  $\pm 0.01$  g'.

For conclusions to be drawn on the basis of the results of an experiment, it is essential that the results of the experiment inspire confidence. Simple statements by students to the effect that they are happy with their results and that their experiments worked may well be true but they are of little value and will earn no credit. If an experiment worked well, it is necessary that this judgement be supported by evidence from the experimental results.

#### 3 Drawing conclusions

This is also a higher-level skill, which will demand of the student a thorough understanding of the basic theory that underpins the chemistry involved.

The conclusions drawn from a set of data must be judged on the basis of the strength or weakness of any support for or against the original hypothesis or on the results of qualitative tests. Students should be able to use the detailed scientific knowledge and understanding they have gained in theory classes in order to make judgements about the validity of the conclusions they have drawn.

Without practice in this area, students are likely to struggle. In order to increase the confidence of students in drawing conclusions, it is recommended that exercises which are set within familiar contexts are used initially. Once confidence has started to grow, then is the time to move to less familiar territory.

### Designing a practical course for the A2 year

The course outlined in Appendix 1 is based simply on syllabus order. It is highly likely that the schemes of work used by different teachers will require topics to be taught in very different sequences. This is fine, as there is no preferred order for performing these practical exercises.

What is important, however, is that your students see the relevance of their practical lessons and that their skills are developed gradually, starting with simple exercises and moving on to more advanced activities. Most of the exercises are designed to provide practical support to the teaching which goes on in theory lessons. Careful scheduling of experiments so that they fit into the normal teaching programme will reinforce the essential nature of practical chemistry. The extra input to the learning process provided by a well-run and successful practical lesson is well worth the effort involved in preparing it.

As the Paper 5 examination approaches it will become necessary to increase the emphasis placed on planning, analysis and evaluation skills. However, it is vital that you introduce these skills right from the beginning of the A2 course, as this allows time for these skills to develop and mature.

Appendix 1 contains thirty-two exercises, fourteen of which have been fully worked up in Appendix 2. Of these fourteen, several contain suites of experiments. Apart from providing 'Student Sheets' and 'Teachers' Notes' the purpose of the fully written up exercises is to provide ideas as to how you may use the remaining exercises in Appendix 1. The intention is



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not to be prescriptive. An approach or exercise that works well in one institution may well be less successful in another. What is important is that when you have used a practical exercise, you evaluate its effectiveness. On the basis of such evaluations, your practical programme will evolve.