A STUDY OF THE IMPACT OF MICROSCALE/ SMALL SCALE CHEMISTRY EXPERIMENTS ON THE ATTITUDES AND ACHIEVEMENTS OF THE FIRST YEAR STUDENTS IN GLASGOW UNIVERSITY

BY

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A thesis submitted in part fulfilment of the
requirements for the degree of M.Sc. in Science
Education (Chemistry) of the University of Glasgow
Scotland U.K.

CENTRE FOR SCIENCE EDUCATION JANUARY 1996

CONTENTS

CHAPTER THREE

CHAPTER FOUR

CHAPTER FIVE

CHAPTER SIX

APPENDICES:

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to Prof. A. H. Johnstone for his supervision, advice, guidance and encouragement during my studies. It was his help, and kindness which helped me in the completion of my study.

I would also like to thank Dr. Fakhir Al-Naeme for his help, guidance, encouragement, suggestions, participation the laboratory and discussion of the material that has been introduced.

I am grateful to the Ministry of Education, Government of the Islamic Republic of Pakistan for their financial support without which this study would have been impossible.

I am also grateful to my parents, my wife and my children who showed great patience during the period of this study when I was away from them.

Finally I would like to thank my colleagues at the Centre for Science Education, Glasgow University for their friendship and support during my stay at the Centre.

ABSTRACT

This study is intended to measure the attitudes of First Year Chemistry Students and Demonstrators at Glasgow University, and also the impact of microscale experiments on the achievements of students.

In 1993-94, two experiments from the First Year Organic Chemistry Course were selected for modification to Small Scale. Small Scale apparatus and techniques were used by the students.

Questionnaires were devised for the students and demonstrators for the measurement of their attitudes toward different aspects of Small Scale Experiments.

In 1994-95 three experiments (partially or fully) from the First Year Inorganic General Chemistry were selected for modification to Small Scale. This year students used the Small Scale apparatus and techniques with modified written instructions in which the procedures of experiments were fully explained.

A questionnaire to measure the attitudes of students toward Inorganic Small Scale Experiments was designed in the same year. Moreover, the achievements of students in the Small Scale Experiments and Normal Scale Experiments were also studied.

This study revealed that students liked Small Scale experiments and would prefer them to the Normal Scale, provided that the following two opportunities are given to them:

a. the students are fully briefed about the usefulness of the Small Scale experiments;

b. the different techniques used in these experiments are fully explained to students before starting any experiments.

CHAPTER ONE

Introduction to Microscale/Small Scale Laboratories

Practical work in a science subject plays important roles by confirming the theory which had already been taught in lectures and as the core of the learning processes of investigation and inquiry.

In recent years the subject of chemical waste has become a major concern everywhere. Disposal of this waste is not only very expensive, but it is becoming increasingly more difficult to find places willing to accept it. In an attempt to help alleviate this problem, a trend is increasing to downscale the amount of chemicals used in chemistry laboratories by instituting microscale/small scale operations. This conversion has educational advantages as well. It teaches the students to be precise in their measurements and very careful with their equipment and products.

A significant change is taking place in today's chemistry lab. This is the steady increase in adoption of the micro chemical lab concept at secondary and post secondary levels. The microscale approach is being adopted to help solve a number of problems.

Cheronis and Stein in 1935 pointed out the techniques such as filtration, distillation and evaporation are general ones, which were recommended on semi-microscale for teaching(1).

In 1945 Cheronis and his co-workers published parts VI and VII of their series "the use of Semi-micro Techniques in organic chemistry"(2,3) with a literature survey going back to 1919. There is not a little doubt that Cheronis was largely responsible for the initial growth of interest in the teaching of organic chemistry by small scale methods. Apart from his papers, his books(4) inspired others to try small scale techniques.

One who was inspired was Kline, Professor Emeritus at the University of Connecticut. Kline still professionally active, makes the following statement in the preface of his book(5).

"In September, 1941, Dr. Nicholas D. Cheronis described his experience with semi-micro procedures at the Atlantic City meeting of the American Chemical Society. He exhibited some of the apparatus used and asked for instructors to volunteer to testing some of the procedures which he had devised and which were described in mimeographed manual which he had prepared".

Kline (an expert glass maker) constructed apparatus and assigned it to 8 students. By the summer 1942, all first year students were equipped with small scale glass ware. According to Kline there was a dramatic rise in student intake after the end of the war. Supplies and bench space was limited(6).

In 1951, Griffin and George introduced the Macroid Semi-micro Organic Set. Soon after, another supplier marketed a slightly different set.

Teaching loads were horrendous, but time was found to work on the manuscript of a small, precollege organic text. The effort , naive by modern standards, attempted to show that the practical curriculum is best served by a combination of macro and semi-micro techniques(7).
In the educational laboratory setting, the reduction in scale (to approximately 50-150 mg of solids and 50-2000 μL of liquids) is known as microscale chemistry (IUPAC uses the term "Small Scale chemistry").

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The application of this concept in industrial research and development laboratories of today is also a viable option. Notably, pharmaceutical research laboratories have worked at this level for years(8).

Prior to 1960, micro-techniques were highly delicate, tedious and specialised, and was used mainly in natural product research and in graduate school studies. In was not until the 1980's when environmental concerns had risen to the forefront and the electronic milligram balance became available that using microscale experiments at the introductory level of instruction became a reality(8).

Microscale chemistry was developed for the introductory laboratory by Mayo and Butcher (from Bowdoin College, Brunswick, Maine, USA) and Pike (Merrimack College, North Andover, Massachusetts, USA)(8).

The necessary techniques and materials were developed for the organic chemistry laboratory in 1982-3. The first teaching tests occurred at Browdoin and Merrimack colleges in 1983. The tests proved to be highly successful with the sophomore level students who rapidly adapted to the new techniques. Preliminary results were reported at the national meeting of the American Chemical Society in 1984(8), and were published in the Journal of Chemical Education(9).

In 1985 (9,10) a series of articles appeared in the Journal of Chemical Education. One of the major reasons for the initial introduction of microscale experiments(9) was to decrease the quantity of potentially hazardous organic solvents in the air in quantity of potentially hazardous organic solvents in the air in organic chemistry laboratories toward the levels recommended by the Occupational Safety and Health Administration(11). This goal is more certainly attainable when reactions are carried out with millilitres of solvents rather than with hundreds of millilitres as usually done previously. Conversion to microscale has also provided many other benefits, both financial and educational.

The microscale experiments that have been developed in organic chemistry are mainly synthetic in nature since much of the laboratory work in organic chemistry involves syntheses. In contrast the diversity required of general chemistry experiments is much greater, since this course material ranges from descriptive chemistry to physiochemical principles. Although qualitative experiments can be used to present the basis of chemical reactions, quantitative activities are often needed to illustrate the principles inherent to such topics as stoichiometry, equilibria, thermodynamics, kinetics and electrochemistry. Fortunately both of these needs can be met by microscale techniques. In fact the ease and speed with which microscale experiments can be done, besides small quantities of materials required for them, make it possible for students to carry out more trials under a greater range of conditions.

The real innovator of microscale techniques in general chemistry was Thompson (1990)(12) of the University of Colorado. In 1977 he modified general chemistry experiments so that only drop size quantities were required. These were mixed on a plastic sheet and the reaction if any, was observed by means of a magnifying glass. Thompson also realised that plastic medical science equipment could have a potential for microscale experiments.

Although Thompson's idiosyncratic laboratory manual gained some following, it was Mills and Hampton (1991)(13) who broadened the appeal of plastic ware for introductory college/university chemistry experiments in the US. At about the same time the impetus for the use of microscale techniques at high school level came from a workshop held at Princeton University (1987)(14). Since then the leaders in school activities have been Maunch and Russo (1989, 1990 and 1992)(15,16) in the US, and Slater (1994)(17) in Canada. A series of microscale experiments have appeared in the North American High School Chemistry Magazine, Chem. 13 News.

The first microscale chemistry textbook, Microscale Organic Laboratory appeared in 1986(18). Many others have followed since then.

This work directly led to the growth of a mini-industry in the area of microscale chemistry, with at least seven glass companies manufacturing microscale kits and glassware.

Szafran, Pike and Singh (at Merrimack College) investigated the implementation of microscale techniques in inorganic chemistry. There had been an increasing tendency toward elimination of laboratories in inorganic chemistry, due to excessive cost of chemicals and equipment, the difficulty of performing interesting experiments in a safe and efficient manner, and the problems of waste disposal. By converting the inorganic laboratory to the microscale level, it became possible to expand the range of experimental coverage to include such important areas as organometallic chemistry of the heavy metals, catalysis, and bioinorganic chemistry(19). The text book of Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience appeared in 1991(20). Further work by Singh, Szafran, Pike, Foster and others has expanded the role of microscale chemistry to the general (introductory) chemistry laboratory(20).

The use of microscale techniques in general chemistry is not limited to the laboratory, since microscale experiments conducted on transparent acrylic spot plates are readily visible, even in large lecture halls, when shown on an overhead projector (reactions in which precipitates are formed are less satisfactory than those that involve colour changes or evolution of gases). This should help to bring lecture demonstration within the reach of all teachers.

A large number of publications on microscale experiments and techniques have appeared in the Journal of Chemical Education during the last decade.

CHAPTER TWO

A Review of the History of Chemistry Laboratory Work

2.1 Practical work in the early eighteenth century

In the early eighteenth century chemistry was taught only by lectures. It was felt at later stages of the same century that some practical work should be introduced in the form of demonstrations in lectures. In 1748 at the University of Glasgow, William Cullen and Joseph Black included some demonstrations in their lectures. Otherwise the practical work was done only by assistants and demonstrators in the laboratories.

At the end of the eighteenth century laboratory based methods of teaching gained rapidly in importance for research schools in chemistry. Therefore, individual practical work was accepted as an essential part of the university chemistry course. Until then laboratory work had been an isolated activity with little support; some of it private instead of institutional and outwith the curriculum i.e. it was not compulsory.

At this time practical work played a great supportive role for confirming the theory which had already been taught in lectures. The experimental procedures were printed along with details in the text book, and any help required during this period was given by well trained staff(21,22).

2.2 Practical work in the late eighteenth and early nineteenth century

Systematic laboratory work by students began at the end of eighteenth century. In 1795(21), the Ecole Polytechnique of Paris (France) introduced laboratory work. In 1806(23) practical work was offered at Gottingen University in Germany by Friedrich Stromeyer who believed that chemistry could only be learned through laboratory practice and that students must be given an opportunity to carry out analysis on their own.

In Stockholm (Sweden) at the Collegium Medium, Berzelius opened his own private teaching laboratory for a few students in 1808(21), first situated in Hisinger's house and then in the Swedish Academy of Sciences, attended by his more famous pupils.

In the beginning of nineteenth century, in Dublin University, Dr. Perceval included practical work in his lectures. During the period of 1810 until 1826 the first laboratory course in chemistry was offered in the U.S.A by William James MacNeven, professor of chemistry in the College of Physicians and Surgeons of New York, where students had an opportunity to practise the techniques, processes and procedures of chemistry(24).

2.3 History of practical laboratories in the UK

The first teaching laboratory in a British university was established by Thomas Thomson in the University of Edinburgh in 1807. In 1819 he introduced it to the University of Glasgow(23). Thomson took up a teaching post in the University of Glasgow, where he tried to establish a research school based on his teaching laboratory(21). Industrial pressure for trained chemists had grown substantially, particularly for analysts to maintain quality control in industry as well as for research chemists. Practical training before this had been on an apprenticeship basis, but Thomson initiated systematic lab training for his students.

In 1824 Liebig's chemistry laboratory was opened at the University of Giessen. It was the first institutional laboratory in which students were deliberately trained for membership of a highly effective research school(21) by systematic research.
The Liebig laboratory was so successful that, according to Morrell(21), 11 out of 30 of Liebig's pupils occupied most of the important posts in chemistry laboratories of British universities.

In 1827(25) Faraday in his book of Chemical Manipulation, which provided instructions for students in chemistry, talked about two main objects of practical work:

i. the extension of present knowledge and

ii. the demonstration of the knowledge previously acquired.

In 1829 Dr. French started practical classes in the University of Aberdeen. There were 115 teaching laboratories in the UK by the year 1876(23). Practical training in chemistry sprang up in universities all over Europe and North America, devoted to the teaching of skills directly usable in industry and research.

2.4 Practical work in the first half of the twentieth century

Until 1930 several investigations comparing individual laboratory instructions with the demonstration method were published. Hunt (1935)(26) argued that demonstration could be done in 5-40% of the time required for individual laboratory, and students would be less likely victims of overzealous instructors who required them to stay after hours and do extra experiments. The

Demonstration method would also make more efficient use of faculty time, not only because they required more effort with concentration but also the teacher who previously tended to neglect laboratory supervision, would be forced to play a more active role,

These arguments (philosophical, educational and economic) tend to favour demonstration over individual methods(27). In the 1930's and 1940's there was an obvious improvement in the use of statistics, with standard tests to pre-test students, and use the results for group comparison etc.

Adam (1942)(28) reported that during this period the literature recorded some 50 studies related to individual versus demonstration laboratories. Of these 45 were applied to high schools and 5 to college classes; 23 dealt with chemistry institutions; 7 investigations of the debate were conducted by means of questionnaires, and 13 were reviews of findings of previous investigations. Fourteen papers expressed the opinion of the authors on the relative merits of individual laboratories versus the demonstration method.

The individual method of laboratory teaching was opposed by those who argued that it was a waste of time and money.

The demonstration method had the advantage of keeping the entire class together and providing students with greater opportunity to think because the instructor could call attention to every point and ensure that certain principles would not be overlooked. Students were, therefore, exposed to a broader experience of chemistry through demonstration by introducing them to methods, apparatus, compounds and uses of chemistry, and so saving a lot of time spent by students performing practical work themselves.

It was also argued by the supporters of the demonstration method that most laboratory manuals of the day were quite useless as far as scientific method was concerned, yet many students gave evidence of their genuine interest in science through their thoughtfully and independently written note-books(26).

Those who were in favour of individual laboratory instruction argued that it facilitated the learning and retention of chemistry facts and principles discussed in the class-room by providing contact with actual materials(29). Moreover, it was also suggested that individual practical work provided the students with some basic insight into elementary laboratory methods and left them with a feeling of the reality of science, thus increasing their interest and enthusiasm, resulting in increased enrolment for chemistry courses.

A movement to re-examine the laboratory work objectives was started after the Second World War. Before it, chemistry had been taught with primary emphasis on knowledge objectives which gradually shifted to a greater concern for process, attitude and interest, and cultural awareness objectives(30).

According to Owen (1949)(31) the normal experiment gave detailed information to students and so he proposed that experiments should allow the students to think by themselves about the experiment.

2.5 Practical laboratories in the second half of the twentieth century

2.5.1 1950's and 1960's

According to Mallison and Buck (1954)(32) there was no critical thinking done in the laboratory, merely "cook-book" manipulation which the students had to follow. So after the Second World War

the discussion moved from two forms of practical work to a greater concern for the objectives of laboratory instructions.
The advent of curricular changes in chemistry was seen in many countries of the English speaking world during the 1960s'. CHEM. study and CBA (Chemical Bond Approach) appeared in the U.S.A and Nuffield became the trend-setter in England and Wales and was exported to several parts of the world.

The new science curriculum of the 1960's resulted in several changes in the role of traditional laboratory work. The curriculum stressed the processes of science and placed emphasis upon the developments of higher cognitive skills. Laboratory work required a central role as the core of the science learning process, not just a place for demonstration or confirmation. It was that laboratory ought to provide students with opportunities to engage in the process of investigation and enquiry(33).

In Scotland the Alternative Chemistry Syllabus appeared in 1962 and was rapidly adopted in all schools. Several curricular packages were tried, with varying success, in Australia and New Zealand and some of their new thinking found its way into Britain.

A less structured course was reported by Newmen and Gassman (1963)(34). They devised it for chemistry majors and allowed students to plan their own experiments based on objectives which were discussed in laboratory lectures - in an attempt to develop a research atmosphere. The laboratory techniques were taught as required. Their evaluation of the courses with open-ended experiments showed that students expressed positive enjoyment and exhibited a truer reflection of their ability and potential. The students were able to undertake original research successfully and also to develop the qualities of independence and motivation. However, their enjoyment was closely related to their interest in the subject, independence, and need for guidance.

It was argued by Young(1968)(35) that there was a failure to find out what students were getting from practical work. He thought it valid to present the students with a detailed experimental plan to work through, to teach principles and techniques. Therefore, he proposed that laboratory work should be more than manipulation of apparatus. However, he maintained, from the first year onward, this method should be supplemented by an approach that allowed students to make their own investigations.

2.5.2 1970's and 1980's

It was by the 1970's that laboratory teaching was beset by "enquiry-discovery" methods and "problem-solving" approaches, with the aim that students should discover for themselves much of what was previously taught to them in lectures. Therefore, laboratory courses during this period stressed that students should learn how to deal with systems as they actually behave in the real world, in contrast to the "ideal" behaviour normally portrayed in lectures.

Cochran et. al. (1972)(36) stressed that chemistry experiments should include various topics and techniques. Students were not initially completely free to choose their experiments. In the research experiments, students could participate in ongoing research projects conducted by faculty members. Supporters of this approach claimed that enthusiasm was generated among the students and staff because of the individuality of each programme and the research nature of the high level experiments which imparted a degree of realism.

A learning-challenge cycle of laboratory work in which the learning stage was followed by a challenge stage, was proposed by Rodolph and Vanketchelam(1974)(37). In the learning stage students were given background reading material in the manual and a bibliography for the more motivated students, which familiarised them with techniques and equipment. After

completing the learning cycle, the students were given the challenge cycle, mainly comprised of variations of cookbook lab experiments phrased in terms of open-ended questions.
The influence of open-ended experiments led to the development of more integrated courses based on modules, which consisted of techniques grouped on a natural or essentially non-classical basis, e.g. the synthesis of an inorganic compound with characterisation which would be allowed by appropriate physical method and by measurement of its reactivity(38).

Over the years many researchers who recognised the existence of problems in laboratory teaching had attempted to redesign their courses; putting forward hybrid schemes involving various degrees of student participation and concentrating on one particular aspect of it. For example, "Chemical measurement" was used by Atkinson (1972)(39) "art of observation" was emphasised by Swinehart (1979)(40); methods of class participation where the students were more actively involved by being asked to do things for themselves. From then on students should be encouraged to acquire specific skills in order to answer questions which they posed in the laboratory(35).

The literature reported a number of courses, where the students were given greater freedom after initial instruction in basic techniques(34,41,42). These courses ran with fairly low student numbers and involved standard experiments and experimental procedures.

A unified laboratory programme was suggested by Aikens(1975)(43), in which the students received instructions about experimental techniques, experimental procedures, evaluation of results, planning design and executing laboratory projects that required a significant degree of judgement.

Wade (1979)(44) argued that for students, the purpose of practical work with detailed experimental procedures was to follow the prescribed procedure as carefully and closely as possible to obtain the optimum result. Therefore, he suggested a practical course without a "cookbook" ; istead students were provided with background material on the techniques and synthetic methods that might be useful.

Johnstone and Wham (1980)(45) asserted that it is important to do laboratory work in a systematic manner; the skills of personal decision, experiment planning, self criticism, evaluation of errors and overcoming practical problems. For this they suggested Mini-Projects, i.e., small open-ended exercises with the minimum of instruction and maximum freedom within the limitations of the present state of the student's knowledge with the objective of reinforcing the learnt skills. This was also supported by Pickering (1988)(46), who argued that a puzzle laboratory (of project-type) could provide much more opportunity for creativity and therefore, would be likely to be more successful in the task of laboratory teaching. They also asserted that practical work reaches its highest form when done by pupils themselves rather than by demonstration, because pupils are then in a position to engage in discovery learning (although guided discovery).

Hodson (1985)(47) asserted that students need a prior conceptual framework to be able to discover anything. He advocated that the discovery method could legitimately investigate the relationship between concepts, but they could not lead to new concepts.

Now-a-days it is advocated that the idea of the pursuit of science solely for the sake of knowledge be abandoned to give way to growing concern about social, practical and technological issues(47) .

2.6 Aims and objectives of practical work

The aims, purpose and effectiveness of practical work in science had been the subject of much debate since the beginning of the

nineteenth century, but after the Second World War much more attention was given to it by researchers.
There are two broad approaches with regard to aims and objectives of laboratory work. The first one is termed as the traditional approach of pure science and is based on a view of the structure of the discipline. Its starting point is in questions such as "what is required for an appreciation of this aspect of organic chemistry?" or "what does a student need to understand about experimentation". These require analyses of the demands of the subject and regard it as the central organising feature around which the course is built.

In the discipline centred approach, laboratory topics and problems are chosen for the purpose of teaching important techniques and methods or concepts.

The second approach is based on needs and takes a view of what is required of the student either for employment or in a subsequent course. Its starting point is in questions such as "what skills does a graduate physicist need?" or "what are the requirements of a chemist working in an analytical laboratory?" Some of these skills may be related to a discipline but others may relate to professional practice or to more general needs of graduates, such as in the area of communication skills.

Hofstein and Lunetta (1982)(48) pointed out that many goals and objectives of practical work are synonymous with those defined for science courses in general and there is a need to define goals for the areas in which laboratory work makes a significant contribution and to capitalise on the uniqueness of this mode of instruction(42). There exists a substantial task of clarity of purpose in this area because of many different ways in which the aims and objectives of practical work can be formulated.

Anderson (1976)(49) focussed particularly on laboratory teaching in his book "The Experience of Science" and he proposed the following purposes for it:

1. The laboratory is a place where a person or group of persons engage in a human enterprise of examining and explaining natural phenomena.

2. The laboratory provides an opportunity to learn generalised systematic ways of thinking that should transfer to other problem situations.

3. The laboratory experience should allow each student to appreciate and in part emulate the role of a scientist in enquiry.

4. The result of laboratory instruction should be a more comprehensive view of science including not only the orderliness of its interpretations of nature but also the sensitive nature of its theories and models.

2.7 Scientific enquiry

One of the most important general goals of a laboratory teaching course is scientific enquiry. Although laboratory teaching may have other purposes, such as inculcation of specific skills or the appreciation of particular aspects of a subject, the characteristic of laboratory work is an active enquiry. It includes such things as observing and measuring, seeing problems and seeking ways to solve them, interpreting data and making generalisations, and building explanatory models to make sense of findings.

Coulter (1966)(50) was the first to compare the outcomes of different types of laboratory practical. He referred to the traditional approach as a deductive laboratory, where the aim was usually to demonstrate or verify some physical principles or to determine the values of some constants. This method was criticised(51) for its failure to develop in pupils, the ability to plan an investigation by applying the scientific method. To counter this criticism, the inductive laboratory was developed where pupils design and develop their own experiments to solve suggested problems.

Lucas (1971)(52) provided the following useful summary of the use of the term 'enquiry' as discussed in the Science Education literature :

1. What scientists do in obtaining answers from nature (their techniques and procedures).
2. The logical process used in science.
3. The teaching techniques that;
   (a) Enable the student to solve problems by asking questions, gather information, or
   (b) use a semi-structured discussion intended to develop skills in probing or searching.
4. A combination of the meanings using the techniques to teach about enquiry.

Faraday(53) stated that an experiment had two principal objectives; (a) the extension of our present knowledge and (b) the proof or demonstration of the knowledge previously acquired. his book on Chemical Manipulation was produced to provide instruction for studentsin chemistry. He felt there was an absence of such material and the object of the volume was to ‘ facilitate to young chemist the acquirement of manipulation’. He did not claim to teach ‘a habit of reasoning’ but only the ‘art of experimenting’.

There was a little attention to safety precautions in his book as his prime concern was for the success of the experiment but there is still a lot of value that can be learned from his book today.

Kempa and Ward (1975)(54) stressed the necessity to evolve at least some broad qualities with reference to the judgement of students' performance in experimental work. They described the process of practical work as follows;

i. Planning and design of investigation in which the student predicts results, formulates hypotheses and designs procedures.

ii. Carrying out the experiments in which the student makes decisions about investigative techniques and manipulates materials and equipment.

iii. Observation of particular phenomena and

iv. Analysis, application and explanation in which the student processes data, discusses results and explores relationships, and formulates new techniques new questions and problems.

They also pointed out that setting up the experiment and conducting of measurements and observations, are genuinely practical in nature, in that they involve handling of chemicals and apparatus. The other activities have a strong theoretical orientation and although they are an integral part of experimental work, they do not involve or depend upon the exercise of manipulative and observational skills.

On the basis of literature on practical objectives in school chemistry Swain (1974)(55) pointed out that:

i. authors differ in what they think to be "desirable" practical objectives;

ii. there is often no detailed breakdown of objectives and vague titles are predominant;

iii. pupils' attitudes to practical work are neglected.

He attempted to produce a list of objectives directly from the practical situation and analysis of the experiment itself. The resulting objectives were, however, based upon those given by previous authors and sometimes modified to produce a new list. Moreover, he proposed three main areas for concentration:

i. The road to the experiment consisting of comprehension of purpose, planning and set-up of the experiment;

ii. The experiment consisting of performing manipulation, observation and recording;

iii. The conclusion of the experiment consisting of analysis, interpretation, organisation and evaluation of results, and presentation of reports.

Johnstone and Wood (1977)(56) examined practical work in secondary schools from the point of view of teachers and of pupils and showed that practical work should not only be used for theory illustration but should stand on its own as part of the chemistry course, with its own objectives.

Shulman and Tamir (1973)(57) proposed a classification of purposes for laboratory instruction in secondary education as follows:

i. To arouse and maintain interest, attitude, satisfaction, open mindedness and curiosity in science;

ii. To develop creative thinking and problem solving ability;

iii. To promote aspects of scientific thinking and the scientific method;

iv. To develop conceptual understanding and intellectual ability; and

v. To develop practical abilities.

An elaborated list of objectives for biology, chemistry and physics was put forward by Hellingman (1982)(58). The objectives were as follows;

i. Preparation for an experiment, which consisted of formulating a research question, planning and handling sources of information;

ii. Performing the experiment, which consisted of performing, manipulation; observation, making notes and repeat activities;

iii. Elaboration of observations, which consisted of investigations and repeating the experiment if necessary.

Whittaker (1974)(59) states that "The integration of practical work with the factual and theoretical basis of a subject and the development of courses involving open-ended practical investigations rather than routine practical operations, results in blurring of distinctions between practical and the theoretical work".

Boud and Thorley (1980)(60) investigated the perception of laboratory work amongst practising scientists, recent graduates and undergraduates. They found a higher degree of concordance between practising scientists and recent graduates, who regarded laboratory experience as important to the acquisition of practical skills, equipment familiarity, observational skills, interpretation of data and critical approach to experimentation. On the other hand, undergraduate students have a different perception of laboratory aims, tending to rate highly those activities associated with educational processes, for example, the linking of theoretical material and laboratory work.

Gunning and Johnstone (1976)(61) also noticed a gap between teachers' objectives and their achievement by pupils. There was a lack of correlation between teachers' and pupils' ranking of the importance of objectives. There was evidence that pupils gave more importance to psychomotor skills, while teachers felt that these skills were less important than objectives in the affective domain.

All the above aims and objectives about laboratory work show that there is consensus amongst the researchers in science education that objectives should be meaningful and helpful to learners and teachers.

2.8 Learning through practical work

Research studies have found that students often fail to connect their practical work with the rest of the subject(62). Attempts to investigate the learning involved in practical work(63-65) have shown that, in fact, little is learned of the theoretical information which such work is alleged to illustrate.

Johnstone and Wham (1982)(66) have highlighted the difficulties involved in learning from traditional class experiments. They contend that in many class practical work situations, the learning process is severely hampered by too much new information being presented at once. The effect is that the working memory becomes overloaded, an outcome of which being, that many students resort to following a 'recipe approach' to conclude the experiment. Often much of the information presented is unnecessary and for the purpose of understanding, contributes as extraneous 'noise'. They suggested that the load on working memory of students could be decreased by increasing the 'signal-noise' ratio by;

i. Giving a clear statement of the point of the experiment.;

ii. Making clear what is preliminary, peripheral, and preparatory in an experiment;
iii. Redesigning experiments and
iv. Avoiding the teaching of manipulative/interpretative skills at the same time as data is being sought.

According to Driver et al (1985)(67) it is important to pay attention to the psychology of learning, and pupils' understanding of science, when designing schemes of practical work. Considering learning psychology, White (1979)(62) proposed that laboratory work should include memorable dramatic experiments, experiments utilising everyday objects, so as to forge a link with common experience.

Tamir (1976)(68) contended that even completing a conventional practical exercise, the students do not understand why they did it or what they found.

Case (1977)(69) recommended the following three things for designing effective instructions with a minimum load on working memory of students:

i. There would be minimum number of items of information that require the student attention in order to minimise the load on working memory of students(70);

ii. Make familiar all cues to which the student must attend and all responses he or she must exhibit. The more familiar a cue, the less working memory is needed for the task of extracting it from the context. Similarly, the more familiar a response, the less working memory is needed for its execution(71).

iii. Highlight all stimuli to which the subject must attend, making them salient, either because their physical characteristics make them stand out from their context on because they are pointed out verbally by the instructor. Therefore, the more salient a stimulus, the less working memory is needed for the task of extracting it from the context(72).

Letton (1987)(73) put forward the following suggestions for reducing the 'noise' in existing laboratories;

i. Giving a clear statement of objectives;

ii. Giving clear instructions, on the requirements for the laboratory report;

iii. Identifying which instructions matter and which are peripheral and make this obvious in the material;

iv. Redesigning the experiment with regard to the content;

v. Dividing the written material into sections which are easily managed by the students;

vi. Making the management of the laboratory efficient and giving a map of the layout of the laboratory with location of all equipment and material; and

vii. Ensuring that relevant skills are taught separately from the actual experiment in order that the student should gain confidence.

CHAPTER THREE

Conversion of a Chemistry Laboratory to Small Scale

3.1 Introduction

There is no doubt about the importance of practical work in a chemistry course. This is generally agreed upon by most chemistry teachers and researchers. Practical work is playing largely a supportive role, that of confirming the theory which has already been taught in lectures. However, laboratory work has acquired a central role as the core of the science learning process, not just a place for demonstration or confirmation. It provides students with opportunities to engage in the process of investigation and enquiry.

Despite the importance of practical work in chemistry courses, there has been an increasing tendency toward elimination of experiments from the chemistry teaching labs in which costly and toxic materials are used. One of the more serious problems is the cost associated with the disposal of hazardous chemicals, due to current governmental regulations. It is generally more expensive to dispose of inorganic and organic wastes than it is to purchase the chemicals themselves. The traditional methods of waste disposal, flushing the waste materials down the drain or burial in land-fills, has resulted in damage to the surrounding environment and has caused concern about public health. These methods of waste disposal are now illegal or tightly regulated. Many compounds of toxic metals (such as lead, mercury, cadmium and barium) have been largely eliminated from instructional laboratory use due to environmental toxicity restrictions.

Laboratory air quality and exposure of students to toxic chemicals is also an area of concern. Based on the current trends, it is clear that all chemical users can look forward to a rising tide of legislation in this area. Colleges and universities are faced with an expensive modification of existing laboratories to meet current air quality regulations(9,74). Insurance costs are also rising, not only for the students but for the instructors as well.

In an attempt to decrease the above problems it has become necessary to decrease the scale of the amount of chemicals used in the chemistry (organic, inorganic and general chemistry) laboratories. This conversion may have educational advantages as well. It can teach the students to be precise in their measurements and very careful with equipment and products.

3.2 Advantages of a small scale laboratory

3.2.1 Small scale impact on waste generation

In many well thought out laboratory experiments the product must be used in various characterisation tests or have utility in a subsequent reaction. According to the current analytical techniques, the amount of product needed is quite small. It has been observed that most of the product generated in the laboratories is never employed for any useful chemical purpose.

The small scale technique sharply reduces the percentage of product for disposal. Many chemistry laboratories have excluded the use of well known toxic metals (like lead, barium, chromium etc.). This is due to the environmental hazards posed by disposal of the wastes and to a lesser degree due to dangers posed to the chemist in the laboratory. This, in turn, affects the possibility of offering a comprehensive inorganic chemistry laboratory. By reducing the quantity of toxic compounds used, the students exposure to these compounds is reduced. Therefore, by suitable safety precautions and by judicious selection of reagents, the small scale techniques allow the interesting area of heavy metal chemistry to be reintroduced to the laboratory. The products in many cases, could be easily recycled.

3.2.2 Laboratory safety

One of the major advantages of small scale chemistry is greatly enhanced safety in the laboratory. It is obvious that air quality is markedly upgraded, as the quantity of solvents and other volatile substances is reduced from the conventional work scale. This is especially beneficial to those laboratories that do not possess high efficiency ventilation systems and have limited funds for upgrading their present facilities.

Students' and instructors' safety increases when the amount of reagent used is reduced. The following most common reasons for accidents in chemistry laboratory(75) become minimal in small scale laboratory:

1. Spills from dropped, broken, or tipped-over containers

2. Cuts from broken glass-ware or reagent containers. When a 1-L bottle is dropped on the floor, there is a virtual certainty that it will break. Test with a 30 mL bottle show there is only a 1% probability of breakage under similar circumstances.

3. Escape of vapours from the reaction.

4. Unsafe storage or handling of chemicals.

5. Unsafe or improper disposal of chemicals.

6. The reaction getting out of hand.

7. Risk of fire.

8. Use of hoods.

3.2.3 Use of wider variety of reagents

There are additional reasons for recycling products to reclaim starting materials. Many materials that could be used in the inorganic laboratory preparations are prohibitively expensive. Converting to the small scale level lowers the reagent costs significantly on a student basis. Recycling the products brings the net cost of experimental procedure down to a minimum, with the only losses corresponding to problems of technique and overall yield.

3.2.4 Laboratory efficiency

Due to the reduced amounts of material used in the micro lab experiments, the length of time necessary for process manipulations is substantially reduced. For example, chromatography, filtration, crystallisation, sublimation, distillation and dissolution are all more readily accomplished. The net effect is that the amount of time required for lab workups is reduced allowing the students to concentrate more on the actual chemistry involved. Reaction times are also somewhat decreased, due to factors including greater relative surface area for reaction and reduced mass transfer requirements. Therefore, more experiments can be done in the same length of time.

3.2.5 Reduction in storage space

The quantities of reagents required in micro labs will be less, and the space needed for small sized apparatus will be far less than that required for conventional apparatus. Moreover, the small scale laboratory can be a more comfortable environment than the traditional lab. Similarly, with small scale equipment the lab is less cluttered.

3.2.6 Students performance in the laboratory

Conscientiousness becomes a way of life in the micro lab(9). Since the starting material is measured in milligrams and microlitres, and the products are obtained in very small quantities, there is no room for error. Therefore, the student must learn very quickly that a small mistake can cost him/her a day's work and prove detrimental to his/her grade for the day. Greater student awareness results in fewer chemical spills and much cleaner balances and work areas. The area around the waste bottles in micro labs is almost free of spills unlike the traditional labs.

Breakage is considerably less with the smaller equipment. Greater care involved in the handling of small scale quantities of chemicals is carried over into the overall performance of the student, resulting in fewer accidents.

In 1985 a series of articles(9,11) appeared in the Journal of Chemical Education. These articles include the concept of microscale organic experiments for the academic community and have led to major changes in the laboratory work in organic chemistry done by many college and university students. In addition, they appear to have prompted teachers of other courses at both secondary and university level to examine the nature of laboratory programmes and to consider the introduction of small scale experiments.

One of the major reasons for the initial introduction of small scale experiments(9) was to decrease the quantity of potentially hazardous organic solvents in the air in organic chemistry laboratories toward the levels recommended by Occupational Safety and Health Administration(76). This goal is more certainly attainable when reactions are carried out with millilitres of solvents rather than with hundreds of millilitres as usually done previously. Conversion to small scale has also provided many other benefits, both financial and educational.

The small scale experiments that have been developed in organic chemistry are mainly synthetic in nature since much of the laboratory work in organic chemistry involves syntheses. In contrast the diversity required of general chemistry experiments is much greater. Although qualitative experiments can be used to present the basis of chemical reactions, quantitative activities are often needed to illustrate the principles inherent to such topics as stoichiometry, equilibria, thermodynamics, kinetics and electrochemistry. Fortunately both of these needs can be met by small scale techniques. In fact the ease and speed with which small scale experiments can be done (besides small quantities of materials required for them) make it possible for students to carry out more trials under a greater range of conditions.

The real innovator of small scale techniques in general chemistry was Thompson(77) of the University of Colorado. In 1977 he modified general chemistry experiments so that only drop size quantities were required. These were mixed on plastic sheet and the reaction if any, was observed by means of a magnifying glass. Thompson also realised that plastic medical science equipment could have a potential for small scale experiments.

Although Thompson's idiosyncratic laboratory manual gained some following, it was Mills and Hampton(78) who broadened the appeal of plastic ware for introductory college/university chemistry experiments in the US. At about the same time the impetus for the use of small scale techniques at high school level came from a workshop held at Princeton University (1987)(79) Since then the leading school activists have been Mauch and Russo (1990 and 1992)(80,81) in the USA, and Slater (1994)(82) in Canada. A series of small scale experiments have appeared in the North American high school chemistry magazine, Chem. 13 News.

The use of small scale techniques in general chemistry is not limited to the laboratory, since small scale experiments conducted on transparent acrylic spot tiles are readily visible, even in large lecture halls, when shown on an overhead projector (reactions in which precipitates are formed are less satisfactory than those that involve colour changes or evolution of gases). This should bring lecture demonstration within the reach of all teachers.

3.3 Small scale organic laboratory

A notable feature of current teaching in the organic laboratory is the reawakening of interest in small scale techniques(83). This is associated with chemical safety, storage and waste disposal, which can be substantially ameliorated by reduction in scale.

So far, small scale technique has had its greatest impact on the college scene in the organic chemistry laboratory. One obvious financial advantage is the saving achieved on chemicals when each student is issued a few hundred milligrams of starting material instead of several grams. This level of chemical usage even makes it feasible to employ more expensive reagents and thus makes possible many experiments that might be ruled out on the basis of cost if carried out on an ordinary conventional scale. The other fiscal benefit is due to 80% reduction in the waste generation(84). One unexpected benefit appears to be a decrease in glassware breakage(85), which could be due to one or more of the following factors;

1. The smaller components are less fragile because of their lower mass-to-thickness ratio.

2. Better connection between assembled components make them less likely to separate during experiments. or

3. Students work more carefully with small scale glassware.

It should be pointed out that these savings apply to the long term and must be balanced against the initial cost of equipping a laboratory with small scale glassware.

3.4 Small scale organic equipment and techniques

The introduction of small scale experiments also reduces to some extent the amount of capital equipment necessary:

a. Fewer rotary evaporators are required in the laboratory: in small scale experiments solvents are evaporated off without recourse to a rotary evaporator due to the smallness of scale;

b. Fewer magnetic hot plate stirrers would also be required as several experiments can be carried out using a single hot plate stirrer.

All the small scale equipment and techniques for organic laboratories will be explained in detail in chapter four.

3.5 Small scale general inorganic chemistry

In 1977, Thompson, of the University of Colorado, modified traditional general chemistry experiments so that only drop size quantities were required(77). Therefore, there is an advantage that introductory level Small Scale experiments enjoy relative to their organic counterparts, their low initial cost. Since the majority of the experiments done in the university first year courses are carried out in aqueous solution, acrylic spot plates can serve as reaction vessels, while polyethylene pipettes (Beral pipettes) can be used to transfer and store reagents. These items can be used repeatedly with a simple water rinse between uses, making the average cost of equipment per experiment even lower than their organic counterpart. The matrix arrangement of the spot plates also provides a convenient means of keeping track of solutions and doing series of experiments.

3.6 Small scale general inorganic equipment and techniques

Microscale in the context of the general chemistry laboratory is doing chemistry by the drop instead of by the millilitre. All the detail regarding the equipment and techniques will be explained in chapter five.

CHAPTER FOUR

Small Scale Organic Chemistry as a New Element in the Laboratory for First Year Chemistry University Students

4.1 The aims of the modification of chemistry-1 organic experiments into small scale

As small scale experiments are a completely new feature of the organic chemistry laboratory, it is intended, therefore, from this research to concentrate only on the reaction of students and demonstrators toward this field of practical chemistry.

The main aims of this study could be summarised as follows:

1. To study the attitude of students towards Small Scale Organic Chemistry experiments based upon a questionnaire.

2. To study the attitude of demonstrators towards Small Scale Experiments on the basis of a questionnaire.

4.2 Microscale equipment and techniques

In this section, it is intended to present what apparatus or techniques are necessary to use in order to perform organic microscale experiments.

Most of the illustrations are adapted from the books (Microscale Organic Laboratory, written by Dana W. Mayo, Ronald M. Pike and Samual S. Butcher and Microscale Practical Organic Chemistry by Stephen W Breuer)(88).

4.2.1 Equipment

Small scale equipment is designed to take into consideration the problems of scale reduction. With a 100-fold reduction in the quantity of solvent, the problem of surface wetting and reagent handling requires serious consideration. Reactions are therefore carried out in 3 to 5 mL conical vials. The micro-kit which contains the conical vials is shown in Figure 4.1.

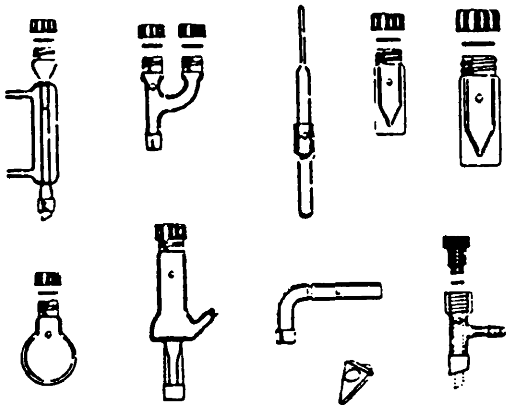


Fig. 4.1 Microscale-kit (Adapted from Kemia-kemi, 8, 1995)

i. Conical vial

The flat bottom of the vial gives it more stability than a round bottom flask would, crucial when the equipment is small. They are used for chemical reactions, having a volume of 3-5 mL (Figure 4.2). The tapered cone within the vial gives a greater height to the same quantity of liquid relative to a non-tapered vial. Even when the quantity of one phase is relatively small, the taper allows a sharp interface to be seen, improving the ease of the extraction process. Condensers and other equipment are attached to the conical vial by use of a greaseless, vacuum tight glass male joint, which fits within the female joint. A threaded plastic Septum cape supported by an O- ring sitting on the shoulder of the male joint screws onto the thread of conical vial. Collectively this is called an O-ring Cap Seal Connector (Figure 4.3). Since the glassware is small, only one micro clamp is necessary for a complete assembly (Figure 4.4).

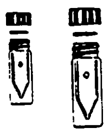


Fig. 4.2 3 and 5 mL conical vials

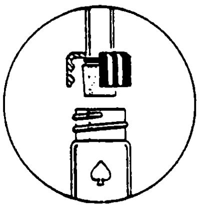


Fig. 4.3 O-ring Cap Seal connector

(Adapted from Kemia-kemi, 8, 1995)

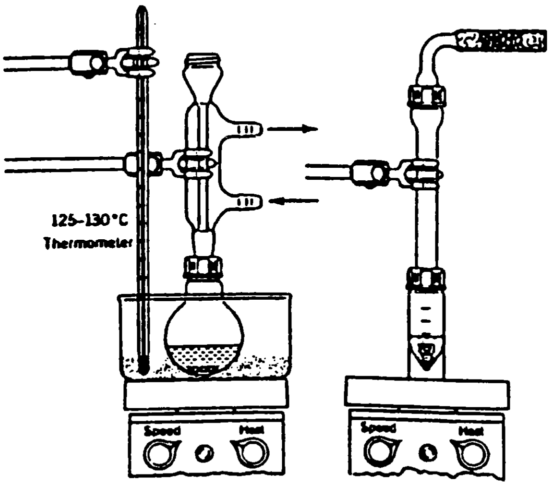


Fig. 4.4 A typical small scale assembly (Adapted from Kemia-kemi, 8, 1995)

ii. Small scale claisen head adapter

The adapter, as shown in figure 4.5, has a vertical screw-threaded standard taper joint that will accept a septum cap. The septum seal allows syringe addition of reagents and avoids the necessity of opening the apparatus to the laboratory atmosphere. The use of the Claisen head adapter with a balloon substituting for the drying tube provides a satisfactory sealing of the system (Figure 4.6).

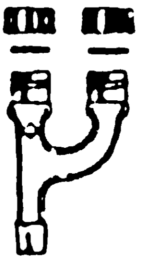


Fig. 4.5 Claisen head adaptor with septum

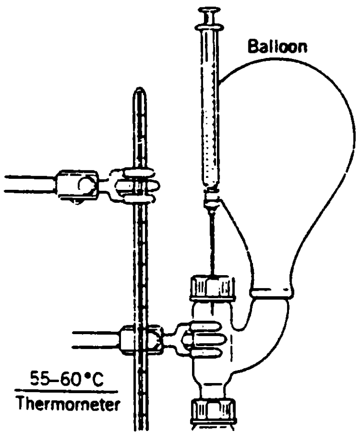


Fig. 4.6 Claisen head adaptor with balloon and syringe

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

iii. Craig tube

Small scale crystallisation (10-75 mg of solid) can be conventionally carried out in a Craig tube. The Craig tube is displayed in figure 4.7.

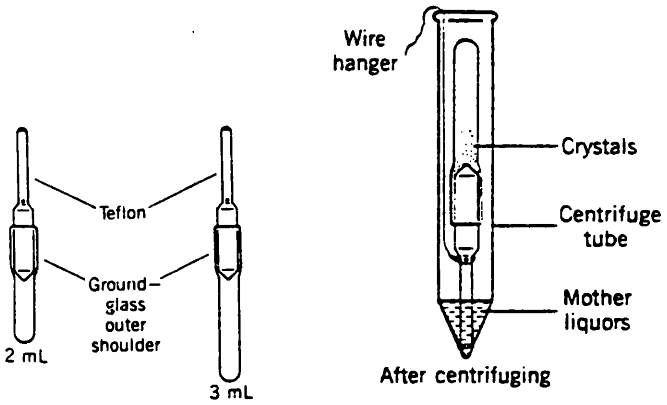


Fig. 4.7 Craig tubes (2 an 3 mL) and Craig tube with centrifuge tube

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

iv. Hickman still

Distillation is accomplished in Hickman stills and semimicroscale spinning band columns as shown in figure 4.8. The spinning band is made of Teflon, and has a magnet embedded in it at the tip, which in turn is cut to fit the cone of a conical vial. The band is spun magnetically using a magnetic-stirring hot plate, which also serves as the heat source for the distillation.

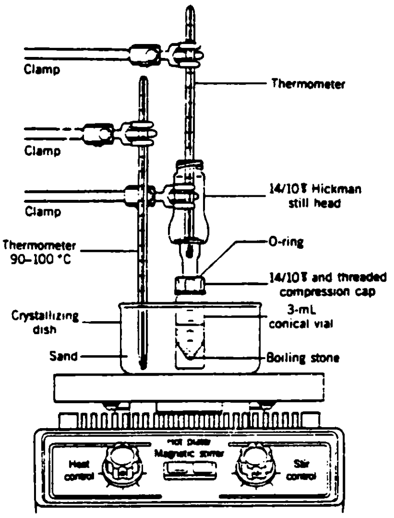


Fig 4.8 Hickman still with conical vial and magnetic stirring hot plate

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

v. The air condenser

The air condenser operates as its name implies, by condensing vapours on the cool vertical wall of an extended glass tube that dissipate the heat by contact with laboratory room air. This simple arrangement functions quite effectively with liquids boiling above 150^{O}C. Air condensers can occasionally be used with water-boiling systems. The air condenser is exhibited in figure 4.9.

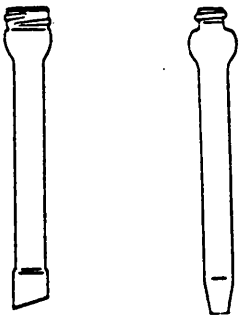


Fig. 4.9 Air condensers

vi. Water-jacket condenser

The water-jacket condenser as shown in figure 4.10, which employs cold water to remove heat from vertical column and thus facilitates condensation, is more often employed for low-boiling systems.

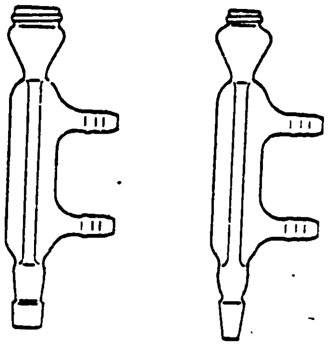


Fig. 4.10 Water-jacket condenser

vii. Small scale pipettes

a. Pasteur pipette

It is a simple glass tube with the end drawn to a fine capillary. These pipettes can hold several millilitres of liquids, and are filled by using a rubber bulb or one of the very handy commercially available pipette pumps. See figure 4.11.

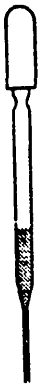


Fig. 4.11 Pasteur pipette

b. Pasteur filter pipette

This pipette is constructed by adaptation of a Pasteur pipette. A small optimum size plug is placed in the drawn section of the tube with a piece of copper wire. Compression of the cotton will build enough pressure against the walls of the capillary to prevent the plug slippage while the pipette is filled with liquid. Once in place, the plug is rinsed with 1-mL of methanol and 1-mL of hexane and dried before use (Figure 4.12).

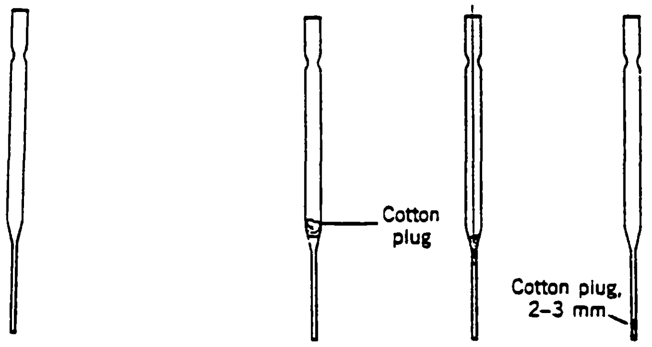


Fig 4.12 Preparation of Pasteur filter pipette

c. Automatic pipettes

At the small scale level these pipettes measure and dispense specific volumes quickly, safely and reproducibly. These pipettes provide considerable insurance for the success of an experiment, as any liquid can be efficiently measured, transferred, and delivered to the reaction flask. The pipettes become essential for laboratories with large number of students.

The automatic pipette system consists of a calibrated piston pipette with a specially designed disposable plastic tip. It is designed so that the liquid comes in contact with the special tip only. The tip is never immersed in the liquid being pipetted. The pipette is filled with the liquid when the tip is in place and then it is kept vertical.

Before inverting the tip into the liquid the piston is depressed to the first top, otherwise bubble formation will result in a filling error. If an air bubble is formed in the tip during filling it is necessary to return the liquid, discard the tip, and repeat the sampling process (Figure 4.13).

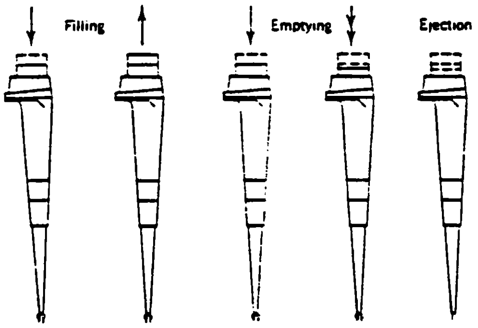


Fig. 4.13 Automatic pipettes

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

viii. Syringes

Syringes are used for transferring liquid reagents or solutions to sealed reaction systems from sealed reagents or solvent reservoirs. They can be inserted through a septum, which avoids opening the apparatus to the atmosphere. See figure 4.14.

Fig. 4.14 Syringe

ix. Equipment for collection of gaseous product

The trapping or collection of gaseous products is conveniently carried out by using the capillary gas delivery tube. The delivery tube is directly attached to a 1- or 3-mL conical vial or to a condenser connected to a reaction flask or vial. The tube leads to a collection system, which is a simple inverted graduated cylinder, blank threaded septum joint or air condenser. The trapping and collection of gas chromatographic liquid fractions becomes particularly important at the Small Scale level of experimentation (Figure. 4.15 a, b, c, & d)

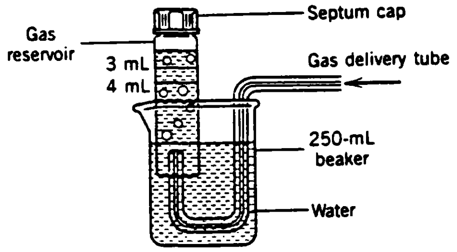


Fig. 4.15 (a) 1-mL vial and capillary gas delivery tube,

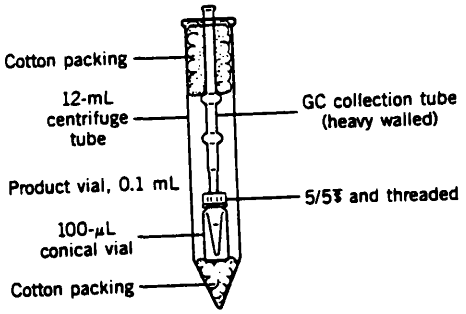


Fig. 4.15 (b) Gas chromatographic collection tube

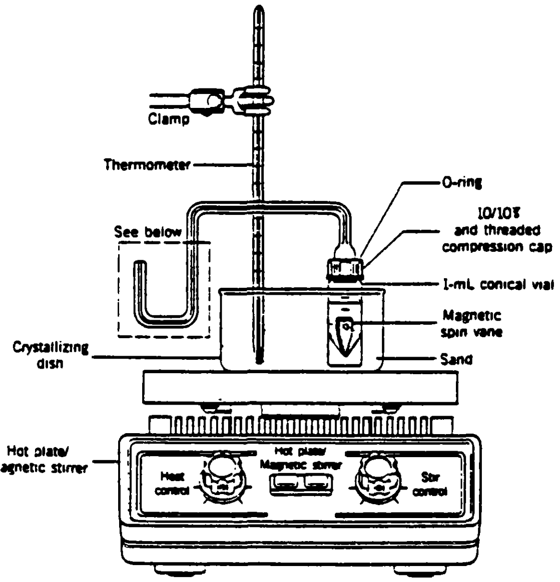


Fig. 4.15 (c) Equipment for collection of gaseous product

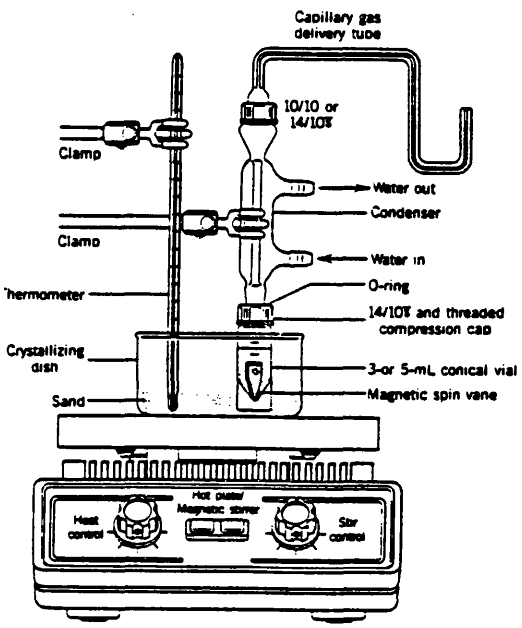


Fig. 4.15 (d) Water jacket condenser with 3- or 5-mL conical vial and capillary gas delivery tube, arranged for heating and magnetic stirring

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and s.s Butcher 2nd Ed.)

4.2.2 Small scale techniques

The specific techniques used to deal with the microscale quantities are as follows;

i. Measurement and material transfer

a. Working with small scale liquids

In small scale experiments, liquid substances are transferred by pipette or syringe. The best technique for transfer is to hold both containers with the fingers of one hand, with the mouths as close together as possible. The free hand is then used to operate the pipette (or syringe) to withdraw the liquid and make the transfer. This approach reduces to a minimum the time that the open tip is not in or over the reservoir or the reaction flask.

b. Working with small scale solids

Working with a crystalline solid is much easier than working with the equivalent quantity of liquid. Unless the solid is in solution, a spill on a clean glass working surface usually can be recovered quickly and efficiently. However, more care is required when working with solution, and the same precautions are used, as for handling pure liquids.

c. Transferring of solids

Solids are normally transferred with micro spatulas, a technique which is not difficult to develop.

d. Weighing solids at small scale level

The current generation of single pan electronic balances has removed much of the drudgery from weighing solids. The weighing is conventionally done on a single pan, top loading balance with an accuracy of 0.001g (1 mg). These systems can automatically tare an empty vial. Once the vial is tared, the reagent is added in small portions. The weight of each addition is instantly registered; material is added until the desired quantity has been transferred. Liquids can be measured with variable volume with glass, graduated 1mL pipettes.

e. Volume-weight conversion of liquids

The volume of liquid can be converted to weight measure by the following relationship;

$$\text{Volume (mL)} = \frac{\text{Mass (g)}}{\text{Density (g/mL)}}$$

ii. Carrying out the small scale reaction

The procedure for carrying out the reaction can vary from simple mixing of two liquids in a specimen tube at room temperature to stirring and heating a two phase system under reflux conditions in an inert gas atmosphere with exclusion of moisture and the slow addition of a reactant. The variables in determining the reaction conditions are the following:

a. Addition of reagent

The success of a reaction may depend on the reagent being added dropwise to the system. Problems may arise from sudden evolution of heat resulting the reaction mixture boiling out of the container, sudden evolution of a gas in large amounts with similar effects or the separation of the reaction mixture into phases owing to low solubility of one of the reactants. In such cases slow addition of a reagent will keep the reaction under control. In large scale experiments this is usually done with a dropping funnel. In small scale a Pasteur pipette, or in closed system hypodermic syringe inserted through a septum cape, easily be used to deliver the necessary amounts dropwise.

b. Stirring

Compounds can only react with each other if their molecules come in contact. If both are soluble in the same solvent this is easy but it often happens that an organic compound is treated with an inorganic reagent in water solution in which the compound is not soluble. In such cases vigorous stirring is necessary so that the reaction can take place at the phase boundary which is continuously regenerated. Stirring is best provided in these reactions by the use of a magnetic stirrer which can create effective agitation in either the tapered flask or round-bottomed flask. The container should be clamped securely with the magnetic stirrer at or near the centre of the stirrer-hotplate, not too far above it.

c. Heating and cooling

Reactions are often carried out with heating or with cooling. This is because at room temperature the reaction may be too slow or too fast or because a reactant may only be able at low temperature or one is seeking greater selectivity in the action of a reagent.

Heating usually speeds up a reaction. In the small scale experiment heating is usually done with a sand bath on an electric hotplate. This combines maximum safety with great versatility. The hot plate can be controlled reasonably precisely. In addition the sand bath has the advantage that sand is a poor conductor of heat and there is a considerable temperature gradient. The temperature can vary by as much as 30-40 °C or more from the bottom to top of the sand bath. Consequently it is quite easy to boil off the volatile solvent carefully by simply resting the specimen tube on the top of the hot sand and then drive of the last traces of the solvent by pushing the tube right into the sand. See figure 4.16.

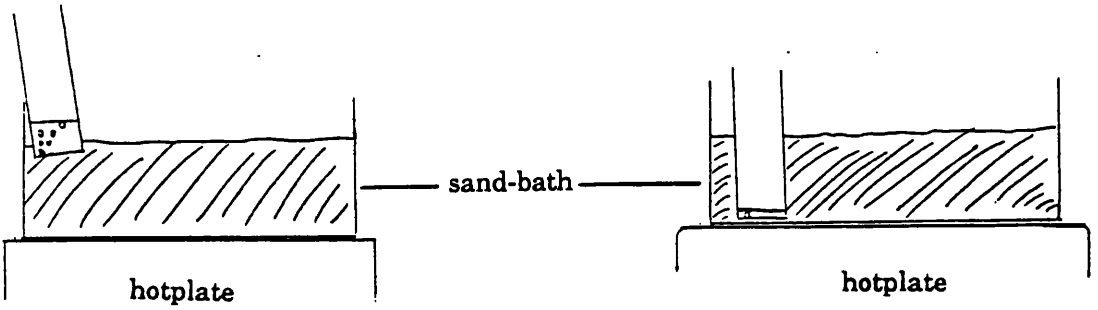


Fig. 4.16 Set-up of heating in sand bath on an electric hotplate

d. Reaction in a dry set up

Atmospheric moisture may have an undesirable effect on the reagents or the course and outcome of the reaction. Therefore, it is necessary to keep the reaction set up dry. To dry the glass ware, it is kept in an oven at 120 °C for at least two hours and allowed to cool. To keep the reaction protected from atmospheric moisture a $CaCl_2$ drying tube is used as shown in figure 4.17. If it is necessary to carry out the reaction in an inert atmosphere, the set-up shown in figure 4.18 is used.

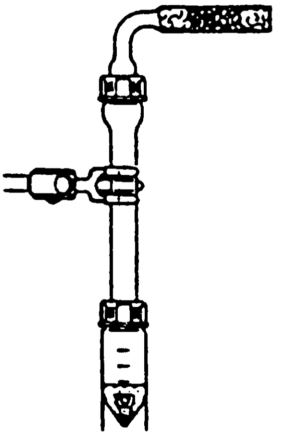


Fig. 4.17 Set-up for reaction protected from atmospheric moisture

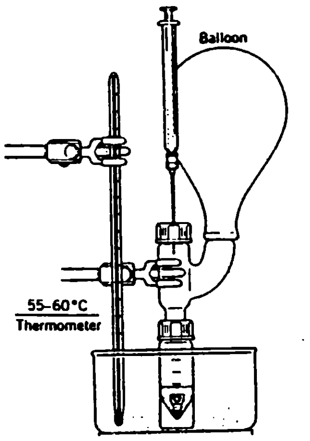


Fig. 4.18 Set-up for reaction in an inert atmosphere

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

iii. Isolation of the crude product

The isolation procedure depends on whether the product is solid or liquid. If the solid product crystallises out of the reaction mixture or the aqueous suspension, it can often be isolated by filtering and washing it on the filter with water to remove all the water soluble reagents or by-products. If the organic solvent is miscible with water, it is necessary to remove it before the partitioning. The removal of these solvents is conventionally done by boiling them off or evaporating them in a stream of air or nitrogen and then the partitioning can be carried out.

iv. Extraction and partition

Small scale/microscale extraction and partition can be carried out using the various containers; the mixing is done by sucking the solution into the pipette and squirting it back into the flask or tube. To separate the layers it is generally easier to pipette out the bottom layer (Figure 4.19) than the top one and with a little care one can achieve complete separations. If the objective is to purify the compound in a solution by adding an immiscible solvent that will dissolve and remove it leaving the impurities behind, the process is called *extraction*. On the other hand, if the objective is to remove the impurities while leaving the desired compound behind, the process is called ***washing***. The purification/separation by this technique is quite a powerful one because it gives two substances separating in different layers of a pair of immiscible liquids. Therefore, it is much more effective than methods dependent on smaller differences in the properties of the material concerned. For example, a mixture containing a neutral and an acidic substance. If the neutral material is required, then the mixture in the organic solvent is washed with aqueous alkali solution, which will cause the acidic substance to ionise and so to become water soluble, while the required neutral material stays behind in the organic layer. On the other hand, if the acidic material is required to be separated from the mixture, then the aqueous layer is acidified to reduce the solubility of the acid in water and extract it into organic layer.

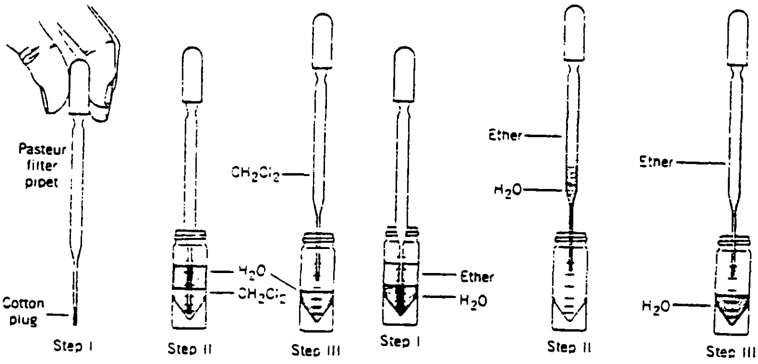


Fig. 4.19 Separation and extraction

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

v. A simple method of filtration and recystallization in a small scale organic laboratory

In the microscale organic chemistry laboratory, reactions are generally performed in small, threaded vials in ground glass vials. These reactions can also be carried out in test tubes(86) to which a condenser can be adapted.

When the reaction products are crystallized, it is important to perform the filtration, washing, and recrystallization without loss. Therefore, the following simple and inexpensive method has been found quite useful (Figure 4.20).

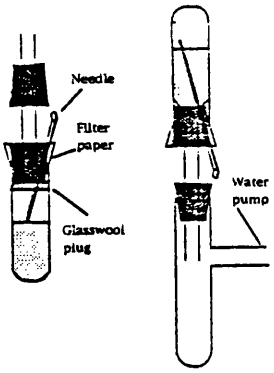


Fig. 4.20 Experimental set-up(left) and arrangement during filtration(right)

a. Filtration

The left part of the figure shows the complete assembly, in which a single-bore rubber stopper is fitted to the reaction vial and pierced by hypodermic syringe needle, which reaches the bottom of the vial. A second single-bore rubber stopper is connected top to bottom to the reaction vial stopper by a short length of glass tubing. On the reaction vial stopper is placed a thin glass wool plug, which is connected by a filter paper that is folded to envelop the stopper and to fit the reaction tube.

This assembly is inverted and fitted on a small filtering flask. The liquid is then filtered off while the solid materials are deposited on the filter and on the walls of the tube, shown in the right hand side of figure 4.20.

b. Washing

The filtering flask is disconnected and the assembly is arranged as shown in the left hand side of figure 4.20. With a syringe, washing solvent is introduced into the vial, via the needle. After shaking, the liquid is filtered off as before. This process may be repeated several times.

c. Recrystallization

The "filtering stopper" is carefully disconnected while the crystals remaining on the filter are pushed down into the tube with a micro spatula. Then a condenser may be fitted, and the product is recrystallized from a suitable hot solvent. In a similar way filtration is accomplished using a new filtering plug. The crystals are dried by water-pump suction for a few minutes.

vi. Method for rapid and efficient determination of recrystallization solvents at the small scale level

With the introduction of small scale organic chemistry laboratory experiments, students frequently encounter crude solid product yield of less than 25 mg. Therefore, it is essential to adapt a rapid and efficient method for the determination of recystallization solvents. The following method(87) has been found useful in first year university level organic chemistry laboratory, particularly in the organic qualitative analyses.

Procedure

Using a nine-well pyrex glass spot plate (Figure 4.21), weigh 1-2 mg of your impure solid into each well and powder each of the samples with a stirring rod. Add 3-4 drops of a given solvent to the first well and observe whether solution occurs immediately. If not, stir the sample/solvent for 1.5-2 min, and again observe whether solution has occurred. Record the solubility of the first solvent at the recorded room temperature. Proceed to each of the remaining solvents stepwise as per the figure. The solvents chosen and the choice of arrangement on the spot plate, polar to non polar, high boiling to low boiling, etc., is optional. 'When the solubility in the cold (room temperature) is obtained, transfer your "spot plate" with above sample to the hood , add additional solvent to the "volatile solvent wells" if necessary, then carefully warm the spot plate on the lowest setting of the hot plate. Record the solubility characteristics in hot solvent'. Allow the spot plate to cool, and observe if crystallization has occurred in any of the wells. Based on the solubility in hot and cold solvents an appropriate single solvent or solvent pair for recrystallization may be chosen. The samples are retained. Allow the solvents to evaporate, then dissolve the solid from each well using a solvent in which the solid is infinitely soluble. Transfer this solution into the Craig recrystallization tube, evaporate the solvent off using N_2 gas, and proceed with you recrystallization using your recently determined recrystallization solvent or solvent pair.

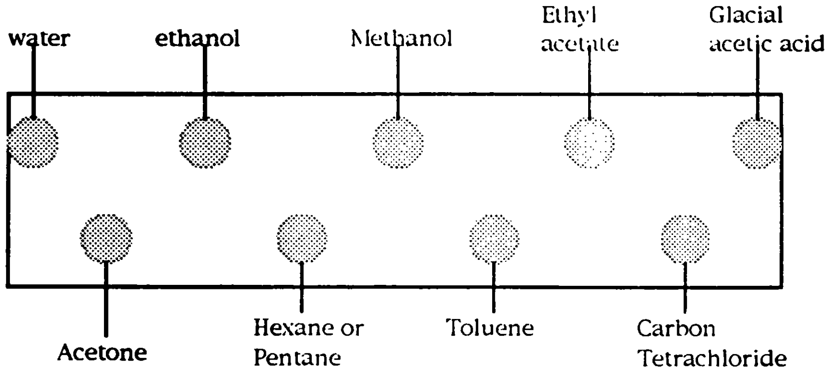


Fig. 4.21 Recrystalliztion solvent "spot-plate" technique

(Apapted from . Chem. Educ; 1989, 66(1), 88)

vii. Determination of boiling point in small scale

Since small scale preparations generally yield quantities of liquid products in the range 30-70 μL, the collection of 5-μL or less to boiling point measurements becomes highly desirable.

Procedure

Micro boiling points can conveniently be determined in standard (90 mm long) Pyrex glass capillary melting point tubes. The sample (3-4 μL) is placed into capillary (sealed at one end) via 10 μL syringe and centrifuged to the bottom.

A small glass bell is formed by heating 3-mm Pyrex tubing with a micro burner and drawing it out to a diameter small enough to be readily accepted by the melting point capillary. A section of the drawn capillary is fused and then cut to yield two small glass bells approximately 5-mm long (Figure 4.22 a). It is important that the fused section be reasonably large. This section is more than just a seal. The fused glass must add sufficient weight to the bell so that it will firmly seat itself in the bottom of the melting point tube. One end of the glass bell is inserted into the loaded melting point capillary, open end down, and allowed to fall to the bottom. The assembled system (Figure 4.22 b) is then inserted into the melting point apparatus.

The temperature is readily raised to 15-20 °C below the expected boiling point (the temperature should be monitored carefully in the case of unknown substances) and then adjusted to a 2 °C/min rise rate until a fine stream of bubbles is emitted from the glass bell. The heat control is then adjusted to drop the temperature. The boiling point is taken at the point where the last escaping bubble collapses (i.e. when the vapour pressure of the substance equals the atmospheric pressure). This procedure may then be repeated several times.

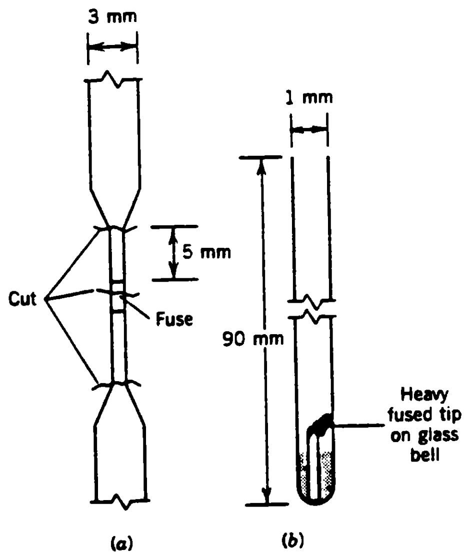


Fig. 4.22 (a) Preparation of small glass bell for ultra micro boiling point determination (b) Assembled system

(Adapted from Microscale Organic Laboratory by D.W Mayo, R.m Pike and S.S Butcher 2nd Ed.)

4.3 Procedure used in the small scale organic laboratory

4.3.1 The sample selected

A sample of 140 Glasgow University Chemistry-1 students of 1993-94 Session was selected to study their attitudes towards small scale experiments.

4.3.2 Experiments selected

From the Organic Chemistry First Year experiments the following two experiments were modified to microscale (**See Appendices** 1a and 1b) .

i. **Experiment-2** The aldehyde and ketone functional group (oxidation of an unknown secondary alcohol and then preparation of solid imine derivative).

ii. **Experiment-3** The carboxylic acid and ester functional groups (identification of an unknown ester).

4.3.3 Small scale manual

In the microscale manual only the amounts of chemicals were changed to microscale. Therefore, pages 10 and 11 (for experiment 2) and pages 16 and 17 (for experiment 3) of the existing manual were replaced by pages containing changes for microscale experiments **(See appendices 2a and 2b).**

Students were asked to do their Small Scale experiments according to the instructions given in the additional pages of the existing manual, using the Small Scale kit (Quickfit and Quartz Limited) in pairs for experiments 2 & 3.

4.4 Measurement of the attitudes of students towards small scale organic experiments

The main aim of this research was to find the attitudes of students towards small scale organic experiments and so to determine that small scale organic experiments are workable in terms of, handling of apparatus, risk involved, measurement of quantities, the help of lab staff needed by students and procedures involved in it. The responses about all these aspects were obtained through a questionnaire for students as well as one for demonstrators.

4.4.1 The design of the questionnaires

Questionnaires for students (Figure 4.23) and demonstrators (Figure 4.24) were designed containing six questions meant to measure, the overall attitudes of students about different aspects of the small scale experiments. Students' responses to the questionnaire are displayed in figure 4.25.

STUDENT'S EVALUATION OF MICROLAB. WORK

This questionnaire is seeking information about your experience in the present laboratory(microscale organic laboratory) work.

Please answer each question carefully and accurately. If you feel you cannot answer a particular question, leave it and go to the next question. Your views are anonymous.

Circle the number which closely corresponds to your view about each statement.

1- I found the handling of the apparatus(i.etime spent on setting up, operation and cleaning)

very easy	easy	reasonable	difficult	very difficult
1	2	3	4	5

2- I found the risks(of spillage, breakage, skin exposure and jamming of glassware)when working in the microlab

very high	high	reasonable	difficult	very difficult
1	2	3	4	5

3- I found that measuring the quantities of chemicals required was

very easy	easy	reasonable	difficult	very difficult
1	2	3	4	5

4- When working in the microlab. the help I needed from the lab. staff was

a lot	considerable	a little	v. little
1	2	3	4

5- I found that separating and purifying the product in the microlab was

very easy	easy	neither easy nor difficult	difficult	v. difficult
1	2	3	4	5

6- Overall as a lab. experience, I would rate this work as, (circle any three of the following)

Interesting	fiddly	simple	challenging	time consuming	time saving
1	2	3	4	5	6

Fig 4.23 Questionnaire for the students

DEMONSTRATOR'S DIARY

We need your help in evaluating the work of students working in the microscale lab. compared to those in the ordinary scale lab. The students are not in a position to make such a comparison.

1- Compared to ordinary scale lab. the handling(time spent on setting up, cleaning up, and completing the experiment) in the microscale lab. was
much shorter shorter similar longer much longer

2- Compared to ordinary scale lab. the microscale lab. risks(spillage, fire, breakage, skin exposure and jamming of glassware) were
much higher higher similar lower much lower

3- Compared to the ordinary scale lab. the time for measuring chemicals in microscale lab. was
much shorter shorter same longer much longer

4- Compared to ordinary lab. the help asked by the students in the microscale lab. was
a lot more a little more similar less a lot less

5- Compared to ordinary lab. the need for careful work in the experiments in the microscale lab. was
much higher higher same lower much lower

6- Compared to the students doing this experiment in the ordinary scale lab. the students doing the same experiment in the microscale lab. have
gained a lot more gained a little more neither gained nor lost lost out a little more lost out a lot more

Fig. 4.24 Questionnaire for the demonstrators

Q1 Handling of apparatus	Very easy 06(4%)	Easy 26(19%)	Reasonable 77(55%)	Difficult 22(16%)	Very difficult 08(8%)	
Q2 Risk involved	Very high 02(2%)	High 21(17%)	Reasonable 85(67%)	Low 19(15%)	Very low 01(1%)	
Q3 Measurement of chemicals	Very easy 06(5%)	Easy 53(41%)	Reasonable 64(50%)	Difficult 06(5%)	Very difficult Nil	
Q4 Staff help	Very little 07(5%)	A little 68(44%)	reasonable 47(30%)	A lot 05(3%)	Quite a lot Nil	
Q5 Separation/ purification	Very easy 03(2%)	Easy 19(15%)	Neutral 63(49%)	Difficult 39(30%)	very difficult 05(4%)	
Q6 Rating of the overall lab experience (to tick any three)	Interesting 43(17%)	Fiddly 45(18%)	Simple 12(5%)	Challenging 64(25%)	Time consuming 89(35%)	Time saving 04(2%)

Fig 4.25 The overall picture of the students' attitudes toward small scale experiments

4.4.2 Student responses

The sample of students involved in this research showed the following responses:

i. In favour of small scale

Almost half of the total sample of students showed responses in favour of small scale experiments in question 3 and 4. These students agreed that small scale experiments do not require much help from the laboratory staff. They also found that to measure chemicals on such a scale is fairly easy.

ii. Small scale are "reasonable"

A substantial number of students (almost half of the total sample) expressed their views in questions 1, 2, 3 and 5 that small scale experiments are "reasonable". These students agreed that the handling of apparatus, risk involved, measurement of chemicals and separation/purification of a compound obtained in small scale experiments was reasonable.

iii. Against small scale

In questions 1 and 5, a minority of students regarded handling of apparatus and separation/purification of a compound obtained in small scale experiments would be difficult.

iv. The overall lab experience

In question 6, students were asked to express their views with regard to the overall experience gained during their work on organic microscale experiments.

It seems to be that one third of the sample showed interest in doing small scale experiments. Also, one third of the sample regarded organic small scale experiments as fiddly. More than one third of the sample regarded organic small scale experiments as challenging. However, two thirds of the sample expressed their views that organic small scale experiments were time consuming.

4.5 The researcher's views about students' responses

In question 1, although it is thought that students would find difficulties in handling small scale apparatus, in fact the outcome of the results were moderate and students found such apparatus was reasonable to handle. However, this was not very clear in question 6, as one third of the sample mentioned that such a scale of apparatus was fiddly.

In question 2, it was expected that students would find low risk involved, in terms of hazards and breakage of apparatus, in small scale experiments. Indeed, the outcome of the measurements supported the expectation.

In question 3, students agreed that small scale experiments are either easy or reasonable in terms of the measurement of chemicals. This was interesting as students, in small scale experiments, deal with very small amounts of chemicals which are not easy to measure. However, students found it easy to deal with such a scale of measurement.

In question 4, the outcome of the students' attitudes were as expected since small scale experiments would require little help from the staff. The students agreed that there was little help required from the lab staff when dealing with small scale experiments.

Question 5 involves a chemical technique to be carried out on the product of any small scale experiments. As the product would be small, therefore, it is thought that students would find this process hard. The outcome of this measurement showed that the majority of students were 'neutral' in this regard. In other words, such a chemical process was no problem even when carried out in small scale. However, there were some students who found such a technique was difficult.

Students were interested in the organic small scale experiments, as displayed in question 6; they also found that such a scale of experiments is challenging. This could have a certain advantage on the overall performance of students in the laboratory and on the learning process(89).

But, students also found that small scale organic experiments were time consuming and fiddly. However, this may present no disadvantage to the process of learning. It could also have been because they were meeting the techniques for the first time. Experimental time did not allow students to meet the techniques more often.

4.6 Opinion of demonstrators about small scale experiments

Besides the students' attitude the demonstrators' attitude toward small scale experiments was also important in order to gain a complete picture about small scale experiments. Seven demonstrators were involved in the running of small scale organic experiments in the laboratory. As mentioned in the previous section of this chapter, a questionnaire was designed (Figure 4.24) containing six questions aimed at studying the demonstrators point of views toward small scale experiments. The responses of demonstrators are shown in figure 4.26.

Q1 The time taken in completing the experiment in the small scale lab compared to ordinary scale lab was	Much shorter –	Shorter 01	Similar 06	Longer –	Much longer –
Q2 The risk involved in small scale lab compared to ordinary scale lab was	Much higher –	Higher –	Similar 04	Lower 03	Much lower –
Q3 The time for measuring chemicals in small scale lab compared to ordinary scale lab was	Much shorter –	Shorter –	Same 06	Longer 01	Much longer –
Q4 The help askedby the students in the micro lab compared to ordinary lab was	A lot more –	A little more 01	Similar 06	Less –	A lot less –
Q5 The need for careful work in the small scale lab compared to ordinary lab was	Much higher 01	Higher 04	Same 02	Lower –	Much lower –
Q6 Compared to the students doing this experiment in the ordinary scale lab the students doing the same experiment in the small scale lab have	Gained a lot more –	Gained a little more –	Neither gained nor lost 05	Lost out a little more 02	Lost out a lot more –

Fig 4.26 The overall picture of the demonstrators' evaluation of the small scale lab work compared to ordinary scale lab work

The demonstrators found that there is no difference in time whether students perform small scale organic experiments or normal scale conventional organic experiments. However, demonstrators' opinions were divided with regard to the risk involved in small scale organic experiments. 3 demonstrators agreed low risk would be involved in small scale experiments, while 4 others believed that the risk would be the same when compared to ordinary conventional scale organic experiments.

Demonstrators found that the time for measuring chemicals in small scale laboratory would not be much different than the time for measuring chemicals in normal scale laboratory. They could not notice a difference in the amount of help needed by students in the small scale laboratory. Demonstrators found that more careful work is needed if small scale experiments are carried out. They thought that students would not gain or lose in general if working on small scale experiments.

4.7 The researchers' views about demonstrators' responses

In question 1, the demonstrators' responses were not astonishing as in every organic laboratory, whether it is macro or micro, a product is required to be produced at the end of each experiment and therefore would take a similar time.

In question 2, although the demonstrators' opinions were scattered, it was apparent that almost half of the demonstrators agreed that the risk involved in small scale experiments would be lower than normal scale experiments.

In questions 3 and 4, it was expected that students would take more time to measure very small amounts of chemicals required for experiments. It was also rational to think that students, as for the first time confronting microscale techniques, would approach the demonstrators seeking for help.

As a small scale laboratory, it is thought that students would require more careful work especially as they were presented with such a lab for the first time. The outcome of the demonstrators' opinions in question 5 was expected.

Finally, demonstrators thought that small scale experiments did no harm to students. In other words, it did not bring any confusion or practical difficulty expected from the first year students in the organic laboratory. This could be considered as a score for small scale experiments as being performed in such a laboratory for the first time.

4.8 Summary

As it was the first time that students were required to perform two experiments in a small scale, it is to be expected that students and staff will experience some difficulty. However, the findings of this chapter were in support of or at least not against small scale experiments. Students experienced some challenging tasks when working on such a scale, besides a good number of them were interested in performing organic microscale experiments. On the other hand, demonstrators found themselves more busy when working with organic microscale due to the novelty of such a scale for everyone involved.

In general, microscale organic chemistry would do no harm to the teaching and learning process, as supported by the findings in the present chapter. It would be prudent to use such a technique or at least to promote and encourage it for its other logistical advantages of cost and safety.

CHAPTER FIVE

Modification of General Chemistry-1 Inorganic Experiments to Small Scale

5.1 Introduction

In the early 1980's, it was suggested(9) that much of the lab programme should be replaced with video tapes, computer simulations and demonstrations in order to overcome a number of problems like chemical hazards, storage space, chemical costs, wastes disposal etc. that beset chemistry teachers and students.

For those who believe the students should still do their chemistry practical in the laboratory, the microscale chemical approach offers an attractive alternative. One noticeable feature in the novelty of microscale experimentation is that students can work while seated. Once they have collected their reagents, they can sit comfortably to perform their experimental work and complete their lab report book. Since many microscale inorganic experiments are performed by spot methods on a polythene transparent sheet, in well plates or petri dishes the bench is not cluttered. Moreover, since the quantity of material used in microscale experiments is quite small, hazardous materials can easily be handled on an open bench instead of fume-hoods. Hence, students do not need to wait in queues to perform their experiments involving hazardous materials, in fume-hoods. In this way they save not only time but avoid frustration as well.

5.2 Aims of the modification of General Chemistry-I inorganic experiments into small scale

Since it appears that no study has been done about the impact of small scale inorganic chemistry on student learning, it is prudent to state the main research aims or purposes at the beginning of the present chapter. These aims lead to the main research approaches which were used in the study.

Three main aims for this research:

I. To study whether by using small scale techniques students will obtain similar marks in their experiments as their marks when doing experiments on conventional scale. In other words, to check whether inorganic microscale chemistry is a hindrance to their laboratory achievement.

II. To study the attitude of students toward small scale inorganic experiments based upon a questionnaire and on the basis of the extensive observations of the researcher.

These aims were assigned at the beginning of the research hoping that the picture would be complete and all the queries would be clarified and solved at the end of the present study.

5.3 Apparatus used

The main apparatus used in the present study were:

a. transparent polythene sheets (4 x 4 inch)
b. Pasteur pipettes (Capillary pipettes)
c. Beral pipettes
d. small test tubes (4 mL)
e. well-plates

5.3.1 Transparent polythene sheet

Each polythene sheet is a quarter of A4 sheet size (10 x 10 cm). See figure 5.1. By means of Pasteur pipette drops of solutions are placed on the polythene transparent sheets. Students are asked to mix drops of different solutions for chemical reactions on the polythene sheets and observe the change of colour and formation of precipitates. Coloured precipitates could be clearly seen by students by putting white paper under the polythene sheet and white precipitates could be clearly seen by putting the sheet on the surface of bench (dark brown).

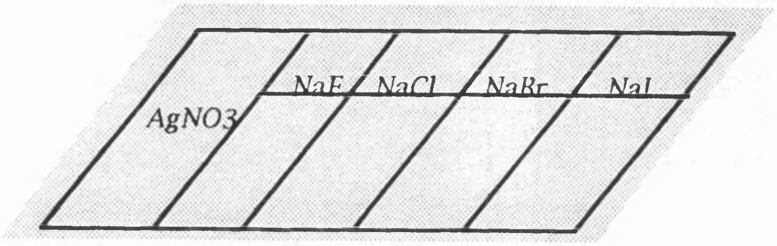


Fig. 5.1 Transparent polythene sheet

5.3.2 Pasteur pipette

It is also called capillary pipette. It is a simple glass tube with the end drawn to a fine capillary and are filled using a small rubber bulb or one of the very handy commercially available pumps. These pipettes can hold 0.5 to 2.0 mL of liquid (Figure 5.2). They are used for the transfer of liquids from one vessel to another and for placing drops of solutions on polythene sheets.

Fig. 5.2 Pasteur pipette

5.3.3 Beral pipettes

This is found as standard, jumbo, microtip, and graduated. It is a one-piece plastic disposable pipette used for the transfer of liquids. They are used to deliver a number of drops per millilitre (Figure 5.3)

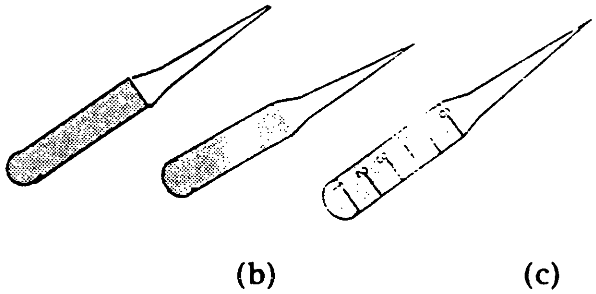

(a) (b) (c)

Fig. 5.3 Beral pipettes (a) Jumbo (b) Microtip and (c) Graduated

5.3.4 Small test tube

These test tubes can hold from 2.0-4.0 mL of liquids and are used for mixing different liquids for chemical reactions, and obtaining layers of immiscible liquids. They can easily be used as a set of five tubes in an audio cassette transparent box. See figure 5.4.

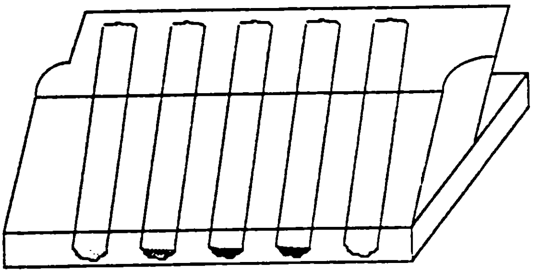


Fig. 5.4 A set of five small test tubes in an audio cassette

5.3.5 Well-plate

These plates of dimensions 12 cm by 8 cm are available in different sizes and number of wells. The preferred reaction vessels are the 0.3 mL wells also available in strips of 8 or 12 wells, which can be mounted in a frame. Simple reactions such as single or double displacement reactions, can be accomplished in the wells satisfactorily with volumes of 0.1- 0.2 mL. The larger well-plates are used for collecting reagents. See figure 5.5.

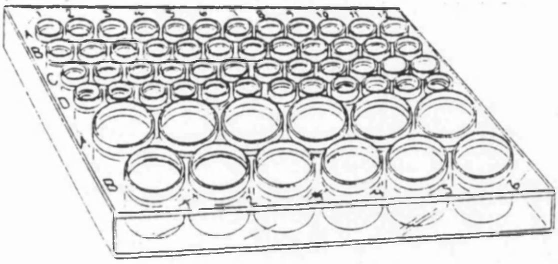


Fig. 5.5 A well-plate with 48 x 0.3 mL wells and 12 x 3.5 mL wells

5.4 Practical procedures used in the inorganic small scale laboratory

5.4.I Experiments selected

From the general chemistry-I inorganic experiments the following experiments were selected for modification from normal scale to microscale (**Appendix 3 (a), (b), and (c)**):

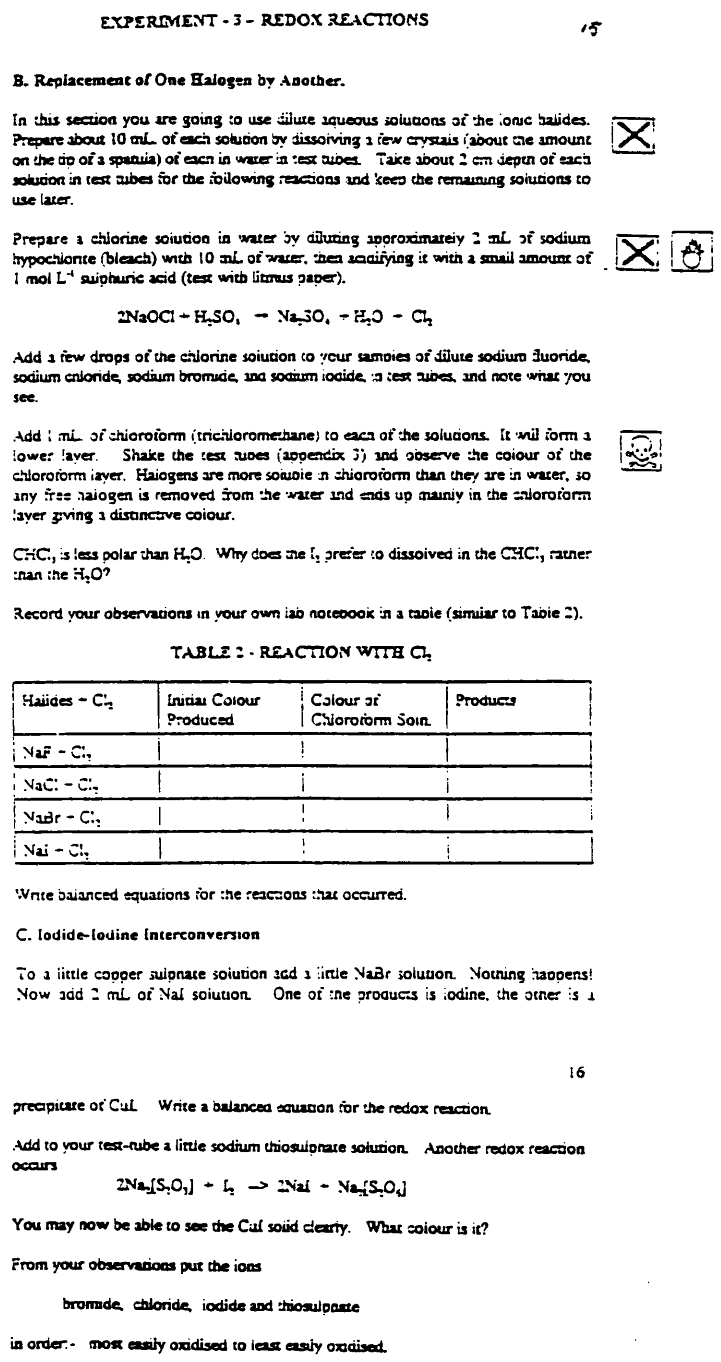
a. Experiment 3 part B and C
b. Experiment 5 all parts
c. Experiment 7 part A

5.4.2 Modifications of the manual

The selected experiments were designed and modified to small scale and therefore, a new modified manual was introduced to the students in place of the old one (**Appendices 4a, b, and c**) .

a. The old manual

Experiment-3 part B and C from the old is shown in figure 5.6 as follows:



EXPERIMENT - 3 - REDOX REACTIONS

15

B. Replacement of One Halogen by Another.

In this section you are going to use dilute aqueous solutions of the ionic halides. Prepare about 10 mL of each solution by dissolving a few crystals (about the amount on the tip of a spatula) of each in water in test tubes. Take about 2 cm depth of each solution in test tubes for the following reactions and keep the remaining solutions to use later.

Prepare a chlorine solution in water by diluting approximately 2 mL of sodium hypochlorite (bleach) with 10 mL of water, then acidifying it with a small amount of 1 mol L^{-1} sulphuric acid (test with litmus paper).

$$2NaOCl + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + Cl_2$$

Add a few drops of the chlorine solution to your samples of dilute sodium fluoride, sodium chloride, sodium bromide, and sodium iodide, in test tubes, and note what you see.

Add 1 mL of chloroform (trichloromethane) to each of the solutions. It will form a lower layer. Shake the test tubes (appendix 3) and observe the colour of the chloroform layer. Haiogens are more soluble in chloroform than they are in water, so any free halogen is removed from the water and ends up mainly in the chloroform layer giving a distinctive colour.

$CHCl_3$ is less polar than H_2O. Why does the I_2 prefer to dissolved in the $CHCl_3$ rather than the H_2O?

Record your observations in your own lab notebook in a table (similar to Table 2).

TABLE 2 - REACTION WITH Cl_2

Halides + Cl_2	Initial Colour Produced	Colour of Chloroform Soln.	Products
$NaF + Cl_2$			
$NaCl + Cl_2$			
$NaBr + Cl_2$			
$NaI + Cl_2$			

Write balanced equations for the reactions that occurred.

C. Iodide-Iodine Interconversion

To a little copper sulphate solution add a little NaBr solution. Nothing happens! Now add 2 mL of NaI solution. One of the products is iodine, the other is a

16

precipitate of CuI. Write a balanced equation for the redox reaction.

Add to your test-tube a little sodium thiosulphate solution. Another redox reaction occurs

$$2Na_2[S_2O_3] + I_2 \rightarrow 2NaI + Na_2[S_4O_6]$$

You may now be able to see the CuI solid clearly. What colour is it?

From your observations put the ions

bromide, chloride, iodide and thiosulphate

in order:- most easily oxidised to least easily oxidised.

Fig. 5.6 The design of experiment-3 part B and C in the old manual.

b. The new laboratory manual

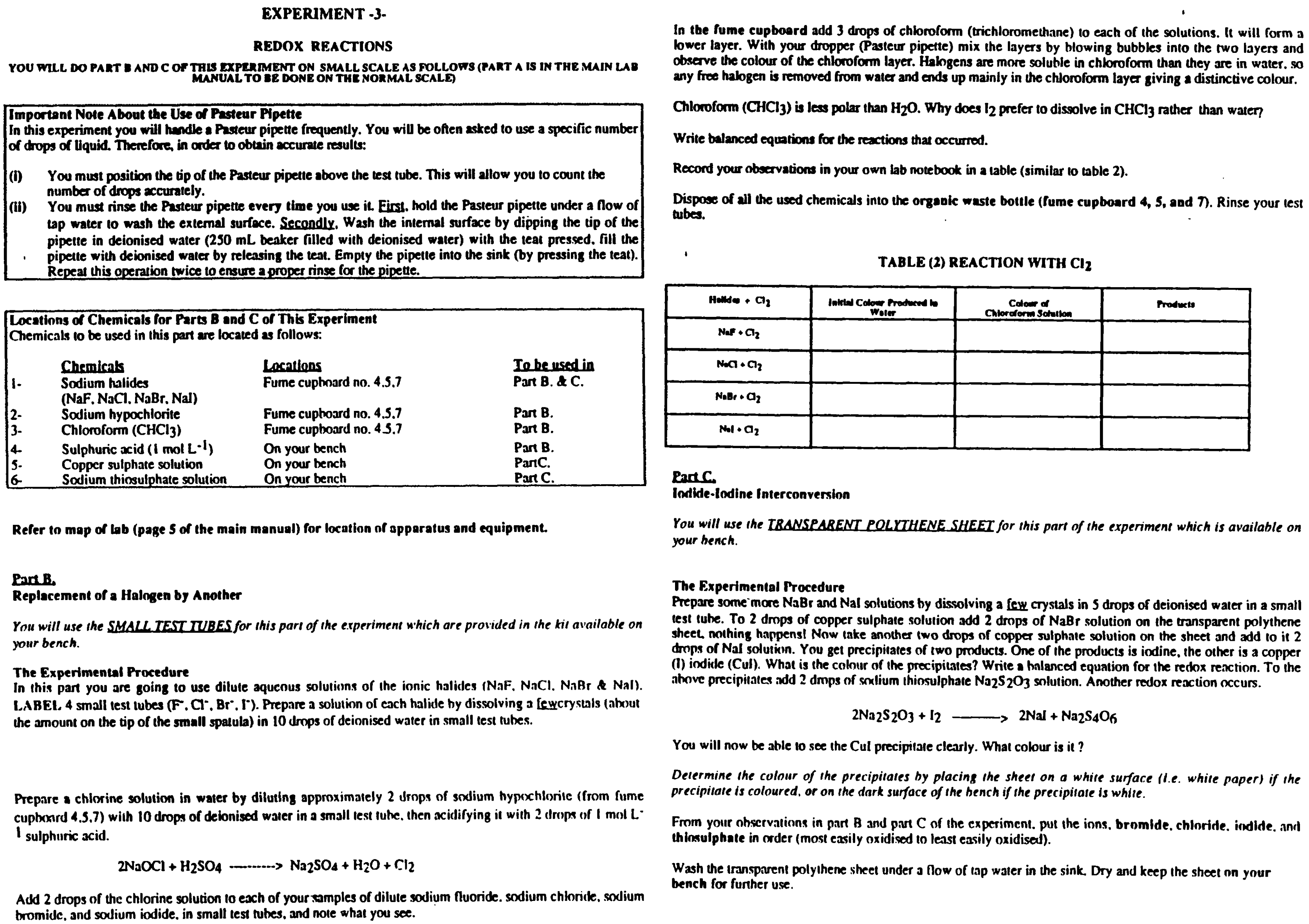
A new lab manual for the selected experiments modified to microscale was introduced to the general chemistry-I students. Since general chemistry-I students vary in their background of chemistry from those who have very little knowledge of chemistry to those who have satisfactory background, the new manual was prepared with a view to providing students with good written instructions which give them information not only about the experimental procedures but also about the lab techniques involved in the experiments.

Generally, the modifications were partly about small scale experiments and partly about writing the manual to incorporate other educational advantages.

It is possible to summarise which techniques were used in the modifications of the manual in the following main points:

1. Dividing the written material into sections which are easily managed by students.
2. Giving complete notes about the use of apparatus, location of reagents and their use in the relative experiment.
3. Showing special instruction by *Italics*, emphasis by underlining, **bold** type and CAPITAL letters.

The Design of Experiment 3 part B and C in the new manual is shown in figure 5.7. The rest of the selected experiments modified to small scale (experiment 5 and experiment 7 part A) in the new manual could be seen in Appendix (2a) and (2b).



EXPERIMENT -3-

REDOX REACTIONS

YOU WILL DO PART B AND C OF THIS EXPERIMENT ON SMALL SCALE AS FOLLOWS (PART A IS IN THE MAIN LAB MANUAL TO BE DONE ON THE NORMAL SCALE)

Important Note About the Use of Pasteur Pipette
In this experiment you will handle a Pasteur pipette frequently. You will be often asked to use a specific number of drops of liquid. Therefore, in order to obtain accurate results:

(i) You must position the tip of the Pasteur pipette above the test tube. This will allow you to count the number of drops accurately.

(ii) You must rinse the Pasteur pipette every time you use it. First, hold the Pasteur pipette under a flow of tap water to wash the external surface. Secondly, Wash the internal surface by dipping the tip of the pipette in deionised water (250 mL beaker filled with deionised water) with the teat pressed, fill the pipette with deionised water by releasing the teat. Empty the pipette into the sink (by pressing the teat). Repeat this operation twice to ensure a proper rinse for the pipette.

Locations of Chemicals for Parts B and C of This Experiment
Chemicals to be used in this part are located as follows:

	Chemicals	Locations	To be used in
1-	Sodium halides (NaF, NaCl, NaBr, NaI)	Fume cupboard no. 4,5,7	Part B. & C.
2-	Sodium hypochlorite	Fume cupboard no. 4,5,7	Part B.
3-	Chloroform ($CHCl_3$)	Fume cupboard no. 4,5,7	Part B.
4-	Sulphuric acid (1 mol L^{-1})	On your bench	Part B.
5-	Copper sulphate solution	On your bench	PartC.
6-	Sodium thiosulphate solution	On your bench	Part C.

Refer to map of lab (page 5 of the main manual) for location of apparatus and equipment.

Part B.
Replacement of a Halogen by Another

You will use the SMALL TEST TUBES for this part of the experiment which are provided in the kit available on your bench.

The Experimental Procedure
In this part you are going to use dilute aqueous solutions of the ionic halides (NaF, NaCl, NaBr & NaI). LABEL 4 small test tubes (F^-, Cl^-, Br^-, I^-). Prepare a solution of each halide by dissolving a few crystals (about the amount on the tip of the small spatula) in 10 drops of deionised water in small test tubes.

Prepare a chlorine solution in water by diluting approximately 2 drops of sodium hypochlorite (from fume cupboard 4,5,7) with 10 drops of deionised water in a small test tube, then acidifying it with 2 drops of 1 mol L^{-1} sulphuric acid.

$$2NaOCl + H_2SO_4 \text{ ---------> } Na_2SO_4 + H_2O + Cl_2$$

Add 2 drops of the chlorine solution to each of your samples of dilute sodium fluoride, sodium chloride, sodium bromide, and sodium iodide, in small test tubes, and note what you see.

In the fume cupboard add 3 drops of chloroform (trichloromethane) to each of the solutions. It will form a lower layer. With your dropper (Pasteur pipette) mix the layers by blowing bubbles into the two layers and observe the colour of the chloroform layer. Halogens are more soluble in chloroform than they are in water, so any free halogen is removed from water and ends up mainly in the chloroform layer giving a distinctive colour.

Chloroform ($CHCl_3$) is less polar than H_2O. Why does I_2 prefer to dissolve in $CHCl_3$ rather than water?

Write balanced equations for the reactions that occurred.

Record your observations in your own lab notebook in a table (similar to table 2).

Dispose of all the used chemicals into the organic waste bottle (fume cupboard 4, 5, and 7). Rinse your test tubes.

TABLE (2) REACTION WITH Cl_2

Halide + Cl_2	Initial Colour Produced in Water	Colour of Chloroform Solution	Products
$NaF + Cl_2$			
$NaCl + Cl_2$			
$NaBr + Cl_2$			
$NaI + Cl_2$			

Part C.
Iodide-Iodine Interconversion

You will use the TRANSPARENT POLYTHENE SHEET for this part of the experiment which is available on your bench.

The Experimental Procedure
Prepare some more NaBr and NaI solutions by dissolving a few crystals in 5 drops of deionised water in a small test tube. To 2 drops of copper sulphate solution add 2 drops of NaBr solution on the transparent polythene sheet, nothing happens! Now take another two drops of copper sulphate solution on the sheet and add to it 2 drops of NaI solution. You get precipitates of two products. One of the products is iodine, the other is a copper (I) iodide (CuI). What is the colour of the precipitates? Write a balanced equation for the redox reaction. To the above precipitates add 2 drops of sodium thiosulphate $Na_2S_2O_3$ solution. Another redox reaction occurs.

$$2Na_2S_2O_3 + I_2 \text{ ---------> } 2NaI + Na_2S_4O_6$$

You will now be able to see the CuI precipitate clearly. What colour is it ?

Determine the colour of the precipitates by placing the sheet on a white surface (i.e. white paper) if the precipitate is coloured, or on the dark surface of the bench if the precipitate is white.

From your observations in part B and part C of the experiment, put the ions, **bromide**, **chloride**, **iodide**, and **thiosulphate** in order (most easily oxidised to least easily oxidised).

Wash the transparent polythene sheet under a flow of tap water in the sink. Dry and keep the sheet on your bench for further use.

Fig. 5.7 The design of experiment-3 part B and C in the new manual.

5.4.3 The sample of the students selected for the study

A sample of 214 first year university general chemistry students was selected for the present study to do the small scale experiments. Twelve students of the sample had been exempted from doing labs because they were repeaters of first year general chemistry. Therefore, the sample comprised of 202 students, but 197 students actually attended the lab during the study. These students had the following range of academic backgrounds:

I. None

These students have no chemistry background whatsoever when they join university.

II. Module

These students have achieved at least a limited course of study or a module including some chemistry before joining university.

III. Standard

These students have four years of study of chemistry before joining university having passed Standard Grade.

IV. Higher

These students have five years of studies of chemistry before joining university having passed Higher Grade.

V. Others

These students have different levels of chemistry background. Some have good and some have poor chemistry background when they join university.

The picture of the qualifications and the respective number of all students in first year general chemistry is shown in table 5.1.

TABLE -5.1-
The Qualifications of all Students in the First Year General Chemistry

QUALIFICATIONS	NO. OF STUDENTS
None	24
Module	34
Standard	38
Higher	83
Other	18
Total	197

5.4.4 The empirical work

The results obtained from the general chemistry students with regard to their achievements in small and normal scale experiments are explained as follows (experiment 5 comes before 3 as it was come first in the actual lab work);

I. Experiment (5) "Periodic Table Trends"

This experiment was performed on small scale by only 19 students (i.e. one group only), while the rest of students did this experiment on normal scale.

The researcher could not find an access to more than one lab. session for this particular experiment and therefore 48 students were excluded from his sample.

The achievement of the 19 students (marks % they obtained) in the experiment on the small scale was **84%**, while the achievement of the rest of students (130 students) who did the same experiment but on normal scale was **81%.**

There was apparantly no difference between the two groups in the students' achievement.

II. Experiment (3) "Redox Reactions"

This experiment was performed by **all** the 7 groups of the general chemistry students (results obtained from 129 students) on small scale. The achievement of students in this experiment on small scale was **92%.** It is obviously high when compared to other experiments which have been done on normal scale and so the microscale approach has not disadvantaged them.

III. Experiment (7) "Complexes"

Only Part A of this experiment was modified into small scale. The experiment was performed by **all** the 7 groups of general chemistry students (results obtained is from 111 students). The achievement of students was **93%.** The achievement is again high when compared to other experiments as shown in **figures 5.8 to 5.12** which have been done on normal scale.

5.4.5 Points for discussion

It is worth to mentioning some points concerning what has been done so far in the lab:

1. The only experiment which could be considered as it has been properly done on small scale is experiment 3 as compared to experiments 5 and 7.

2. Experiment 5 was performed by very few students on small scale which made the comparison to normal scale difficult. Also, experiment 7 has been performed in a way which

prevented a significant comparison to occur. Part B of this experiment was done on paper only while Part A was performed on small scale.

5.4.6 Results obtained from all General Chemistry students

The aim of this section is to exhibit and to discuss the achievements obtained from each group of the General Chemistry Students in the three microscale experiments.

I. Achievements obtained from all General Chemistry students groups in Experiment-3

It is very noticeable that the best marks obtained by students in this experiment on small scale belonged to the "HIGHER" group. These students came with a good background in chemistry which could be the reason why they managed to show a good performance in doing this experiment.

The "STANDARD" group gained the second place in performing experiment 3. These students have also a good background in chemistry. The "MODULE" group is third and this could be due to what chemistry modules they took during their previous years in education(fig. 5.9 and 5.10 page 78)

The groups which fell behind were "NONE" and "OTHER". It is apparent from the achievements of these groups in this experiment that the "NONE" group, a group with no chemistry background, did better than the group "OTHER" which comprises of some students who did chemistry before. The picture of the achievements of all groups separately in this experiment is displayed in figures 5.8, 5.9, 5.10, 5.11, and 5.12 successively.

TABLE -5.2-

The achievement of all General Chemistry students in the experiments (No. 197)

	NONE. (No. of Students)	MOD. (No. of Students)	HIGH. (No. of Students)	STAND. (No. of Students)	OTHER (No. of Students)
Expt.1 (N) % Marks	91.0 (21)	88.5 (28)	90.5 (67)	91.6 (34)	90.3 (14)
Expt.2 (N) % Marks	86.9 (20)	88.0 (27)	82.8 (71)	83.6 (33)	87.2 (16)
Expt.3 (S) % Marks	87.7 (17)	90.9 (27)	92.7 (53)	91.8 (20)	82.7 (12)
Expt.4 (N) % Marks	81.3 (23)	81.4 (29)	81.4 (69)	82.9 (28)	82.7 (15)
Expt.5 (S) % Marks	85.7 (1)	92.9 (1)	88.8 (14)	—	54.8 (3)
Expt.5 (N) % Marks	73.8 (18)	81.3 (24)	83.6 (51)	90.9 (27)	77.8 (10)
Expt.7 (S) % Marks	87.5 (14)	97.5 (21)	94.5 (45)	88.8 (20)	92.0 (11)
Expt.7 (N) % Marks	81.2 (14)	85.1 (21)	90.0 (45)	88.1 (20)	86.4 (11)

II. Achievements obtained from all General Chemistry students groups in Experiment-5

Although the picture of the groups' achievement in experiment 5 (small scale) looks similar to the picture in experiment 3, it is not possible to draw a good comparison due to the limited number of students who did experiment 5 on the small scale. The picture of the achievements of all groups separately in this experiment is displayed in figures 5.8, 5.9, 5.10, 5.11, and 5.12 successively.

III. Achievements obtained from all General Chemistry students groups in Experiment-7

The “MODULE” group of students have obtained the highest marks in this experiment and the “HIGHER” group comes in the second place, while the “OTHER” comes in the third place. The picture of the achievements of all groups separately in this experiment is displayed in figures 5.8, 5.9, 5.10, 5.11 and 5.12 successively. In general, the students in all of these groups have obtained high marks as compared to their achievements in experiment 7 Part A on the normal scale. However, not all students performed experiment 7 Part A on the normal scale (apart from Monday lab students), the rest did it on paper only!

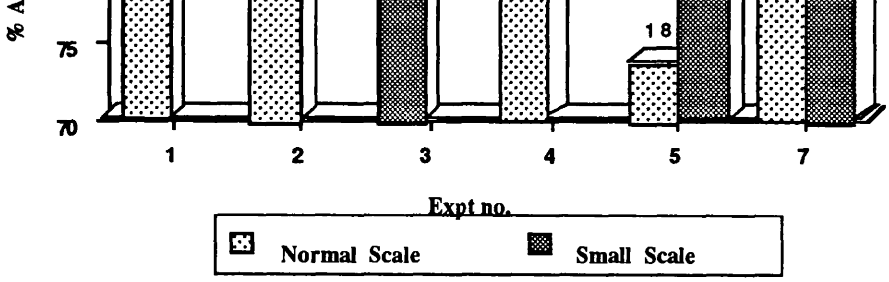


Fig. 5.8 The achievement of the “NONE” group in the experiments

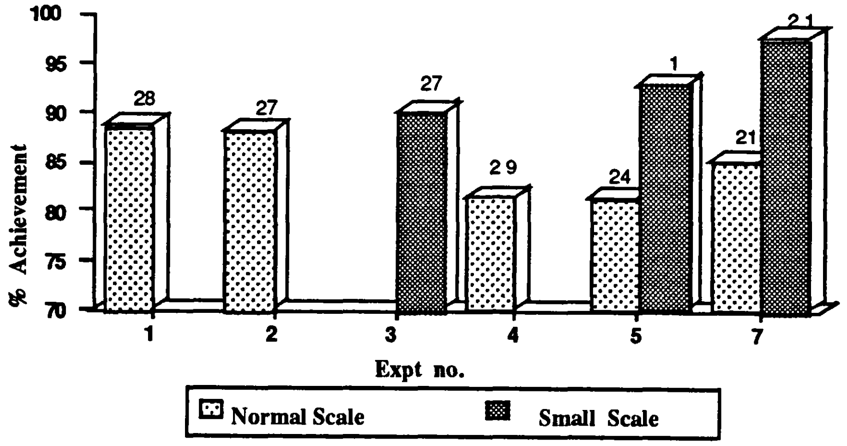


Fig. 5.9 The achievement of the "MODULE" group in the experiments

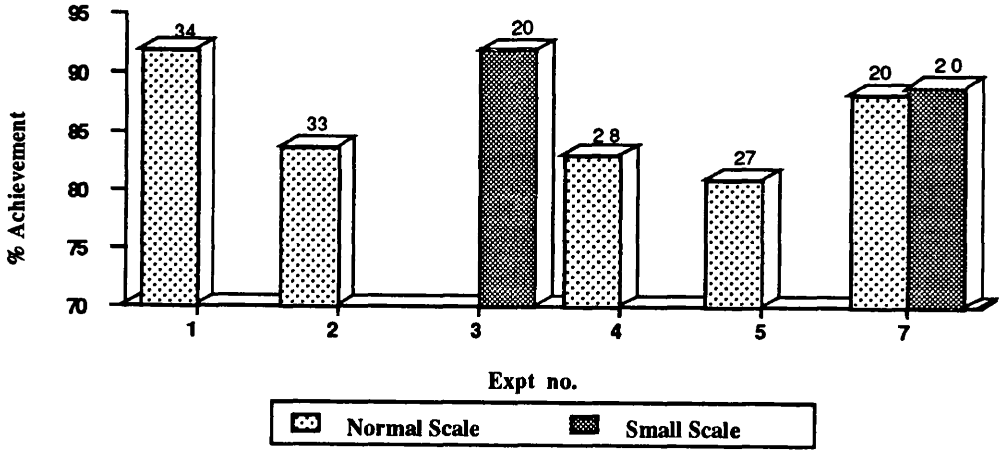


Fig. 5.10 The achievement of the "STANDARD" group in the experiments

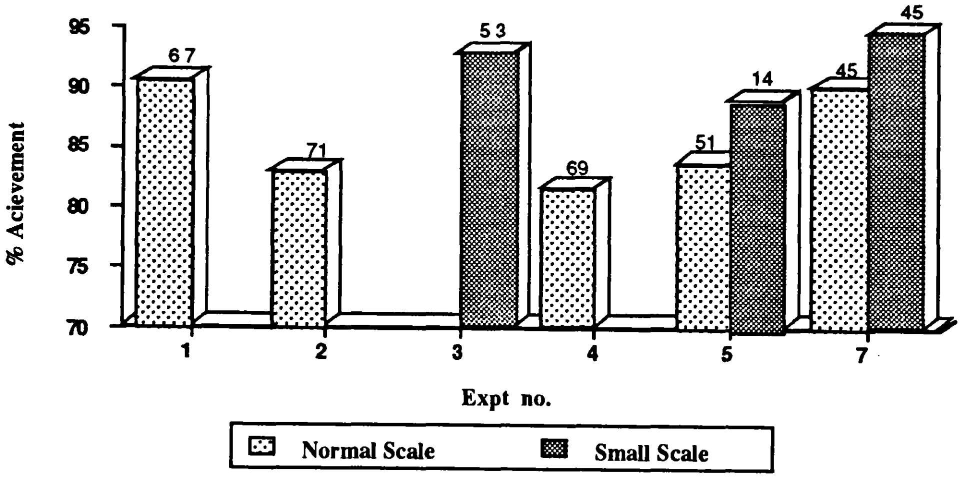


Fig. 5.11 The achievement of the "HIGHER" group in the experiments

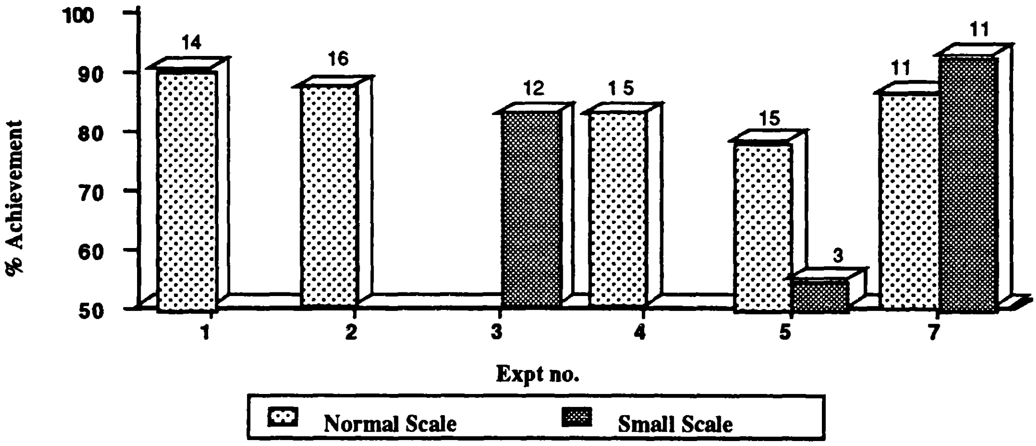


Fig. 5.12 The achievement of the "OTHER" group in the experiments

5.4.7 Summary of the findings

The overall picture of the students achievement on normal and small scale experiments could be shown in table 5.2. **First**, the students achievement which were obtained from their lab note books, in both experiments 3 and 7 on small scale could be seen to be as good as those which were done on normal scale. **Secondly**, the achievement of students in the "MODULE" group has been improved in the case of small scale experiments. Also, the "NONE" group achieved well when small scale experiments were applied. Could it be that small scale experiments give no disadvantage when working in the inorganic chemistry lab?

5.5 Student attitudes towards small scale experiments

It would not be possible to draw a picture of the findings of this research without studying the general attitudes of students toward microscale experiments. One of the aims of this research is to find out whether these small scale experiments are workable, feasible and would sustain the educational aims for a first year inorganic chemistry lab!
If everybody, using small scale techniques, is able to learn, understand and make the progress, why do we not modify all possible experiments in that lab?

5.5.1 The questionnaire design

A questionnaire of 15 questions measuring the different aspects of students attitudes about small and normal scale experiments was designed. The different aspects involved in the questionnaire are those that the researcher intended to study (all aspects are shown in table 5.3). The questionnaire was completed by each student on the last day of each lab. A copy of this questionnaire is to be found in **Appendix 5.**

The researcher managed to obtain 101 responses from his sample. However, on another occasion, he gathered some 67 extra forms (without names) which makes the total of responses 168. A form with the total responses of 101 students is shown in **Appendix 6.** Another form with the total responses of 168 students is shown in **Appendix 7.**

The overall picture (combination of 101 and 168 responses) of the students' attitudes toward small/normal scale experiments is displayed in table 5.3.

TABLE-5.3-
An Overall Summary of Student Attitudes toward Small/Normal Scale Experiments

	Normal Scale rather than Small Scale	Small Scale rather than Normal Scale	No Difference	Normal Scale rather than Small Scale	Small Scale rather than Normal Scale	No Difference
	Total of 168	Total of 168	Total of 168	Total of 101	Total of 101	Total of 101
Q1: Exp as more fun	36	**73**	59	28	**36**	**37**
Q2: Easiness/simplicity	30	**106**	32	20	**61**	20
Q3: Spillage/Breakage	12	**110**	46	8	**66**	27
Q4: Less staff help	11	48	**109**	7	29	**65**
Q5: Interest in chemistry	31	47	**90**	20	27	**54**
Q6: Less time	15	**120**	33	10	**68**	23
Q7 Understanding	21	39	**108**	14	22	**65**
Q8 Less apparatus	7	**136**	25	4	**85**	12
Q9 Safety and relaxation	12	**78**	**78**	7	**46**	**48**
Q10 Concentration	15	55	**98**	12	32	**57**
Q11 Tidiness	7	**131**	30	3	**79**	19
Q12 Understanding of lab manual	19	**76**	**73**	14	**40**	**47**
Q13 Learning	19	47	**102**	13	25	**63**
Q14 Care in handling apparatus	**90**	47	31	**57**	26	18
Q15 Choice between Normal/small Scales	30	**106**	32	20	**60**	21

5.5.2 The student attitudes towards small scale experiments

The responses which have been obtained from the first year General Chemistry students could be divided into the following groups:-

Group 1

This consists of those students whose responses are almost the same to "small scale" and "no difference" in questions 1, 9 and 12 (Table 4.3). They have also shown a very small attitude trend towards the "normal scale", particularly in case of questions 9, and 12.

Although, students did enjoy chemistry experiments on both scales, there is still some attitude trend apparent towards the small scale (Q1). In question 9, students showed "no difference" or little attitude trend towards "small scale" due to the nature of the question itself. The question asked for two matters at the same time, safety and relaxation, and that could be the reason for the scattered responses obtained from this group. The instructions of the small scale experiments in the new manual seemed to be satisfactory. Students found this clear but there is still, however, some attitude trend toward the small scale manual compared to the normal scale manual.

If we group the responses, questions 7, 12, 13 are about learning and the main response is "no difference" or in favour of "microscale" and so the microscale has not caused any obvious deterioration in learning.

Questions 1, 2 and 5 are about "enjoyment" and again the main responses are in the "no difference" and "microscale" categories, showing that microscale has not received a negative response.

Group 2

This consists of those students who have shown high response in favour of "no difference" and moderate response to normal and small scale experiments. This trend of high response to the "no difference" category took place when questions were asked about 'less staff help', 'interest', 'understanding', 'concentration' and 'learning' (i.e. 4, 5, 7, 10, and 13 in table 5.3).

In general, a better picture would have been obtained from the students' attitudes in this group if all experiments in the lab had been performed on small as well as on normal scale!

However, the lesson which could be learnt from this group is that students would need an equal amount of help from lab staff (Q4) in performing normal as well as small scale experiments. 'Interest in chemistry' could also be seen as being no difference whether students are performing small or normal scale experiments (Q5).

Moreover, the students found "no difference" in learning, concentrating and understanding both normal and small scale experiments (Q7, 10, and 13). This might be due to the factor that not all students performed the small scale experiments and also that not all experiments were modified to small scale. The students' attitudes, however, are more in favour of the small scale when compared to the normal scale.

Group 3

Normal scale (Q14 in table 5.3). It may reflect that less care in handling apparatus is required in the case of small scale experiments. This seems to be interesting and contrary to what was anticipated, as small scale apparatus would be seen as fiddly and hard to handle when compared to normal scale.

Group 4

This consists of those students who have shown an obviously high response in favour of the small scale experiments. These students' attitudes are shown in questions 2, 3, 6, 8, 11, and 15 (Table 5.3).

The small scale experiments involve the use of 'small test tubes', 'polythene sheets', and 'droppers', they also involve the techniques of using 'a few drops of solutions', 'a few crystals of solids'. Apparently, students found that all these techniques and apparatus would reduce risk when doing practical experiments in inorganic chemistry. The small scale experiments, in accordance with students' attitudes, could also reduce the time spent in carrying out experiments, and could make the task of experiments easier and could also make students' benches less cluttered. Accordingly, the majority of students in this group preferred the small scale experiments rather than the normal scale experiments in the lab.

Question 15 was one of the most important questions. It concerns the students opinion about whether they prefer to carry out normal or small scale experiments. In this question the students responded to Normal Scale, Microscale and No difference, in a ratio 1:3:1 respectively, which means that the general student attitude trend is in favour of small scale experiments and is three times higher than that for normal scale experiments or that for "no difference" attitude.

5.5.3 Students' attitudes related to their previous qualifications

From the number of students who completed the attitude questionnaire (101 students) and with regard to their known chemistry qualifications, it has been found that the ultimate picture for each group individually is premature to produce and therefore, is left to future work.

5.6 Summary

On the basis of the above data regarding the students marks in the small and normal scale experiments and their attitudes toward them, it is possible to conclude that the small scale experiments could work in the first year inorganic lab and would be equally effective as normal scale experiments. These small scale experiments scored an even better success in some cases (as can be seen in experiment 3 Part B & C and experiment 7 Part A on small scale) when compared to normal scale experiments, especially, when they are carried out within the three hours of the lab period. Students obtained high marks in experiments 3 Part B & C and 7 Part A on small scale as compared to other normal scale experiments.

In the attitude survey most of the students also seemed to prefer small scale rather than normal scale experiments in the lab. This could add another significant factor to the present study.

CHAPTER SIX

Conclusions and Discussions

6.1 Problems confronted by the research

It is essential to state, at the beginning of this chapter, and before the findings, what this study had to confront of problems concerning the use of microscale experiments and techniques:-

(a) Microscale chemistry was introduced to the first year university students for the first time and therefore some problems were anticipated. One of these problems was the modification of the existing lab manuals from macroscale into microscale lab manuals. In order to apply microscale techniques, all experiments in the first year organic and inorganic manuals were needed to be modified making microscale apparatus and techniques possible to be employed. It was therefore inevitable that some experiments in both manuals had to be avoided as it was not possible to modify them due to a certain use of apparatus or technique or procedure.

(b) Microscale chemistry was introduced to the first year university students. Most of these students, had just arrived from schools, and had little experience in practical chemistry. It was not easy for them to deal with certain aspects of microscale chemistry techniques. For instance, in the organic microscale experiments, some students faced difficulties in dealing or processing small yields. Also, filtration or recrystalisation of yields was another hard procedure for students.

(c) The demonstrators have also met Microscale chemistry for the very first time. It was not easy for every demonstrators to adopt the new techniques. The researcher, however, arranged a preliminary session for organic and inorganic laboratory staff to explain and demonstrate the new techniques and experiments which were intended for use. Also, be made himself available in every laboratory session when help was required by demonstrators or students.

(d) Both laboratories, organic and inorganic, were not previously used as microscale chemistry laboratories. For example, students performed microscale experiments in the inorganic laboratory side by side with other students who carried out the same experiments but on macroscale. It would be preferable to have microscale experiments performed independently with regard to the staff, technicians, demonstrators and arrangements of equipment and chemicals.

(e) As mentioned, microscale chemistry has been introduced to the students and staff for the very first time. There was not any in advance theoretical background or introduction given to the students regarding such a field apart from a brief introduction made by the researcher at the beginning of each lab session. Students found themselves suddenly presented with some techniques and equipment which have not been used before. This would obviously have a negative impact on at least some of them and would also have a similar impact on some members of staff.

6.2 The findings obtained from this study

The immediate section of this research is devoted to study the impact of microscale chemistry on the first year chemistry students in their work in the organic and inorganic laboratories.

Lots of advantages, regarding the use of microscale experiments and techniques, have been discussed in the previous chapters of this thesis, but the main aims of this study were to analyse what effect microscale chemistry would have on the attitude of students and on the learning of practical chemistry.

The findings which were obtained from this study could be categorised in two subsections; organic microscale and inorganic microscale findings.

6.2.1 The organic microscale findings

The findings obtained from the attitudes measurements of students when they used microscale organic techniques in the first year organic chemistry laboratory could be summarised as follows:-

(a) Students found that microscale organic apparatus is not difficult to handle in comparison to normal scale organic apparatus. It was accepted that small scale apparatus would have some complication in use since students had never been trained in how to use such apparatus in doing organic chemistry experiments. This finding may promote the use of microscale techniques in organic laboratories.

(b) In executing organic tasks, students found that microscale organic apparatus and techniques have a reasonable amount of risk as do normal scale apparatus or techniques. In fact, students forgot that using such small scale apparatus will result in the use of small amounts of chemicals which would eventually have a low risk, especially when things go wrong in any experiments. Also, such small amount of chemicals will produce very small yields at the end of any microscale experiments when compared to normal scale experiments. It is important to create a teaching laboratory which involves low risk and produces less hazardous chemicals. This should be encouraged for the sake of our environment, safety and money especially if the university organic laboratory is designed for learning and not for industrial production.

(c) Students also found that to measure small scale amounts of chemicals is not difficult. Indeed, they found such a procedure to be easier than to measure large amounts of chemicals. This could score another positive or significant point in favour of the use of microscale organic chemistry in the university laboratory. It was expected that small amounts of chemicals would be difficult to weigh and handle, but the attitude measurements of students suggested the contrary.

(d) When working on small scale organic experiments students indicated that the amount of help needed from staff was little as compared to normal scale experiments. It should be borne in mind that students expressed such an attitude even when they had never been trained in microscale techniques or apparatus before. This may indicate that organic microscale techniques or apparatus are not complicated when they have employed in the laboratory.

(e) One of the main difficulties of the use of small scale organic techniques is when students obtain a yield (very small) and such a yield needs to be processed e.g. separation or purification etc. Findings in this regard indicated that students were 'neutral' between the use of macroscale or microscale organic techniques. This could be considered as a positive attitude toward microscale organic chemistry because all students, as mentioned earlier, had never been trained to do such procedures on such a small scale but yet they managed.

(f) Students found that small scale organic experiments are interesting and challenging. The immediate and apparent impacts of such an attitude will be toward the enhancement of learning. Several educational theories hinted at the importance of making any teaching process interesting and challenging in order to enhance the learning process of the learners. Students tend to learn better when they are interested in a particular task or subject. Also, some students tend to learn better in a certain atmosphere when they have to compete with their colleagues in solving problems. This fact is related to some students' styles of thinking and motivation.

(g) Students, however, found that small scale organic experiments are time consuming. This was due to the fact that the sample of students used in this study faced, for the first time, small scale experiments without any preliminary training. On the other hand, most of those students had done very little practical chemistry in schools. Because of these reasons it would not be a total surprise to see students regarding small scale organic experiments as time consuming.

(h) The demonstrators thought that small scale organic experiments or techniques would have the same impact on students in; the time for a completion of any experiments, the time for measuring chemicals, and for the help which was needed by students during performing small scale experiments. However, half of the demonstrators indicated that low risk would be involved in small scale organic experiments, but careful work was also needed when doing such experiments.

6.2.2 The general inorganic microscale findings

The findings obtained from the attitudes' measurements of students when used the microscale inorganic techniques in the first year inorganic chemistry laboratory could be summarised as follows:-

(a) Most students found that microscale inorganic experiments are more fun and easy when compared to normal scale experiments. They also found that less apparatus, spillage, breakage, care in handling apparatus and time were needed when doing microscale experiments. They indicated that their benches were more tidy and less cluttered in the case of microscale experiments when compared to normal scale ones.

(b) In doing microscale inorganic experiments, most students indicated that "no big difference" was notified in the amount of help needed, 'interest', 'understanding of lab manual', 'concentration' and 'learning' when compared to normal scale experiments. It is expected that a better picture could be obtained from the students' attitudes if all inorganic experiments had been performed on small scale as well as on normal scale!

(c) Students' attitudes were divided with regard to safety and relaxation when doing microscale experiments. Half of the students felt safer and more relaxed when performing small scale experiments, while the other half felt 'no difference' between small or normal scale experiments. It is also anticipated that a better attitude picture would be gained by this study with regard to the safety of small scale experiments if all experiments were designed to be done on both scales.

(d) The most significant and obvious picture of students' attitude toward small scale experiments emerged when students were asked to choose between either small or normal scale experiments. Most students were in favour of small scale rather than normal scale experiments. This would support the general trend of this study toward inorganic microscale experiments.

(e) One of the main findings in this research and in both organic and inorganic microscale experiments was when students found that small scale apparatus is not fiddly or hard to handle. This certain attitude was even more obvious in the inorganic microscale experiments. Such a finding was a surprise since the researcher was expecting the contrary. Students had to deal with very small apparatus e.g. "small test tubes", "droppers", "small spatula", and "microscale organic kit", however, it was apparent that such a scale of apparatus was easy to handle by students and did not present any difficulty.

6.2.3 Findings obtained from students' achievements in general inorganic microscale experiments

The findings obtained from the sample of students used in this study in the microscale inorganic experiments could be summarised as follows:-

(a) Students' achievements on small scale experiments were as good as those which were obtained on normal scale. However, students obtained, in general, slightly better marks when doing small scale experiments compared to their achievements in normal scale experiments.

(b) When students studied in categories with regard to their background in chemistry, it appeared that students with chemistry background ('Higher', 'Standard', 'Module') would achieve better than students who had no or very little chemistry background ('Other', 'None'). However, the group 'Other' managed to come third among the rest in one of the microscale experiments.

(c) The main finding which could be drawn from the picture of students' achievements in inorganic microscale experiments would give no disadvantage when applied in the laboratory. Moreover, it is expected that small scale experiments would have shown their explicit effectiveness with regard to the picture of students' achievements if all inorganic experiments were to be designed and used in both small and normal scale forms.

6.3 Suggestions for further research

In every study a number of suggestions for further research surface. This particular research has been carried out for the very first time in the Centre for Science Education at the Department of Chemistry in the University of Glasgow. As mentioned earlier in this chapter, there were many hurdles confronting this research which made it impossible to cover all microscale aspects. Therefore, it is prudent to suggest the following studies:-

(a) One of the main problems which faced this research was that not all experiments in both chemistry manuals (organic and inorganic) were able to be modified into microscale experiments. Therefore, it would be absolutely necessary to carry out another study on the effectiveness of microscale techniques when most or all experiments are modified into microscale. In ideal circumstances, students could be divided into two groups; a group to do microscale experiments; while the other group to do same experiments but on macroscale techniques. Then, groups should swap to do the other technique which they have not experienced yet. Such a study would be essential to show how far have students enjoyed, learnt, from and experienced microscale experiments. Moreover, such a study would be even helpful for students allowing them to gain a proper picture of comparison between the ordinary and microscale techniques. It is expected that such a proposed study would support the findings which emerged from the present research.

(b) Research is necessary to focus on whether students will obtain better marks in the laboratory when doing organic microscale experiments rather than organic macroscale experiments.

(c) Research should also be carried out on students who are in second, third, and fourth years in the university. Microscale chemistry could have a different impact on those students who study chemistry at advanced level in the university.

(d) An attempt could be made for further research in other areas of chemistry apart from organic and inorganic chemistry i.e. physical, analytical, and biochemical. Such research should concentrate on the possibility of creating and developing new experiments which employ the microscale techniques. Also, students' attitudes and achievements would be possible to measure and study.

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APPENDICES

APPENDIX-1a

APPENDIX-1a

10

PROCEDURE :

1. OXIDATION OF AN UNKNOWN SECONDARY ALCOHOL TO A KETONE

All the apparatus for steps 1 and 2 is in the fume hood. At the end, clean it and put it back there.
Choose one of the alcohols. Take a note of its number.
Using the graduated pipette or burette provided transfer 1.2 mL of your alcohol into the 10 mL tapered flask.
In a FUME HOOD, clamp the flask and attach a reflux condenser (see APPENDIX D).
Have a beaker of cold water handy and lower the flask into it if the reaction mixture gets too warm. If you have alcohol 1, pass water through the condenser. In the other cases this is not necessary.
Using a clean measuring cylinder, measure out 2 mL of 2 $molL^{-1}$ chromic acid.
Add the acid, drop by drop, down the condenser over a period of 5 minutes.
The mixture will warm up but should not be allowed to boil.
Occasionally swirl the flask to mix the contents and to allow heat to be transferred to the water bath.
The reaction is complete when there is no further evolution of heat.
Note any change in the colour or appearance of the mixture.

2. DISTILLATION OF THE KETONE/WATER AZEOTROPE

After the oxidation is complete, remove the condenser and rinse it with water. Add 2 mL of water (measuring cylinder) and 2 boiling stones to the tapered flask.
See APPENDIX E and set up the flask as part of the distillation apparatus.
Pass a moderate flow of water through the condenser and make sure that all the ground glass joints are tightly fitting.

CAUTION :

The distillate contains a ketone which is highly flammable.
Do not light the Bunsen burner until the apparatus is assembled
with the ground glass joints securely fitting.
Extinguish the burner before dismantling.
Do not let the flask or receiver hang unsupported.

Heat the flask gently with the micro Bunsen flame and record the temperature at which liquid begins to drip off the thermometer bulb. Continue the distillation as instructed below and record the range of temperature over which you were collecting the distillate. Stop the distillation when the temperature starts to rise steeply. You should have collected approximately 1.2 mL distillate for alcohol '1', 1.4-1.6 for alcohol '2' or '3' and 1.8-2.0 mL distillate for alcohol '4'.
If you stop then, your ketone will be almost completely free of water. Add a little sodium chloride from the end of a spatula to dry it totally.
Because sodium and chloride ions become strongly solvated by the water present, this leaves little water to solvate the (less polar) dipole of the ketone.
Allow the tube to stand in a test tube rack for a couple of minutes.
Using a clean Pasteur pipette, transfer the upper layer of fairly pure ketone to a clean test tube.

3. PREPARATION OF THE SEMICARBAZONE

A ketone can react with a primary amine to produce an imine. However, amines are easily oxidised and hence difficult to store. Frequently amines are kept as salts (which are crystalline solids and easy to work with) and liberated by the addition of a weak base when required in a reaction.
Here sodium ethanoate is used as the base and semicarbazide hydrochloride is the salt of the amine.

CAUTION :

Semicarbazide hydrochloride is thought to be a carcinogen and mutagen (causing genetic changes).
Protect your skin and avoid inhalation. Limit handling to one area of the bench and report any spillage to laboratory staff.
On no account should semicarbazide hydrochloride be allowed to contaminate cupboards, drawers or balances.

Weigh out 0.8 g sodium ethanoate and 0.5 g semicarbazide hydrochloride into separate clean 50 mL beakers on a top-pan balance.
Dissolve the two solids in about 3 mL of water, in the wide boiling tube by heating on a water or steam bath and swirling until the solid dissolves.
Add around 0.6 mL (about 12 drops) of the ketone you prepared and continue to heat for 10-15 minutes.
If the ketone is insoluble, stir it frequently until there is no oil left.
Allow the tube to cool and the semicarbazone derivative should begin to crystallise.
Cool the tube further in a little ice or cold water.
If necessary, scratch the sides of the tube with a clean glass rod to induce crystallisation.
Collect the crystals (see APPENDIX B) and press them between filter papers.
Place some of the crude semicarbazone in a labelled sample tube to be handed in when your report is marked.
Use the bulk of the solid for recrystallisation.

4. RECRYSTALLISATION OF THE SEMICARBAZONE

Read APPENDIX A. Transfer the semicarbazone to a clean, boiling tube.
Place the tube in the steam bath. Using a clean Pasteur pipette, transfer BOILING water into the tube drop by drop until the solid just dissolves.
Remove the tube and allow it to cool so that the semicarbazone crystallises out.
Collect the solid by vacuum filtration as before.
Transfer the pure semicarbazone to a labelled sample tube and hand this in during your assessment.

PROCEDURE :

The apparatus for the hydrolysis is set up on the bench or in the fume hood. At the end clean it and reassemble it.
Choose one of the esters. Make a note of its number and whether it is a methyl or ethyl ester.
If you have chosen a solid ester weigh out 0.4 g (using a top-pan balance) and place this into a 10 mL round-bottomed flask.
If the ester is a liquid, use a clean Pasteur pipette to place 10 drops of the liquid into a 10 mL round-bottomed flask.

Add about 4 mL of 5 $molL^{-1}$ sodium hydroxide solution and drop in two boiling stones to prevent bumping. Note any changes.

Set up a reflux apparatus (see APPENDIX D), using a <u>heating mantle</u> (use setting 6 to heat up and about 2 to maintain boiling) and an <u>air</u> condenser. Insert a teflon sleeve between the condenser end and the flask to prevent these jamming together.
One clamp round the neck of the flask or condenser is enough.

Warm the flask until the liquid just begins to boil.
Continue to heat the flask gently, maintaining vigorous reflux for 20-25 minutes.

Allow the apparatus to cool. When the flask is cool enough to handle, detach the flask from the condenser. Carefully decant the solution into a clean flask or boiling tube leaving the boiling stones behind.

When the flask is cool, hold it in a water bath and slowly add hydrochloric acid from a measuring cylinder (about 4 mL). Swirl the flask to help dissipate the heat caused by the addition of acid. Check the pH of the solution by spotting some of it on pH paper with a clean spatula or glass rod and, if necessary, add more hydrochloric acid until the pH value is approximately 1.

Once the acid has been added and the flask left to cool, the carboxylic acid should crystallise out. If crystals don't form on cooling, check the pH of the solution - it may not be low enough. Collect the crystals by vacuum filtration (see APPENDIX B).
Wash them, in the funnel, with cold water and continue suction until no more liquid can be seen coming from the bottom of the funnel.

2. RECRYSTALLISATION OF THE CARBOXYLIC ACID

Transfer the carboxylic acid to a boiling tube (see APPENDIX A).
Add a small volume (1 mL) of water and heat the tube on a water or steam bath.
If the solid does not all dissolve add, drop by drop by pipette, just enough ethanol to get it all into solution, keeping the solution hot.

Allow the tube to cool until the carboxylic acid crystallises out.
Collect the crystals by filtration as before, and dry them between filter papers.
Transfer the crystals to a sample tube, labelled with your name and the number of the ester you used.
Retain this sample for inspection when your report is being assessed.

3. MELTING POINT AND MIXED MELTING POINT

See APPENDIX C.
Follow the procedure and determine the melting point of the acid you have made.
Examine the table of acids and their melting points on page 18.
Your acid is one of these. Choose which melting point fits yours and ask your demonstrator for a sample of that acid.
Using this authentic sample take a mixed melting point, as detailed in the appendix.
If your acid is the same as the authentic sample then you have correctly identified it - if not consult your demonstrator.

CLEAN UP :

At the end of the practical work, pour the aqueous solutions and solvents used in crystallisation down the sink.
Rinse the glassware and pipette, using a little ethanol, if necessary.
Reassemble the hydrolysis apparatus.
Put the other apparatus and clean pipettes in your drawer.
Put papers in the plastic bin bag (not into the sink).
Place capillaries into the "broken glass" bin.

APPENDIX-1b

PROCEDURE :

The apparatus for the hydrolysis is set up on the bench or in the fume hood. At the end clean it and reassemble it.
Choose one of the esters. Make a note of its number and whether it is a methyl or ethyl ester.
If you have chosen a solid ester weigh out 0.2 g (using a top-pan balance) and place this into a 10 mL round-bottomed flask.
If the ester is a liquid, use a clean Pasteur pipette to place 4-5 drops of the liquid into a 10 mL round-bottomed flask.

Add about 3 mL of 5 $molL^{-1}$ sodium hydroxide solution and drop in two boiling stores to prevent bumping. Note any changes.

Set up a reflux apparatus (see APPENDIX D), using a heating mantle in place of the heating bath. Insert a teflon sleeve between the condenser end and the flask to prevent these jamming together.
One clamp round the neck of the flask is enough. Pass water gently through the condenser.

Warm the flask until the liquid just begins to boil.
Continue to heat the flask gently, maintaining vigorous reflux for 20-25 minutes.

Allow the apparatus to cool. When the flask is cool enough to handle, detach the flask from the condenser. Using the spatula carefully remove the boiling stones from the solution and leave the flask to cool.

When the flask is cool, hold it in a water bath and, using a Pasteur pipette, slowly add hydrochloric acid from a measuring cylinder (abaout 3 mL). Swirl the flask to help dissipate the heat caused by the addition of acid. Check the pH of the solution by spotting some of it on pH paper with a clean spatula and, if necessary, add more hydrochloric acid until the pH value is approximately 1.

Once the acid has been added and the flask left to cool, the carboxylic acid should crystallise out. If crystals don't form on cooling, check the pH of the solution - it may not be low enough. Collect the crystals by vacuum filtration (see APPENDIX B).
Wash them, in the funnel, with cold water and continue suction until no more liquid can be seen coming from the bottom of the funnel.

2. RECRYSTALLISATION OF THE CARBOXYLIC ACID

Transfer the carboxylic acid to a boiling tube (see APPENDIX A).
Add a small volume (0.5 mL) of water and heat the tube on a water or steam bath.
If the solid does not all dissolve add, drop by drop by pipette, just enough ethanol to get it all into solution, keeping the solution hot.

Allow the tube to cool until the carboxylic acid crystallises out.
Collect the crystals by filtration as before, and dry them between filter papers.
Transfer the crystals to a sample tube, labelled with your name and the number of the ester you used.
Retain this sample for inspection when your report is being assessed.

3. MELTING POINT AND MIXED MELTING POINT

See APPENDIX C.
Follow the procedure and determine the melting point of the acid you have made.
Examine the table of acids and their melting points on page 18.
Your acid is one of these. Choose which melting point fits yours and ask your demonstrator for a sample of that acid.
Using this authentic sample take a mixed melting point, as detailed in the appendix.
If your acid is the same as the authentic sample then you have correctly identified it - if not consult your demonstrator.

CLEAN UP :

At the end of the practical work, pour the aqueous solutions and solvents used in crystallisation down the sink.
Rinse the glassware and pipette, using a little ethanol, if necessary.
Reassemble the hydrolysis apparatus.
Put the other apparatus and clean pipettes in your drawer.
Put papers in the plastic bin bag (not into the sink).
Place capillaries into the "broken glass" bin.

APPENDIX-2a

EXPERIMENT 2

THE ALDEHYDE AND KETONE FUNCTIONAL GROUPS

PRE-LABORATORY WORK

Read the experiment and APPENDICES A to E.

Attempt the following questions before you come to the laboratory.
Write your answers in your lab report book and let your demonstrator mark them.
For background reading, see lectures on: aldehydes, ketones and imines.
See also: Hart - Chapter 9.

1. *Propene and ethanal have similar structures and nearly identical formula weights, but different physical properties. Explain the differences shown in the table.*

Compound	Soluble in water	Boiling point (°C)
propene CH_3-$CH=CH_2$	no	-48
ethanal CH_3-$CH=O$	yes	+21

2. Draw the structures of propanal, cyclohexanone, and benzaldehyde. Which of these would you expect to be soluble in water?

3. When nucleophiles add to carbonyl groups why do they always add to the carbon atom?

4. Aldehydes and ketones can react with primary amines to give imines ($R_2C=NR$) and water.
Draw the structure of semicarbazide and of the semicarbazone of propanal.

5. Draw general structures to represent a primary alcohol and a secondary alcohol. Explain why an excess of a powerful oxidising agent can be used to oxidise secondary alcohols to ketones whereas milder conditions are required to oxidise primary alcohols to aldehydes.

LABORATORY WORK

AIM OF THE EXPERIMENT :

In this experiment the aim is to oxidise an unknown secondary alcohol, isolate the ketone, prepare a solid imine derivative and purify it, then use its melting point and distillation information to identify the ketone (and so identify the starting alcohol).

EXPERIMENTAL DETAILS :

The unknown alcohol is oxidised using aqueous chromic acid (H_2CrO_4) to give a ketone. The ketone is separated from the reaction mixture by distillation. Part of this ketone is then converted by reaction with semicarbazide to a solid imine, called a semicarbazone. Semicarbazones are generally solids, easy to purify by crystallisation and usually with sharp melting points. This makes them a good choice of derivative to prepare to characterise the ketone.

When the ketone/water mixture is heated the distillate might be pure ketone, pure water or a mixture. Even with a tall column the components of a mixture might distill together if the vapour pressure of the particular mixture is higher than that of either component alone.

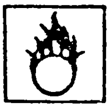
This happens in several cases and the lowest boiling mixture is called an azeotrope (Greek = no boiling change). It has fixed composition and boils at a fixed, steady temperature - until one of the components has completely distilled over.

In all cases, if you keep on distilling, the temperature on the thermometer will eventually rise as pure water comes over (and dilutes your ketone).

When it condenses, the azeotrope vapour mixture may separate to give two immiscible liquid layers (ketone and water).

CAUTION :

All alcohols and ketones are highly flammable. All are skin and eye irritants if swallowed or inhaled.
They must be kept away from naked flames.
Chromic acid is thought to be a carcinogen (cancer causing agent).
It is very toxic and very corrosive (acidic and powerful oxidant).
It can react violently with oxidisable materials. To reduce the hazard you will use small quantities, and wear gloves. Dispose of all chromium solutions into the chromium residues bottle,
<u>not down the sink.</u>



PROCEDURE :

1. OXIDATION OF AN UNKNOWN SECONDARY ALCOHOL TO A KETONE

All the apparatus for steps 1 and 2 is in the fume hood. At the end, clean it and put it back there.
Choose one of the alcohols. Take a note of its number.
Using a measuring cylinder transfer 6 mL of your alcohol into the 50 mL round-bottomed flask.
In a FUME HOOD, clamp the flask and attach a reflux condenser (see APPENDIX D). Have a beaker of cold water handy and lower the flask into it if the reaction mixture gets too warm. If you have alcohol 1, pass water through the condenser. In the other cases this is not necessary.
Clean the measuring cylinder and use it to measure out 10 mL of 2 $molL^{-1}$ chromic acid. Pour the acid, about 1 ml at a time, down the condenser over a period of 5 minutes. The mixture will warm up but should not be allowed to boil.
Occasionally swirl the flask to mix the contents and to allow heat to be transferred to the water bath.
The reaction is complete when there is no further evolution of heat.
Note any change in the colour or appearance of the mixture.

2. DISTILLATION OF THE KETONE/WATER AZEOTROPE

After the oxidation is complete, remove the condenser and rinse it with water. Add 10 mL of water (measuring cylinder) and 2 boiling stones to the round-bottomed flask.
See APPENDIX E and set up the flask as part of the distillation apparatus.
Pass a moderate flow of water through the condenser and make sure that all the ground glass joints are tightly fitting.

CAUTION :

The distillate contains a ketone which is highly flammable.
Do not light the Bunsen burner until the apparatus is assembled
with the ground glass joints securely fitting.
Extinguish the burner before dismantling.
Do not let the flask or receiver hang unsupported.

Heat the flask gently and record the temperature at which liquid begins to drip off the thermometer bulb. Continue the distillation as instructed below and record the range of temperature over which you were collecting the distillate.

If you used alcohol 1: collect about 6 mL of distillate. If you stop then, your ketone will be almost completely free of water. Add a few spatulae of sodium chloride to dry it totally.
The sodium and chloride ions become strongly solvated by the water present. This leaves little water to solvate the (less polar) dipole of the ketone and so the ketone is forced out of solution.

If you used alcohol 2 or 3: collect about 7-8 mL of distillate. Add roughly 4 g of sodium chloride (weighed out on the top-pan balance) to the collector tube. This will result in separation into two layers.
If you used alcohol 4: collect about 9-10 mL of distillate. It will begin to separate into two layers immediately.

Allow the tube to stand in a test tube rack for a couple of minutes.
Using a clean Pasteur pipette, transfer the upper layer of fairly pure ketone to a clean test tube.

3. PREPARATION OF THE SEMICARBAZONE

A ketone can react with a primary amine to produce an imine. However, amines are easily oxidised and hence difficult to store. Frequently amines are kept as salts (which are crystalline solids and easy to work with) and liberated by the addition of a weak base when required in a reaction.
Here sodium ethanoate is used as the base and semicarbazide hydrochloride is the salt of the amine.

$$R\text{-}NH_3^{\oplus} + CH_3CO_2^{\ominus} \rightleftharpoons RNH_2 + CH_3CO_2H$$

CAUTION :

Semicarbazide hydrochloride is thought to be a carcinogen and mutagen (causing genetic changes).
Protect your skin and avoid inhalation. Limit handling to one area of the bench and report any spillage to laboratory staff.
On no account should semicarbazide hydrochloride be allowed to contaminate cupboards, drawers or balances.

Dissolve 3.8 g (top-pan balance) of sodium ethanoate in about 15 mL of water, in the wide boiling tube in your drawer, then add 2.5 g (top-pan balance) of semicarbazide hydrochloride.
Heat the boiling tube on a water or steam bath and swirl until the solid dissolves.
Add around 3 mL of the ketone you prepared and continue to heat for 10-15 minutes.
If the ketone is insoluble, stir it frequently until there is no oil left.
Allow the tube to cool and the semicarbazone derivative should begin to crystallise.
Cool the tube further in a little ice or cold water.
If necessary, scratch the sides of the tube with a clean glass rod to induce crystallisation.
Collect the crystals (see APPENDIX B) and press them between filter papers.
Place some of the crude semicarbazone in a labelled sample tube to be handed in when your report is marked.
Use the bulk of the solid for recrystallisation.

4. RECRYSTALLISATION OF THE SEMICARBAZONE

Read APPENDIX A. Transfer the semicarbazone to a clean, boiling tube.
Place the tube in the steam bath. Using a clean Pasteur pipette, transfer BOILING water into the tube drop by drop until the solid just dissolves.
Remove the tube and allow it to cool so that the semicarbazone crystallises out.
Collect the solid by vacuum filtration as before.
Transfer the pure semicarbazone to a labelled sample tube and hand this in during your assessment.

5. MELTING POINT OF THE CRUDE AND RECRYSTALLISED SEMICARBAZONE

See APPENDIX C.
Follow the procedure and determine the melting point of the crude semicarbazone.
Allow the apparatus to cool by about 30° and determine the melting point of the pure semicarbazone.

CLEAN UP :

When practical work is finished, rinse all the apparatus containing chromium solutions into the chromium residue bottle, NOT into the sink.
Wash all glassware (using a little acetone if necessary). Return the distillation set to the fume hood and tubes and funnels *etc.* to your drawer.
Put papers in the plastic bin bag (not in the sink).
Place capillaries into the "broken glass" bin.
Rinse Pasteur pipettes with water and ethanol and put them in your drawer for re-use.

EXPERIMENTAL REPORT :

Describe what you observed during the oxidation reaction.
Record the temperature range over which your mixture distilled.
Describe the appearance of the crude and pure semicarbazone derivative.
Report the melting points of both the crude and pure compounds.
What can you deduce from these results.

From the data given in the table, deduce which ketone/semicarbazone you produced.
Draw the structures of the ketone, and the secondary alcohol it was made from, and name both. Draw the semicarbazone you made.
Hand in the pure and crude semicarbazones with your report.

DATA TABLE

ALCOHOL	KETONE	KETONE-WATER AZEOTROPE BOILING POINT (°C)	MELTING POINT OF KETONE SEMICARBAZONE (°C)
Propan-2-ol	Propanone	Up to 70	190
Butan-2-ol	Butan-2-one	73-80	146
Pentan-2-ol	Pentan-2-one	83-90	112
Cyclohexanol	Cyclohexanone	95-100	167

see over

QUESTIONS :

1. The ion-electron half equation for the oxidation of the alcohol in acid is:

$$R_2CHOH \rightarrow R_2CO + 2H^+ + 2e^-$$

From your observations, deduce the oxidation state of the chromium at the end of the reaction.
Write an ion-electron equation for the reduction of CrO_4^{2-} in acid solution.
Write a balanced equation for the overall redox reaction.

2. In the experiment a ketone reacted with a primary amine to produce an imine. Aldehydes and ketones react in the same way with hydroxylamine ($HO\text{-}NH_2$) to produce an oxime.
Draw the structure of the oxime derived from propanone. Suggest why the oxime and the semicarbazone of propanone are solids whereas the ketone is liquid.

3. How could your ketone be converted back into a secondary alcohol?
Suggest a possible reagent for this process.

4. Why were no primary or tertiary alcohols used in this experiment?

APPENDIX-2b

EXPERIMENT 3

THE CARBOXYLIC ACID AND ESTER FUNCTIONAL GROUPS

PRE-LABORATORY WORK

Read the experiment and APPENDICES A-D.

Attempt the following questions before you come to the laboratory.
Write your answers in your lab report book and let your demonstrator mark them.
For background reading, see lectures on : carboxylic acids and esters, pH and $\underline{pK_a}$ *and reaction rate. See also : Hart, Chapter 10.*

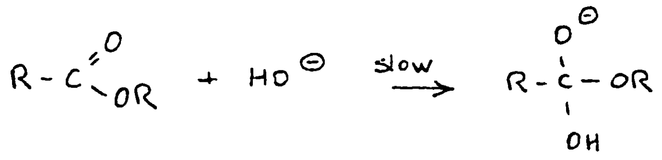
1. Draw the structures of ethanoic acid, benzoic acid and their ethyl esters.

2. Of these four compounds, explain why only ethanoic acid is soluble in cold water.

3. On thin-layer silica chromatography, with ethyl acetate as the eluent, methyl benzoate has a much higher R_f than benzoic acid.
Explain this observation.

4. Write down the equilibrium equation for the dissociation of a general carboxylic acid, RCOOH, and the definitions of K_a and pK_a.
If $pK_a = 5$ for a carboxylic acid, then calculate the pH of a 0.1 $molL^{-1}$ solution of the acid in water.
Calculate the ratio $[RCOO^-]/[RCOOH]$ in aqueous solution at pH 9 and pH 1.
What are the implications of your answers for the variation of solubility of benzoic acid in water at different pH values?

5. The slow step in the hydrolysis is addition of hydroxide ion to the ester.



What two other stages follow this to form the alcohol and the anion?

LABORATORY WORK

AIM OF THE EXPERIMENT :

In this experiment the aim is to identify an unknown ester (it will be either a methyl or ethyl ester) by hydrolysing it totally, isolating the acid produced, purifying the acid and identifying it from its melting point.

EXPERIMENTAL DETAILS :

Esters can be hydrolysed under alkaline conditions to give alcohols and carboxylic acids (as their sodium salts).

In this experiment, the unknown ester will be hydrolysed using hot aqueous alkali, to produce the salt of the acid and methanol or ethanol (depending on the ester chosen).

As considered in your pre-lab work, acidification of this reaction mixture will give the (water insoluble) carboxylic acid.

This acid is collected and purified and its melting point is found thus identifying it.

From this the identity of the unknown ester can be established.

CAUTION :

Although the quantities you are handling are small, remember that all carboxylic acids are flammable, corrosive and poisonous and esters are highly flammable, irritant and poisonous.

Hot 5 molL^{-1} sodium hydroxide and 5 molL^{-1} hydrochloric acid are extremely corrosive and must be handled with great care.

PROCEDURE :

The apparatus for the hydrolysis is set up on the bench or in the fume hood. At the end clean it and reassemble it.
Choose one of the esters. Make a note of its number and whether it is a methyl or ethyl ester.
If you have chosen a solid ester weigh out 1 g (using a top-pan balance) and place this into a 50 mL round-bottomed flask.
If the ester is a liquid, use a clean Pasteur pipette to place 20-25 drops of the liquid into a 50 mL round-bottomed flask.

Add about 15 mL of 5 $molL^{-1}$ sodium hydroxide solution and drop in two boiling stones to prevent bumping. Note any changes.

Set up a reflux apparatus (see APPENDIX D), using a heating mantle in place of the heating bath. Insert a teflon sleeve between the condenser end and the flask to prevent these jamming together.
One clamp round the neck of the flask is enough. Pass water gently through the condenser.

Warm the flask until the liquid just begins to boil.
Continue to heat the flask gently, maintaining vigorous reflux for 20-25 minutes.

Allow the apparatus to cool. When the flask is cool enough to handle, detach the flask from the condenser. Using the spatula carefully remove the boiling stones from the solution and leave the flask to cool.

When the flask is cool, hold it in a water bath and, using a measuring cylinder, slowly add hydrochloric acid. Swirl the flask to help dissipate the heat caused by the addition of acid. Check the pH of the solution by spotting some of it on pH paper with a clean spatula and, if necessary, add more hydrochloric acid until the pH value is approximately 1.

Once the acid has been added and the flask left to cool, the carboxylic acid should crystallise out. If crystals don't form on cooling, check the pH of the solution - it may not be low enough. Collect the crystals by vacuum filtration (see APPENDIX B).
Wash them, in the funnel, with cold water and continue suction until no more liquid can be seen coming from the bottom of the funnel.

2. RECRYSTALLISATION OF THE CARBOXYLIC ACID

Transfer the carboxylic acid to a boiling tube (see APPENDIX A).
Add a small volume (2 mL) of water and heat the tube on a water or steam bath.
If the solid does not all dissolve add, drop by drop by pipette, just enough ethanol to get it all into solution, keeping the solution hot.

Allow the tube to cool until the carboxylic acid crystallises out.
Collect the crystals by filtration as before, and dry them between filter papers.
Transfer the crystals to a sample tube, labelled with your name and the number of the ester you used.
Retain this sample for inspection when your report is being assessed.

3. MELTING POINT AND MIXED MELTING POINT

See APPENDIX C.
Follow the procedure and determine the melting point of the acid you have made.
Examine the table of acids and their melting points on page 18.
Your acid is one of these. Choose which melting point fits yours and ask your demonstrator for a sample of that acid.
Using this authentic sample take a mixed melting point, as detailed in the appendix.
If your acid is the same as the authentic sample then you have correctly identified it - if not consult your demonstrator.

CLEAN UP :

At the end of the practical work, pour the aqueous solutions and solvents used in crystallisation down the sink.
Rinse the glassware and pipette, using a little ethanol, if necessary.
Reassemble the hydrolysis apparatus.
Put the other apparatus and clean pipettes in your drawer.
Put papers in the plastic bin bag (not into the sink).
Place capillaries into the "broken glass" bin.

EXPERIMENTAL REPORT :

Record what you observed when the ester and alkali were mixed in the flask, before heating.
Compare this to the appearance of the flask's contents at the end of the refluxing.
When the HCl was added, what did you see? Why was this stage exothermic?

What was the appearance of your ester and its carboxylic acid?
Report the melting point of the acid and deduce which acid you produced.
From this draw the structure of the ester you chose. Name this ester. Hand in your acid in a labelled sample tube with your report.

QUESTIONS :

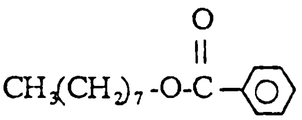
1. Why are some of the esters liquids, while all the carboyxlic acids in this experiment are solid?

2. Why is the hydrolysis faster:

   a) at 100°C?
   b) at pH 14 than at pH 7?
   c) when bubbling vigorously?

3. What happened to the alcohol (methanol or ethanol) which was also formed in the hydrolysis i.e. where did it end up?

4. In this experiment only ethyl and methyl esters were used. Predict the outcome if the hydrolysis reaction had been carried out as above, but using the ester, 1-octyl benzoate, with a larger alcohol unit?



MELTING POINTS OF SELECTED SOLID CARBOXYLIC ACIDS

Alternative trivial names are given in brackets.

ACID	MELTING POINT (°C)
Benzoic	122-123
2-Hydroxybenzoic (salicylic)	158-160
3-Hydroxybenzoic	201-203
4-Hydroxybenzoic	215-217
2-Methylbenzoic (o-toluic)	103-105
3-Methylbenzoic (m-toluic)	108-110
4-Methylbenzoic (p-toluic)	180-182
2-Methoxybenzoic	98-100
3-Methoxybenzoic	106-108
4-Methoxybenzoic	182-185

APPENDIX-3a

APPENDIX-3a

EXPERIMENT - 3 - REDOX REACTIONS

Purpose

The purpose of this experiment is to investigate oxidation-reduction reactions.

The Experimental Report should contain:

the balanced equations for the reactions which occur in the experiment;
answers to questions in these written instructions.

Outline of the Experiment

A. The decomposition of ammonium dichromate (ammonium dichromate volcano) to produce chromium(III) oxide.
B. Replacement of one halogen by another.
C. Iodide ⇌ Iodine interconversion

THE EXPERIMENTS

The Experimental procedure

Chemicals for this experiment are on benches 'A' or 'B'.
Refer to map of lab (page 5) for location of apparatus and equipment.

Safety Precautions

Chromium salts are toxic, particularly by skin absorption.

A. The Ammonium dichromate 'Volcano'

Using a rough balance (appendix-1) weigh out approximately 3.5g of ammonium dichromate $(NH_4)_2Cr_2O_7$. Also weigh a 100 mL beaker and record its weight. It will be used to collect and weigh the product of the reaction. ☠

Place a large (24 cm) filter paper (bench 'C') on the bench in the fume cupboard and on top of this your "asbestos" centered wire gauze. Pour the ammonium dichromate on to the gauze so that it forms a cone shaped pile in the centre. Light the apex of the cone with a match. It may take two or three attempts, but once the reaction has started it will continue by itself. Record your observations in your own lab notebook. The solid product is chromium(III) oxide. The other products are nitrogen (N_2) and water (steam). Write a balanced equation for the reaction. Was there any sign of the nitrogen and water? Where did they go?

From your equation work out the weight of Cr_2O_3 you expect to get from your 3.5g of $(NH_4)_2Cr_2O_7$. Collect and weigh your Cr_2O_3 and calculate what fraction of the predicted weight you actually got [i.e. your yield]. ☠

B. Replacement of One Halogen by Another.

In this section you are going to use dilute aqueous solutions of the ionic halides. Prepare about 10 mL. of each solution by dissolving a few crystals (about the amount on the tip of a spatula) of each in water in test tubes. Take about 2 cm depth of each solution in test tubes for the following reactions and keep the remaining solutions to use later.

Prepare a chlorine solution in water by diluting approximately 2 mL of sodium hypochlorite (bleach) with 10 mL of water, then acidifying it with a small amount of 1 mol L^{-1} sulphuric acid (test with litmus paper).

$$2NaOCl + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + Cl_2$$

Add a few drops of the chlorine solution to your samples of dilute sodium fluoride, sodium chloride, sodium bromide, and sodium iodide, in test tubes, and note what you see.

Add 1 mL. of chloroform (trichloromethane) to each of the solutions. It will form a lower layer. Shake the test tubes (appendix 3) and observe the colour of the chloroform layer. Halogens are more soluble in chloroform than they are in water, so any free halogen is removed from the water and ends up mainly in the chloroform layer giving a distinctive colour.

$CHCl_3$ is less polar than H_2O. Why does the I_2 prefer to dissolved in the $CHCl_3$ rather than the H_2O?

Record your observations in your own lab notebook in a table (similar to Table 2).

TABLE 2 - REACTION WITH Cl_2

Halides + Cl_2	Initial Colour Produced	Colour of Chloroform Soln.	Products
NaF + Cl_2			
NaCl + Cl_2			
NaBr + Cl_2			
NaI + Cl_2			

Write balanced equations for the reactions that occurred.

C. Iodide-Iodine Interconversion

To a little copper sulphate solution add a little NaBr solution. Nothing happens! Now add 2 mL of NaI solution. One of the products is iodine, the other is a precipitate of CuI. Write a balanced equation for the redox reaction.

Add to your test-tube a little sodium thiosulphate solution. Another redox reaction occurs

$$2Na_2[S_2O_3] + I_2 \rightarrow 2NaI + Na_2[S_2O_6]$$

You may now be able to see the CuI solid clearly. What colour is it?

From your observations put the ions

bromide, chloride, iodide and thiosulphate

in order:- most easily oxidised to least easily oxidised.

APPENDIX-3b

APPENDIX-3 b

EXPERIMENT - 5 - PERIODIC TABLE TRENDS

Purpose

The purpose of this experiment is to do three series of reactions on halogen compounds and to compare the reactions within each series looking for gradations in behaviour. In particular you are looking for the effects of oxidation of the halide ion compared to other reactions. The reactions of the halogen group of elements and compounds should give an indication of more general trends in the periodic table of the elements.

Safety Precautions

Unlike sodium chloride, the sodium salts of the other halides are poisonous and should be handled with care. Many of the other chemicals in this experiment are toxic and corrosive. Pay attention to symbols in the right margin. They indicate the hazards of the substances used.

The Experimental Report

For your own benefit, draw up tables of comparisons in your own lab notebook while doing the following experiments and make a note of each observation. (examples of the tables are included in the instructions).
Remember that 'no reaction' is a valid observation. The balanced equations for the reactions should be reported along with the tabular comparison that you make. Check with your demonstrator that you have interpreted your observations correctly. Answers to the questions in these written instructions should be given.

Outline of the Experiment

The three series of reactions are;

A. reaction of chlorides with water.
B. reaction of halides with sulphuric acid.
C. redox reactions of halides.
D. reaction of halides with silver nitrate.

THE EXPERIMENTS

The experimental procedure

Chemicals for this experiment are in fume cupboards 5; 6; 11 and 12.
Refer to map of lab (page 5) for location of apparatus and equipment.

A. Reaction of Chlorides with water

This experiment should be carried out in the fume cupboard.

In the following experiments you are going to observe how some **CHLORIDE** compounds react when water is added to them drop by drop. Before you start - think what might happen. Will a gas be evolved? If so - what is it likely to be? What will the other products of the reaction be? If you mix **XCl** + **HOH**, there is a possibility that you will get **HCl** + **XOH**, BUT it does not happen in every case.

See what happens in the following cases and record your observations in a table in your own lab notebook (similar to table below). Take small samples of the following chlorides in clean dry test tubes (either 1 cm of a liquid in a test tube or the amount of a solid that will fit on the tip of a spatula). Carefully add water drop by drop.

1. Sodium chloride ($NaCl$)
2. Magnesium chloride ($MgCl_2$)
3. Aluminium chloride ($AlCl_3$)
4. Silicon tetrachloride ($SiCl_4$)
5. Phosphorus pentachloride (PCl_5)

When any reaction has ceased, add more water - up to about 5 mL.

What effect do these solutions have on litmus paper? Look at the periodic table and note the relative positions of sodium, magnesium, aluminium, silicon, phosphorus. How would you describe their positions?
Explain any differences in behaviour you have observed in terms of the type of bonding in the original halides.
Write equations for any reactions that happened.
Suggest a name for the type of reaction observed.

B. Reaction of halides with sulphuric acid.

You are going to compare the reactions of conc. sulphuric acid on a few crystals of sodium fluoride, sodium chloride, sodium bromide and sodium iodide. (Note the relative positions of fluorine, chlorine, bromine and iodine in the periodic table).

This experiment should be carried out in the fume cupboard.

Have some wet litmus paper ready to use. Line up 4 test tubes, one for each of the halide compounds. Use a few crystals of each. Carefully add about 2 mL of concentrated H_2SO_4 (18 mol L^{-1}) to each test tube. If necessary warm each test tube gently. Observe what happens. Note your observations in your own lab notebook, looking especially for any gradation in behaviour. Test each gas evolved for its reaction with litmus paper by holding a piece of moist litmus paper in the mouth of the test tube

Note: In the case of sodium fluoride the gas evolved may react with glass.
Rinse out that test tube with water and look for evidence of this on the walls of the test tube. What is happening in this reaction? Remember that glass is a chemical substance. This reaction is one of the methods used for etching glass.

C. Redox reactions of halides.

The experiment should be carried out in the fume cupboard.

In a test tube mix a few crystals of sodium chloride with a small sample of MnO_2 (a good oxidizing agent), then add about 2 mL of concentrated H_2SO_4 and gently warm the test tube. Compare this result with that in the previous section in which sodium chloride by itself was allowed to react with H_2SO_4. What gas has been evolved? What made the difference? Why?

D. Reaction of halides with silver nitrate.

You are going to study the reaction of silver nitrate with solutions of sodium fluoride, sodium chloride, sodium bromide, and sodium iodide.
Take about 2 cm depth of each halide solution (prepared previously) in test tubes, and to each add a <u>few</u> drops of aqueous silver nitrate (obtainable from the bench 'A' - it is expensive!). Record your observations in your own lab notebook (similar to table below). Note especially any gradation in colour.

<u>In the fume cupboard</u> add a <u>few</u> drops of concentrated ammonia (ammonium hydroxide) to any of the silver halides which are precipitated, and shake the tube. The ammonia acts as a ligand forming the complex ion $[Ag(NH_3)_2]^+$. What do you conclude about the solubility of <u>its</u> halide?

At the end of your report summarise your conclusions to this experimeny by answering the following questions:

1. Describe any trends along a period of the periodic table you have observed.
2. Describe any trends down a group of the periodic table you have observed.

APPENDIX-3c

APPENDIX-3c

EXPERIMENT - 7 - COMPLEXES

Purpose

To carry out some reactions involving complex formation, to make observations and answer questions on the reactions and compounds you have made. The structure of one of the complexes you have made will be examined by means of model building.

Outline of the experiment.

In Part A you will make four metal complexes. In Part B you will prepare a chromium (III) complex and examine its structure and possible stereoisomerism.

Part A.

This part concerns complex compound formation. <u>Reminder</u>: a <u>complex compound</u> is a compound in which either a the cation or the anion (or sometimes both) is a complex ion. A <u>complex ion</u> is an ion containing a central atom bonded via coordinate bonds to two or more ligands. Complex compounds may be water soluble or insoluble.

(1) Make an insoluble compound starting with copper (II) sulphate ($CuSO_4$) solution. To this solution add dilute sodium hydroxide solution. What is the blue precipitate? Repeat this experiment but <u>first</u> add 1 mL of tartaric acid solution to the copper (II) sulphate solution, then add the sodium hydroxide solution. Is a blue precipitate formed this time? You have formed a <u>soluble</u> complex between the Cu^{2+} ions and tartrate anions and this prevents reaction between the Cu^{2+} ions and OH^- ions since there are very few simple Cu^{2+} ions left.

(2) Another soluble complex of copper (II) is easily made. Add excess ammonia solution ('bench' ammonium hydroxide) to some copper (II) sulphate solution. The blue ion is $[Cu(NH_3)_4]^{2+}$. Name the complex cation.

(3) Now make an insoluble complex, $K_3[Co(NO_2)_6]$ (potassium cobaltinitrite or tripotassium hexanitrito cobaltate (III). This compound is unusual in that it is one of the few insoluble compounds of potassium. To some potassium chloride (KCl) solution add a few drops of sodium cobaltinitrite solution (yes, the sodium salt <u>is</u> soluble). The yellow precipitate is potassium cobaltinitrite.

(i) Complete the equation for the reaction:

$$3KCl + Na_3[Co(NO_2)_6] \rightarrow$$

(ii) What is the oxidation state of the cobalt in the complex?

(iii) Draw a sketch to illustrate the geometrical arrangement of the ligands around the cobalt.

(4) A common test to detect the presence of Fe^{3+} (ferric ion) in solution is to add potassium ferrocyanide solution. Try this for yourself by using some ferric chloride solution and adding a few drops of potassium ferrocyanide (systematic name : tetrapotassium hexacyanoferrate(II)). The intense blue complex that is precipitated is known as Prussian Blue. It has the formula $Fe_4[Fe(CN)_6]_3$

$$4FeCl_3 + 3K_4[Fe(CN)_6] \rightarrow Fe_4[Fe(CN)_6]_3 + 12KCl$$

See if you can assign the oxidation states to the iron atoms in the complex.

Part B

Preparation of $K_3[Cr(C_2O_4)_3].3H_2O$

Purpose

The purpose of this experiment is to prepare a chromium(III) complex, and then to examine its structure by model building.

Safety Precautions

Oxalate salts are toxic and should be handled with care. Chromium compounds are potential skin irritants and can cause cancer. Pay attention to the symbols in the right margin. They indicate the hazards of the substances used.

The Experimental Report should contain:

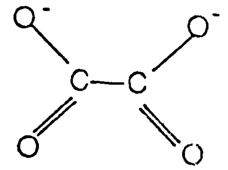
the balanced equations for the reactions in the experiment;
answers to questions in these written instructions.

Outline of the Experiment

A. Preparation of the complex.
B. Model building.

THE EXPERIMENTS

Basic ideas behind the formation of the complex



Metal ions can accept electrons from electron pair donors to form bonds. These donors are molecules or ions called **LIGANDS**. An example is the oxalate (ethanedioate) ion. The oxygen atom at each end of the ion has a lone pair of electrons which it can donate i.e. this ligand is bidentate.

The new complex ion has an overall charge of 3^- since the Cr^{3+} ion is surrounded by three oxalate ions each of which has a 2^- charge.

The Experimental Procedure

Chemicals for this experiment are on bench A or B.

A. Preparation of the complex:

1. Dissolve about 4.5 g of oxalic acid dihydrate, $(COOH)_2.2H_2O$ in 10 ml of warm water.

☠

2. Using a rough balance weigh out 1.5g of potassium dichromate and then add it, a little at a time, to the oxalic acid solution. There will be a fairly vigorous reaction. What is the gas? Why does the colour change?

☠ 🔥

3. While the reaction is subsiding, weigh out about 1.75 g of potassium oxalate ($K_2C_2O_4.H_2O$). Gently heat the reaction mixture (from part 2) until it is just beginning to boil and add the potassium oxalate and allow it to dissolve.

Note: We have now completed two operations.

(a) The chromium in the dichromate ion was in the 6+ oxidation state and the oxalic acid has reduced it to the 3+ oxidation state.

(b) More oxalate and potassium ions have now been added to complete the formation of the complex $K_3[Cr(C_2O_4)_3].3H_2O$.

4. Cool the solution and add 2 mL of ethanol. Blue-green crystals of the complex now grow in the nearly black solution. The ethanol reduces their solubility.

🔥

5. Filter off the crystals on a paper in a Buchner apparatus. (See appendix 3).
6. Wash the crystals (which are still on the filter) with a mixture of 5 mL of ethanol and 5 mL of water. Finally wash the crystals with 5 mL of pure ethanol. Continue to draw air through the filter to dry the crystals, but finally dry them by pressing them between two sheets of filter paper.

Note: To be clear in your mind about what has been done so far, write ion/electron half equations for:

Dichromate ---> Chromium(III) (reduction) in acid solution
Oxalate ion ---> Carbon dioxide (oxidation)
and an equation for the complex formation, which is not a redox step.
Oxalate ion + Chromium(III) ---> complex ion

B. Model building.

1. The formula of the crystals you have made is $K_3[Cr(C_2O_4)_3].3H_2O$. The water molecules are not part of the complex but are components of the crystal lattice, as in $Na_2[SO_4].10H_2O$.

2. Using the model building materials provided, make a model of the complex ion.

3. Are any stereoisomers of this complex ion possible? State the number you think are possible after comparing with your neighbours' models..

4. How are the isomers related to one another.

APPENDIX-4a

APPENDIX-4a

EXPERIMENT -3-

REDOX REACTIONS

YOU WILL DO PART B AND C OF THIS EXPERIMENT ON SMALL SCALE AS FOLLOWS (PART A IS IN THE MAIN LAB MANUAL TO BE DONE ON THE NORMAL SCALE)

Important Note About the Use of Pasteur Pipette

In this experiment you will handle a Pasteur pipette frequently. You will be often asked to use a specific number of drops of liquid. Therefore, in order to obtain accurate results:

(i) You must position the tip of the Pasteur pipette above the test tube. This will allow you to count the number of drops accurately.

(ii) You must rinse the Pasteur pipette **every time** you use it. First, hold the Pasteur pipette under a flow of tap water to wash the external surface. Secondly, Wash the internal surface by dipping the tip of the pipette in deionised water (250 mL beaker filled with deionised water) with the teat pressed, fill the pipette with deionised water by releasing the teat. Empty the pipette into the sink (by pressing the teat). Repeat this operation twice to ensure a proper rinse for the pipette.

Locations of Chemicals for Parts B and C of This Experiment

Chemicals to be used in this part are located as follows:

	Chemicals	Locations	To be used in
1-	Sodium halides (NaF, NaCl, NaBr, NaI)	Fume cupboard no. 4,5,7	Part B. & C.
2-	Sodium hypochlorite	Fume cupboard no. 4,5,7	Part B.
3-	Chloroform ($CHCl_3$)	Fume cupboard no. 4,5,7	Part B.
4-	Sulphuric acid (1 mol L^{-1})	On your bench	Part B.
5-	Copper sulphate solution	On your bench	Part C.
6-	Sodium thiosulphate solution	On your bench	Part C.

Refer to map of lab (page 5 of the main manual) for location of apparatus and equipment.

Part B.

Replacement of a Halogen by Another

You will use the SMALL TEST TUBES for this part of the experiment which are provided in the kit available on your bench.

The Experimental Procedure

In this part you are going to use dilute aqueous solutions of the ionic halides (NaF, NaCl, NaBr & NaI). **LABEL** 4 small test tubes (F^-, Cl^-, Br^-, I^-). Prepare a solution of each halide by dissolving a few crystals (about the amount on the tip of the **small** spatula) in 10 drops of deionised water in small test tubes.

Prepare a chlorine solution in water by diluting approximately 2 drops of sodium hypochlorite with 10 drops of deionised water, then acidifying it with 2 drops of 1 mol L^{-1} sulphuric acid.

$$2NaOCl + H_2SO_4 \text{ ---------> } Na_2SO_4 + H_2O + Cl_2$$

Add 2 drops of the chlorine solution to each of your samples of dilute sodium fluoride, sodium chloride, sodium bromide, and sodium iodide, in small test tubes, and note what you see.

Add 3 drops of chloroform (trichloromethane) to each of the solutions. It will form a lower layer. With your dropper (Pasteur pipette) mix the layers by blowing bubbles into the two layers and observe the colour of the chloroform layer. Halogens are more soluble in chloroform than they are in water, so any free halogen is removed from water and ends up mainly in the chloroform layer giving a distinctive colour. ☠

Chloroform ($CHCl_3$) is less polar than H_2O. Why does I_2 prefer to dissolve in $CHCl_3$ rather than water?

Write balanced equations for the reactions that occurred.

Record your observations in your own lab notebook in a table (similar to table 2).

Dispose of all the used chemicals into the **organic waste bottle (fume cupboard 4, 5, and 7)**. Rinse your test tubes.

TABLE (2) REACTION WITH Cl_2

Halides + Cl_2	**Initial Colour Produced in Water**	**Colour of Chloroform Solution**	**Products**
$NaF + Cl_2$			
$NaCl + Cl_2$			
$NaBr + Cl_2$			
$NaI + Cl_2$			

Part C.

Iodide-Iodine Interconversion

You will use the TRANSPARENT POLYTHENE SHEET for this part of the experiment which is available on your bench.

The Experimental Procedure

Prepare some more NaBr and NaI solutions by dissolving a few crystals in 5 drops of deionised water in a small test tube. To 2 drops of copper sulphate solution add 2 drops of NaBr solution on the transparent polythene sheet, nothing happens! Now take another two drops of copper sulphate solution on the sheet and add to it 2 drops of NaI solution. You get precipitates of two products. One of the products is iodine, the other is a copper (I) iodide (CuI). What is the colour of the precipitates? Write a balanced equation for the redox reaction. To the above precipitates add 2 drops of sodium thiosulphate $Na_2S_2O_3$ solution. Another redox reaction occurs.

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

You will now be able to see the CuI precipitate clearly. What colour is it?

Determine the colour of the precipitates by placing the sheet on a white surface (i.e. white paper) if the precipitate is coloured, or on the dark surface of the bench if the precipitate is white.

From your observations in part B and part C of the experiment, put the ions, **bromide**, **chloride**, **iodide**, and **thiosulphate** in order (most easily oxidised to least easily oxidised).

Wash the transparent polythene sheet under a flow of tap water in the sink. Dry and keep the sheet **on your bench** for further use.

APPENDIX-4b

APPENDIX-4b

EXPERIMENT-5-

PERIODIC TABLE TRENDS

THIS EXPERIMENT IS TO BE DONE ON SMALL SCALE ACCORDING TO THE INSTRUCTIONS GIVEN BELOW

Purpose

The purpose of this experiment is to do three series of reactions on halogen compounds and to compare the reactions within each series looking for gradations in behaviour. In particular you are looking for the effects of oxidation of the halide ion compared to other reactions. The reactions of the halogen group of elements and compounds should give an indication of more general trends in the periodic table of elements.

Safety Precautions

Unlike sodium chloride, the sodium salts of other halides are poisonous and should be handled with care. Many of the other chemicals in this experiment are toxic and corrosive. Pay attention to the symbols in the right margin. They indicate the hazards of the substances used.

The Experimental Report

For your own benefit, draw up tables of comparisons in your own lab notebook while doing the following experiments and make a note of each observation. (Examples of the tables are included in the instructions).

Remember that 'no reaction' is a valid observation. The equation for the reaction should be reported along with the tabular comparison that you make. Check with your demonstrator that you have interpreted your observations correctly.

Answers to the questions in these instructions should be given.

Outline of the Experiment

The four reactions are;

A. reactions of chlorides with water.
B. reactions of halides with sulphuric acid.
C. redox reactions of sodium chloride.
D. reactions of halides with silver nitrate.

THE EXPERIMENT

Important Note About the Use of Pasteur Pipette

In this experiment you will handle a Pasteur pipette frequently. You will often be asked to use a specific number of drops of liquids. Therefore, in order to obtain accurate results:

(i) You must position the tip of the Pasteur pipette above the test tube. This will allow you to count the number of drops accurately.

(ii) You must rinse the Pasteur pipette **every time** you use it. First, hold the Pasteur pipette under a flow of tap water to wash the external surface. Secondly, Wash the internal surface by dipping the tip of the pipette in deionised water (250 mL beaker filled with deionised water) with the teat pressed, fill the pipette with deionised water by releasing the teat. Empty the pipette into the sink (by pressing the teat). Repeat this operation twice to ensure a proper rinse for the pipette.

Locations of Chemicals for This Experiment
Chemicals to be used in this experiment are located as follows:

	Chemicals	Locations	To be used in
1-	Sodium chloride	Fume cupboard no. 4,5,7	Part A., B. & D.
2-	Magnesium chloride	Fume cupboard no. 7	"
3-	Aluminium chloride	Fume cupboard no. 4,5,7	"
4-	Silicon tetrachloride	Fume cupboard no. 4,5,7	"
5-	Phosphorus pentachloride	Fume cupboard no. 4,5,7	"
6-	Sodium fluoride	Fume cupboard no. 4,5,7	Part B. & D.
7-	Sodium bromide	Fume cupboard no. 4,5,7	" "
8-	Sodium iodide	Fume cupboard no. 4,5,7	" "
9-	Sulphuric acid (conc.)	Fume cupboard no. 4,5,7	Part B. & C.
10-	Manganese dioxide	Fume cupboard no. 4,5,7	Part C.
11-	Silver nitrate	Fume cupboard no. 5,7	Part D.
12-	Ammonium hydroxide	On your bench	Part D.
13-	Blue litmus paper	On your bench	Part A., B., & C

Refer to map of lab (page 5 in the main manual) for location of apparatus and equipment.

IN PARTS A., B., AND C. OF THIS EXPERIMENT YOU WILL USE SMALL TEST TUBES PROVIDED IN A KIT AVAILABLE ON YOUR BENCH.

Part A.

Reaction of Chlorides with Water

(THIS REACTION MUST BE DONE IN THE FUME CUPBOARD)

In the following experiments you are going to observe how some chloride compounds react when water is added to them. Before you start, think what might happen. Will a gas be evolved? If so, what is it likely to be? What will the other products of the reaction be? If you mix XCl + HOH, there is a possibility that you will get HCl + XOH, BUT it does not happen in every case.

The Experimental Procedure

See what happens in the following cases and record your observations in a table (similar to table 3 on the next page) in your own lab notebook.

Take very small samples of the following chlorides in clean dry small test tubes (either 2 drops of a liquid by a Pasteur pipette or a few crystals of a solid that will fit on the tip of a **small** spatula) and **LABEL** them. Then add carefully 5 drops of deionised water, drop by drop, to each test tube.

1. Sodium chloride (NaCl)
2. Magnesium chloride ($MgCl_2$)
3. Aluminium chloride ($AlCl_3$)
4. Silicon tetrachloride ($SiCl_4$)
5. Phosphorus pentachloride (PCl_5)

When any reaction has ceased, add more deionised water up to about 10 drops.

What effect do these solutions have on blue litmus paper? Look at the periodic table and note the relative positions of sodium, magnesium, aluminium, silicon, and phosphorus.

Explain any differences in behaviour you have observed in terms of the type of bonding in the original halides. Write equations for any reactions that happened. Suggest a name for the type of reaction observed.

Dispose of all the used chemicals into the **the sink** and wash them away with water. Rinse your test tubes. In the case of $SiCl_4$ you may not be able to clean the test tube. In that case dispose of it in the **glass bin** (refer to map of lab in the main manual page 5).

TABLE (3) REACTION WITH DEIONISED WATER

Chlorides	**What gas and its colour if any**	**Effect on blue litmus paper**	**Any other observations**
Sodium chloride(NaCl)			
Magnesium chloride($MgCl_2$)			
Aluminium chloride ($AlCl_3$)			
Silicon tetrachloride($SiCl_4$)			
Phosphorus pentachloride(PCl_5)			

Part B.

Reactions of Halides with Sulphuric Acid

(THIS REACTION MUST BE DONE IN THE FUME CUPBOARD

You are going to compare the reaction of concentrated sulphuric acid on a few crystals of sodium fluoride, sodium chloride, sodium bromide and sodium iodide. (Note the relative position of fluorine, chlorine, bromine and iodine in the periodic table).

The Experimental Procedure
Have some wet blue litmus paper ready to use. Place a few crystals of each of the halide compounds (NaF, NaCl, NaBr, NaI) in a small test tube and **LABEL** them. Carefully add 3 drops of concentrated sulphuric acid to the crystals of each halide. Observe what happens. Note your observations in your own notebook in a table like table 4, looking especially for any gradation in behaviour.

Test each gas evolved for its reaction with litmus paper by holding the paper at the mouth of each test tube. Record your results.

TABLE (4) REACTION WITH H_2SO_4 (Conc.)

Halides + H_2SO_4	**What gas and its colour if any**	**Effect on blue litmus paper**	**Any other observations**
$NaF + H_2SO_4$			
$NaCl + H_2SO_4$			
$NaBr + H_2SO_4$			
$NaI + H_2SO_4$			

Retain the test tube of sodium chloride for use in part C.

Dispose of all the rest of used chemicals into the **sink** and wash them away with water.. Rinse your test tubes.

Part C.

Redox Reactions of Halides (NaCl)

(THIS REACTION MUST BE DONE IN THE FUME CUPBOARD)

The Experimental Procedure
Add a few granules of manganese dioxide (MnO_2 is a good oxidising agent) to the test tube of sodium chloride you have used in part B, and then add a further 3 drops of concentrated H_2SO_4. Compare this result with that in the previous part, in which sodium chloride alone was allowed to react with the concentrated sulphuric acid (H_2SO_4). What gas has been evolved this time? Test with wet blue litmus paper. What made the difference? Why?

Dispose of all the used chemicals into the **sink** and wash it away with water. Rinse your test tubes.

Part D.

Reaction of Halides with Silver Nitrate

You will use the TRANSPARENT POLYTHENE SHEET for this part of the experiment which is available on your bench. Plus 4 small test tubes.

You are going to study the reaction of silver nitrate with solutions of sodium fluoride, sodium chloride, sodium bromide, and sodium iodide.

The Experimental Procedure
Make up solutions of sodium fluoride, sodium chloride, sodium bromide, and sodium iodide in your labelled test tubes by dissolving a few crystals of each halide (on the tip of a **small** spatula) in 5 drops of deionised water (use a Pasteur pipette).

Place 1 drop of each halide side by side on the transparent polythene sheet and add 1 drop of silver nitrate solution to each.

Record your observations in your own lab notebook (similar to table 5). Note especially any gradation in the colour of the products (you may have to try it against a **dark** background as well as the white backing sheet provided).

Add 2 drops of ammonium hydroxide (4 mol L^{-1}) by Pasteur pipette to the silver halides precipitated, and observe any change. The ammonia acts as a ligand forming the complex ion $[Ag(NH_3)_2]^+$ (a ligand is a negative ion or any other electrons donor molecules). What do you conclude about the solubility of Ag halides in ammonium hydroxide? Record your observations in your own lab notebook (similar to table 5).

TABLE (5) REACTION WITH SILVER NITRATE AND SOLUBILITY OF HALIDE OF SILVER-AMMONIA COMPLEX ION

$NaX + AgNO_3$	**Colour of the precipitate**	**Product**	**Solubility of AgX in ammonia solution**
$NaF + AgNO_3$			
$NaCl + AgNO_3$			
$NaBr + AgNO_3$			
$NaI + AgNO_3$			

At the end of your report summarise your conclusions to this experiment by answering the following questions:

1. Summarise any trend along a period of the periodic table you have observed.
2. Summarise any trend down a group of the periodic table you have observed.

Wash the transparent polythene sheet under a flow of tap water into the **sink**. Dry it and keep the polythene sheet **on your bench** for further use.

APPENDIX-4c

EXPERIMENT -7-

COMPLEXES

YOU WILL DO PART A OF THIS EXPERIMENT ON SMALL SCALE ACCORDING TO THE INSTRUCTIONS (PART B IS IN THE MAIN MANUAL TO BE DONE ON THE NORMAL SCALE).

Purpose

In part A., the purpose is to carry out some reactions involving complex formation, to make observations and answer questions on the reactions and compounds you have made.

In part B., the purpose is to prepare a chromium (III) complex, and then to examine its structure by model building.

Safety Precautions

In part B. oxalate salts are toxic and should be handled with care. Chromium compounds are potential skin irritants. Pay attention to the symbols in the right margin. They indicate the hazards of the substances used.

The Experimental Report

The experimental report should contain:

a. The balanced equations for the reactions in the experiments.
b. Answers to the questions in these written instructions.

Outline of the Experiment

In part A. you will make four metal complexes.

In part B. you will prepare a chromium (III) complex and examine its structure and possible stereoisomerism.

Important Note About the Use of a Pasteur Pipette

In this experiment you will handle a Pasteur pipette frequently. You will often be asked to use a specific number of drops of liquids. Therefore, in order to obtain accurate results:

(i) You must position the tip of the Pasteur pipette above the test tube. This will allow you to count the number of drops accurately.
(ii) You must rinse the Pasteur pipette **every time** you use it. First, hold the Pasteur pipette under a flow of tap water to wash the external surface. Secondly, Wash the internal surface by dipping the tip of the pipette in deionised water (250 mL beaker filled with deionised water) **with the teat pressed,** fill the pipette with deionised water by releasing the teat. Empty the pipette into the sink (by pressing the teat). Repeat this operation twice to ensure a proper rinse for the pipette.

Locations of Chemicals for This Experiment

Chemicals to be used in this experiment are located as follows:

	Chemicals	Locations	To be used in
1-	Copper (II) sulphate	On your bench or bench 'B'	Part A.
2-	Sodium hydroxide (1 mol L^{-1})	On your bench	"
3-	Tartaric acid (20%)	On bench 'B'	"
4-	Ammonium hydroxide (4 mol L^{-1})	On your bench	"
5-	Sodium cobaltinitrite	On bench 'B'	"
6-	Potassium chloride	On bench 'B'	"
7-	Ferric chloride	On bench 'B'	"
8-	Potassium ferrocyanide	On bench 'B'	"
9-	Oxalic acid dihydrate	Fume cupboard no. 4,5,7	Part B.
10-	Potassium dichromate	Fume cupboard no. 5,7	"
11-	Potassium oxalate	Fume cupboard no. 5,7	"
12-	Ethanol	On your bench	"

Refer to map of lab (page 5 of the main manual) for location of apparatus and equipment.

Part A.

Complex Compound Formation

You will use the TRANSPARENT POLYTHENE SHEET which is available on your bench in this part of the experiment. You may also use the small test tubes which are provided in the kit.

A **complex compound** is a compound in which either the cation or the anion (or sometimes both) is a complex ion. A **complex ion** is an ion containing a central atom bonded via co-ordinate bonds to ligands. Complex compounds may be water soluble or insoluble.

In this part of the experiment you are asked to form 4 different complex compounds. These are:-

1- Complex of copper and tartrate ions (soluble).
2- Complex of ammonia and copper (II) ions (soluble).
3- Potassium cobltinitrite (insoluble).
4- Ferric ferrocyanide (insoluble).

The Experimental Procedure

1- Place 2 drops of copper (II) sulphate ($CuSO_4$) solution on the <u>transparent polythene sheet</u>. To this solution add 1 drop of dilute sodium hydroxide solution (1 mol L^{-1}). What is the blue precipitate? Repeat this experiment, but **first** add 2 drops of tartaric acid solution (20%) to the copper (II) sulphate solution, then add 1 drop of the sodium hydroxide solution. Is a blue precipitate formed this time? You have formed a soluble complex between the Cu^{2+} ions and tartrate ions and this prevents reaction between the Cu^{2+} ions and OH^- ions, since there are very few simple Cu^{2+} ions left for reaction with OH^- due to reaction with tartrate ions.

2- Another soluble complex of copper (II) is easily made by adding 2 drops of ammonia solution (ammonium hydroxide) to 2 drops of copper (II) sulphate solution on the polythene sheet. The blue ion is $[Cu(NH_3)_4]^{2+}$. Name the complex cation.

DIX-5

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STUDENTS' POINTS OF VIEW

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conducting a pilot study to investigate the scale for doing experiments in our ories. During this term you will have done some of your experiments on normal scale me on small scale and we would value your responses to the following statements ormal scale and small scale experiments. Your views will help us in our future g. Thank you for your help.

ndicate your views about each statement by ticking ONE box for each:-

	NORMAL SCALE rather than on SMALL SCALE	SMALL SCALE rather than on NORMAL SCALE	No Difference
chemistry experiments are more fun when doing them on			
experiments easier and simpler on the			
llage of chemicals and breakage of apparatus in the experiments s in case of the			
less help from the lab staff to do an experiment on the			
re interested in chemistry when doing experiments on the			
ne less time to complete the experiments in case of the			
oing chemistry experiments, I can understand the chemistry more hen working on			
that less apparatus is needed when doing experiments on the			
re relaxed and safe when doing experiments on the			
eriments have enabled me to concentrate on the chemistry more in the			
rk on the bench was more tidy and less cluttered when doing ents on the			
manual instructions are easier and clearer to follow in case of the			
that I could learn chemistry better when doing experiments on the			
ed more care in handling apparatus in case of the			
given a choice between small scale and normal scale experiments I refer to do the			

te any additional COMMENTS you may have, regarding small scale experiments, on the back of this sheet your opinion about the use of polythene sheets, small test tubes and droppers):

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STUDENTS' POINTS OF VIEW

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ndicate your views about each statement by ticking ONE box for each:-

101 STUDENT	NORMAL SCALE rather than on SMALL SCALE	SMALL SCALE rather than on NORMAL SCALE	No Difference
hemistry experiments are more fun when doing them on	28	36	37
experiments easier and simpler on the	20	61	20
llage of chemicals and breakage of apparatus in the experiments s in case of the	8	66	27
l less help from the lab staff to do an experiment on the	07	29	65
re interested in chemistry when doing experiments on the	21	27	54
ne less time to complete the experiments in case of the	10	68	23
oing chemistry experiments, I can understand the chemistry more hen working on	14	22	65
that less apparatus is needed when doing experiments on the	4	85	12
re relaxed and safe when doing experiments on the	7	46	48
eriments have enabled me to concentrate on the chemistry more in the	12	32	57
rk on the bench was more tidy and less cluttered when doing ents on the	03	79	19
manual instructions are easier and clearer to follow in case of the	14	40	47
that I could learn chemistry better when doing experiments on the	13	25	63
red more care in handling apparatus in case of the	57	26	18
given a choice between small scale and normal scale experiments I refer to do the	20	60	21

te any additional COMMENTS you may have, regarding small scale experiments, on the back of this sheet your opinion about the use of polythene sheets, small test tubes and droppers):

IX-7

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CENTRE FOR SCIENCE EDUCATION

STUDENTS' POINTS OF VIEW

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conducting a pilot study to investigate the scale for doing experiments in our ries. During this term you will have done some of your experiments on normal scale ne on small scale and we would value your responses to the following statements ormal scale and small scale experiments. Your views will help us in our future g. Thank you for your help.

ndicate your views about each statement by ticking ONE box for each:-

168 STUDENTS	NORMAL SCALE rather than on SMALL SCALE	SMALL SCALE rather than on NORMAL SCALE	No Difference
hemistry experiments are more fun when doing them on	36	73	59
experiments easier and simpler on the	30	106	32
llage of chemicals and breakage of apparatus in the experiments s in case of the	12	110	46
l less help from the lab staff to do an experiment on the	11	48	109
ore interested in chemistry when doing experiments on the	31	47	90
ne less time to complete the experiments in case of the	15	120	33
loing chemistry experiments, I can understand the chemistry more vhen working on	21	39	108
that less apparatus is needed when doing experiments on the	07	136	25
ore relaxed and safe when doing experiments on the	12	78	78
periments have enabled me to concentrate on the chemistry more in the	15	55	98
rk on the bench was more tidy and less cluttered when doing nents on the	07	131	30
manual instructions are easier and clearer to follow in case of the	19	76	73
that I could learn chemistry better when doing experiments on the	19	47	102
ired more care in handling apparatus in case of the	90	47	31
given a choice between small scale and normal scale experiments I prefer to do the	30	106	32

ite any additional COMMENTS you may have, regarding small scale experiments, on the back of this sheet y your opinion about the use of polythene sheets, small test tubes and droppers):

DIX-5

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STUDENTS' POINTS OF VIEW

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ndicate your views about each statement by ticking ONE box for each:-

	NORMAL SCALE rather than on SMALL SCALE	**SMALL SCALE rather than on NORMAL SCALE**	**No Difference**
chemistry experiments are more fun when doing them on			
experiments easier and simpler on the			
llage of chemicals and breakage of apparatus in the experiments s in case of the			
l less help from the lab staff to do an experiment on the			
re interested in chemistry when doing experiments on the			
ne less time to complete the experiments in case of the			
loing chemistry experiments, I can understand the chemistry more hen working on			
that less apparatus is needed when doing experiments on the			
re relaxed and safe when doing experiments on the			
eriments have enabled me to concentrate on the chemistry more in the			
rk on the bench was more tidy and less cluttered when doing ents on the			
manual instructions are easier and clearer to follow in case of the			
that I could learn chemistry better when doing experiments on the			
red more care in handling apparatus in case of the			
given a choice between small scale and normal scale experiments I refer to do the			

te any additional COMMENTS you may have, regarding small scale experiments, on the back of this sheet your opinion about the use of polythene sheets, small test tubes and droppers):